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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

because of differences in weights of the filtering mats, especially when the asbestos mats are used. If sediment concentrations are greater than 1,000 mg/l, then it may not be necessary to obtain a new tare weight each time a crucible with a glass-fiber filter mat is used.

14. Compute the net weight of sediment in the container and deduct, if necessary, the dissolved-solid correction.
15. Compute the concentration of the sample in parts per million determined on the basis of one million times the ratio of the net weight of sediment to the net weight of the water-sediment mixture. The computed parts per million can then be expressed in the common units of milligrams per liter by application of the conversion factor given in table 1. It is convenient to make the conversion from parts per million to milligrams per liter when the data are transferred from the form *Sediment Concentration Notes* to other uses. Usually concentrations are less than 16,000 parts per million in which case parts per million equals milligrams per liter. However, when the concentration does exceed 16,000 ppm the appropriate conversion factor should be penciled plainly below the concentration on the form as a reminder to the data user that milligrams per liter is different from parts per million.

Determination of Particle-Size Distribution of Suspended Sediments

Complete definition of the variations of particle-size distribution for a stream throughout the ranges of discharge and with time is presently not economically feasible. It is feasible, however, to analyze a sufficient number of samples to provide representative data for a range of the many conditions occurring throughout the year.

Particle-size data have many uses. One use is to compute the probable specific weight of sediment as it will deposit in reservoirs. This computation requires plotting the median particle size against sediment load in tons per day. Information concerning the behavior of sediment particles in different environments may be studied and, therefore, requires analyses for particle-size distribution of fine particles in both native and dispersed settling media. The size distribution of both suspended and bed material has an important use in the computations of total (measured plus unmeasured) sediment discharge. The frequency and kind of particle-size analyses should be adequate to describe the pertinent characteristics of sediment particles so that satisfactory comparisons can be made between sediments collected from different places or from the same place at different times.

Recommended frequency of analyses

The following recommendations are for the determinations of ultimate sizes by use of a distilled-water settling medium and a chemical dispersant for the silt and clay fractions. The recommended frequency of native-water analysis is discussed in the section "Native water settling medium" in respect to the theories, problems, and methods of such analysis. Each sample should be depth integrated and represent the true size distribution in the cross section at the time of sampling. Thus, for most streams, samples must be collected by the ETR (equal-transit-rate) method or by sampling at an adequate number of the centroids of equal discharge. If the "daily" samples collected at one vertical near midstream are representative of the sediment characteristics in the entire cross section, the selection should normally be adequate to cover a wide range of flow conditions.

Judgment concerning the selection of samples will be enhanced by a study of the conditions for which particle-size samples of past records for the given station were selected, if available; and (or) by a study of the sample information recorded on the form *Sediment Concentration Notes* (fig. 4) for the current water year. For many streams, information on sediment characteristics is usually lacking during the first year of sampling on a given stream; therefore, some samples, particularly from high-stage flows, should be set aside to be analyzed at a later time either for particle size or concentration at the end of the water year when the facts are known concerning the range and other conditions of the year's flow. These extra samples should, of course, be tightly sealed, tared and stored in a dark place.

Size analyses of suspended sediment for the so-called daily station should be made on a minimum of five or six samples per year if selected to represent various flow conditions and range of concentrations. Additional samples, up to a

maximum of about 30, may be desirable for new stations or to define the size distribution during unusually high runoff periods to represent a range of stream discharge or some of the unusual areal patterns of runoff. The minimum number of samples may be reduced by necessity because of low runoff due to a prolonged drought or in the event of extremely low sediment concentration.

Particle-size analyses should be made on a minimum of one-fifth of the observations for stations operated on a periodic basis. The number depends on the frequency of sampling, the stream conditions, and the projected use of the data. Whenever possible, a desirable number should be set as a guide at the time of programing for the station.

Computations of total sediment load (measured plus unmeasured) require the use of particle-size distribution data of the suspended sediment included in the average concentration at the time of the observation. The above programs for determining particle size must then include consideration of the desired times for the total load computations.

Method of analysis

Limitations

Specific limitations of the various methods of analysis will be discussed as each method is presented in the following sections. The tabulation at side gives a guide to the size, analysis concentration, and (or) the quantity of sediment needed for each kind of analysis. This knowledge will facilitate making the necessary decisions on how many sample bottles are needed, how much splitting is required, or which size-analysis technique is best to use.

Dispersed Settling Medium

The program involving stream sediment size analysis should place major emphasis on the determination of particle size by use of methods that minimize floccule formation and thus represent "standardized" conditions. The recommended method of analysis for determining particle-size distribution in the dispersed settling medium is, by necessity, a combination of methods. For example, laboratories analyzing a large number of samples use the VA tube-pipet method for most of the samples. The VA tube is preferred to sieves because the results of the anlyses are expressed in sedimentation diameters, which are more suitable for studies of sediment transport and deposition. However. to compare the sedimentation diameter with that of actual sizes as determined by sieves, two or three analyses (or 10 or 15 percent of the total, whichever is more) per station for the first year or two of operation should also be made with sieves for comparison. The results of both methods are then published for a given sample. Likewise, the "small-scale" laboratory not equipped with a VA tube, but using the sieve-pipet method should send the sand fraction from three or four samples for each station to a laboratory having a VA tube for comparative analysis.

	Size range (mm)	Analysis concentration (mg/l)	Quantity of sediment (g)
Sieves	0.062-32		<0.05
VA tube [1]	0.062- 2 0		0.05-15.0
Pipet	0.002- 0.062	2,000-5,000	1.0 - 5.0
BW tube [2]	0.002- 0.062	1,000-3,500	0.5 - 1.8

[1] See table 5 for more detail.
[2] If necessary, may be expanded to include sands up to 0.35 mm, the accuracy decreasing with increasing size. This required concentration and quantity can be increased accordingly.

Table 5.—Guide to selection of VA tube size

Quantity of sample		Maximum particle size		Sedimentation tube	
Dry weight (g)	Volume of sand (ml)	Fall diameter (mm)	Sieve diameter (mm)	Length (cm)	Diameter (mm)
0. 05- 0. 8	0. 03-0. 5	0. 25	0. 25	120	2. 1
0. 4 - 2. 0	0. 2 -1. 2	. 35	. 40	120	3. 4
0. 8 - 4. 0	0. 5 -2. 4	. 50	. 60	120	5. 0
1. 6 - 6. 0	1. 0 -4. 0	. 70	1. 00	120	7. 0
5. 0 -15. 0	3. 0 -9. 0	----------	2. 00	180	10. 0

Many laboratories must retain the use of the more time-consuming, and possibly less accurate, BW tube method because of an insufficient quantity of sediment in most of the samples. Whenever possible, a sufficient quantity of sediment should be collected to permit analysis by the VA tube-pipet method. The increased time to obtain the additional material by accumulating more samples is usually more than compensated by the decreased time required for analyses and computation.

The BW tube can theoretically define the sedimentation diameter for sands up to 0.350 mm, but the analysis is simplified considerably if all sand is removed by wet sieving leaving only the silt and clay sizes for analysis by the BW tube. Particles larger than 0.350 mm and preferably larger than 0.062 should be removed by wet sieving and analyzed by the sieve method if a VA tube is not available. (See section on "Sieving.")

Other parts of this chapter give detailed procedures for making the analyses by various combinations of sieve and sedimentation methods. For most stations, when the VA tube-pipet or the sieve-pipet methods are used, only the following size divisions need be determined: 0.002, 0.004, 0.016, 0.062, 0.125, 0.250, 0.500, 1.00, and 2.00 mm. This is especially true when the median size is in the sand range. If the median size is in the silt range, then the 0.008 and 0.031 sizes should be determined. When samples are split for analyses, all the above sizes, including the 0.008 and 0.031 sizes, should be determined in both native water and a chemically-dispersed settling medium. If the BW tube method is used, additional sizes may be determined from the Oden curve with little additional effort.

Pipet analyses are made using either 500 or 1,000 ml of suspension with a suspension range of 2,000 to 5,000 mg/l. However, if sufficient sediment is not available to obtain a concentration of 2,000 mg/l for a 500 ml suspension, less than 500 ml can be used for the suspension. Though not recommended for general use, such abbreviated pipet analyses, in which only the 0.002, 0.004, and 0.016 mm determinations are made, can conveniently be performed using as little as 200 ml of suspension. By decreasing the volume of suspension and the number of sizes determined, and by using withdrawal depths of 5 or 3 cm (centimeters) or less, sufficiently accurate pipet analyses can be made using as little as 0.4 g of silt and clay. By reducing the volume of suspension in this manner, pipet analyses can be made on samples containing a small amount of sediment which otherwise would have to be analyzed by the more time-consuming and possibly less accurate BW tube method.

When samples contain some kinds of clay, considerable difficulty is encountered with the sieving, especially the initial separation of the fines from the sands, because it is not possible to obtain complete dispersion and cleaning of the sand particles. Treatment by ultrasonic methods prior to sieving has been found helpful (Kravitz, 1966; Moston and Johnson, 1964). A device having a range of power settings, such as the Branson "Sonifier" model S–75 is recommended. The procedure using this device would require only that the "Sonifier" horn be submersed in the water and sediment to a depth of 1 cm and operated for 3–5 minutes. The contents would then be immediately poured onto a 3-inch (7.5 cm) diameter sieve and the material on the sieve washed with a spray of distilled water. Silt and clay so treated will pass through the sieve without clogging it, and this procedure will reduce the amount of time and the quantity of water required for wet sieving. The ultrasonic treatment should not be used on sediment to be analyzed in the native water settling medium.

Native water settling medium

Prediction of the fall behavior of sediment particles in different environments is difficult because there are many types of fluvial sediments being transported in media having a wide variation in kind and concentration of chemical constiuents. The incomplete understanding of the characteristics and behavior of small particles, especially clay minerals, complicates the problem of predicting particle behavior. The problem is most acute in situations where clay and perhaps fine silt tend to form floccules in native water. Generally, if the native water is low in dissolved solids and (or) contains sodium as the dominant cation in solution, the fine parti-

cles will behave as discrete units. Many stream waters either are low in dissolved solids or contain considerable sodium, and therefore the problems under discussion have little application to mechanics of sediment transport within the flowing stream.

Use of settling methods for size analysis is based on the definition that the sedimentation diameter of a discrete particle is the diameter of a sphere having the same specific gravity and the same uniform terminal fall velocity as the given particle. Size analyses in which native water is the settling medium may violate this theory in two ways:

1. The particles in suspension loosely join to form masses in which both size and density are changing with time. Such masses fall faster than the component particles but, because of their loose aggregation, fall slower than a quartz sphere of the same nominal diameter.
2. The floccules may be in the process of accretion while settling and therefore would have an increasing and accelerating velocity rather than the constant terminal fall velocity.

In connection with item 1, a floccule would have a density between that of water and that of the discrete component particles. These large irregular floccules probably are retarded by form drag in addition to a viscous drag. Thus, the settling velocity and, consequently, the sedimentation diameter for many particles in native water result from complicated and probably indeterminable factors.

In the absence of the disruptive forces of turbulence or shear, the stability of a suspension is mainly controlled by the magnitude of the electrokinetic or zeta potential associated with the individual particles. This potential can be defined as the potential between the layer of immobile ions adsorbed on the particle surface and the last mobile ion associated with the particle. Most clay particles have a negative charge and migrate to the positive pole if placed between two electrodes of unlike sign. The migration velocity is proportional to the negative potential. The zeta potential is an important key to the properties of colloids. When this potential is sufficiently high, particles will repel each other, but when lowered to a point called the critical potential (by addition of an electrolyte or by a change in water characteristics), the electrical double layer associated with the particle collapses and the material flocculates.

In a natural stream, the stability of particles in a water-sediment mixture is controlled by continuous turbulent motion as well as the zeta potential; whereas, in the laboratory, the stability is controlled only by the zeta potential. Information concerning the degree of flocculation of sediment suspended in natural streamflow is thus lacking because the natural conditions cannot be simulated during size analysis in the laboratory.

Other variables that influence the amount of flocculation occurring with analyses of sediment in native water settling media are (1) water characteristics in terms of the kind and concentration of dissolved constituents, (2) kind of clay mineral, (3) nature of the ions associated with the particle before it entered the stream, and (4) concentration of the sediment in the analysis suspension. The complex interaction and effects of these variables makes it obvious that data obtained by size analyses in native water is difficult to interpret and, therefore, not easily applied to the solution of sediment problems. The results of such analyses for some streams are found to be highly variable depending on stage, season, or other factors; however, for other streams the results are relatively uniform, indicating a similar degree of flocculation under varying hydrologic conditions. In a like manner, samples from some streams show a marked difference in particle-size distribution determined in native water and dispersed media; whereas, samples from other streams show little or no difference between the two methods of analysis.

Native water size analyses are not necessary for some streams if it is apparent that flocculation is highly improbable throughout the discharge range. However, during the first 2 years of determining sediment transport at a site, it is desirable that about one-fifth as many analyses should be made using native water as using distilled water. Thereafter, the number of native water size analyses can be greatly reduced un-

less a significant change in the regimen of the river is in progress.

Split portions of a sample should be used when analyses are made in both native water and in a dispersed settling medium. The collection of "duplicate" samples and the analysis of one sample in native water and the other sample in a dispersed medium is not recommended. If two or more consecutive samples are necessary to obtain a sufficient quantity of sediment, they should be combined and then split for each type of analysis. The pipet method is recommended for native water size analyses, using the depths of withdrawal shown in table 6. The concentration of material finer than 0.062 mm should be in the range of 2,000 to 5,000 mg/l.

The 0.062 mm size should not be determined by the pipet method in the native water media if this value can be determined either by the VA tube or sieve method. The reason for this is that residual turbulence, caused by the preparatory mixing prior to the first withdrawal, lasts throughout most of the theoretical settling time required for the 0.062 mm withdrawal; in other words, true terminal fall velocity is not achieved because any tendency for natural flocculation is retarded.

Except for rare instances, it is recommended that the 0.002 mm size be determined in order to more accurately define correlations with respect to the clay mineral content of the sample.[2] Although some primary minerals exist as particles smaller than 0.002 mm, and some clay minerals exist as particles larger than 0.002 mm, separation at this point is believed to give an approximation of the clay mineral content of the sample.

If information concerning the chemical quality of the water is essential to a sediment investigation, standards and methods currently followed in chemical quality laboratories (Rainwater and Thatcher, 1959) should be used to make determinations for total dissolved solids, specific conductance, pH, calcium, magnesium, sodium, potassium, bicarbonate, or alkalinity. These analyses of the water can supplement knowledge obtained by other kinds of sediment examination.

Particle-size determinations by three different methods may be desirable for some streams. In addition to the standard methods using native water and dispersed medium, a third method would utilize a "weighted yearly average water" with respect to dissolved chemical constituents as the settling medium. Preparation of the "average water" would require past records of chemical quality data for the stream and would require that chemical laboratory facilities be available. The extra analyses would require the availability of samples with sufficient sediment for a three-way split.

Sample preparation for native water analysis

The preparation of samples for dispersed conditions is discussed in connection with each method of analysis in later sections. The preparation of a sample of which part is to be analyzed in native water needs further consideration. All compositing, splitting, wet sieving, or other steps involved in preparation of the portion of the sample to be analyzed in native water must be accomplished with native water. Distilled water, dispersing agents, or flocculating agents should not be added. Organic matter should not be removed by use of oxidizing agents or other methods. Mechanical dispersion of the sample should be avoided, and only a small amount of agitation should be used to remove the sediment from the sample bottle. Immediately prior to the analysis, the suspension should be stirred by hand for one-half the recommended time for dispersed conditions—30 seconds with the hand stirrer for the pipet method, and 90 seconds of tilting for the BW tube method.

Either the modified Jones-Otto splitter or the BW tube sample splitter is recommended for samples that contain no sand. The qualitative and quantitative accuracy of the modified Jones-Otto splitter is greater than that of the

[2] The hydraulic engineer thinks of "clay" primarily as a particle-size term. However, in some scientific fields, "clay" is primarily a mineralogical term with particle size a secondary consideration. As pointed out by Grim (1953), "The nonclay minerals usually are not present in particles much smaller than about 0.001 to 0.002 mm. A separation at 0.002 mm is frequently about the optimum size for the best split of the clay-mineral and nonclay-mineral components of natural materials."

BW tube splitter if the sample contains sand. Some samples may require several splittings in order that the desired quantity of sediment can be obtained to prepare a suspension having a concentration between 2,000 and 5,000 mg/l for the pipet method and 1,000 and 3,500 mg/l for the BW tube method. Skill and experience are necessary to estimate the number of splittings necessary to yield the required concentrations. The alternative is to collect extra samples for concentration determinations prior to the required splitting for size analysis.

Samples containing suspended material that settles extremely slow create an undesirable laboratory time lag. Usually a "sample" consists of 2–12 bottles (about 400 g each) of water-sediment mixture. After the compositing has been accomplished, the sediment in the composite must settle before an analysis can be made in a dispersed medium or before splitting can be accomplished prior to analyses in both native and dispersed media. Therefore, two settling periods are usually necessary prior to analysis: one before compositing, the other before splitting. It is recommended that suspended material be allowed to completely settle prior to each of these steps. In the event that the sample data is needed immediately, the following procedure is recommended:

1. Allow the samples to settle as much as practical.
2. By use of a Berkefeld filter tube (or a similar filter tube) and a vacuum apparatus, remove as much of the native water from each bottle as possible without disturbing the sediment in the bottom of the container. The fine sediment remaining in suspension adheres to the outside of the filter tube and the water passes through the walls.
3. Back flush the material on the outside of the tube with native water and carefully rinse it into the container to be used for compositing the sediment.

As an alternative procedure for treatment of samples that do not settle, the following method has been used successfully. Allow the samples to settle for several days and then, using extreme care not to agitate the sample, siphon off the top two-thirds to three-fourths of the water and fine sediment mixture. Determine the weight of sediment in an aliquot of the decanted water and, from this weight, compute the concentration of fine sediment particles removed from the sample. Split the sample if necessary to obtain the desired quantity of sediment, transfer to the analysis cylinder, fill with filtered sediment-free native water, and make the analysis as usual. Each withdrawal must then be corrected for the fine sediment previously decanted and not included in the analysis cylinder. The correction would be made in a manner similar to that for the dissolved solids correction except that the correction would be added to the weight of the withdrawal instead of being subtracted. Obviously, the same correction would also have to be applied to each withdrawal for the analysis made in the dispersed settling media using an alternate split of the same sediment. This method has an undesirable aspect because all the sediment sizes present in the original sample are not present in the part of the sample to be analyzed. Therefore, the analyses will not show the chemical and (or) physical effect of the very fine particles in suspension on the larger particles present.

Procedure for the Sieve-Pipet Method of Particle-Size Analysis

The pipet method of determining particle-size gradation for sizes finer than 0.062 mm is one of the most widely accepted techniques utilizing the Oden theory and the *dispersed system* of sedimentation. The upper size limit of sediment particles which settle in water according to Stokes law and the lower size limit which can be determined readily by sieves is about 1/16 mm or 0.062 mm. This size is the division between sand and silt (table 2) and is an important division in many phases of sediment transport phenomena.

The fundamental principle of the pipet method is to determine the concentration of a suspension at a predetermined depth as a function of settling time. Particles having a settling velocity greater than that of the size at which separation is desired will settle below the point of withdrawal after elapse of a certain time. The

Table 6.—Time of pipet withdrawal for given temperature, depth of withdrawal, and diameter of particles

[The values in this table are based on particles of assumed spherical shape with an average specific gravity of 2.65, the constant of acceleration due to gravity=980, and viscosity varying from 0.010087 at 20° C to 0.008004 at 30° C]

Diameter of particle, mm	0.062		0.031				0.016		0.008		0.004		0.002			
Depth of withdrawal, cm	15	10	15		10		10		10		5		5		3	
Time of withdrawal	(sec)	(sec)	(min)	(sec)	(min)	(sec)	(min)	(sec)	(min)	(sec)	(min)	(sec)	(hr)	(min)	(hr)	(min)
Temperature (° C):																
20	44	29	2	52	1	55	7	40	30	40	61	19	4	5	2	27
21	42	28	2	48	1	52	7	29	29	58	59	50	4	0	2	24
22	41	27	2	45	1	50	7	18	29	13	58	22	3	54	2	20
23	40	27	2	41	1	47	7	8	28	34	57	5	3	48	2	17
24	39	26	2	38	1	45	6	58	27	52	55	41	3	43	2	14
25	38	25	2	34	1	42	6	48	27	14	54	25	3	38	2	11
26	37	25	2	30	1	40	6	39	26	38	53	12	3	33	2	8
27	36	24	2	27	1	38	6	31	26	2	52	2	3	28	2	5
28	36	24	2	23	1	35	6	22	25	28	50	52	3	24	2	2
29	35	23	2	19	1	33	6	13	24	53	49	42	3	19	1	59
30	34	23	2	16	1	31	6	6	24	22	48	42	3	15	1	57

time and depth of withdrawal are predetermined on the basis of Stokes law. Table 6 gives recommended times and depths of withdrawal to determine concentrations finer than each of six size increments from 0.002 mm to 0.062 mm and for a range of water temperatures. Values are given for the standard depths of withdrawal of 10 cm for the 0.062 to 0.008 sizes and 5 cm for the 0.004 and 0.002 sizes as well as 15 cm for 0.062 and 0.031 sizes and 3 cm for the 0.002 size. The alternate depths allow greater flexibility in programing; greater depth for the coarse sizes increases accuracy and shallower depth for fines decreases time required to complete the set.

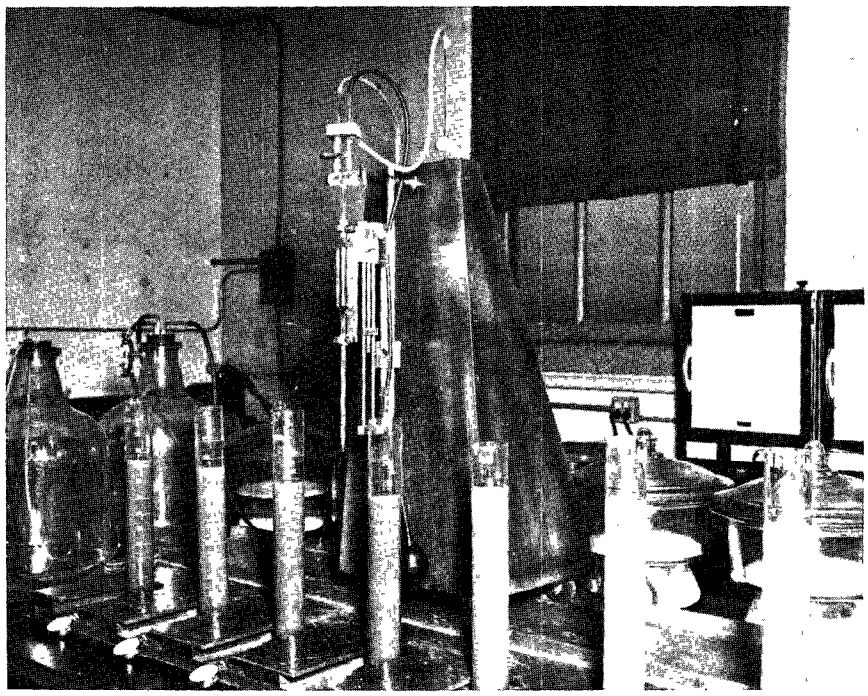
Equipment

The pipet equipment described by Krumbein and Pettijohn (1938, p. 165–167) is satisfactory when only relatively few samples are analyzed. Most sediment laboratories, however, need adequate equipment for analyses of many hundreds of samples each year. To facilitate the analysis of so many samples, the apparatus shown in figure 7, consisting of a rack built to accommodate eight sedimentation cylinders, is suggested. A movable carriage containing the pipet is installed behind the rack to enable pipetting from all the sedimentation cylinders. A mechanism is also attached to the carriage for conveniently lowering and raising the pipet.

In laboratories where the temperature varies considerably, it is desirable to use a constant-temperature water bath for the sedimentation cylinders. If a temperature is chosen that is always above the laboratory air temperature, then only a heating element and control is required. The constant-temperature operation allows the use of the same withdrawal schedule for all analyses. It is then possible to preprint the temperature, fall distance, and settling time on the pipet form (fig. 8).

Satisfactory use of the pipet method requires careful and precise operation to obtain maximum accuracy in each step of the procedure. Shown also in figure 7 is a sketch of the arrangement of the apparatus indicating the use of a 25 ml pipet equipped with a three-way stopcock. Rubber tubing of sufficient length to allow the pipet carriage to traverse the length of the rack is connected to the left stem of the stopcock and to a vacuum bottle and pump. A small, adjustable screw clamp on a short length of rubber tubing just above the pipet forms a constriction which helps to maintain a uniform rate of withdrawal in the proper length of time.

Attached to the right stem of the stopcock with rubber tubing is an inverted Y-shaped glass tube. A length of rubber tubing, with a pressure bulb at one end, is attached to the top stem of the Y. A length of rubber tubing is attached to the short stem of the Y connecting the apparatus to a distilled water supply with a head of 1–1.5 meters. The flow of distilled water is controlled by a stopcock.



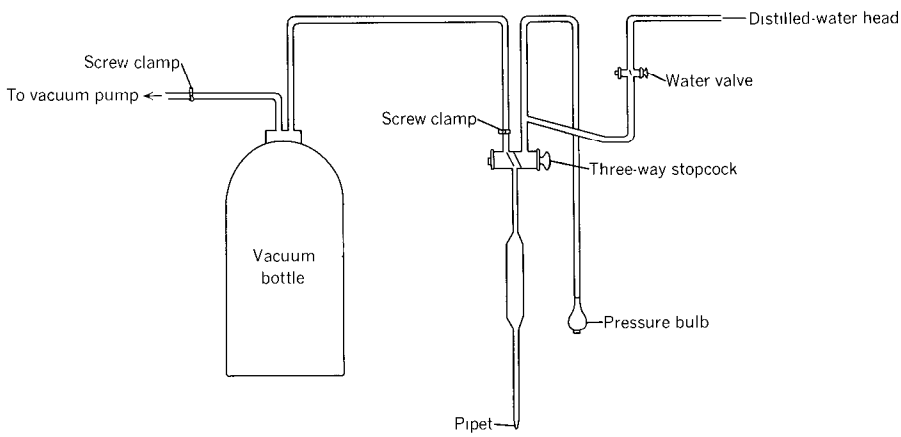


Figure 7.—Apparatus for pipet particle-size analysis.

After a sample is drawn into the pipet, the three-way stopcock plug is rotated 180°, and the sample is allowed to drain freely into an evaporating dish. The drainage can be accelerated by use of the pressure bulb. To insure complete removal of all sediment in the pipet, the distilled-water valve is then opened, and the pipet is washed out from the top. When the rinse is complete, it may be necessary to blow the remaining one or two drops of water from the tip of the pipet with the pressure bulb. At this time, any small quantity of the mixture that may have collected in the vacuum line near the three-way stopcock (resulting from accidental overfilling of the pipet) should be removed by allowing a small quantity of air to be sucked through the line from the open pipet. Removal of this water is essential to clear the constriction controlling the vacuum on the pipet and insure a uniform rate of withdrawal of the next sample.

A more simple "constant-vacuum device" is being used for pipet analyses in some laboratories. This device replaces the constriction (screw clamp) shown in figure 7 and insures a more uniform vacuum during a single withdrawal as well as for all analyses. The arrangement consists of three lines instead of two to the vacuum bottle. The third line is connected to a constant-head cylinder that has been sealed with a two-hole stopper and partly filled with water. The cylinder then acts as a "bleeding" device because the vacuum in the bottle cannot be in excess of the head of water around the tube that extends from the outside through the stopper to the bottom of the cylinder. This head of water can be adjusted to give a desirable rate of filling of the pipet.

Preparation of sample

Recording and decanting

The procedure for analysis of the wet sample requires the net weight of the water-sediment mixture for the entire sample. This weight is usually recorded on the form *Sediment Concentration Notes* (fig. 4 or 5) before samples are selected for particle-size analysis. The number of bottles and their composite net weight are recorded in the upper righthand block of the *Sieve-Pipet* form. (See fig. 8.) The name of the stream, the date, and other pertinent data are also recorded in this block.

After the sediment has settled to the bottom of the bottles, decant as much clear supernatant native water as possible. In the event that the sediment does not settle in a reasonable time and if the particle-size information is needed immediately, see "Note A (p. 11)" concerning special procedures for handling unsettled samples. The sediment in the individual sample bottles is then composited.

Mechanical dispersion

Sample splitting may be required because of excess silt and clay in the sample, but very rarely because of excess sand. Experience shows that splitting inaccuracies are much more likely to occur in splitting sand fractions than in splitting silt-clay fractions. Therefore, the following procedure is recommended whether or not splitting is required. If the sample contains significant organic matter, the organic matter should be removed using the procedure discussed in the following note.

NOTE B.—In close agreement with accepted methods, the following procedures set forth for removal of most forms of organic material from soil and sediment samples: Add 5 ml of 6 percent solution of hydrogen peroxide for each gram of (dry) sample which is contained in 40 ml 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.

It should be remembered that if a native-water analysis is to be made, then the organic material must not be removed. After completion of the necessary steps in this procedure, the composited sediment is placed in the soil dispersion cup and diluted to about 250–300 ml with distilled water. The sample is then mixed for 5 minutes with a commercial milk-shake mixer. The stirring paddle should operate not less than three-fourths nor more than 1½ inches above the bottom of the dispersion cup and turn about 10,000 rpm without load. An automatic timer

Form 9.265h (REV. 10-62)

UNITED STATES DEPARTMENT OF THE INTERIOR

Geological Survey-Water Resources Division

PARTICLE SIZE ANALYSIS, SIEVE-PIPET METHOD

File no. _____

ANALYSIS DATA

Date _____ by _____

Portion used _____

Disp. agent _____ cc.

Pipet suspen.: Sed. _____ gm.; Vol. _____ cc.; Concen. _____ ppm

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

Weight: Sieve fract. _____ gm.; Sand fract. _____ gm.; Pipet fract. _____ gm.; Silt-clay _____ gm.; Total sed. _____ gm.

DISSOLVED SOLIDS

Volume _____ cc. dispersed / native

Dish no. _____

Gross _____ gm.

Tare _____ gm.

Net _____ gm.

WEIGHT OF PORTION NOT ANALYZED

Portion _____ Dish no. _____

Gross _____ gm.

Tare _____ gm.

Net _____ gm.

Dis. solids _____ gm.

Net _____ gm.

TOTAL SAMPLE DATA

Stream _____

Location _____

Date _____ Time _____

G.H. _____ Sta. _____ Temp. _____

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

Dis. solids _____ ppm

Spec. cond. _____ pH _____

Other chem. qual. _____

Remarks

SIEVE

Size, mm	4.0	2.0	1.0	0.50	0.25	0.125	0.062	Pan
Container no.								
Weight-gm: Gross								
Weight-gm: Tare								
Weight-gm: Net								
% of total								
% Finer than								

PIPET

Pipet no. Volume: Volume factor:

Size, mm	Conc.	0.062	0.031	0.016	0.008	0.004	0.002	Resid.
Clock time								
Temperature								
Fall distance								
Settling time								
Container no.								
Weight-gm: Gross								
Weight-gm: Tare								
Weight-gm: Net								
Weight-gm: D.S. Corr.								
Weight-gm: Net sediment								
Weight-gm: Finer than								
% finer than								

Figure 8.—Sample of laboratory form, Particle-Size Analysis, Sieve-Pipet Method.

will help assure a consistent mixing time for each sample; the wet-sieve separation of sand from silt and clay immediately follows the mixing.

Sieving

Only in exceptionally rare cases is it necessary or desirable to split the suspended-sediment sand fraction prior to sieve analysis. The samples usually contain a very high concentration of fine material and only small amounts of sand. A minimum of about 0.02 g of sand is required for an accurate sieve analysis—more is required if the sample contains particles of 1.0 mm or larger; therefore, the following procedure is recommended in which all of the sand is removed from a sample before the fine material is split.

Separation of sand from fines

After the mechanical dispersion is completed, the sediment should be wet-sieved using distilled water and a 250-mesh (0.062 mm) sieve. The sieve is tilted, rotated, and tapped gently to facilitate the washing procedure. The washing must be continued until there is no sediment passing through the screen. The material passing through the sieve can be temporarily stored in a beaker of suitable size. The material retained on the sieve is then washed into an evaporation dish and oven-dried for 1 hour after all visible water has been evaporated. Drying time can be shortened if practically all the supernatant water in the dish is carefully poured off the sand before placing the dish in the oven.

Dry method

The dried sand is then removed from the oven, cooled in a desiccator, and weighed; the weight is recorded in the *Analysis Data* block of the *Sieve-Pipet* form. The dry sand can then be brushed into a nest of 2½- or 3½-inch diameter certified sieves to obtain separates finer than 4, 2, 1, 0.50, 0.25, 0.125, and 0.062 mm. The sieves are then shaken for 10 minutes on a shaker having vertical and lateral movements. The weight of sand remaining for each size fraction is recorded in the *Sieve* block. The sediment that passes the 0.062 mm sieve (pan material) is added to the silt-clay material that was obtained during the wet-sieve operation.

Wet method

If the sample contains considerable clay that cannot be completely removed from the sand particles (see p. 20 concerning the "Sonifier"), then a wet-sieve method may be more suitable than the dry sieve. The recommended method uses a technique that keeps the sieve and sand completely submerged. The equipment may consist of six or more 10 cm dishes, a set of 3-inch (7.6 cm) sieves, and a thin glass tube. All sieves are washed with a wetting solution such as alcojet and then rinsed with distilled water that will leave a membrane of water across all openings. The first or largest sieve is immersed in a dish with distilled water to a depth of about one-fourth inch (one-half centimeter) above the screen. If the surface tension of the water across the openings is sufficient to trap a pocket of air beneath the screen, the thin tube is used to blow out a small group of the membranes near one edge of the screen. This will allow the air to escape if the open holes are kept above the water until the rest of the screen is immersed. The sediment is washed onto the wet sieve and agitated somewhat vigorously in several directions until it is evident that all of the passable material has had a chance to fall through the sieve. Material retained in the sieve is washed into an evaporating dish to be dried and weighed, and material passing the sieve with its wash water is then poured onto the next smaller size sieve into another dish. This procedure is continued until the 0.062 mm sieve is used, after which the material passing the 0.062 mm sieve is added to the material obtained during the initial separation of the fines from the sands.

A more simple wet-sieve method is often used when the total sand is less than about 20 percent of the total. In this method the sieving is accomplished with a gentle jet of water that washes the finer particles through the successively smaller sieves. Though not as accurate and consistent as the "immersed screen" method, it is sufficiently accurate when the sand is less than 20 percent of the total.

Analysis of silt-clay fraction

Splitting

If a sample contains an estimated 5 g or less of silt-clay, and if a single dispersed analysis is desired, splitting of the material is not necessary. For example, if an estimated 4.5 g of material is present, the entire amount of material should be analyzed in a 1,000 ml cylinder; the material should not be split and analyzed in a 500 ml cylinder.

The following procedure is recommended if the sample contains more than 5 g. The silt-clay fraction, composed of the material passing the 0.062 mm "wet-sieve" plus the "pan material" obtained during the sieve analysis of the sand, is split as required to obtain a concentration of 2,000–5,000 mg/l in a 1,000 ml sedimentation cylinder. Either the modified Jones-Otto splitter or the BW tube splitter is satisfactory for the splitting of wet-silt-clay samples. If the dissolved solids concentration is still high, which is unlikely if the previous recommendations have been followed, it may be necessary to rinse the sediment until the final concentration of dissolved solids is less than about 200 mg/l.

Dispersing

To insure complete dispersion of the particles, add to the sample 1 ml of dispersing agent (see Kilmer and Alexander, 1949 or Note C below) for each 100 ml of the desired volume of the suspension.

NOTE C.—The dispersing agent is made by dissolving 35.70 g of sodium hexmetaphosphate and 7.94 g of sodium carbonate in distilled water and diluting to one liter volume. The sodium carbonate gives a pH between 9 and 9.5 for the mixture and acts as an alkaline buffer to prevent the hydrolysis of the metaphosphate back to orthophosphate. A determination of a dissolved solid's correction should be made each time a new solution of dispersing agent is prepared.

After the dispersing agent is added, transfer the sample to the cup of the mechanical mixer and mix for 5 minutes. The sample is then transferred to the sedimentation cylinder and diluted to the desired volume.

To obtain the dissolved solids correction factor to compensate for the dispersing agent added, the following procedure is recommended. Add 5 ml of the dispersing agent solution to each of two 500 ml cylinders, and add distilled water to make up the 500 ml volume. Using the standard 25 ml pipet, make three withdrawals from each cylinder and place each withdrawal in a separate, previously weighed, evaporation dish. Using the same temperature standards as for sediment drying, evaporate the material in each dish and obtain the weight of residue in each dish. Because very slight variations in weight will be found for the six residues, the average weight is obtained and is used as the correction factor.

Pipetting

After the split portion or the unsplit silt-clay fraction has been transferred to the sedimentation cylinder, and before the actual pipetting begins, the temperature of the suspension, the depth of withdrawal (fall distance), the settling time, and the weights of numbered containers for each withdrawal must be recorded in the *Pipet* block of the form. The suspension is then stirred for 1 minute with a hand stirrer of the plunger type illustrated in Krumbein and Pettijohn (1938, p. 167), and the stopwatch is started when the stirrer is removed. The analysis should be made at nearly constant temperature to minimize convection currents.

The time and depth of pipetting (table 6) is determined from Stokes law on the basis of temperature of suspension and settling diameters for the sizes 0.062, 0.016, 0.004, and 0.002 mm. On some occasions it may be desirable to add withdrawals for the 0.031 and 0.008 mm sizes. The pipet is filled in 8–12 seconds and then emptied into an evaporation dish. One rinse from the pipet is added. The material in each evaporation dish is handled as indicated by steps 11, 12, and 13 (p. 17). The resulting weight is entered in the *Pipet* block of the form.

Calculation of results

Total weight of sediment in sample

The calculation of results from the sieve-pipet method requires the total weight of sediment in the sample; this weight can be determined by one of the following methods:

1. Evaporate the sample to dryness before analysis and determine the oven-dry weight to the nearest milligram. This method is not recommended because sediment when once dried is difficult to disperse and requires special treatment, especially a sediment that contains considerable quantities of clay minerals.
2. Determine the weight of the silt and clay fractions from the mean concentration and volume of the pipet settling suspension. The average concentration of the suspension is determined by making a "concentration withdrawal" immediately after mixing. The weight of sediment in the suspension cylinder is then added to the weight of the sand which was determined separately.
3. Determine the dry weight of sediment remaining in the suspension after all pipetting has been completed. To this add the dry weight of sediment in each pipet withdrawal and the dry weight of the sand fraction if it was separated.
4. Split the sample in two portions by means of a suitable splitting device. Evaporate one-half to dryness to determine the weight of sediment, and use the other half for the sieve-pipet analysis. The accuracy of this method depends on the quantitative and qualitative accuracy of the splitting operation which is subject to considerable error; therefore, the method is not recommended.

Methods 2 or 3, either singly or together, are recommended. Because of speed and ease of determination, method 2 is suitable for most samples. Occasionally, the weight of material in a suspension should be determined by both methods 2 and 3 as a check on method 2. This is accomplished simply by making the "concentration withdrawal" as suggested in method 2 and including the weight of material in this withdrawal in the method 3 determination.

Calculations

The form illustrated in figure 8 facilitates recording the data and calculating the results for the sieve-pipet method. The tabulation of the sand fractions into the usual form of percent finer than the indicated sizes is accomplished by using the total dry weight of all sediment in the sample.

The net dry weight of the sediment in each pipet withdrawal when multiplied by the volume factor, which is the ratio of total volume of suspension to volume of pipet, gives the weight of sediment in the suspension finer than the size corresponding to the time and depth of withdrawal. This latter value divided by the dry weight of the total sediment in the sample gives the percent of total sediment finer than the indicated size.

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

The visual-accumulation tube method fills a fundamental need in the process of obtaining data related to sediment transport. It is a fast, economical, and accurate means of determining the size distribution in terms of the fundamental hydraulic properties of the particles and the fall velocity or fall diameter. Not only does "sieve" diameter fail to indicate the desired hydraulic properties of a sediment sample, but the particle-size distribution may be in error because of irregularities in the size and shape of sieve openings, limitations on the time of sieving required to pass all particles, and possible adhesiveness of clay on the larger particles. Therefore, the VA tube-pipet method is recommended for the determination of the particle-size distribution of sediment for most streams. The VA tube method is especially adapted for size analysis of samples composed mainly of sand. Finer material, when its measurement is required, must be removed from samples by either wet sieving or by sedimentation methods. Sieving is employed to remove particles which are too large for measurement by the VA tube method. The importance of the objections to sieving is of little significance for particles too large for the VA tube.

The VA tube method may not be suitable for some streams that transport large quantities of organic materials such as root fibers, leaf

fragments, and algae. Extra care is also needed when a stream transports large quantities of heavy or light minerals such as taconite or coal. For such streams, the results for both the VA tube and sieve methods should be reported.

The VA tube method employs the *stratified system* of sedimentation as contrasted with the *dispersed system* of the pipet or the BW tube. In the stratified system, the particles start falling from a common source and become stratified according to settling velocities. At a given instant, the particles coming to rest at the bottom of the tube are of one "sedimentation size" and are finer than particles that have previously settled out and are coarser than those remaining in suspension. Specific details covering the equipment and methods of operation are not discussed here because a clear and concise operator's manual (U.S. Inter-Agency Committee on Water Resources, Subcommittee on Sedimentation, 1958) is furnished with each set of the apparatus. Likewise, the details of the development and calibration are contained in Inter-Agency Report No. 11 (U.S. Inter-Agency Committee on Water Resources, Subcommittee on Sedimentation, 1957). It has been shown that particles of a sample in the visual tube settle with greater velocities than the same particles falling individually, because of the effect of mutual interaction of the particles. The VA tube method is calibrated to account for the effects of this mutual interaction and the final results will be given in terms of the individual particles falling alone in a fluid of sufficient extent to avoid the effect of space limitations. The size-gradation analysis, therefore, is based on the *standard fall diameter* of the particles, which may be defined as the diameter of a sphere having a specific gravity of 2.65 and having the same uniform terminal settling velocity as the given particle. The relationship between fall velocity and fall diameter depends only on the relationship between the velocity of fall and the diameter of the sphere of specific gravity 2.65. The fall diameter of a particle is then independent of the type of material or specific gravity, the concentration in which it is found or analyzed, or the method of analysis.

The diameter concept is just as basic as fall velocity, and has the sole advantage of supplying a linear size tag by which the particle may be more readily visualized. For example, when a VA tube analysis shows that 65 percent of the material of a sample is finer than 0.125 mm, it means that if the entire sample were dropped, one particle at a time in a fluid of infinite extent, 65 percent of the particles by weight would have fall velocities less than that of a 0.125 mm diameter sphere with a specific gravity of 2.65 which may be allowed to fall under the same conditions.

The calibration of the recorded was based on the analysis of hundreds of samples having known fall velocities and makes allowance for the increased fall velocities when a sample containing hundreds of particles is settled instead of individual particles. This allowance is in the order of 10 percent for the coarser particles and about 30 percent for the finer particles. The calibration is for natural sands with the recommended size of tube as related to the amount or concentration of sample and particle size. If the VA tube method is properly used, the resulting gradation will be quite accurate.[3]

Equipment

Equipment for the VA tube method of analysis consists primarily of the special settling tube and the recording mechanism in addition to the usual laboratory equipment for sediment investigations. As shown in figure 9, the device consists of (1) a glass funnel about 25 cm long, (2) a rubber tube connecting the funnel and the main sedimentation tube with a special clamping mechanism serving as a "quick acting" valve, (3) glass sedimentation tubes having different sized collectors, (4) a tapping mechanism that strikes against the glass tube and helps keep the accumulation of sediment uniformly packed, (5) a special recorder consisting of a cylinder carrying a chart that rotates at a constant rate and a carriage that can be moved vertically by hand on which is mounted a recording pen and an optical instrument for tracking the accumulation, and (6) the recorder chart which is a printed form incorporating the fall-diameter

[3] For special particle shapes or for samples with specific gravities much different from 2.65, accurate analysis might require a special calibration to relate settling velocity, fall diameter, and linear size.

calibration (fig. 10). Drawings and further details are given in Inter-Agency Report No. 11 (U.S. Inter-Agency Committee on Water Resources, Subcommittee on Sedimentation, 1957) and in the operator's manual furnished with the apparatus.

Preparation of sample

The VA tube method is suitable for samples whose particles are mainly sand. As mentioned previously, if particles larger than about 1.2 mm are present in the sample, they should be removed by sieving. If clay or silt is contained in the sample, it is removed before analysis by wet sieving with a fine sieve, by settling the sample through a sedimentation tube, or by other means such as the ultrasonic treatment mentioned on page 20. The more thoroughly the clays and silts are removed from the sand, the simpler and faster will be the VA tube analysis although some coarse silt in the VA tube does not affect the accuracy of the analysis.

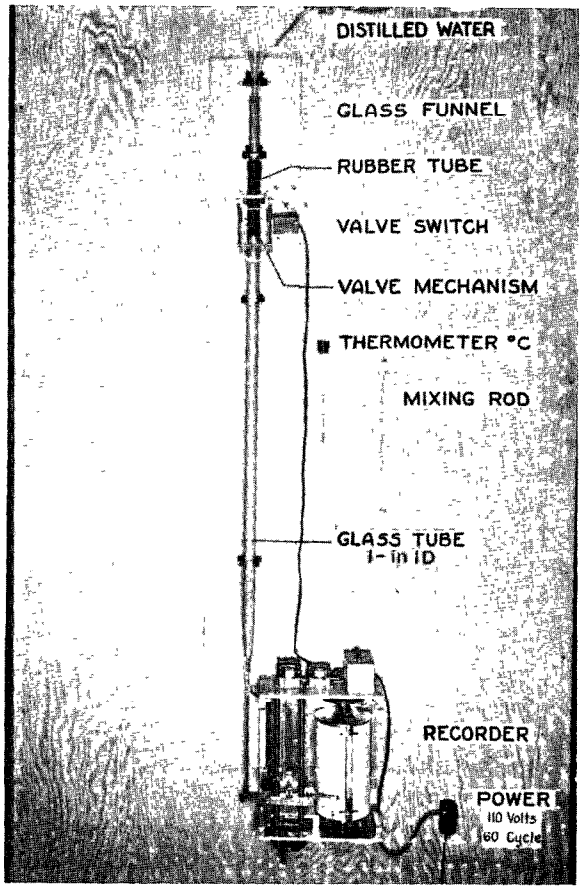


Figure 9.—VA tube and recording apparatus for analysis of sand-sized materials in terms of fall diameter.

Separation of sand from fines

The following method is recommended for the separation of sand from the silt-clay fraction. Separate the sand from the silt-clay fraction by the wet method using a sieve with 0.053 mm openings. If the material contains a large amount of sand, the use of a 0.062 mm or 0.125 mm sieve for an initial separation will help prevent rapid clogging of sieve openings in the 0.053 mm sieve. The material passing the coarser sieve is then wet-sieved through an 0.053 sieve. The amount of sand passing the 0.053 mm sieve will be very minute and the VA tube will give an accurate determination of the percentage finer than 0.062 mm.

An alternate method of separation of sands from the remainder of the sample is by sedimentation. This is accomplished by introducing the sample at the top of a sedimentation tube and allowing the material to settle for a time interval that will permit, for the given water temperature and distance of fall, all the particles with sedimentation diameters greater than 0.062 mm to settle to the bottom of the column. The part of the sample that has settled out can be withdrawn at this time and analyzed in the VA tube and the part remaining in suspension can be analyzed by the pipet method. Separation by simple sedimentation as a routine practice may be fraught with difficulty because (1) a sharp separation of sand from silt and clay during a single sedimentation period is extremely difficult to obtain and (2) the sample may contain a large amount of silt and clay which may move rapidly downward as a turbidity current. The first difficulty can be remedied by multiple sedimentation separations; the second by multiple sedimentation or preferably preliminary separation with the 0.053 mm sieve to remove most of the fine material in the sample prior to the final separation by sedimentation.

Before either the sieving or sedimentation methods can be used for separating the sand from the silt and clay, it is necessary that the net weight of the water-sediment mixture be obtained and recorded on *Sediment Concentration Notes* (fig. 4), that the supernatant native water be decanted, and that necessary compositing be accomplished. Obviously, if the silt-clay fraction is to be analyzed in both native and dispersed media, then the compositing, wet sieving, and splitting must be accomplished using only native water. Prior to analysis of samples selected for particle-size determination, all pertinent sample information previously recorded on *Sediment Concentration Notes* must be transferred to the charts used for the VA tube analysis. (See figs. 10, 11, and 12.)

Splitting and dispersing

A general discussion of methods for sample splitting is presented in a prior section of this chapter; however, some additional explanation is needed here concerning the reasons why splitting and (or) chemical dispersion should be accomplished after the sand is separated from the silt-clay fraction. The purpose of sample splitting is to obtain a concentration of 2,000–5,000 mg/l of silt and clay for the pipet analysis (1.0–5.0 g of silt and clay depending on whether 500 or 1,000 ml cylinder is used) and 0.05–15.0 g of sand for the VA tube analysis (depending on the diameter of the accumulation section of the tube). (See table 6.) The silt-clay fraction must be split but the sand need not be split for samples that contain an estimated 25–30 g of silt and clay and 5 g of sand. Quantitative and qualitative errors are much more apt to occur in the splitting of sand than in the splitting of silt and clay; fortunately, suspended-sediment samples rarely contain sufficient sand to require splitting. The separation of the sand from the silt-clay prior to the splitting of the sample requires little extra effort in computing the results of the analysis. The necessary records are maintained on the back of the VA tube form (fig. 12) in the block *Sample Fractions*.

The chemical dispersing agent should not be added to the silt and clay until the material is ready for the pipet analysis. For example, the dispersing agent in the suspension before separation by the sediment tube would obviously become mixed with the distilled water in the sedimentation tube, and an unknown quantity of the dispersant might be removed when the sand is removed from the bottom of the tube. The purpose of the chemical dispersing agent is not so much to separate the particles but to create a suspension having stability. Because the separation and splitting operations are accomplished rather quickly and do not involve sedimentation procedures, mechanical dispersion alone is an adequate preparatory treatment.

The VA tube analysis

The VA tube-pipet method differs from the sieve-pipet method only by the manner in which the sand is analyzed. The preparation of the sample, separation of sand from the silt-clay fraction, analysis of the silt-clay fraction, and calculation of results was discussed previously in the procedure for the sieve-pipet method. These and other general recommendations noted hereafter apply equally to the pipet analyses made in conjunction with either the sieve or the VA tube analyses.

Sand particles should be in such condition that the grains will fall as individual particles in the sedimentation tube and therefore should be thoroughly wet and free of attached clay particles or air bubbles before analysis. They should be contained in not more than 40 ml of water at a temperature within 2° or 3°C of the water in the VA tube. Organic matter should be removed before the sample is analyzed if present in sufficient volume to interfere with or decrease the accuracy of the analysis. A small number of root hairs, for example, although volumetrically small may have a considerable effect on the results of the analysis. Therefore, unless a sample is reasonably free of organic material, treatment for its removal is recommended. Methods for removal of organic matter are discussed on page 52.

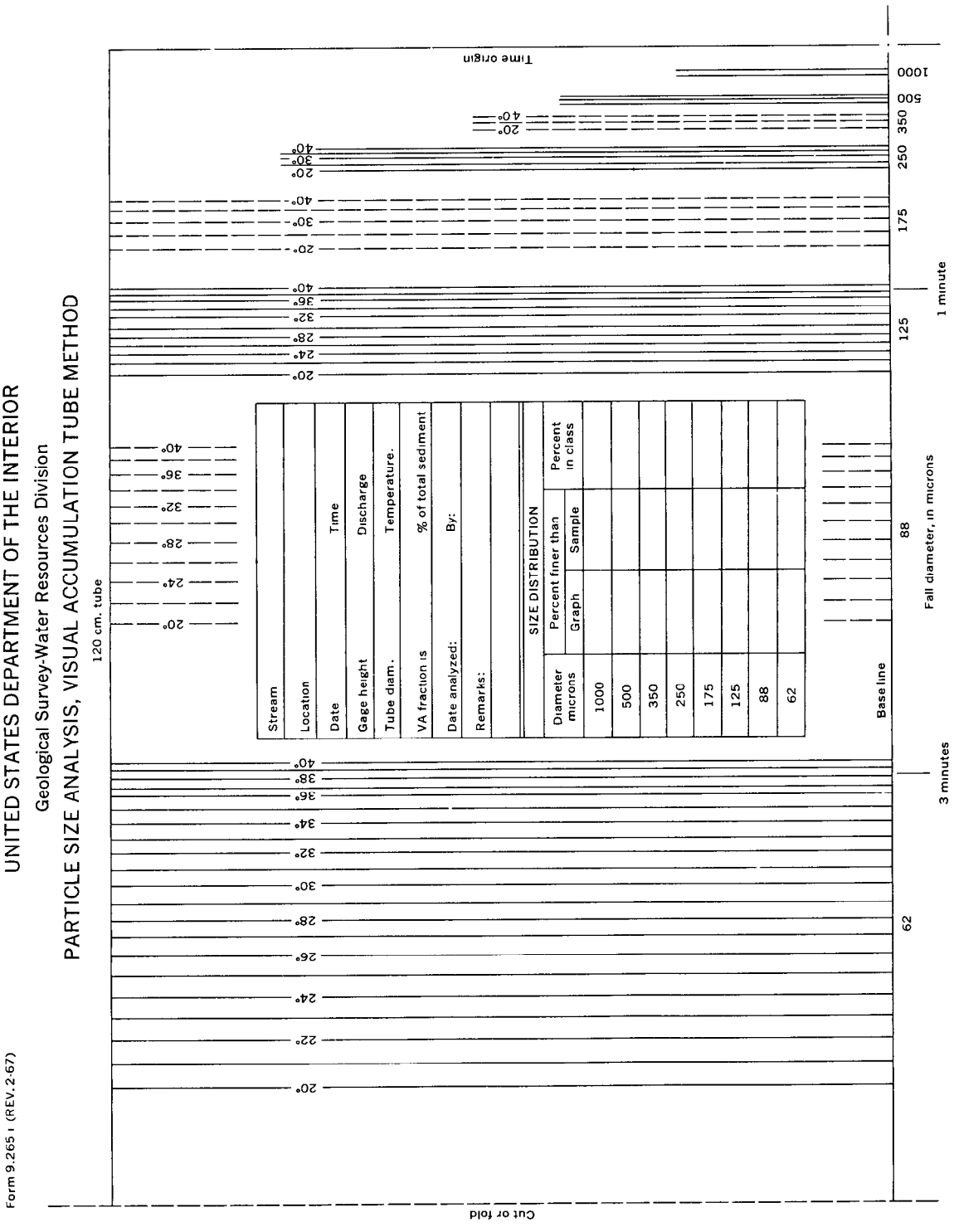


Figure 10.—Sample of laboratory form, Particle-Size Analysis, VA Tube Method for use with a 120-cm tube. The reverse side is designed for sieve and pipet analysis of the same sample. See figure 12.

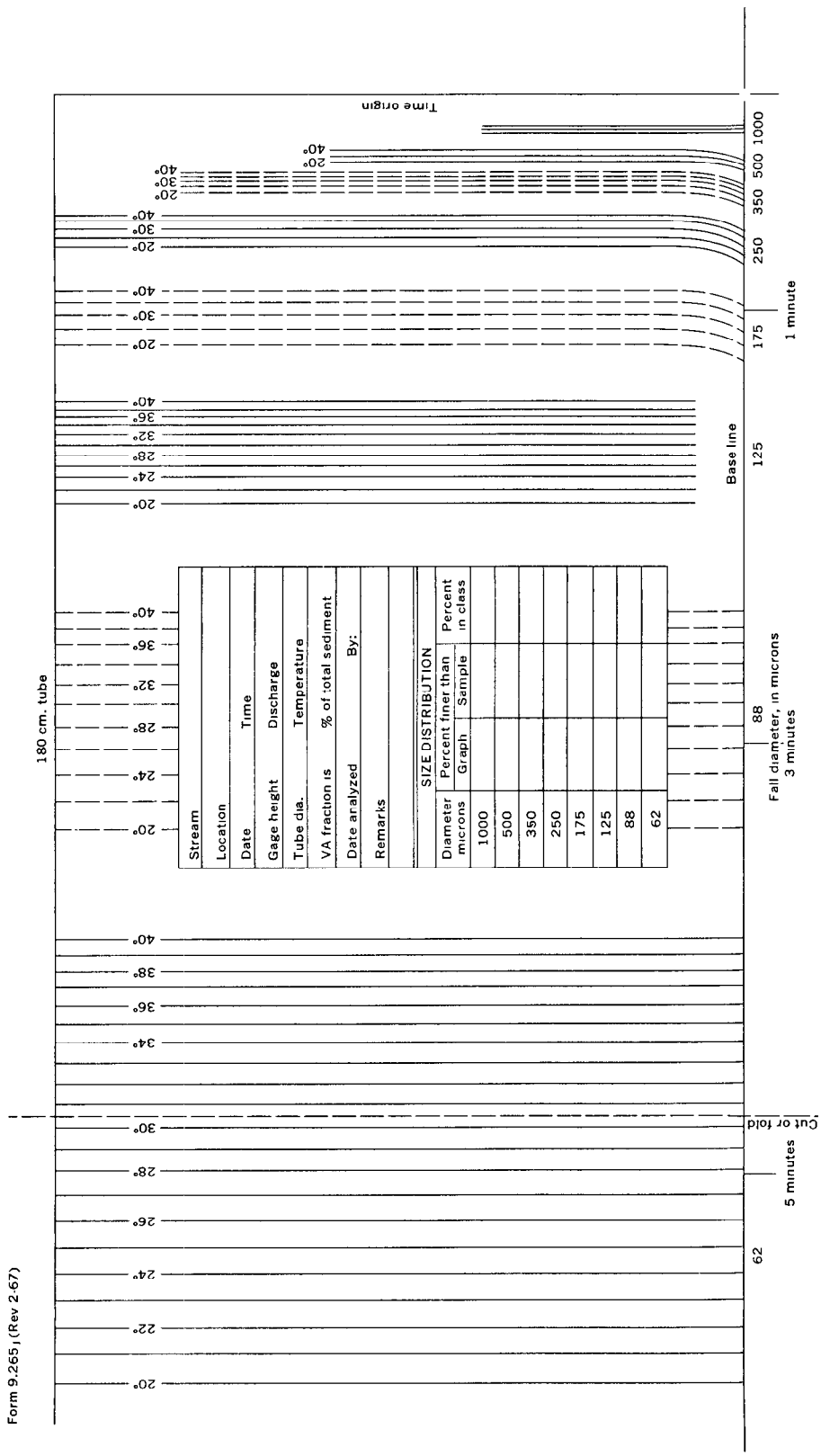


Figure 11.—Sample of laboratory form, Particle-Size Analysis, VA Tube Method for use with the 180-cm VA tube (size reduced about 35 percent). The reverse side is designed for sieve and pipet analysis of the same sample. See figure 12.

SIEVE ANALYSIS								
Size, mm								Pan
Weight gm.	Gross							
	Tare							
	Net							
% of total								
% finer than								
% in class								
Container no.								

Cut or fold

Stream	Date	Lab No.	
Location		Time	Gage height
Sampling stations	Start		
Method of sampling	Finish		
Remarks	Mean		
	Temp.	Mean vel.	
	Stream width	Depth	
	Discharge		

SAMPLE FRACTIONS								
	Total sample	Sieve fraction	VA-tube fraction	Pipet fraction	Remainder from split fractions			
					Total	Pipet	Other	
Portion								
Container no.								
Weight gm. Gross								
Tare								
Net								
% of total								

CONCENTRATION DETERMINATIONS								
	Total		VA-tube	Pipet		Dissolved solids		
Wt sample, gm.								
Container no.								
Weight gm. Gross								
Tare								
Net								
Concentration								
Analysis remarks								

PIPET ANALYSIS								
Pipette no.					Volume		Volume factor:	
Size, mm		0.062	0.031	0.016	0.008	0.004	0.002	Residue
Clock time								
Temperature								
Fall distance								
Settling time								
Container no								
Weight gm. Gross								
Tare								
Net								
D. S. Corr.								
Net sed.								
Finer than								
% finer than								
% in class								

Figure 12.—Sample of laboratory form for recording steps in computing particle size when the VA tube is used with sieve, pipet, and (or) other methods of analysis. Reverse side of 120- and 180-cm VA tube charts (figs. 10 and 11).

Tube selection

Best results from the VA tube analysis are obtained if the total height of accumulation in the bottom of the tube is between 4 and 12 cm. If a sample is predominately coarse or has a very limited size range, the maximum accumulation should be less than 10 cm. The choice of a suitable tube is not difficult because the usable limits of the respective tubes overlap, and even if a satisfactory size is not selected the first time, the samples can be rerun in another size of tube. Table 5 (p. 19) is arranged according to quantity and maximum particle size and should prove helpful in selecting the correct tube size. The maximum particle sizes in this table are those that should not be exceeded by a significant percentage of the sample. This significant percentage may be greater if the sample is small in relation to the capacity of the tube or if the analysis of the coarser portion is not highly important.

Tube selection is made easier in some laboratories by use of an "initial break tube" that is similar to the VA tube except the fall distance is about 50 cm. Use of this tube makes it possible to obtain (1) the size of the largest one or two particles by timing their fall, (2) the amount of sand accumulated or sample size, and (3) a cleaner sand sample by removal of silt and clay particles. The disadvantages of using the "initial break tube", though not serious, involve the problems of additional handling and chance for loss of sediment, and the fact that the fines are dispersed in another volume of water.

Procedure

The following procedure for making an analysis by the VA tube is reported in Inter-Agency Report No. 11 (U.S. Inter-Agency Committee on Water Resources, Subcommittee on Sedimentation, 1957, p. 118–120). It should be noted that analysis can be made in less than 10 minutes for samples with particles greater than 0.062 mm. The recommended chronological procedure for VA tube analysis is as follows:

1. The chart is chosen for the length of tube; after notes to identify the sample, operator, and analysis are recorded, the chart is placed on the cylinder. The base line of the chart should be parallel to the bottom of the cylinder so that the pen trace will be parallel to the base line except when sediment is accumulating. (If the chart is used for two or more samples, a different base line, somewhat above the original, may be used by drawing it with the recorder pen. The 180 cm and the 120 cm tubes require different charts because of the unequal distances through which the sample must settle.)
2. The recorder pen is oriented on the zero-accumulation and zero-time lines of the chart. The pen should be started to the right of the zero-time line and brought to the line by the motor-driven rotation of the cylinder.
3. The recorder is adjusted to bring the horizontal hair in the eyepiece level with the top of the tube plug where the accumulation of sediment begins.
4. When the apparatus, including the proper sedimentation section, is assembled, the tube is filled with distilled water to just above the valve. The temperature of the water in the tube is determined and recorded, and the valve is closed. Normally the water need not be changed after each analysis.
5. The electrical tapping mechanism is started; this operation also closes the electrical circuit to a switch at the valve so that rotation of the cylinder will start when the valve is opened.
6. The sand sample is washed into the funnel above the closed valve; the funnel is filled to the reference mark; then the sample is stirred briskly for 10 seconds with a special stirring rod.
7. The valve is immediately and fully opened. Because opening the valve automatically starts the cylinder, the chart time and settling of the particles in the tube begin simultaneously.
8. The operator watches through the eyepiece and, as soon as the first particles reach the bottom of the tube, he starts moving the carriage vertically at a rate that keeps the horizontal hair level with the top of the accumulation of sediment. This procedure continues until the pen has passed the 0.062 mm size on the chart; then rotation of the cylinder automatically stops. If material is stlil falling, the tracking operation is continued, at least intermittently, until the maximum height of accumulation is determined.
9. While the pen stands at the maximum height of accumulation, the cylinder drive clutch is released and the cylinder is rotated by hand to extend a horizontal line of maximum accumulation back across the chart to the time of zero accumulation.
10. After the valve is closed (and the tube plug is removed), the sample is (extruded from the tube into a beaker by gravity or by squeezing the rubber tube between the valve and the VA tube) * * * The valve (may then be) opened slightly to drain out excess water and to wash out the lower end of the tube more completely.

(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