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LABORATORY THEORY AND METHODS

(Chapter of proposed sediment manual)

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H. P. Guy and others

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INTRODUCTION

(Section I)

General.--A sediment laboratory in the Quality of Water Branch of the U. S. Geological Survey has two principal functions: 1) the determination of suspended-sediment concentration of samples collected from streams; and 2) the determination of particle-size distribution of suspended sediment, stream-bed material, and reservoir deposits. Data from these determinations are used to make computations of suspended-sediment discharge, computations of total sediment discharge, and computations of the probable volume and specific weight of reservoir deposits. These are but a few of the many uses made of concentration and particle size data of fluvial sediment. The reliability of the results of these computations and the utility of the data are dependent on the accuracy and reliability of the laboratory analyses of the samples.

In the determination of suspended-sediment concentration, the quantity and characteristics of the sediment, and the chemical quality of the native water influence the processing of the sample. Samples containing small quantities of sediment may make small errors in weighing and transfer of material especially significant; whereas, large quantities of sediment may result in splitting, drying, and weighing problems. Samples containing colloidal clay and certain types of native water result in difficulties in the separation of sediment from the native water. The quantity of dissolved solids determines if a correction is necessary when the evaporation method is used to determine suspended-sediment concentration. These and other problems encountered in the determination of suspended-sediment concentration are discussed in section II of this chapter.

The determination of the particle-size distribution of suspended sediment offers several problems in addition to those encountered in determining concentration. Again, the quantity of sediment is likely to be a problem; compositing of several samples may be necessary when the stream concentration is low and splitting of the sample may be necessary when the concentration is high so that a suitable quantity of sediment may be analyzed. As little as 0.05 gram of sand can be analyzed in the visual-accumulation tube, and 0.5 gram of sediment in the bottom-withdrawal tube. It is generally necessary to obtain the concentration of samples for which particle-size distribution is determined; therefore, provision must usually be made to obtain the weights of all withdrawals and portions not used in the analysis. Predrying for determination of total solids causes the formation of aggregates that resist redispersion; thus, the sample must be kept wet until after the particle-size analysis is complete. Division of the sample into two portions, one to be used for concentration analysis and the other for particle-size analysis, should be avoided unless there is sufficient quantity of suspended solids for both analyses. Even then, splitting may introduce some quantitative and qualitative errors that will affect either or both the concentration or particle-size results. Procedures for determining particle-size distribution of fluvial sediment by sieve and sedimentation methods are discussed in sections IV, V, and VI. The frequency of the analyses and the need for special procedures or methods for both native and dispersed settling media are discussed in section III.

The determination of particle-size distribution of bed-material samples is different from suspended-sediment samples in that 1) the quantity of material is usually large, 2) the samples are usually sand with a median size considerably larger than that for suspended-sediment samples, and 3) many samples will be in a room-dry condition. Unlike suspended-sediment samples, which may have very limited quantities of material available for analysis, the larger bed-material samples allow the use of optimum quantities of material for all size ranges. Sieves are generally used to determine particle-size distribution of material 2.0 millimeter and coarser. The visual-accumulation tube is generally used to determine particle-size distribution of material from 0.062 to 2.0 millimeter. The size and settling rate relationship is well known for spheres; particle size computed by this concept is referred to as sedimentation diameter. The specific gravity of quartz (2.65) is assumed for the material unless the material is shown to be significantly different. However, both specific gravity and particle shape vary depending on geologic and hydrologic characteristics of any given stream and drainage basin. Specific gravity and shape must be known in order to convert fall diameter to fall velocity or actual size.

Units of measurement and definitions.--The common unit for expressing suspended-sediment concentration is parts per million (ppm), and is computed as one million times the ratio of the weight of sediment to the weight of water-sediment mixture. Other units, such as percent, have occasionally been used to express suspended-sediment concentration but are not recommended. Total sediment concentration has in some cases been expressed on a volume basis, especially for sands and other coarse material. Such volume measurements are generally not reliable because of variations in particle characteristics and compaction, and the necessary assumption of a density for conversion of volume into weight.

Several measures of particle size (see note A) should be defined as the basis for simple, accurate, and practical determination and expression of sediment sizes. Some measures of particle size are difficult to evaluate and are seldom determined. Others are determined only approximately. Before the basic concepts of these measurements are discussed, several definitions relating to particle size are given as set forth in Report No. 12 (1957, p. 10).

Note A: The meaning of the term "particle" is ambiguous in a manner similar to terms in other branches of science. Consider any given sample of soil which contains a considerable number of relatively large aggregates; these aggregates are "particles", and many properties of soil depend on their size and stability. The aggregates in turn consist of grains which are also "particles" and which are commonly referred to as "ultimate particles" of soil. The size of the ultimate particles also determines many soil properties. These so-called ultimate particles are not truly ultimate because the grains may be composed of two or more crystals of the same or different minerals. Obviously it is important to specify which particles are measured. In sediment investigations, the ultimate particle size should be used unless otherwise specified; ultimate particle size can be defined for practical purposes as the particle size obtained by standard methods of preparation and dispersion of sediment samples.

"The NOMINAL DIAMETER of a particle is the diameter of a sphere that has the same volume as the particle.

"The SIEVE DIAMETER of a particle is the length of the side of a square sieve opening through which the given particle will just pass.

"The STANDARD FALL VELOCITY of a particle is the average rate of fall that the particle would finally attain if falling alone in quiescent distilled water of infinite extent and at a temperature of 24°C.

"The STANDARD FALL DIAMETER, or simply FALL DIAMETER, of a particle is the diameter of a sphere that has a specific gravity of 2.65 and has the same standard fall velocity as the particle.

"The SEDIMENTATION DIAMETER of a particle is the diameter of a sphere that has the same specific gravity and has the same terminal uniform settling velocity as the given particle in the same sedimentation fluid.

"The STANDARD SEDIMENTATION DIAMETER of a particle is the diameter of a sphere that has the same specific gravity and has the same standard fall velocity as the given particle.

"SIZE DISTRIBUTION, or simply DISTRIBUTION, when applied in relation to any of the size concepts, refers to distribution of material by percentages or proportions by weight.

"FALL VELOCITY and SETTLING VELOCITY are general terms which may apply to any rate of fall or settling as distinguished from standard fall velocity. . . ."

The basic concept of "size" of a sediment particle is expressed in terms of volume or the nominal diameter as per the definition above. When consideration of the density or mass of the particle is necessary, the specific gravity is used. It is recognized that sieves, frequently used for the separation of sediments into "size" grades, do not grade particles entirely by size but by shape as well. Ideally, sieving should show relative quantities of the sample which could pass sieves with stated sizes of uniform square openings; but actually, many irregular particles with nominal diameters much greater than the sieve openings will pass a sieve. The above definition of sieve diameter means that the sieve diameter of the particle is the length of the side of the smallest square opening through which the particle could pass and that the nominal and sieve diameters of a sphere are always equal.

The physical size of particles may be expressed (Report 12, p. 12) on the basis of the length of three diameters of the particle, a, b, and c, all mutually perpendicular. The axes are differentiated as, a = the longest axis of the particle, b = the intermediate axis of the particle, and c = the shortest axis of the particle. The harmonic mean diameter (Krumbein and Pettijohn, 1938, p. 128) is defined as $d_h = 3\ abc/(ab+bc+ac)$ and is considered to be closely related to diameters calculated from Stokes' law. In addition to the harmonic mean, the arithmetic mean $d_a = (a+b+c)/3$ and the geometric mean diameters $d_g = \sqrt[3]{abc}$ have been used to express size by various investigators. The important value of these physical measurements is in the expression of a particle shape factor. The shape factor $sf = c/\sqrt{ab}$ is useful in relating nominal diameters to fall velocity or fall diameter. Figure 1 shows the relation of naturally worn quartz particles to their fall velocity for shape factors of 0.5, 0.7, and 0.9 and at temperatures of 0°, 10°, 20°, 24°, 30°, and 40°C. The shape factor of 0.7 is about average for natural sediments.

The usefulness of shape factors is limited in routine sedimentation problems by the fact that even if the sieve, nominal, and axis length diameters are known, the fall diameter may be determined more easily and accurately through fall velocity data than by the shape factor. In summary, the factor of shape may be kept in proper perspective by remembering that for smooth quartz spheres all four diameters, nominal, sieve, axis length, and fall are the same; that the relative effect of shape varies with size; and that the effect of shape is more significant with sieve than with the sedimentation type of analyses, at least for the smaller sizes.

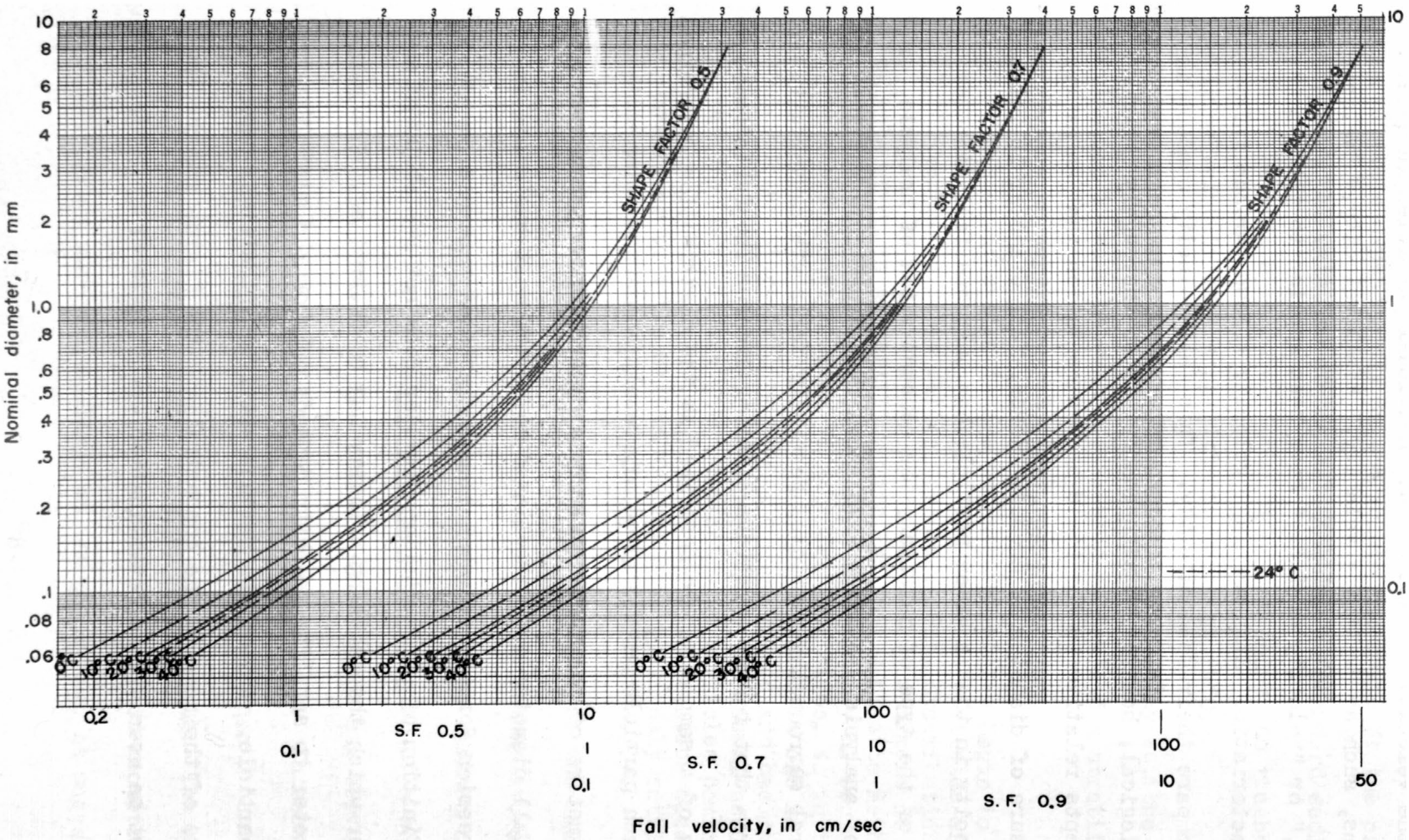


Figure 1.--Relation of nominal diameter and fall velocity for naturally worn quartz particles with shape factors of 0.5, 0.7, and 0.9 (from fig. 2 Report No. 12, 1957)

One of the dynamic properties necessary for sediment transport studies is the velocity of fall of the individual particles in quiet water. Terms, such as "settling velocity," "fall velocity," "velocity of settling," or "velocity of fall," have been applied to this fundamental characteristic. Standard fall velocity allows various investigators to compare their data directly. This fall-velocity concept is simple and logical; however, a size concept is so thoroughly imbedded in the concepts relative to the field of sedimentation that an expression or measure of diameter is needed. Hence, fall diameter is related to fall velocity in that a given particle has only one fall diameter, independent of the type of material, the concentration of analysis, or the method of analysis. Studies have shown that a particle settling as one of several surrounding particles generally falls with a velocity different from that for the particle settling alone. The fall diameter distribution of a sample may be described as the distribution determined as though each particle were dropped separately and a summation made of the fall diameters of the particles. This distribution is based on weight and fall diameter and is believed to be the most basic and desirable expression for the sedimentation analysis of a sample.

The definition of sedimentation diameter does not restrict the conditions under which the settling velocity may be determined. The relation of diameter to settling velocity depends only on the specific gravity of the particle. Any of the various settling velocities, depending on the nature of the sample, the concentration, the fluid, and the apparatus, must be used with a determination or estimate of specific gravity to establish sedimentation diameter. Standard sedimentation diameter of a particle depends only on the volume and shape of the particle. Therefore, the relation of nominal diameter to standard sedimentation diameter becomes a measure of the effect of shape and roughness on the settling velocity of the particle in water at 24°C.

With respect to methods of particle size analyses, the following distinction is made between a dispersed system and a stratified system as reported in Report No. 11 (1957, p. 13).

> "A dispersed system is one in which particles begin to settle from an initial uniform dispersion and in which particles of different sedimentation sizes settle together. Size distribution may be determined by measuring the concentration of sediment at given intervals of depth and settling time, as in the pipette method, or the distribution may be obtained from the quantity of sediment remaining in the suspension after various settling times, as in the bottom-withdrawal-tube method. A stratified system is one in which the particles start falling from a common source and become stratified according to settling velocities, as in the visual-accumulation-tube method. At any given instant, the particles coming to rest at the bottom of the tube are of one sedimentation size only and are finer than the particles that have previously settled out and are coarser than those remaining in suspension. . . ."

The size gradation scale or the distribution of the size classes is a descriptive function which serves to place nomenclature and terminology on a uniform basis. A grade scale has been defined by Krumbein and Pettijohn (1938, p. 76) as an arbitrary division of a continuous scale of sizes, such that each scale unit or grade may serve as a convenient class interval for conducting the analysis. In such a scale of sizes it is recognized that a natural sediment has a continuous size distribution without implication of natural grouping. The Wentworth grade scale is favored in America. This has a fixed geometric interval with each grade one-half as large as the preceding, as suggested by

Udden in 1898. Grade scales are without exception based on unequal class intervals due to the large range of sizes for most sediment. If equal intervals were chosen to give full significance to the smaller sizes, then an excessive number of classes would be necessary with the result of virtually no significance between classes for the larger sizes. The unequal class intervals may make the application of certain statistical analyses to the data more difficult. Table 1 gives the size limits for each class name of the recommended gradation scale.

Table 1.--Recommended scale of sizes for sediment analysis.

Class name	Metric units		English units (inches)
	(millimeters)	(microns)	
Boulders.........	>256	-	>10
Cobbles..........	256 - 64	-	10 - 2.5
Pebbles..........	64 - 4.0	-	2.5 - 0.16
Granules.........	4.0 - 2.0	-	0.16 - 0.078
Very coarse sand.	2.0 - 1.0	2,000 - 1,000	0.078 - 0.039
Coarse sand......	1.0 - 0.50	1,000 - 500	0.039 - 0.020
Medium sand......	0.50 - 0.25	500 - 250	0.020 - 0.0098
Fine sand........	0.25 - 0.125	250 - 125	0.0098 - 0.0049
Very fine sand...	0.125 - 0.062	125 - 62	0.0049 - 0.0024
Coarse silt......	0.062 - 0.031	62 - 31	0.0024 - 0.0012
Medium silt......	0.031 - 0.016	31 - 16	0.0012 - 0.00061
Fine silt........	0.016 - 0.008	16 - 8	0.00061 - 0.00030
Very fine silt...	0.008 - 0.004	8 - 4	0.00030 - 0.00015
Coarse clay......	0.004 - 0.0020	4 - 2	0.00015 - 0.000076
Medium clay......	0.0020 - 0.0010	2 - 1	0.00076 - 0.000038
Fine clay........	0.0010 - 0.0005	1 - 0.5	0.000038 - 0.000019
Very fine clay...	0.0005 - 0.00024	0.5 - 0.24	0.000019 - 0.000010

Specific gravity is the ratio of the weight of a substance to the weight of an equal volume of water at 4°C. The measurement is then dimensionless. It is sometimes confused with specific weight, often called density, which is defined as the weight per unit volume. Water with a weight of 62.4 pounds per cubic foot is an example.

Theory of particles falling in a fluid.--Table 2 illustrates the relative position of the finer particles commonly present in sediment samples with respect to the general subdivisions of matter. The table also contains a list of the properties for a given size range as well as related information such as the limit of the microscope and the diameter of pores of various types of filtering materials. Ordinarily, sediment size analyses are made only for material down to about 2 microns in the coarse suspensions category. Further separations would require excessive time and lack practical value in nearly all hydraulic engineering work; however, in many scientific fields a knowledge of the size distribution of material finer than 2 microns is useful.

Most methods of determining particle size distribution gradation are based upon the action of particles in fluids. The milling, ceramic, cement, abrasive, and paint industries accomplish the separation in a rising column of air. In the fields of geology, soils, and river hydraulics, quiet water is the most common settling medium. Whatever the medium, the principle involved results in the tendency for particles of similar specific gravity to fall at a rate that is related to the size and shape of the particle. Several laws or mathematical expressions have been developed for expressing the relation of particle size to its

Millimeters	1	0.001	0.0001	0.00001	0.000001
Microns (μ)	1,000	1	0.1	0.001	0.0001
Millimicrons (μμ)	1,000,000	1,000	100	1	0.1
Relative number of particles	1	10^9	10^{12}	10^{18}	10^{21}
Relative surface of particles	1	10^3	10^4	10^6	10^7

Qualities Studied	Coarse suspensions	Colloidal suspensions	Colloidal solutions	Molecular solutions
a. Appearance in water	Very cloudy	Turbid	Clear	Clear
b. Particles observed	With naked eye	With microscope	With ultramicroscope	Cannot be observed
c. Rate of settling	Quickly or overnight	Slowly or not at all	Do not settle	Do not settle
d. Particles separated from water	With filter paper	With clay filters	With ultrafilter	Not by filtration
e. Determination of particle sizes	Sieves, microscope, gravity or centrifugal settling	Microscope, centrifugal settling, absorption or dispersion of light	Ultramicroscope, ultracentrifuge	Ultracentrifuge
f. Form after evaporation	Loose powders	Powders and gels	Gels	Crystals
g. Soil separates	Sand ↑ Silt ↑ Clay 50μ 5μ	Suspended clay	Ultra clay	Soil solution

NOTES

Limit of microscope (ultra violet light)	100μμ
Limit of ultramicroscope	10μμ
Limit of ultrafilter	1μμ
Brownian movement starts at about	5μ
Diameter pores of hardened filter paper	1.5 to 2.2μ
Diameter pores of Chamberland filter	0.2 to 0.4μ
Diameter of bacteria	0.5 to 1.2μ
Diameter of 200-mesh particles	74 μ

Table 2.--The subdivision of matter and the resulting properties (from table 1, Report No. 4, 1941).

settling velocity. These, particularly Stokes' law, serve as a basis for many size separation techniques including the pipette and bottom-withdrawal-tube methods. In most analyses, either sieves or the visual-accumulation tube are used for 62 microns and larger. The latter has the advantage of being a hydraulic method and helps to insure continuity from the fine to the coarse particles. The following discussion assumes the settling medium to be water since it is the universal environment of fluvial sediment and it has desirable physical characteristics for analytical use.

The nature of particle movement in a settling medium depends on a balance of the accelerating and retarding forces, which in turn depends on the size, shape, and density of the particle and the viscosity and(or) turbulence induced in the fluid. The resistance to motion can be any one of three types of drag (Rouse, 1938), (Report No. 4, 1941, p. 24): (1) "deformation", (2) "surface", or (3) "form", depending on the interrelation of the characteristics of the particle and fluid. The "deformation" drag is the resistance due entirely to the viscosity of the fluid as it extends far into the fluid. Stokes' law expresses the fall of particles under such conditions. If the fall velocity of a particle is somewhat faster than this, then the inertial effects of the fluid reduces the viscous influence to a thin layer around the particle which produces the effect of "surface" drag. Larger sizes of particles result in the "form" type of resistance by the development of a wake behind the particle in which eddies are formed and the inertial effects of the fluid assume an increasing importance. The low pressure area in this wake results in a force that opposes the motion. Reynolds number is a measure of the ratio of inertial forces to the viscous forces.

Stokes' law as developed in 1851, states that the viscous resistance to fall of a particle in a fluid is equal to the net downward force of the particles. These forces are evaluated as

$$6 \pi r \mu v = 4/3 \pi r^3 g(\rho_s - \rho_f) \tag{1}$$

where r = radius of sphere in cm.
μ = dynamic viscosity of the fluid in dyne-sec./cm.^{2}
v = velocity of fall in cm/sec.
g = acceleration due to gravity (980 cm/sec./sec.)
ρ_s = density of sphere in gm./cm.^{3}
ρ_f = density of fluid in gm./cm.^{3}

The net downward force is the force of gravity on the spherical particle minus the buoyant force of the liquid,

$$\text{or} \quad 4/3(\pi r^3 \rho_s g) - 4/3(\pi r^3 \rho_f g)$$

The fall velocity as defined by Stokes' law is obtained from equation 1,

$$v = 2/9 \frac{(\rho_s - \rho_f)gr^2}{\mu} \tag{2}$$

Assuming that the specific gravity of the particles is 2.65 and that water is the settling medium, most investigators reduce equation 2 to

$$v = \frac{89.83\, d^2}{\mu} \quad \text{or} \quad d = \frac{\sqrt{\mu v}}{9.478} \tag{3}$$

where d = diameter of particle in cm. Figure 2 gives a graphical solution to the equation for a water temperature at 20°C. Viscosity correction factors for use with the graph when other temperatures are used are tabulated below:

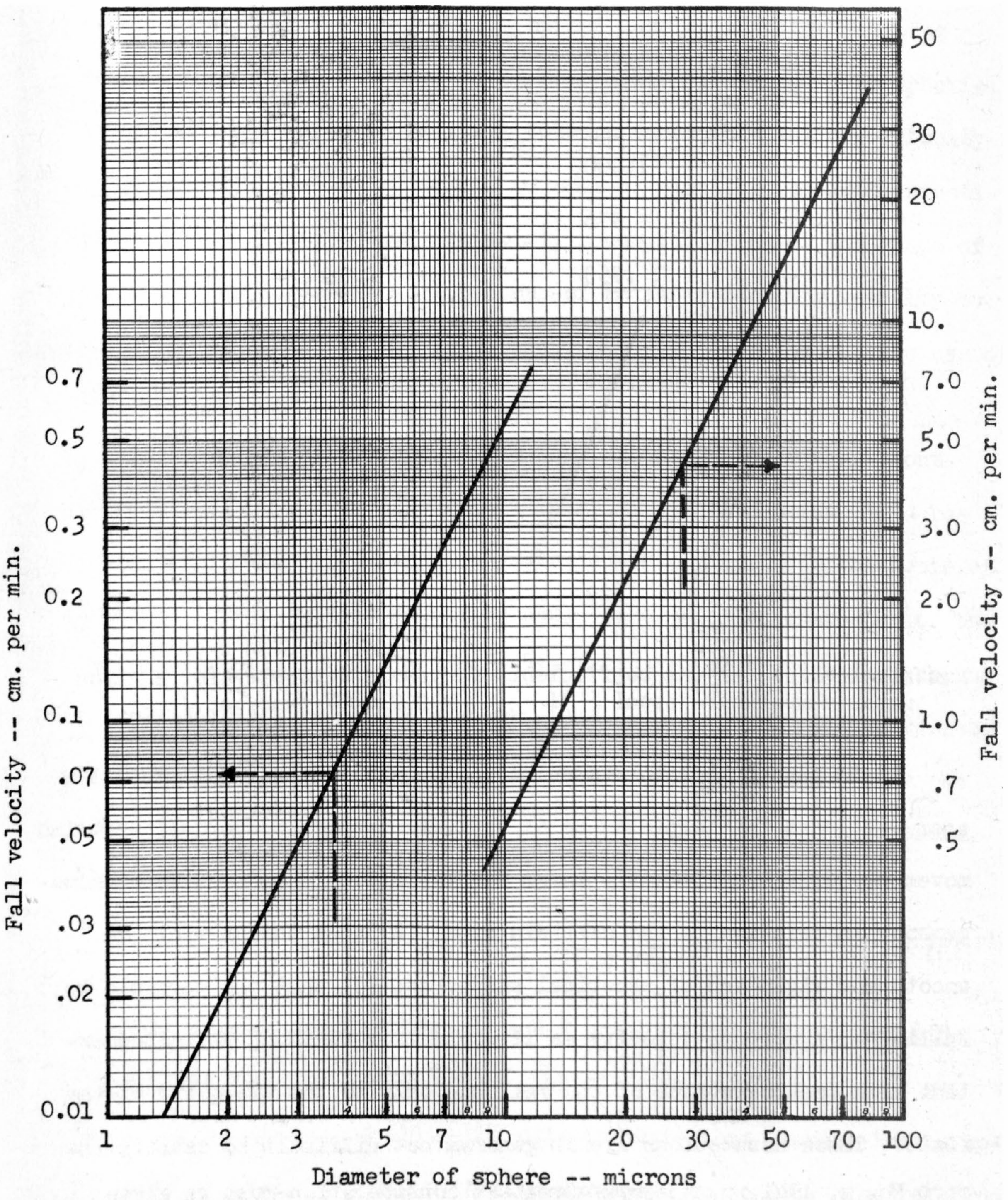


Figure 2.--Relation of fall velocity and diameter for quartz spheres in water at 20°C (redrawn from fig. 2, Report No. 4, 1941).

T (deg. C)	μ_T/μ_{20}	T (deg. C)	μ_T/μ_{20}
18	1.051	29	0.811
19	1.027	30	.794
20	1.000	31	.776
21	.976	32	.760
22	.953	33	.746
23	.930	34	.729
24	.908	35	.714
25	.887	36	.700
26	.867	38	.673
27	.848	40	.648
28	.829		

Numerical values of the coefficient of dynamic viscosity for a given water temperature equal (μ_T/μ_{20}) x 0.010087 dyne-sec./cm.^{2}

Basic assumptions to recognize in the application of Stokes' law are: (1) that the particles be of sufficient size to overcome Brownian movement, or be sufficiently large for the fluid to be considered homogeneous in relation to the particle size; (2) that the particle be smooth and rigid and of spherical shape; and (3) that the particle be falling at a uniform velocity as it would in a fluid of unlimited extent with the resistance of the fall due only to the viscosity of the fluid. These assumptions are in general not difficult to satisfy (Report No. 4, 1941, p. 33-36). Greatest consideration must be given to the effect of the proximity of particles to each other whereby the deformation effects extend into the path of nearby particles. It is also conceivable that a high concentration of a small group of particles, causing a localized area of higher density than the settling medium, may settle as a unit at an abnormally high rate. The sphericity of the particle is of no great concern since the particle velocity is referred to the sedimentation or nominal diameter for most investigations. From a practical viewpoint, a consideration of the shape of these fine particles for the purpose of relating settling velocity to physical size would be nearly an insurmountable task; hence, the use of the sedimentation diameter concept.

Stokes' law is not applicable for particles larger than about 60 microns (0.060 mm), because the inertial effects of the fluid reduces the viscous influence. Reynolds number R indicates the ratio of inertial forces to viscous forces and, therefore, the character of the resistance to fall. It is a dimensionless quantity, which for spherical particles falling in a fluid, is defined as $\frac{v d \rho_s}{\mu}$. The resistance to fall of these larger particles is $\frac{C_D A v^2 \rho_s}{2}$ where C_D is the coefficient of resistance and A = cross-section area of the particle. This resistance is equated to the net downward force, equation 1.

$$\tfrac{1}{2} C_D A v^2 \rho_s = \tfrac{4}{3} \pi r^3 g(\rho_s - \rho_f) \qquad (4)$$

The relationship between C_D and R has been established empirically for spheres and some other shapes (Report No. 4, 1941, p. 39 and Report No. 12, 1957, p. 21 and 51). The data indicate that Stokes' law is applicable up to R = 0.1 and with fair agreement up to 0.4. The relationship between C_D and R is of academic interest only because methods of analysis using sedimentation methods must be calibrated in terms of some definite, easily understood, and readily reproducible unit of sediment size. Standard fall velocity and fall diameter provide a simple foundation for the expression of the size distribution of samples. Particle size for these larger sizes is based on a comparison with actual samples from which the individual particle fall velocities have been determined.

PROCEDURE FOR DETERMINING SUSPENDED-SEDIMENT CONCENTRATION

(Section II)

The determination of concentration and particle-size distribution from suspended-sediment samples offers numerous problems. Such samples may contain only minute quantities of sediment of very small particle size; or, the sample may contain such large quantities of sediment that special procedures are necessary. Samples for particle-size analysis require special processing and computational procedures to insure the determination of the concentration as well as the particle-size distribution with only one drying of the sediment.

Each of the several methods for determining concentration has its advantages and disadvantages. The two most commonly used will be presented. They are the evaporation method and the filtration method. The filtration method may be somewhat faster than the evaporation method for some samples but it imposes a severe limitation on the quantity of sediment that can be analyzed. The evaporation method may require an additional step if the dissolved solids content is high, especially for samples having low concentration.

Evaporation method.-- The determination of total concentration by allowing the sediment to settle to the bottom of the sample bottle, decanting the supernatant liquid, washing the sediment into an evaporating dish, and drying it in an oven has advantages in the simplicity of equipment and technique. The method is satisfactory if the sediment is relatively coarse or if flocculation results in rapid settling of the suspended clay in the sample. Conversely, with suspensions of naturally dispersed clay, the settling time may be so great as to make the method impractical.

A flocculating agent may be used to reduce the settling time of these naturally dispersed clays. This will require the use of a correction factor to be applied to the weight of dried sediment. Such a factor is more of an estimate than an exact value because of the difficulty in determining the amount of flocculant incorporated in the final weight and the amount of dissolved material precipitated. Hence, a flocculant should not be used unless absolutely necessary and then only a minimum amount. As the samples decrease in concentration, the opportunity for significant error increases because of the estimated correction. Dilute HCl or $CaCl_2$ are the flocculants commonly used. The most effective one will depend on the nature of the sediment and the dissolved minerals in the water. However, extreme care is necessary in using either of the above flocculants. Carbonate minerals are readily decomposed in acid solutions and many other minerals are slightly soluble in weak acid solutions; therefore, the use of hydrochloric acid is not recommended. Calcium chloride is highly hygroscopic; therefore, if the sample has been treated with calcium chloride, the use of closed weighing bottles is recommended instead of open evaporating dishes for the determination of concentration.

The weight of natural dissolved solids in the evaporated water is included in the weight of total solids obtained by the evaporation method. The following may be used as a guide to determine if a correction for the dissolved solids is necessary. A 5 percent or more improvement in the results of the sediment determination will be realized when the dissolved solids content is equal to or greater than the sediment content, if 20 cc of native water is used in the evaporation, and if 400 grams of mixture is contained in the original sample. For example, a dissolved solids correction should be made when the dissolved solids content is 200 ppm or higher and the sediment content is 200 ppm or less. Similarly, a 2 percent improvement will result if the dissolved solids content is 40 percent or more of the sediment content.

Filtration method.-- The filtration method for the determination of suspended-sediment concentration utilizes a Gooch crucible in conjunction with various types of filter material. The Gooch crucible is a small porcelain cup of approximately 25 cc capacity with a perforated bottom. The crucible is easily adapted to an aspirator system and vacuum filtration.

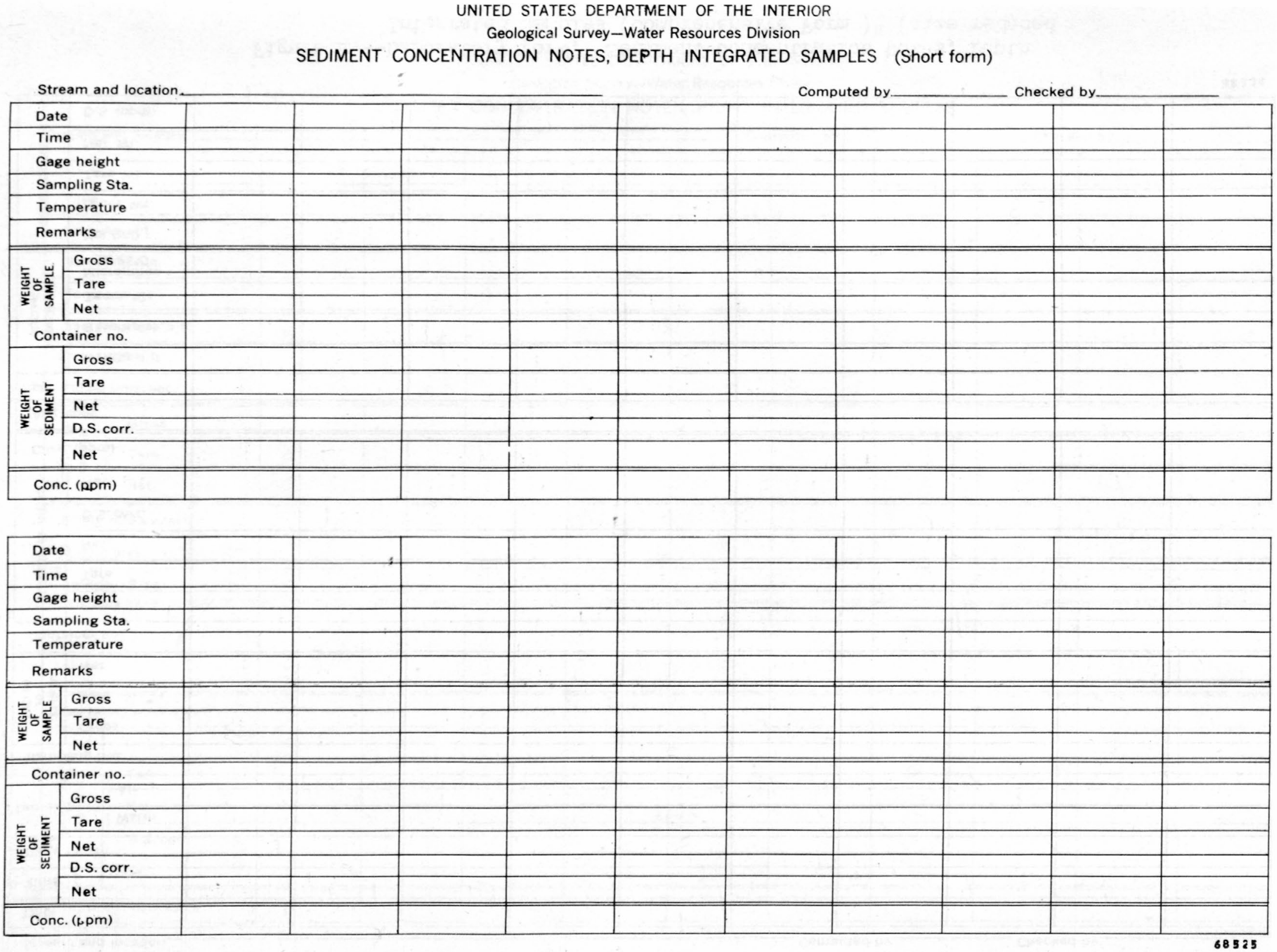
Various types of material can be used for filters in the crucibles. Asbestos is generally satisfactory because of its inertness, resistance to heat, and low hygroscopicity. The asbestos filter mats can be prepared in one of two ways. The simplest and most convenient way is to prepare a slurry of shredded asbestos and distilled water, and pour a small volume of this slurry into the crucible while vacuum is being applied. A uniform mat of asbestos is thus formed in the bottom of the crucible. The mat should be rinsed with distilled water while vacuum is still applied; the crucible is then oven-dried, cooled in a desiccator, and the tare weight determined. Another method for preparation of the mat is to place a small portion of loose, uniform asbestos on the bottom of the crucible, and wet the asbestos with distilled water while vacuum is applied to the crucible.

Glass fiber filter disks, no. 934-AH or similar specifications, have proven satisfactory for filtration of many types of sediment samples. These filter disks show no weight loss during filtration, save considerable preparation time, result in more uniform filtration, and make cleaning of the crucible easier. However, the filter disks become clogged rather rapidly when some types of fine sediment are filtered. Better filtration results have been obtained for these fine grained sediment samples by using glass fiber filter disks in conjunction with an asbestos mat. The crucible with this "double" mat is prepared by placing the filter disk in the crucible while vacuum is being applied and pouring the asbestos slurry on top of the disk also while vacuum is applied. During the filtration process, the somewhat coarse asbestos mat retains much of the sediment that would ordinarily clog the fiber glass disk. The fiber glass disk prevents loss of asbestos or loss of very fine sediment that would pass through the asbestos mat. This filter is recommended if inaccuracy or inconvenience results from the use of a single type of filter because little additional time is required for the preparation of the fiber glass-asbestos type filter.

Gooch crucibles have numerous advantages over evaporating dishes for the determination of concentration. The crucibles are lighter in weight and consume less oven and desiccator space than evaporating dishes; their tare weight is less likely to change during the weighing due to sorption of moisture because less surface is exposed; and dissolved material passes through the crucibles and thus eliminates the need for a dissolved solids correction.

If the sediment sample contains extremely fine material, an actual loss through the filter may result in a turbid filtrate. This problem is generally treated in one of three ways: (1) add a known quantity of an adsorbing agent, such as activated carbon, which adsorbs the colloids as a result of the difference in electric charges, (2) impregnate the filter with sediment and then re-filter the filtrate, and (3) use a flocculating agent of the acid or salt type. In addition to preventing loss through the filter, the adsorbing agent should reduce the settling time and prevent the filter from clogging as quickly. The main advantage of the adsorbing agent over the use of the flocculating agent is that a precise correction, instead of an estimated correction, can be made for the amount retained on the filter. Acids or salts are not recommended because undesirable reactions with the sediment are likely to occur. In many cases, the use of a glass fiber filter disk in conjunction with an asbestos mat will eliminate filtrate problems.

Laboratory forms. -- Figures 3, 4, and 5 show forms used in recording data and making computations for determination of suspended-sediment concentration. These forms (Sediment Concentration Notes, Depth Integrated Samples (Short Form); Sediment Concentration Notes, Depth Integrated Samples (Comprehensive Form); Sediment Concentration Notes, Point Integrated Samples) provide an orderly means of recording the sample information, the necessary weights, and the results of computations for the concentration of the sample. On the form "short form" for depth integrated samples (fig. 3) space



UNITED STATES DEPARTMENT OF THE INTERIOR
Geological Survey—Water Resources Division
SEDIMENT CONCENTRATION NOTES, DEPTH INTEGRATED SAMPLES (Short form)

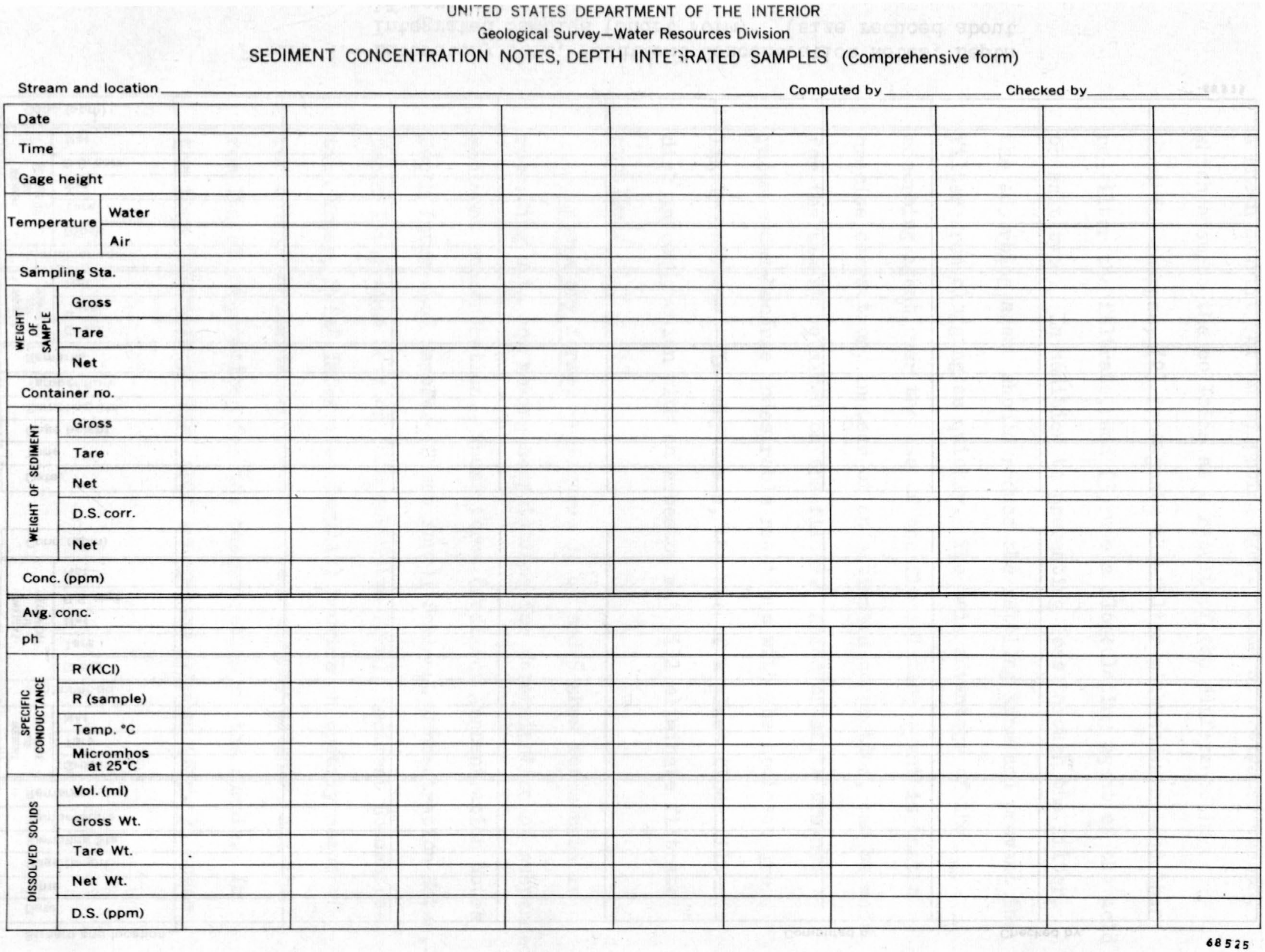
Stream and location__________ Computed by__________ Checked by__________

Date											
Time											
Gage height											
Sampling Sta.											
Temperature											
Remarks											
WEIGHT OF SAMPLE	Gross										
	Tare										
	Net										
Container no.											
WEIGHT OF SEDIMENT	Gross										
	Tare										
	Net										
	D.S. corr.										
	Net										
Conc. (ppm)											

Date											
Time											
Gage height											
Sampling Sta.											
Temperature											
Remarks											
WEIGHT OF SAMPLE	Gross										
	Tare										
	Net										
Container no.											
WEIGHT OF SEDIMENT	Gross										
	Tare										
	Net										
	D.S. corr.										
	Net										
Conc. (ppm)											

68525

Figure 3.--Laboratory form, "Sediment Concentration Notes, Depth Integrated Samples (Short Form)" (size reduced about 15 percent)



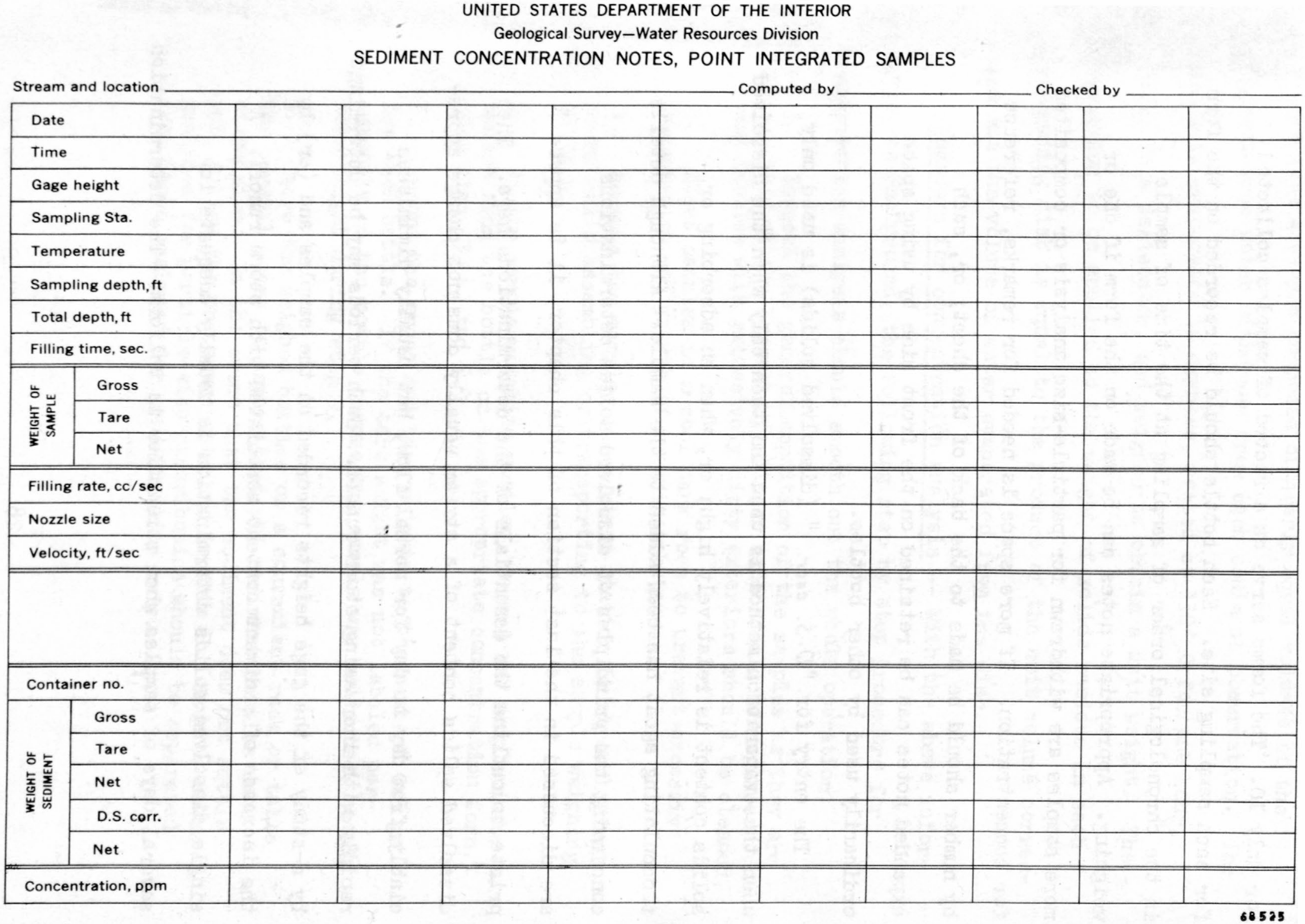
UNITED STATES DEPARTMENT OF THE INTERIOR
Geological Survey—Water Resources Division
SEDIMENT CONCENTRATION NOTES, DEPTH INTEGRATED SAMPLES (Comprehensive form)

Stream and location ______________ Computed by ______________ Checked by ______________

Date											
Time											
Gage height											
Temperature	Water										
	Air										
Sampling Sta.											
WEIGHT OF SAMPLE	Gross										
	Tare										
	Net										
Container no.											
WEIGHT OF SEDIMENT	Gross										
	Tare										
	Net										
	D.S. corr.										
	Net										
Conc. (ppm)											
Avg. conc.											
ph											
SPECIFIC CONDUCTANCE	R (KCl)										
	R (sample)										
	Temp. °C										
	Micromhos at 25°C										
DISSOLVED SOLIDS	Vol. (ml)										
	Gross Wt.										
	Tare Wt.										
	Net Wt.										
	D.S. (ppm)										

68525

Figure 4.--Laboratory form, "Sediment Concentration Notes, Depth Integrated Samples (Comprehensive Form)" (size reduced



UNITED STATES DEPARTMENT OF THE INTERIOR
Geological Survey—Water Resources Division
SEDIMENT CONCENTRATION NOTES, POINT INTEGRATED SAMPLES

Stream and location ______ Computed by ______ Checked by ______

Date											
Time											
Gage height											
Sampling Sta.											
Temperature											
Sampling depth, ft											
Total depth, ft											
Filling time, sec.											
WEIGHT OF SAMPLE	Gross										
	Tare										
	Net										
Filling rate, cc/sec											
Nozzle size											
Velocity, ft/sec											
Container no.											
WEIGHT OF SEDIMENT	Gross										
	Tare										
	Net										
	D.S. corr.										
	Net										
Concentration, ppm											

68525

Figure 5.--Laboratory form, "Sediment Concentration Notes, Point Integrated Samples" (size reduced about 15 percent)

is provided for 20 bottles or samples; the other two forms provide for only 10. The forms serve as a record of samples collected for each sampling site. Each bottle should be recorded on the form in the chronological order of sampling at the time of sample weighing. Appropriate notes can be made on the form if one or more samples are withdrawn for particle-size analysis or composited for concentration. If more space is needed for remarks, reference by number should be made to the back of the sheet; or, such expanded notes can be retained on the front side by using space ordinarily used by other bottles.

The entry for "D.S. corr." (dissolved solids) is used only when the evaporation method is used and then only when the dissolved solids content is relatively high; or, when an adsorbing or flocculating agent has been added to the sample. Although details concerning the principles of dissolved solids determinations are discussed in the last section of this chapter, it is appropriate to outline the essentials of the determinations here. The dissolved solids content of a stream usually does not change appreciably from day to day for normal flow, but usually diminishes rapidly with increasing storm runoff. Such periods may be determined by a study of the gage heights recorded on the samples and (or) by the increase of sediment content associated with storm runoff. A single dissolved solids determination is usually adequate for several days of samples when streamflow is uniform. The determination is made from a sample composed of nearly equal volumes of the sediment-free water withdrawn from each sediment observation. The sample is evaporated to dryness, heated at 110° C for one hour, cooled in a desiccator, and weighed to obtain a unit weight. The correction to be applied to the weight of solids residue in each evaporating dish is equal to the product of the unit volume correction and the volume of water evaporated from the dish.

Procedure for concentration analysis.-- With the above information as background, the following step by step procedure for concentration analysis should smooth out the whole operation:

1. Inspect the general condition of the samples as they are received at the laboratory. Loose caps should be replaced and bottles with excessively dirty exteriors should be cleaned.

2. Store samples in a cool dark room to prevent excessive evaporation and growth of organic substances.

3. Arrange the samples for a given station in chronological order while assembling or transporting to the sample weighing station.

4. At the sample weighing station, record the sample information from the bottle on the appropriate concentration form. (See figs. 3, 4, and 5.)

5. Record the gross and tare weights to the nearest 0.5 gm for each bottle. If the tare weight was not labeled permanently on the bottle before the sample was collected, obtain this weight during step 9.

6. Store the weighed bottles on a convenient rack or table for several hours to assure that all sediment settles from suspension. In the event that the sediment does not settle out, follow the procedure outlined in Note B. Samples to be analyzed for particle-size distribution should be separated from those analyzed for concentration only.

7. Decant most of the sediment-free water using care not to disturb or remove sediment.

8. Wash the remainder into a previously weighed evaporating dish or Gooch crucible and check to be certain that the container number on "Sediment Concentration Notes" corresponds with the given sample or samples.

9. The sample bottles must be thoroughly cleaned, air dried, recapped and packed in carrying cases for reuse.

10. Dry the evaporating dishes or crucibles and, after loss of all visable water, heat at about 110°C for 1 hour. The evaporating dishes must be dried at a temperature low enough to prevent "spattering."

11. Cool the containers in a desiccator.

12. Weigh the containers to the nearest ~~0.001~~ 0.0001 gm. on an analytical balance. The tare weight of the evaporating dish should be obtained before each use because of the loss of small chips from the dishes, the loss of weight due to frequent washing, and changes in weight due to slight differences in washing or cleaning methods. The tare weight of the crucible must be obtained before each use due to differences in weights of the filtering mats.

13. Compute the net weight of sediment in the container and deduct the dissolved solid correction, if applicable.

14. Compute the concentration of the sample in parts per million (ppm) as one million times the ratio of the net weight of sediment to the net weight of the water-sediment mixture.

Note B: In the event that the sediment does not readily settle, the bottles should be stored for several days. The use of even small quantities of dilute hydrochloric acid or calcium chloride as a flocculant is not recommended except in extreme cases. In no case should flocculants ever be added to a sample intended for particle-size analysis. When the use of a flocculant is absolutely necessary, the following procedure is recommended: Decant the turbid suspension into a separate clean container, leaving the previously settled sediment in the original sample bottle. Add a small amount of flocculant to the turbid suspension and allow to settle; thus the flocculant does not come in contact with the main portion of the sediment. The two portions of sediment are analyzed separately; the concentration is then computed during steps 13 and 14.

DETERMINATION OF PARTICLE-SIZE DISTRIBUTION OF SUSPENDED SEDIMENTS

(Section III)

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

Particle-size data have many uses. One such use is to compute the probable specific weight of sediment as it will deposit in reservoirs. This computation may require a plotting of median particle size against sediment load in tons per day. Information concerning the behavior of sediment particles in different environments must be studied in this connection; and, for the fine particles, involves analyses in both native and dispersed settling media. The size of suspended and bed material has an important use in the computations of total (measured and 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 dispersing agent for the silt and clay fractions. The recommended frequency of native water analysis is discussed in another part of this section in consideration of the theories, problems, and method 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 samples collected at one vertical near midstream are representative of the entire cross section, the observers' samples can be used to provide a greater selection in making analyses to cover the wide range of conditions.

Judgment concerning the selection of samples will be enhanced by a study of the conditions for which samples for past records of the given station were selected, if available; and(or) by a study of the sample information recorded on the form "Sediment Concentration Notes" 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, samples, particularly from high stage flows, should be set aside to be analyzed either for particle size or concentration at the end of the water year when the facts are known concerning the year's flow. These extra samples should, of course, be tightly sealed and stored in a dark place.

Size analyses of suspended sediment for the so-called daily station should be made on a minimum of 10 samples per year. Additional samples, up to a maximum of about 30, may be desirable to define the size distribution during unusually high runoff periods to represent a range of stream discharge or the various areal patterns of runoff. The above minimum may be reduced by necessity in the event of low runoff due to a prolonged drought or because of extremely low concentration of sediment.

Particle-size analyses should be made on a minimum of one-fifth of the observations for stations operated on a periodic basis. The number should depend on the frequency of sampling, the stream conditions, and the projected use of the data. Whenever possible, the relative number should be decided at the time of programming for the station.

Computations of total sediment load (measured plus the 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 the desired times for the total load computations.

Method of analysis (dispersed settling medium).-- The program should place major emphasis on the determination of particle size by use of methods that minimize floccule formation. The recommended method of analyses for determining particle-size distribution is, by necessity, usually a combination of methods. For example, laboratories analyzing a large number of samples use the VA (visual accumulation) tube-pipette method for most of the samples. The VA-tube is preferred to sieves because the results of the analyses are expressed in sedimentation diameters which is more suitable for studies of sediment transport and deposition. However, to compare the sedimentation diameter with that of actual sizes as determined by sieves, 3 or 4 (or 10 or 15 percent of the total, whichever is more) of these determinations per station per year 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-pipette method should send the sand fraction from 3 or 4 samples per year for each station to a laboratory having a VA-tube for comparative analysis.

Many laboratories must retain the use of the more time-consuming, and possibly less accurate, bottom-withdrawal-tube method because of an insufficient quantity of sediment in the samples. Whenever possible, a sufficient quantity of sediment should be collected to permit analysis by the VA-tube-pipette method. The increased time to obtain the additional material is usually more than compensated by the decreased time required for analyses and computation. The bottom-withdrawal tube is satisfactory to define the sedimentation diameter for sands up to 350 microns. Samples containing particles larger than this size should be analyzed by the sieve method if a VA-tube is not available. (See "Sieving" in Section IV).

Sections IV, V, and VI in this chapter give detailed procedures for making the analyses by various sieve and sedimentation methods. For most stations, when the VA-tube-pipette or the sieve-pipette methods are used, only the following sizes need be determined: 0.002, 0.004, 0.016, 0.062, 0.125, 0.250, 0.500, 1.00 and 2.00 mm (millimete

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 also be determined. All the above sizes must be determined when samples are split for analyses in both native water and a chemically dispersed settling medium. If the bottom-withdrawal-tube method is used, additional sizes may be determined from the Oden curve with little additional effort.

Pipette analyses are usually made using either 500 or 1,000 ml (milliliters) of suspension. However, if sufficient sediment is not available to obtain a concentration of 2,000 ppm for a 500-ml suspension, less than 500 ml can be used for the suspension. Abbreviated pipette analyses, in which only the 0.002, 0.004 and 0.016 mm determinations are made, can conveniently be performed using 200 ml of suspension. By decreasing the volume of suspension and the number of sizes determined, and by using withdrawal depths of 5 cm (centimeters) or less, accurate pipette analyses can be made on as little as 0.4 g (gram) of silt and clay. By reducing the volume of suspension in this manner pipette 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 bottom-withdrawal-tube method.

Method of analysis (native water settling medium).-- Prediction of the behavior of sediment particles in different environments is difficult considering the many types of fluvial sediments being transported in media having widely varied chemcial constituents. The incomplete understanding of the characteristics and behavior of small particles, especially clay minerals, compounds 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 particles will behave as discrete units. Many stream waters are either low in dissolved solids or high in sodium, and the problems under discussion have little application with respect to mechanics of sediment transport within the flowing stream.

Theory involved in settling velocity methods for size analysis is based on the fact that the sedimentation diameter is calculated for a discrete particle equivalent to the diameter of a sphere having a uniform terminal fall velocity. 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 they fall slower than a quartz sphere of the same nominal diameter.

(2) The floccules, in the process of accretion while settling, would have an 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 as well as by viscous drag. Thus, the settling velocity and, consequently, the sedimentation diameter for many particles in native water, result from complicated and probably indeterminable factors.

Information concerning the degree of flocculation of sediment suspended in natural streamflow is lacking. In a natural stream, the water-sediment mixture is in continuous turbulent motion; whereas, in the laboratory, analyses are made using a motionless column of water-sediment mixture.

Note C: 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.

The degree of flocculation indicated by laboratory analyses of sediment in native water is influenced by several factors: (1) the characteristics of the water, (2) the kind of clay mineral, (3) the nature of the ions associated with the particle before it entered the stream, and (4) the concentration of the sediment in the analysis suspension. A consideration 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 in native water and in dispersed medium; whereas, other streams show little or no difference between the two methods of analysis.

During the first two years of determining sediment transport at a site, about one-fourth 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 unless a change in regimen of the river is in progress. Native water size analyses can be discontinued for some streams if it is apparent that flocculation is highly improbable throughout the range of discharges.

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 settling media should have a suspended solids concentration of about 3,000 to 5,000 ppm of material finer than about 62 microns. The pipette method, using the depths of withdrawal shown in table 3, (Section IV) is recommended.

Percent ~~finer~~ coarser than 0.062 mm should not be determined by the pipette method if this value can be determined either by the VA-tube or sieve method because residual turbulence, caused by the preparatory mixing prior to the first withdrawal, lasts throughout most of the theoretical settling time used for the 0.062 mm withdrawal. Determination of percent finer than 0.002 will define more accurately the clay mineral content of the sample. 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. (See Note D.)

Note D: 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 1 to 2 microns. A separation at 2 microns is frequently about the optimum size for the best split of the clay-mineral and nonclay-mineral components of natural materials."

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 determine total dissolved solids, specific conductance, pH, calcium, magnesium, sodium, potassium, and bicarbonate, and(or) alkalinity.

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 assure the availability of samples with sufficient sediment for a three-way split.

Preparation of sample (native water settling medium).-- The preparation of samples for dispersed conditions is discussed in connection with each method of analysis in sections IV, V, and VI. 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 or 30 seconds with the hand stirrer for the pipette method, and 90 seconds of tilting for the bottom-withdrawal-tube method.

Either the modified Jones-Otto splitter or the bottom-withdrawal-tube sample splitter are recommended for samples that contain no sand. The qualitative and quantitative accuracy of the modified Jones-Otto splitter is greater than that of the bottom-withdrawal-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 of 3,000 to 4,000 ppm. Skill and experience are necessary in estimating the number of splittings necessary to yield the required concentration. The alternative is to collect extra samples for concentration determinations prior to the required splitting.

Samples containing suspended material that settles extremely slow create a difficult laboratory problem. Usually a "sample" consists of from 2 to 10 or more bottles (about 400 grams each) of water-sediment mixture. The sediment in these bottles must settle from suspension before compositing can be accomplished. Then the sediment in the composite must settle before an analysis can be made in a dispersed medium or before splitting can be accomplished preparatory to analyses both in native and dispersed media. Therefore, two settling periods are usually necessary prior to analysis: one prior to compositing and the other prior to splitting and analysis. If centrifugation equipment is not available, it is recommended that the suspended material be allowed to completely settle prior to each of these steps. In the event that the sample data is immediately needed, 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 most of the native water from each bottle. The fine sediment remaining in suspension adheres to the outside of the filter tube and the water passes through the walls.

(3) The material on the outside of the tube is back flushed with native water and carefully rinsed into the container to be used for compositing of the sediment.

As an alternative to the above procedure for samples that do not settle, the following method has also 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 one-third to three-fourths of the water and fine sediment mixture. Determine the weight of sediment in 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 a concentration of 2,000 to 4,000 ppm in the analysis cylinder, transfer to the analysis cylinder, fill with the filtered sediment-free native water, and make the analysis as usual. Each withdrawal would then be corrected for the fine sediment 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 have to be applied to each withdrawal for the dispersed analysis made in conjunction with the native analysis. This method has an undesirable aspect due to the fact that all of the sediment sizes present in the original sample are not present in the part of the sample to be analyzed. Therefore, the chemical and(or) physical effect of the very fine particles in suspension on the larger particles is not indicated.

PROCEDURE FOR THE SIEVE-PIPETTE METHOD OF PARTICLE-SIZE ANALYSIS OF FLUVIAL SEDIMENT IN DISPERSED CONDITION

(Section IV)

The pipette method of determining particle-size gradation for sizes finer than 62.5 microns is one of the most widely accepted techniques utilizing the Oden theory. The upper size limit of sediment particles which settle in water according to Stokes' law and the lower size limit which can be determined by sieves is about 1/16 mm or 62.5 microns. This size is the division between sand and silt (Wentworth grade scale) and is an important division in many phases of sediment transportation.

The fundamental principle of the pipette method is to determine the concentration of a suspension at a given depth as a function of 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 time and depth of withdrawal are predetermined on the basis of Stokes' law (Section I). Table 3 gives the time for each withdrawal for 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 0.62 to 0.008 sizes and 5 cm for the 0.004 and 0.002 sizes as well as 15 cm for 0.62 and 0.31 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

Table 3.--Time of pipette withdrawal for given temperature, depth of withdrawal, and diameter of particles*

	Diameter of particle in millimeters															
	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															
Temperature (°C)	(sec)	(sec)	(min)	(sec)	(min)	(sec)	(min)	(sec)	(min)	(sec)	(min)	(sec)	(hr)	(min)	(hr)	(min)
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

* 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.

depth for fines decreases the time required to complete the set.

Equipment.-- The pipette equipment described by Krumbein and Pettijohn (p. 165-167) is satisfactory when only relatively few samples are analyzed. However, most sediment laboratories need adequate equipment for analyses of many hundreds of samples each year. To facilitate operations to analyze such numerous samples, the apparatus described by Davis, John R., U.S. Geological Survey, personal communication (~~1951~~) is suggested. The apparatus, shown in figure 6, consists of a rack capable of accommodating 8 sedimentation cylinders. A movable carriage containing the pipette is installed immediately 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 pipette.

Satisfactory use of the pipette method requires careful and precise operation to obtain maximum accuracy in each step of the procedure. Shown also in figure 6 is a diagrammatical sketch of the arrangement of the apparatus indicating the use of a 25-milliliter pipette equipped with a 3-way stopcock. Rubber tubing of sufficient length to allow the pipette 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 pipette forms a constr~~u~~iction which helps to maintain a uniform rate of withdrawal in the proper length of time.

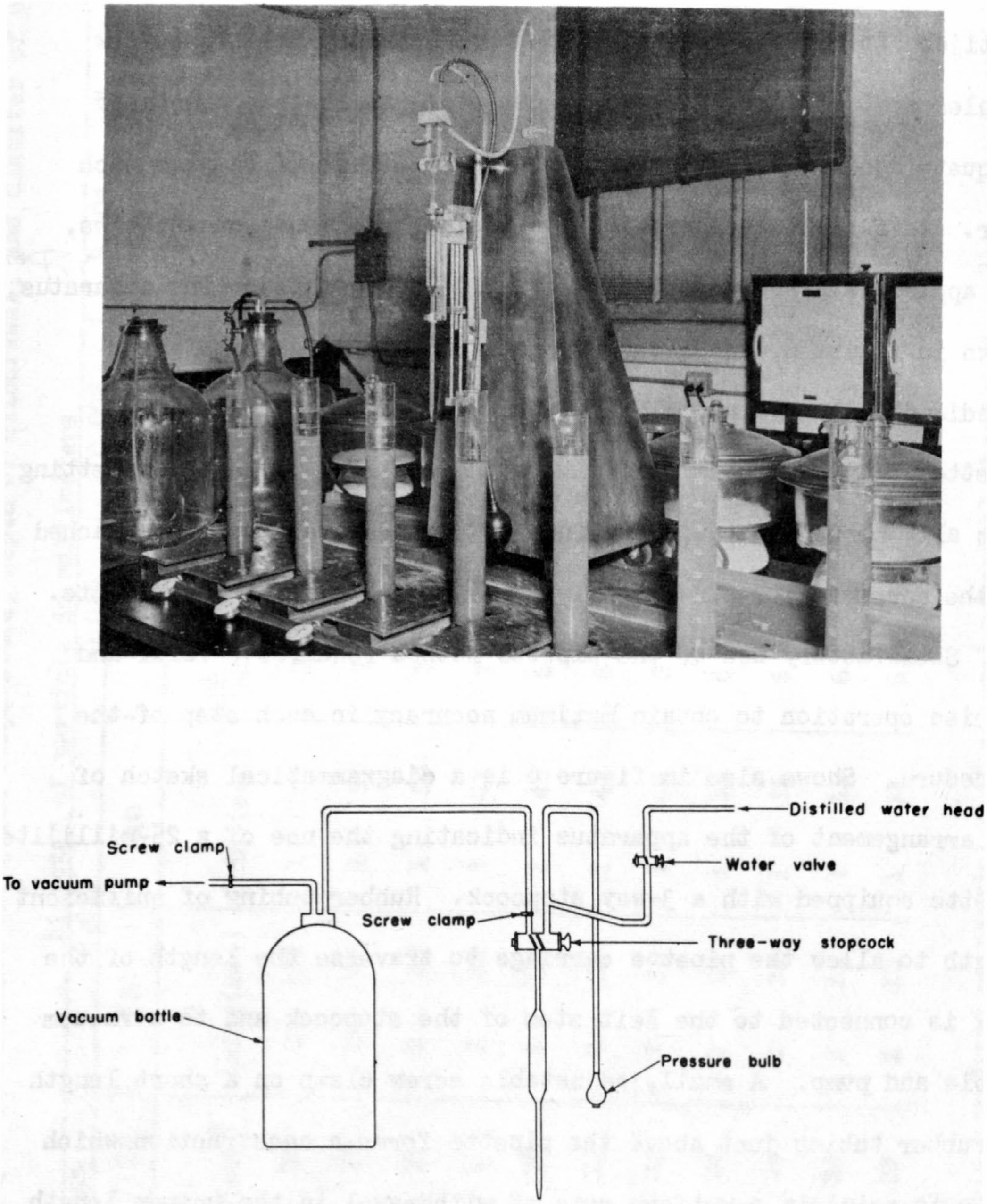


Figure 6.--Apparatus for pipette particle-size analysis.

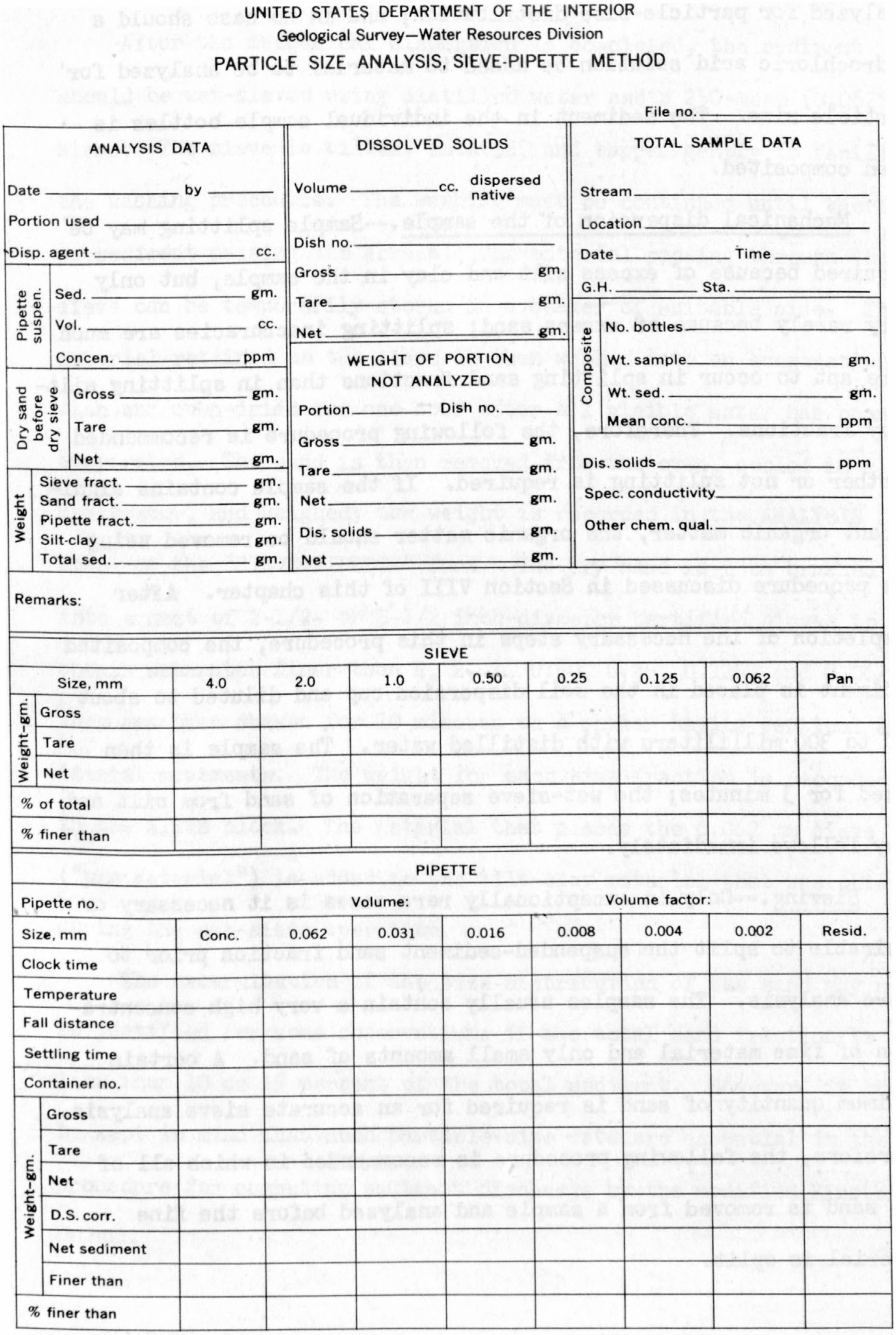
Attached to the right stem of the stopcock with rubber tubing is an inverted Y-shaped glass tube. A length of rubber tubing, with a pressure bulb at one end, is attached to the top stem of the Y. To the short stem of the Y is attached a length of rubber tubing connecting the apparatus to a distilled water supply with a head of 3 to 5 feet. The flow of distilled water is controlled by a stopcock.

After a sample is drawn into the pipette, the 3-way stopcock plug is rotated 180 degrees, and the sample is allowed to drain freely into an evaporation dish. The drainage can be accelerated by use of the pressure bulb. To insure complete removal of all sediment in the pipette, the distilled-water valve is then opened, and the pipette 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 pipette with the pressure bulb. At this time, remove the small quantity of the mixture that may have collected in the vacuum line near the 3-way stopcock (resulting from overfilling the pipette) by allowing a small quantity of air to be sucked through the line from the open pipette. Removal of this water clears the constriction controlling the vacuum on the pipette and thereby a uniform rate of withdrawal of the next sample.

A simple "constant-vacuum device," different from that described by Davis, is being used for pipette analyses in some laboratories. The device insures a more uniform vacuum during a single withdrawal as well as a constant vacuum for all analyses. It replaces the constriction (screw clamp) shown in figure 6. The necessary change in the arrangement consists of 3 lines instead of 2 to the vacuum bottle. The third line is connected to a constant-head cylinder; sealed with a 2-hole stopper; partly filled with water; and, having a copper tube open to the outside and extending to the bottom of the cylinder. The cylinder then acts as a "bleeding" device against a vacuum in the bottle being in excess of the head of water around the copper tube.

<u>Preparation of sample</u>.--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 (figure 3 or 4) 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-PIPETTE" form. See figure 7. 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 immediately needed, a small amount of calcium chloride solution or other suitable calcium solution may be added to cause flocculation and settling. In most cases, the use of a flocculating agent is not recommended for any sample to be



UNITED STATES DEPARTMENT OF THE INTERIOR
Geological Survey—Water Resources Division
PARTICLE SIZE ANALYSIS, SIEVE-PIPETTE METHOD

File no. ____

ANALYSIS DATA

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

Pipette 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.
Pipette 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. ____

Composite
No. bottles ____
Wt. sample ____ gm.
Wt. sed. ____ gm.
Mean conc. ____ ppm

Dis. solids ____ ppm
Spec. conductivity ____
Other chem. qual. ____

Remarks:

SIEVE

	Size, mm	4.0	2.0	1.0	0.50	0.25	0.125	0.062	Pan
Weight-gm.	Gross								
	Tare								
	Net								
% of total									
% finer than									

PIPETTE

Pipette 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								
	Tare								
	Net								
	D.S. corr.								
	Net sediment								
	Finer than								
% finer than									

Figure 7.--Laboratory form, "Particle-Size Analysis, Sieve-Pipette Method" (size reduced about 15 percent)

analyzed for particle-size distribution; and in no case should a hydrochloric acid solution be added to material to be analyzed for particle size. The sediment in the individual sample bottles is then composited.

Mechanical dispersion of the sample.--Sample splitting may be required because of excess silt and clay in the sample, but only very rarely because of excess sand; splitting inaccuracies are much more apt 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 Section VIII of this chapter. After completion of the necessary steps in this procedure, the composited sediment is placed in the soil dispersion cup and diluted to about 250 to 300 milliliters with distilled water. The sample is then mixed for 3 minutes; the wet-sieve separation of sand from silt and clay follows immediately.

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 certain minimum quantity of sand is required for an accurate sieve analysis. Therefore, the following procedure is recommended in which all of the sand is removed from a sample and analyzed before the fine material is split.

After the mechanical dispersion is completed, the sediment should be wet-sieved using distilled water and a 250-mesh (0.0625 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 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 one hour after all visible water has been evaporated. The 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-PIPETTE" form. The dry sand is then brushed into a nest of 2-1/2- or 3-1/2 inch-diameter certified sieves to obtain separates finer than 4, 2, 1, 0.50, 0.25, 0.125, and 0.0625 mm. They are then shaken for 10 minutes on a shaker having vertical and lateral movements. The weight for each size fraction is recorded in the SIEVE block. The material that passes the 0.062 mm sieve ("pan material") is added to the silt-clay material that was obtained during the wet-sieve operation.

The determination of the size distribution of the sand may not be justified for some observations if the total sand fraction is less than 10 or 15 percent of the total sediment. However, it should be kept in mind that such particle-size data are essential in the procedure for computing total sediment discharge by the modified Einstein method.

Splitting of the silt and clay fraction.-- If a sample contains an estimated 5 grams 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 grams 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 grams. 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 to 5,000 ppm in a 1,000-ml sedimentation cylinder. Either the modified Jones-Otto splitter or the bottom-withdrawal tube splitter are satisfactory for the splitting of silt and clay. If the dissolved contents is still high, which is highly unlikely if the previous recommendations have been followed, it may be necessary to rinse the sediment to reduce the dissolved solids concentration in the final suspension to less than 200 ppm. For the rare cases, the sample should be rinsed with distilled water until there appears to be a minimum of flocculating tendency in the suspension.

Dispersion of the pipette fraction.--To insure complete dispersion of the particles, add to the sample 1 milliliter of dispersing agent for each 100 milliliters of the desired volume of the suspension. After the dispersing agent is added, transfer the sample to the mixing cup of a soil dispersion mixer and mix for 10 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 dispersing agent to each of two 500-milliliter cylinders, and add distilled water to a volume of 500 milliliters in each. Using the standard 25 milliliter pipette, 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.

The dispersing agent is made by dissolving 35.70 grams of sodium hexametaphosphate and 7.94 grams of sodium carbonate in distilled water and diluting to one liter volume. A determination of a dissolved solids correction must be made each time a new solution of dispersing agent is prepared.

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 PIPETTE block of the form. The suspension is then stirred for 60 seconds with a hand stirrer of the plunger type illustrated in Krumbein and Pettijohn (p. 167), and the stop watch is started when the stirrer is removed from the suspension. The analysis should be made at nearly constant temperature to minimize convection currents.

The time and depth of pipetting (Table 3) is determined from Stokes' law on the basis of temperature of suspension and settling diameters. The pipette is filled in 8 to 12 seconds and then emptied into an evaporation dish; one rinse from the pipette is added. The material is dried at 110° C, cooled in a desiccator, and weighed.

Determination of total weight of sediment in sample.-- The calculation of results from the sieve-pipette 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 that containing considerable quantities of clay minerals.
2. The weight of the silt and clay fractions can be determined from the mean concentration and volume of the pipette

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 pipettings have been completed. To this add the dry weight of sediment in each pipette withdrawal and the dry weight of the sand fraction if it was separated.

4. Split the sample in two portions by means of the bottom-withdrawal-tube sediment splitter or other splitting device. Evaporate one-half to dryness to determine the weight of sediment, and use the other half for the sieve-pipette analysis. The accuracy of this method will depend on the quantitative and qualitative accuracy of the splitting operation; therefore, the method is not recommended.

Method 2 or method 3, either singly or in combination, are recommended. Because of the speed and ease of the determination, method 2 is recommended for most samples. Occasionally, the weight of material in a suspension should be determined by both methods 2 and 3. 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.

Calculation of results.-- The form illustrated by figure 7 facilitates recording the data and calculating the results for the sieve-pipette 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 pipette withdrawal when multiplied by the volume factor, $\frac{\text{total volume of suspension}}{\text{volume of pipette}}$, 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 VISUAL-ACCUMULATION-TUBE-PIPETTE METHOD OF PARTICLE-SIZE ANALYSIS OF FLUVIAL SEDIMENT

(Section V)

The visual-accumulation-tube method fills a fundamental need in the process of obtaining data relating 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 measurement, but the sieve method does not yield accurate results because (1) inaccuracies exist in size and shape of sieve openings, (2) sieving can seldom be continued until all particles which might pass a given sieve have the necessary opportunity to do so, and (3) to a minor degree, fine material may cling to larger particles. Therefore, the visual-accumulation-tube-pipette method is recommended for the determination of the particle-size distribution. The visual-accumulation method is especially adapted for size analysis of samples composed mainly of sand. The finer material must be removed from samples by either wet sieving or by sedimentation methods when its measurement is required. Sieving is employed to remove particles which are too large for measurement by the visual-accumulation.

The visual-accumulation-tube (or VA-tube) method employs the <u>stratified system</u> of sedimentation as contrasted with the <u>dispersed</u>

system of the pipette or the bottom-withdrawal tube. The stratified system is one in which 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 is furnished with each of the apparatus. Likewise, the details of the development and calibration are contained in Report No. 11 and only some of the highlights are mentioned herein.

Further consideration of the results obtained by the method should be made before describing the equipment and process. It has been shown that particles of a sample in the visual tube settle with greater velocities than for the same particles falling individually. The calibration of the method is then based on the fall velocities of individual particles falling alone in a fluid of sufficient extent to avoid the effect of space limitations. Therefore, the size frequency analysis is based on the 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 for 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 recorder was based on the analysis of hundreds of samples having known fall velocities and makes allowance for the increased fall velocities when a sample 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 sample concentration and particle size. If the VA-tube method is properly used, the resulting gradation will be quite accurate. See note E.

Note E: For special particle shapes, or 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.

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 by figure 8, 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 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 with a carriage that can be moved vertically by hand on which are mounted a recording pen and optical instrument for tracking the accumulation as well as a cylinder carrying a chart that rotates at a constant rate; and (6) the recorder chart which is a printed form incorporating the fall-diameter calibration. (See fig. 9.) Drawings and further details are given in Report No. 11 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. If many particles larger than about 1.2 mm. are present in a sample, they should be removed by sieving. If any clay or much silt is contained in the sample, it is removed before analysis by wet sieving with a fine sieve or by settling the sample through a sedimentation tube. Although some coarse silt does not affect the accuracy, the more thoroughly the clays and silts are removed from the sand, the simpler and faster will be the VA-tube analysis.

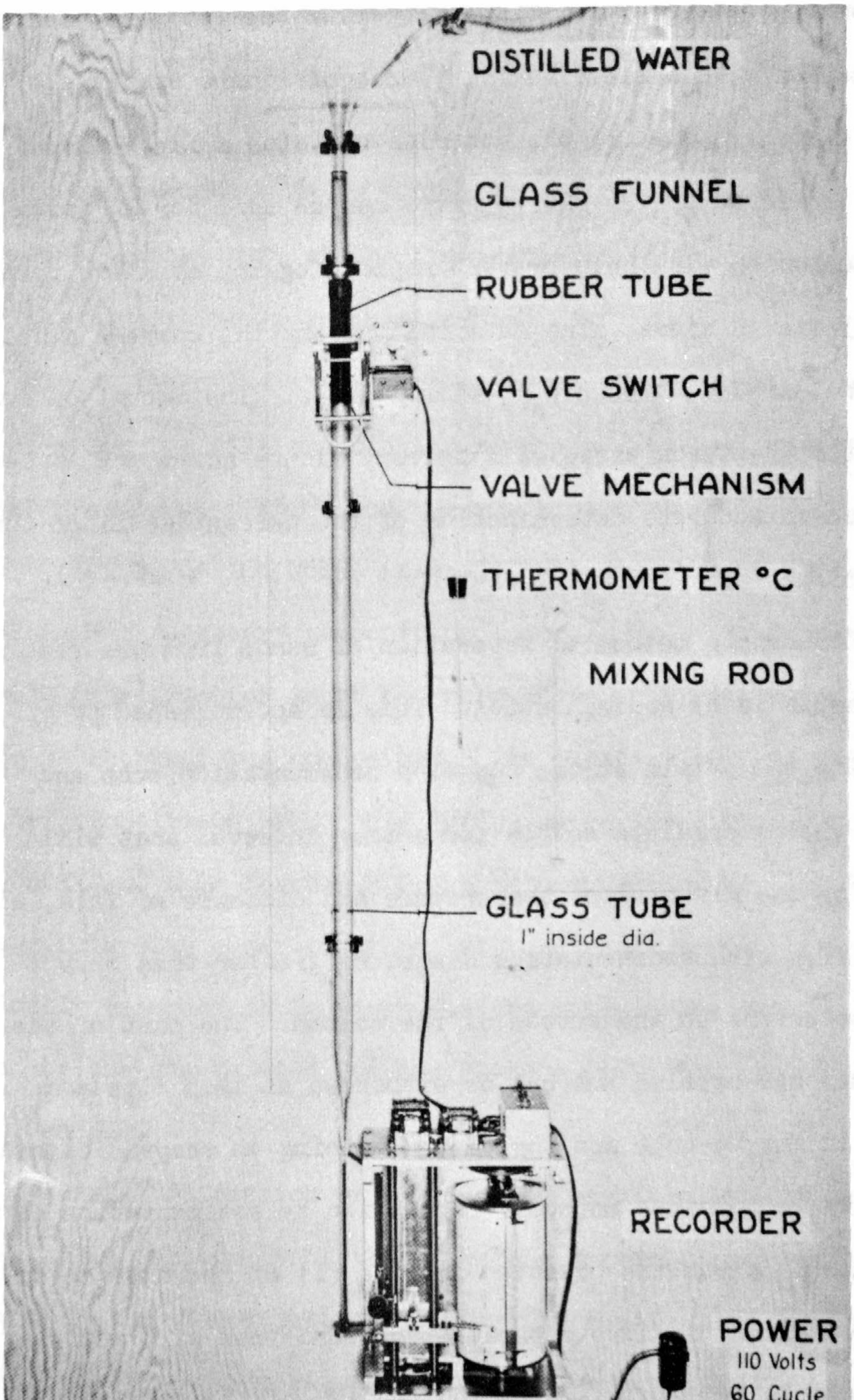


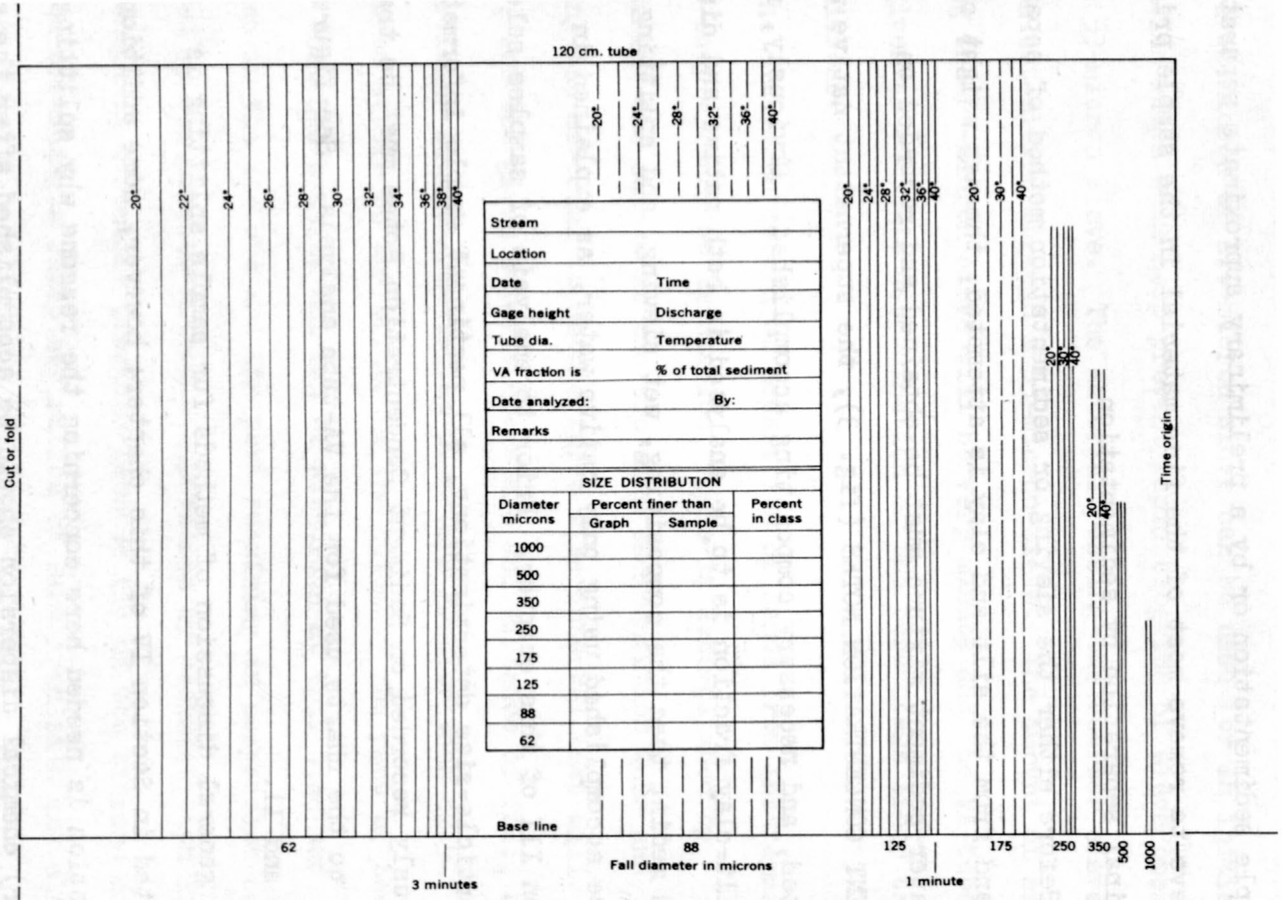
Figure 8.--Apparatus for visual-accumulation-tube particle-size analysis.

The following method is recommended for the separation of sand from the silt-clay fraction. Separate the sand from the silt-clay fraction with a sieve having 53-micron square openings by the wet sieve method. If the material contains a large amount of sand, a 62-micron or 125-micron sieve can be used for an initial separation which will help prevent rapid clogging of sieve openings in the 53-micron sieve. The material passing the coarser sieve is then wet-sieved through a 53-micron sieve. The amount of sand passing the 53-micron sieve will be very minute and the VA-tube will given an accurate determination of the percentage finer than 62.5 microns.

The 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 62.5 microns 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 pipette method. Separation by sedimentation is not recommended as a routine practice because (1) of the extreme difficulty in obtaining a sharp separation of sand from silt and clay during a single sedimentation period and (2) of the difficulties and problems involved if the sample contains 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 difficulty can be remedied either by multiple sedimentation or by a preliminary approximate separation by sieve to remove most of the fine material in the sample prior to the final separation by sedimentation.

Before either the sieving or sedimentation method of separating the sand from the silt and clay is attempted, the net weight of the water-sediment mixture must be obtained and recorded on SEDIMENT CONCENTRATION NOTES (fig. 3), the supernatant native water decanted, and necessary compositing 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, as explained in Section III of this chapter. Prior to analysis of samples selected for particle-size determinations, all pertinent sample information previously recorded on Sediment Concentration Notes must be transferred to the charts used for the VA-tube analysis. See figures 9, 10, and 11.

A general discussion of methods for sample splitting is presented in Section IV 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



Stream	
Location	
Date	Time
Gage height	Discharge
Tube dia.	Temperature
VA fraction is	% of total sediment
Date analyzed:	By:
Remarks	

SIZE DISTRIBUTION

Diameter microns	Percent finer than		Percent in class
	Graph	Sample	
1000			
500			
350			
250			
175			
125			
88			
62			

Figure 9.--Illustration of chart for use with the 120-cm. visual-accumulation tube (size reduced about 35 percent)

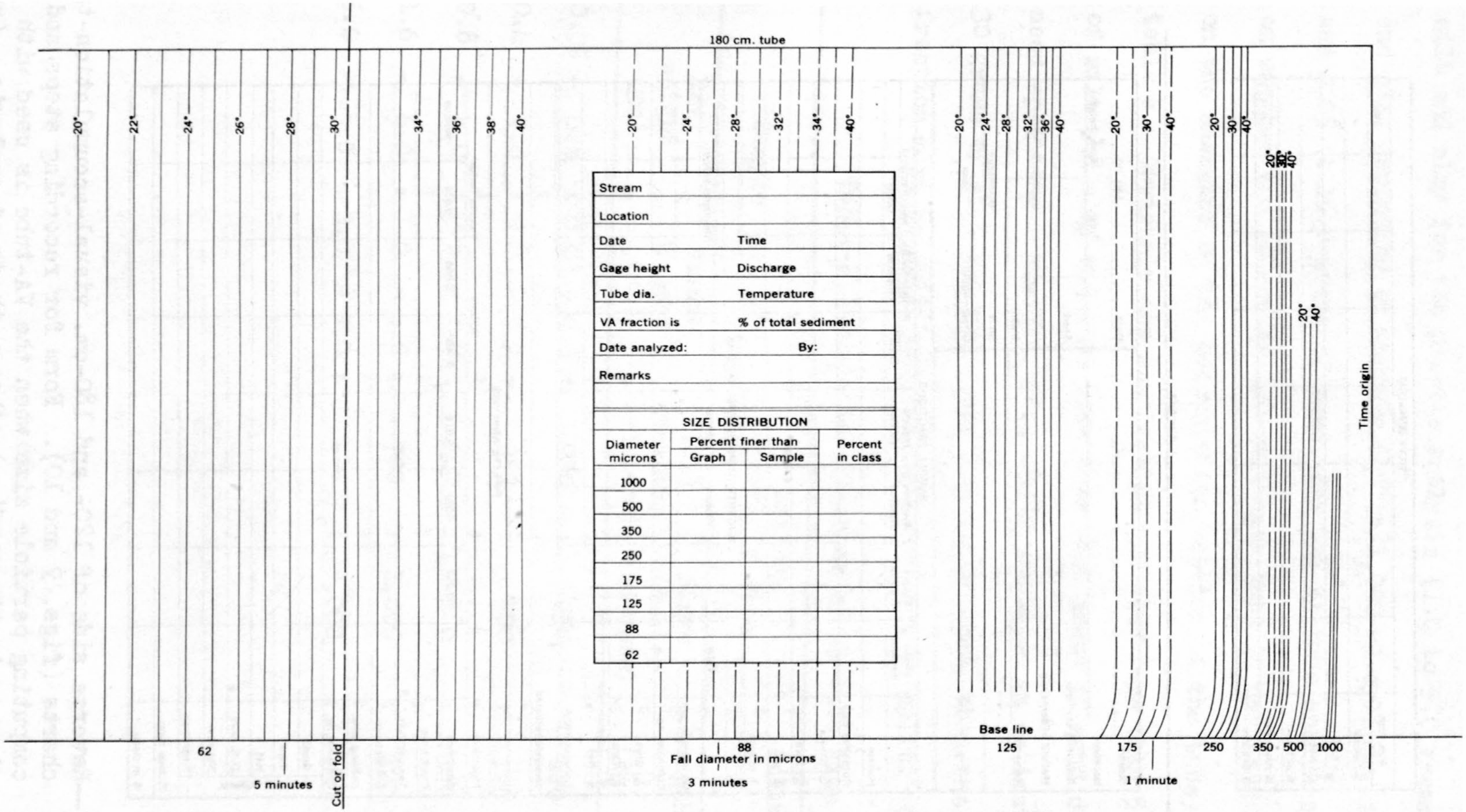
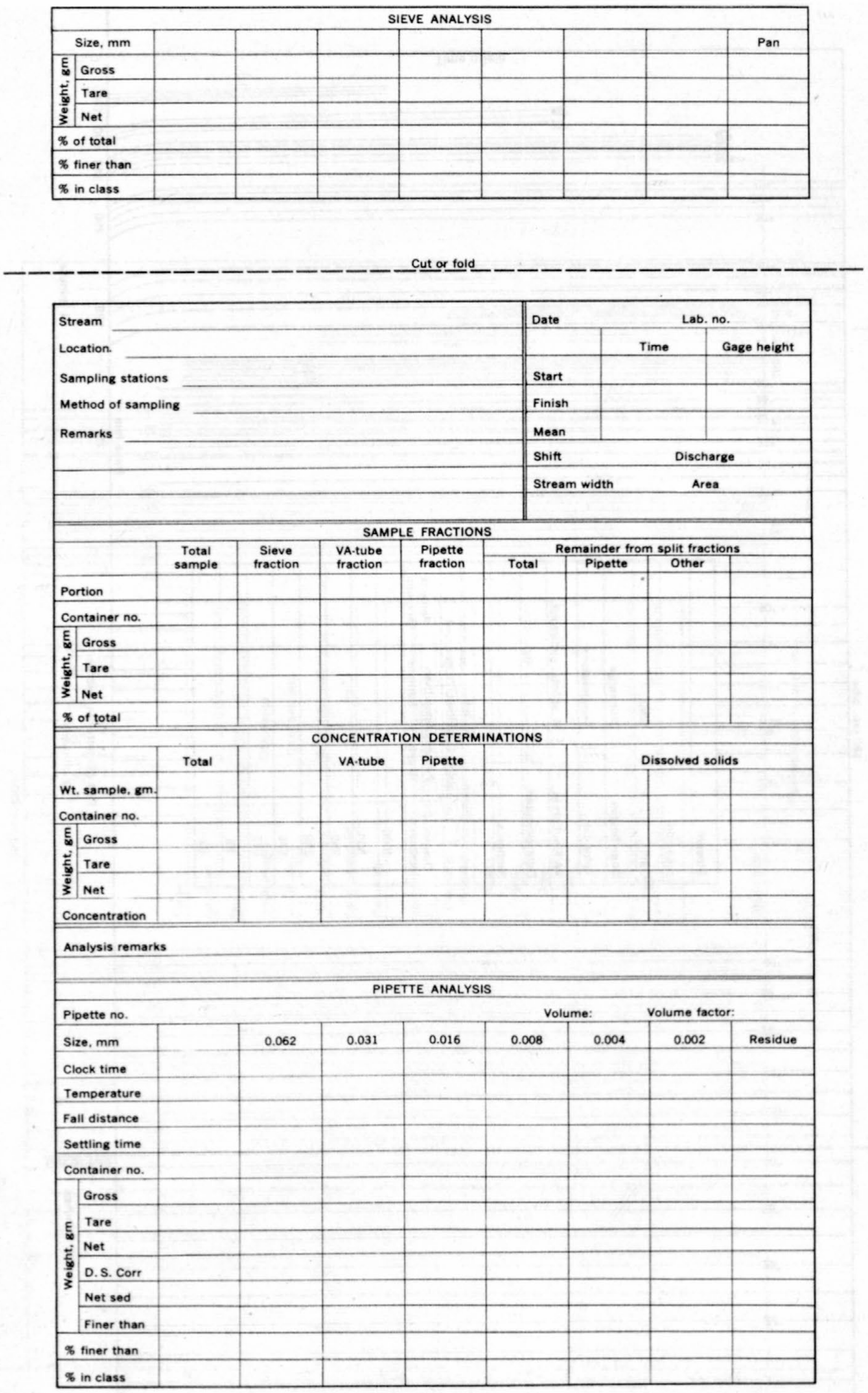


Figure 10.--Illustration of chart for use with the 180-cm. visual-accumulation tube (size reduced about 35 percent)



SIEVE ANALYSIS

Size, mm									Pan
Weight, gm	Gross								
	Tare								
	Net								
% of total									
% finer than									
% in class									

Cut or fold

Stream

Location.

Sampling stations

Method of sampling

Remarks

Date	Lab. no.	
	Time	Gage height
Start		
Finish		
Mean		
Shift	Discharge	
Stream width	Area	

SAMPLE FRACTIONS

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

CONCENTRATION DETERMINATIONS

		Total		VA-tube	Pipette		Dissolved solids		
Wt. sample, gm.									
Container no.									
Weight, gm	Gross								
	Tare								
	Net								
Concentration									

Analysis remarks

PIPETTE 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 11.--Reverse side of 120- and 180-cm. visual-accumulation-tube charts (figs. 9 and 10). Form for recording steps and computing particle size when the VA-tube is used with sieve, pipette, and(or) other methods of analysis. (size reduced about 35 percent)

splitting is to obtain a concentration of 2,000 to 5,000 ppm of silt and clay for the pipette analysis (1.0 to 5.0 grams of silt and clay depending on whether 500 or 1,000 ml cylinder is used) and 0.05 to 15.0 grams of sand for the VA-tube analysis (depending on whether the 180-cm or 120-cm length tube is used and depending on the diameter of the accumulation section of the tube). See table 4. Obviously any sample that contains less than 5.0 grams of silt and clay and (or) less than 15.0 grams of sand does not need splitting. However, if a sample contains an estimated 25 to 30 grams of silt and clay, and 5 grams of sand, then the silt-clay fraction must be split and the sand need not be split. Quantitative

Table 4.--Guide to selection of VA-tube size

Sample		Maximum particle size		Sedimentation tube	
Dry weight (gm)	Volume of sand (ml)	Fall diameter (microns)	Sieve diameter (microns)	Length (cm)	Diameter (mm)
0.05 - 0.8	0.03 - 0.5	250	250	120	2.1
0.4 - 2.0	0.2 - 1.2	350	400	120	3.4
0.8 - 4.0	0.5 - 2.4	500	600	120	5.0
1.6 - 6.0	1.0 - 4.0	700	1,000	120	7.0
5.0 - 15.0	3.0 - 9.0	---	2,000	180	10.0

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. 11) 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 pipette analysis. For example, the dispersing agent in the suspension before separation by the sedimentation 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 ultimate particles but to create a suspension having stability. Because the separation and splitting operations are relatively short-term procedures, mechanical dispersion alone is an adequate preparatory treatment for the material.

The VA-tube pipette method differs from the sieve-pipette method only in the way in which the sand is analyzed. Section IV (SIEVE-PIPETTE METHOD) discusses in some detail the preparation of the sample, separation of sand from the silt-clay fraction, splitting of the silt-clay fraction, dispersion of the sample, pipetting, and calculation of results. These general recommendations apply equally to pipette analyses made in conjunction either with sieve or VA-tube analysis.

Sand particles should be in such condition that the grains will fall as individual particles in the sedimentation tube and therefore should be thoroughly wetted before analysis. They should be contained in not more than 40 ml. of water at a temperature within 2° or 3°C of that of the water in the VA tube. If organic matter is present in sufficient volume to interfere with analysis or to decrease the accuracy of the analysis, such organic matter should be removed before the sample is analyzed. 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 in Section VIII.

<u>The VA-tube analysis</u>.-- The best results are obtained if the total height of accumulation in the bottom of the tube is between 1 and 4 inches. If a sample is predominately coarse or has a very limited size range, the maximum accumulation should be less than 4 inches. The choice of a suitable tube is not difficult because the usable limits of the respective tubes overlap; and, if a satisfactory size is not selected the first time, the samples can be

rerun in another size of tube. Table 4, set up on the basis of quantity and maximum particle size, 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. Synthetic samples may be used as a guide in judging the pertinent characteristics of samples from new sources. For example, a sample can be analyzed in a 2.1 mm. tube if it does not exceed in quantity or particle size a synthetic sample containing 0.8 gm. of sand and having a maximum particle size of 250 microns.

The following procedure of making an analysis is reported in Report No. 11, p. 118-120. It should be noted that analysis can be made in less than 10 minutes for samples with particles greater than 62 microns. "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. (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 analys~~e~~is.

"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 the 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 62-micron size on the chart. Then rotation of the cylinder automatically stops. If material is still 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 the line of maximum accumulation across the chart.

"10. After the valve is closed, the sample is drained into a beaker by removing the tube plug. The valve is opened slightly to drain out excess water and to wash out the lower end of the tube more completely. The plug is replaced.

"11. The chart is removed from the recorder."

Interpretation of chart.-- 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 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 those sizes on the chart 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 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, say 30 percent of the original sample, then the 30 on the scale is held on the total-accumulation line and direct reading made as above. Similarly, if material coarser has been removed, then the percentage coarser to the total is subtracted from the total or 100 and held on the zero-accumulation line. The results of these readings in percent finer than the given size are tabulated on the form in the block for this purpose.

The equipment and procedure for making the pipette analysis of the fine 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 pipette 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 BOTTOM-WITHDRAWAL-TUBE AND VISUAL-ACCUMULATION-TUBE MET OF PARTICLE-SIZE ANALYSIS OF FLUVIAL SEDIMENT

(Section VI)

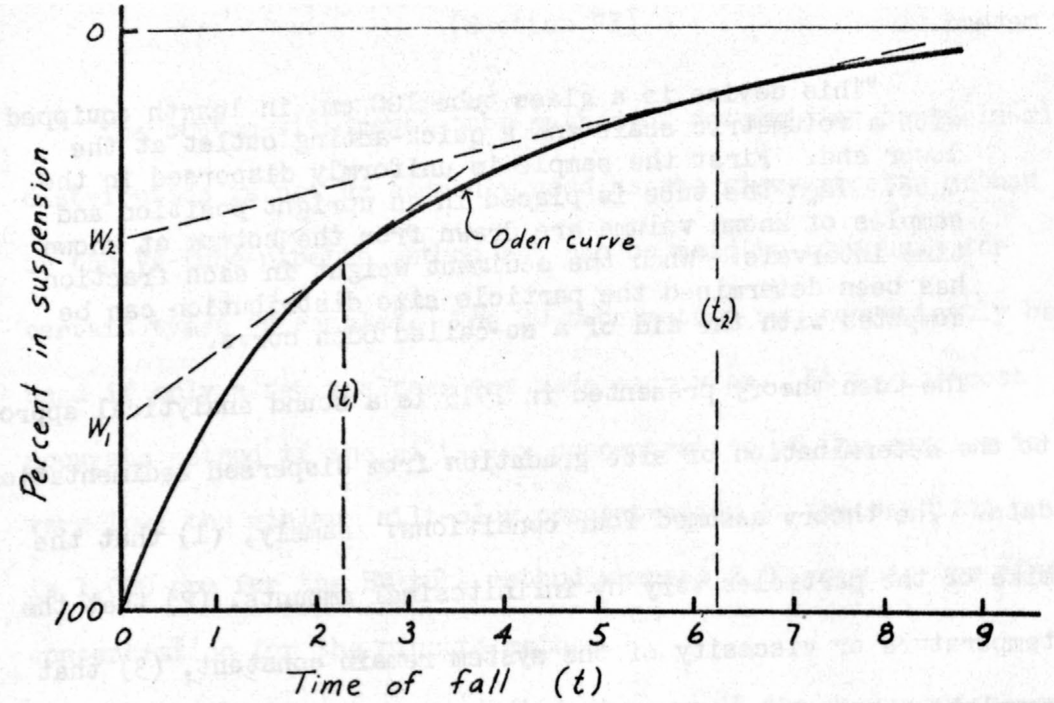
The bottom-withdrawal-tube method of determining particle-size distribution is not as commonly used as the sieve-pipette method or the VA-tube-pipette method but can be used to advantage for certain types of samples. The BW-tube method can conveniently be used if only a few analyses are made each year. It is the most accurate method if the silt-clay concentration of the samples is very low; the minimum silt-clay concentration in the settling process is 1,000 ppm for the BW-tube method, whereas 2,000 ppm is the minimum concentration for the pipette 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 ppm 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 determinations are made at concentrations of 120 ppm or less. Therefore, it is obvious that the accuracy of the particle-size analysis by either the BW-tube method or pipette method depends not only on the original concentration of suspension but on the particle-size distribution of the material in the sample.

The bottom-withdrawal tube method makes direct application of the Oden theory which is, in turn, dependent on Stokes' law. The following quotation (Report No. 7, p. 5) gives a synopsis of the method.

> "This device is a glass tube 100 cm. in length 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 presented in 1915 is a sound analytical approach to the determination of size gradation from dispersed sedimentation data. The theory assumed four conditions: namely, (1) that the size of the particles vary by infinitesimal amounts, (2) that the temperature or viscosity of the 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 the start of the settling of the particles, the 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 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 time t_1 and t_2.

Equipment.--The necessary special equipment beyond that ordinarily found in the sediment laboratory would consist of the bottom-withdrawal tube or tubes with adequate provisions for mounting. Figure 12 shows such an arrangement (after fig. 10 and 12, Report No. 7). The following are specifications for the manufacture of the tube:

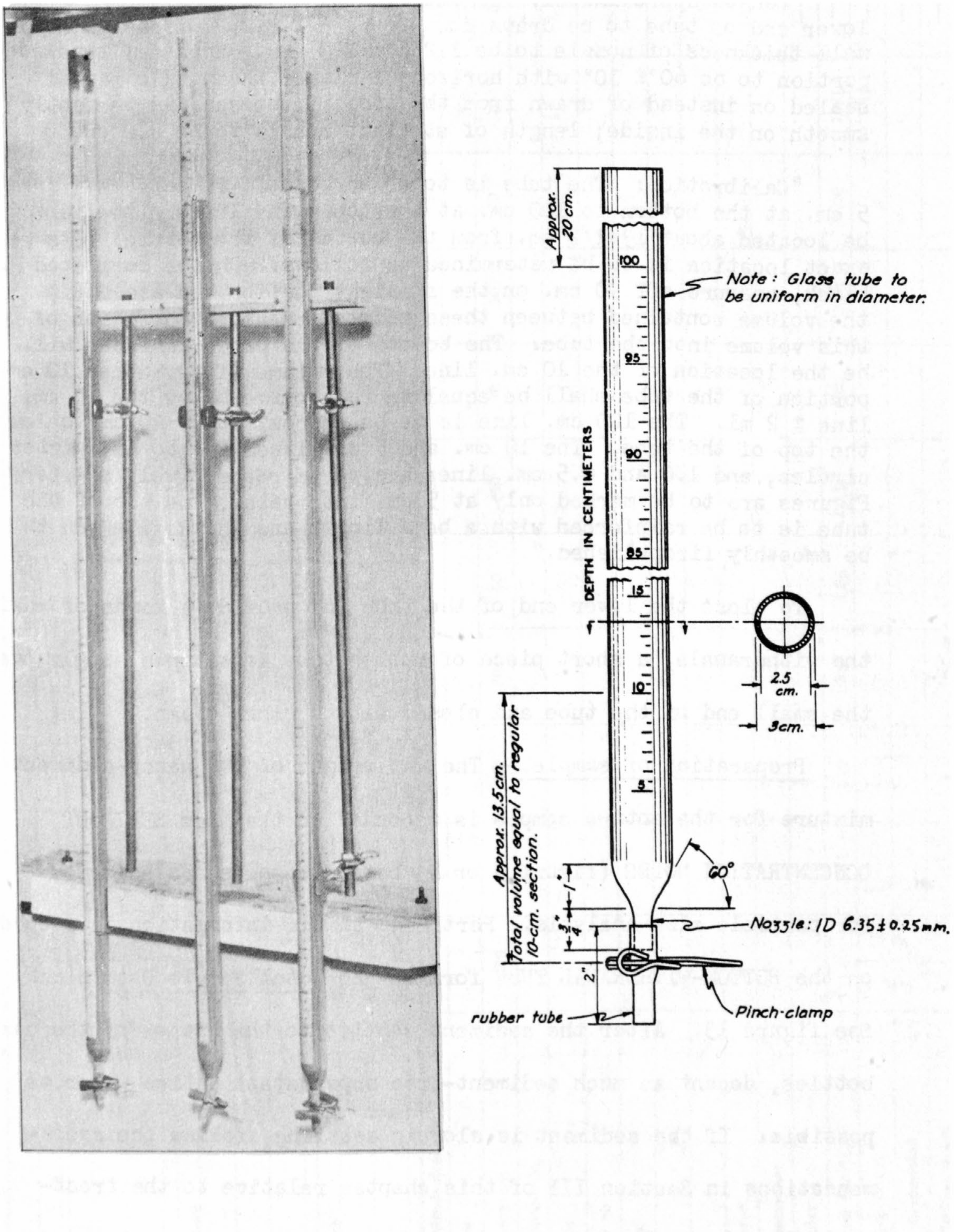


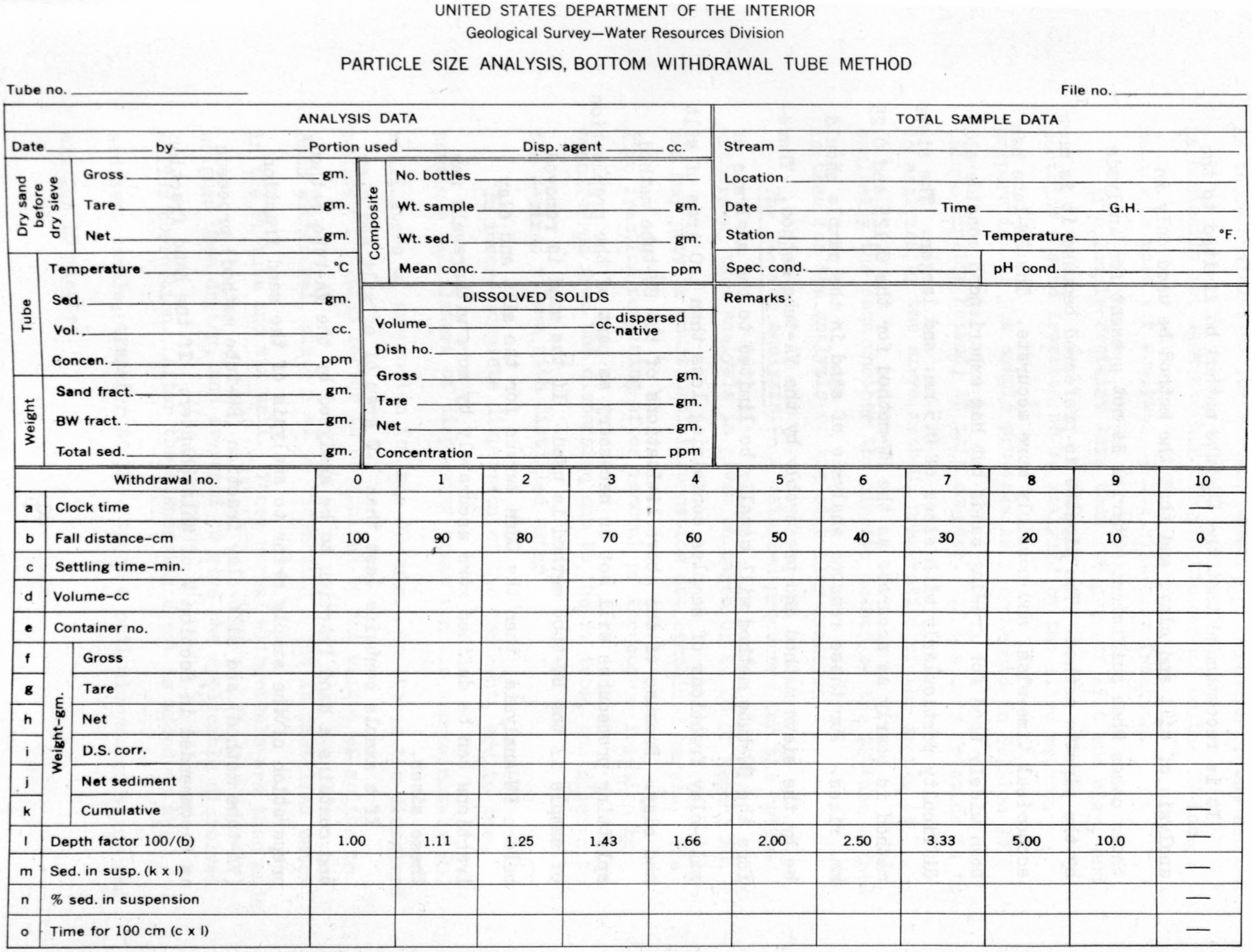
Figure 12.--The bottom-withdrawal tube and stand.

"Length approximately 122 cm.; inside diameter 25 to 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 to 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 perfectly 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 13-1/2 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, and 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.

Preparation of sample.-- The net weight of the water-sediment mixture for the entire sample is recorded on the form SEDIMENT CONCENTRATION NOTES (figure 3 or 4) before samples are selected for particle size analysis. Pertinent sample information is recorded on the BOTTOM-WITHDRAWAL TUBE form in the Total Sample Data block. See figure 13. After the sediment settles to the bottom of the sample bottles, decant as much sediment-free supernatant native water as possible. If the sediment is slow in settling, follow the recommendations in Section III of this chapter relative to the treatment of such samples.



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

Weight
Sand fract. ____________ gm.
BW fract. ____________ gm.
Total sed. ____________ gm.

DISSOLVED SOLIDS
Volume ____________ cc. dispersed / native
Dish no. ____________
Gross ____________ gm.
Tare ____________ gm.
Net ____________ gm.
Concentration ____________ ppm

TOTAL SAMPLE DATA

Stream ____________
Location ____________
Date ____________ Time ____________ G.H. ____________
Station ____________ Temperature ____________ °F.
Spec. cond. ____________ pH cond. ____________
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		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 (c x l)											—

Figure 13.--Laboratory form, "Particle-Size Analysis, Bottom-Withdrawal-Tube Method" (size reduced about 15 percent

It is recommended that the BW-tube method be limited to the analysis of silt and clay, and that the method be used only in those cases when sufficient material is not present for analysis by the pipette method. The pipette is preferred because it is more economical time-wise, and possibly more accurate. The BW-tube has been widely used for grading sands but has experienced considerable difficulty particularly with sizes of 0.5 mm. and larger. The sieve method is nearly as accurate as the BW-method for the 0.125 and 0.25 mm. sizes. 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 gram of silt and clay. Because of the above limitations of the BW-tube method, splitting procedures will not be necessary as part of the preparation of sample if the BW-tube method is used. If the sand is removed before BW-analysis, then the Oden curves for the silt and clay fractions can be defined more accurately by more withdrawals for these sizes.

If a sample contains less than 1.0 gram of of silt and clay, and contains a sand fraction to be analyzed by the VA-tube method, preparation of the sample prior to analysis of the sand fraction (VA-tube method) and silt-clay fraction (BW-tube method) proceeds as recommended in Section V of this chapter. If the sand fraction is to be analyzed by the VA-tube method, preparation of the sample is basically the same whether the pipette method or the BW-tube method is used for analysis of the silt-clay fraction.

If a sample contains less than 1.0 gram of silt and clay, and contains a sand fraction to be analyzed by the sieve method, preparation of the sample proceeds as recommended in Section IV (Sieve-Pipette Method) of this chapter. If the sand fraction is to be analyzed by the sieve method, preparation of the sample is basically the same whether the pipette method or the BW-tube method is used for the analysis of the silt-clay fraction.

The BW-tube analysis.-- A detailed procedure for the bottom-withdrawal tube analysis is given on pages 82 to 88 of Report No. 7 and may provide additional information not contained herein. The fine fraction remaining after removal of the coarse fraction is transferred from the dispersion cup to the BW tube, and diluted to the desired volume with distilled water.

To insure complete dispersion of the sediment, add to the sample 5 milliliters of dispersing agent for the approximately 500 milliliters of suspension for the BW-tube. To obtain the dissolved solids correction factor to be applied to the weight of solids in each withdrawal, place 5 ml of dispersing agent in distilled water in the BW-tube and mix well. Three normal withdrawals are then made, weighed upon drying, and averaged to give the applicable dissolved solids correction. After the dispersing agent is added to the sediment sample, transfer to the cup of a soil dispersion mixer and mix for 10 minutes.

Before placing the tube in the rack to start the settling operation, further mild mechanical mixing is accomplished by placing a cork in the open end of the tube and tilting the tube with the bottom up about 10° from the horizontal. Hold in this position and shake to wash the coarse particles from the constriction. The air bubble should then be at the constricted end and all coarse particles should be distributed as uniformly as possible along the tube. The tube is then held in an upright position to allow the bubble to travel the full length of the tube (about 5 sec.). Invert the tube from end to end for 3 minutes. 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. If a cork is used for the mixing, it must be removed before the first fraction is withdrawn but after the bubble has reached the top.

Equal-volume fractions are withdrawn using time intervals chosen in such a way as to accurately define the Oden curve. Because the tube has an effective length of 100 cm, each withdrawal corresponds to a column height of about 10 cm.

However, the method is entirely flexible, and 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 about 4 minutes to about 430 minutes. The detailed schedule of withdrawal times may be derived from Table 5.

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 the cone at the constriction of any deposited sediment. Based on reasoning that 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 not taken at the time the pinch clamp is opened, but at the time that it is closed.

Samples are withdrawn into a 100-cc graduate or flask in order to eliminate the possibility of losing any of the sample from splashing. The graduates are poured into evaporating dishes and washed 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 result in spattering. The small flask may be used for drying the sediment, if feasible to weigh, and if cleaning is not too difficult. When the evaporating dishes or

Table 5. Bottom-withdrawal-tube sedimentation timetable to be used with the Oden Curve

(Time in min. required for spheres having a specific gravity of 2.65 to fall 100 cm. in water at varying temperatures. Terminal fall velocity for particles 0.00195 to 0.0625 mm. computed according to Stokes' law; terminal fall velocity for coarser particles taken from curves prepared at the California Institute of Technology.)

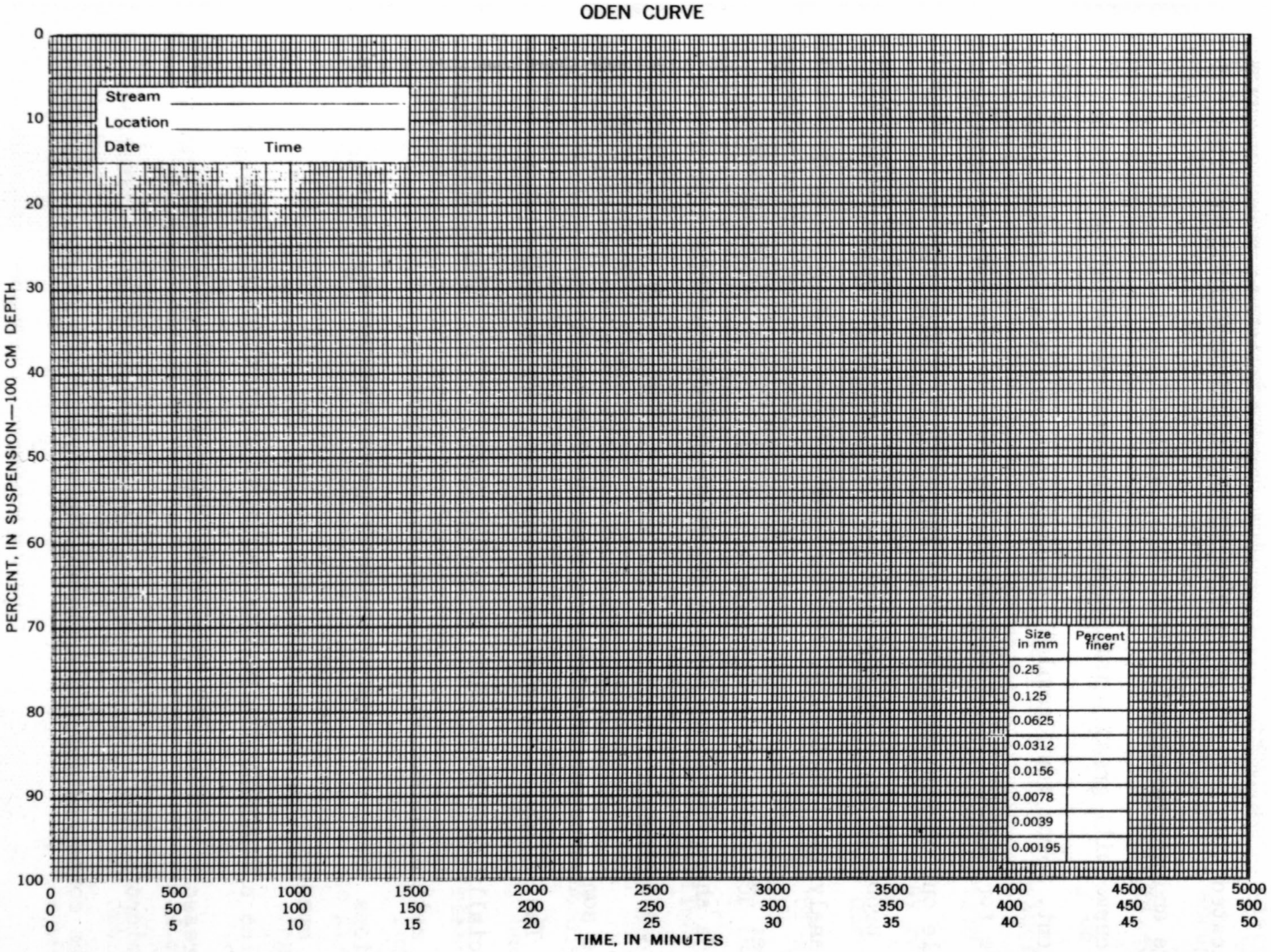
Temp. °C.	Particle Diameter in mm.							
	0.25	0.125	.0625	.0312	.0156	.0078	.0039	.00195
18	0.522	1.48	5.02	20.1	80.5	322	1288	5154
19	.515	1.45	4.88	19.6	78.5	314	1256	5026
20	.508	1.41	4.77	19.2	76.6	306	1225	4904
21	.503	1.39	4.67	18.7	74.9	299	1198	4794
22	.497	1.37	4.55	18.3	73.0	292	1168	4675
23	.488	1.34	4.45	17.8	71.3	285	1141	4566
24	.485	1.32	4.33	17.4	69.6	279	1114	4461
25	.478	1.30	4.25	17.0	68.1	273	1090	4361
26	.472	1.28	4.15	16.7	66.6	266	1065	4263
27	.467	1.26	4.05	16.3	65.1	260	1042	4169
28	.462	1.24	3.97	15.9	63.7	255	1019	4079
29	.455	1.22	3.88	15.6	62.3	249	997	3991
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	3825
32	.442	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	3494

flasks are visibly dry raise the temperature to 110°C for 1 hour, after which the containers are transferred directly from the oven to a dessicator and allowed to cool to room temperature. The weighing procedure is the same as that for sediment concentration determinations.

Since the temperature of the dispersion in the tube greatly influences the settling velocity of the particles, the temperature reading of the suspension should be made between the 6th and 7th withdrawals. If the room temperature is not reasonably constant, then more frequent readings will be necessary.

The recorded data together with the computation required to obtain the coordinates of the Oden curve are shown on the illustrated "BOTTOM-WITHDRAWAL TUBE" form of figure 13. Entries a to g, inclusive, are recorded during the analysis. The weight of sediment (h) is obtained by subtracting the tare from the gross. The dissolved solids correction (i) is obtained by computing the weight of dissolved solids per cc of suspension, on the basis of the determination as recorded in the "DISSOLVED SOLIDS" block, and multiplied by the volume of the withdrawal in the evaporating dish. The net sediment (j) is then determined by subtracting (i) from (h). By adding the net weights cumulatively (k), starting with the last withdrawal, the total sediment weight in suspension above each indicated depth is obtained. The depth factor (l) has been obtained by dividing the fall heights (*b*) into the 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 that would be present in a 100-cm. depth at the same average density (m). The percent of sediment in suspension (n) is obtained by the ratio of sediment in suspension (m) to the total sediment weight including the fraction sieved out as coarse sand. The time required for the average density above each observed height to be reached at the equivalent 100 cm (o) is the result of applying the depth factor (l) to the settling time (c). In effect, then, these computations reduce the observed times of settling and weights in suspension to a constant depth of 100 cm.

The Oden curve is plotted on rectangular coordinate paper as shown on the printed form (fig. 14). The upper curve is the result of the complete plotting of the data from entires (n) and (o) to the enlarged scale (0 to 5,000 min.), while the lower curves represent the expanded scales (0 to 500 and 0 to 50 min.) for better definition of the coarser fractions. Other horizontal (time) scales may be used so long as it is convenient to draw smooth curves through the plotted points. Points of tangency to the curves are determined on the basis of the desired sizes for the grading and the temperature of the suspension from table 5. (From page 55, Report 7).



Stream ______________________

Location ______________________

Date Time

Size in mm	Percent finer
0.25	
0.125	
0.0625	
0.0312	
0.0156	
0.0078	
0.0039	
0.00195	

Figure 14.--Laboratory form for plotting and drawing of Oden Curve from bottom-withdrawal-tube method of particle-size analysis (size reduced about 15 percent)

The intercept of the tangent from this point to the ordinate (percent in suspension) can then be read as the percent finer than the indicated size. The care needed in the construction of the Oden curve and the drawing of tangents is apparent since the shape of the curve will greatly influence the percentage intercept of the tangent. From most samples, the settling of particles will continue for several hours or days before many of the particles will settle out of the suspension and therefore the slope of the curve will probably never become zero over the period of time covered by the analysis. Obviously, the curve will never have a negative slope. A tangent from a curve with too steep a slope or a curve with a sharp curvature cannot be drawn to the intercept with the desired accuracy. The use of the expanded time scales will alleviate some of this difficulty.

The following discussion is based on comprehensive studies, especially for the coarser particle sizes, of the bottom-withdrawal tube method (Report No. 10). In consideration of settling concentrations outside of the recommended 1,000 to 5,000 ppm, it was found that concentrations to 10,000 ppm may be used for sands while samples containing mostly silt and clay about 3,500 ppm is the limit for reasonable accuracy. With the nominal size ranging from 30 to 350 microns, the results become more accurate on a percentage basis as the concentration of the sample increases; the average error decreasing from +5.2 percent at a concentration of 200 ppm to +0.5 percent at a concentration of 10,000 ppm. A lower limit of concentration for fine materials is assumed to be governed by the accuracy of laboratory methods such as volume determinations, weighing, etc. The first withdrawal containing the coarsest particles may be subject to considerable error and in some cases cannot be included on a smooth Oden curve having a straight line from the origin to this point and having no reversal of curvature. Errors may result from poor distribution of coarse particles along the tube due to particles sliding along the wall and for the first 5 sec. of settling due to the action of the bubble as it travels the length of the tube. It is also assumed that the general technique sometimes fails to obtain a sedimentation regimen consistent with the Oden theory of sedimentation in a dispersed system. Such errors undoubtedly are carried into the remainder of the Oden curve, but gradually become decreasingly important in later withdrawals. For a given reasonable concentration of suspension as indicated above, it has been found that with a maximum size of 246 microns, the results of the first withdrawal become somewhat erratic, and at 350 microns and larger, the results are so undependable as to be almost useless.

As indicated above the sum of the errors of the method may be attributed to the influence of operational techniques plus the limitations of the apparatus. In a general statistical sense, a given analysis may give the correct median grain size and still be in error at many points, it may give the correct amounts of many of the size fractions even though the percent finer curve is seriously in error, and the average results of several analyses may be quite accurate but the individual analysis may be erratic. It has also been found that, regardless of size or concentration of suspension, the results obtained with the bottom-withdrawal method show a smaller mean grain size than actually exists in the samples.

DETERMINATION OF PARTICLE-SIZE DISTRIBUTION OF DEPOSITED SEDIMENT AND SOIL SAMPLES

(Section VII)

The particle size distribution of deposited sediment and soil samples representing transport conditions on stream beds, deposition conditions in streams and reservoirs, and erosion conditions for the sources of fluvial sediment is assuming increased importance. Currently used formulae for the computation of total sediment discharge and bed load discharge, for example, require such information.

Bed material samples in streams are usually collected by means of a piston-type core sampler, a BM-48 clam-shell sampler, or a BM-54 sampler. Samples from reservoirs are obtained by various types of clam-shell and spud samplers. Streams having a wide gradation of sizes ranging from fine sands to pebbles or cobble, can best be sampled when dry or at a very low stage by shovel. In most cases, any desired quantity of bed material can be collected conveniently and rapidly from the stream bed. Therefore, in contrast to many suspended-sediment samples, an adequate quantity of material is usually available for analysis.

Equipment.-- The equipment required for particle-size analysis of stream bed and soil material 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 is usually found in suspended-sediment samples. Whereas a nest of 3-inch diameter sieves is satisfactory for analysis of sand from suspended-sediment samples, a nest of 8-inch diameter sieves and a "Rotap" sieving machine are more convenient for analysis of bed material samples. Also, 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 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 pebbles or cobble 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 measururable quantities of silt and clay. If both "fine" and "coarse" samples are obtained at a site, as is often the case in many stream systems, the distribution should be defined for each sample. Often, however, the difference in size distribution across a stream is small and only the mean distribution is required. In this case the samples can be composited 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.

Preparation and analysis of sample.-- The chart used for the VA-tube analysis, see figures 9, 10, and 11, has been designed to include sufficient recording spaces for a complete analysis of 8 coarse pebble and sand sizes, the usual VA-tube sizes, and the pipette. The form may not be sufficient if it is necessary to separate more than 8 sizes by sieve. In these few cases, the end may be cut from another form and attached to the master form giving 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 immersion technique is a rapid and convenient way of obtaining particle volume. The nominal diameter in cm., $d_n = \sqrt[3]{1.92V}$ where V is the volume in cu. cm. 2. The nominal diameter disregards the important aspect of particle shape. This is overcome by measurement of long, intermediate, and short diameters and designating each a, b, and c, respectively. The mean diameter is then computed,

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

Additional details regarding the direct measurement of large particles 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 about 100 grams, use the Jones-type splitter to obtain a portion weighing from 50 to 100 grams. Enter the weight of the entire sample and of the split portion in the appropriate place on the analysis form. Place the split portion of the sample in a nest of 8-inch 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 the "Rotap" sieving machine is not available and a nest of 3-inch 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 around 400 grams 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 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 about 15 grams and is analyzed by the VA-tube method. If the original sample weighs more than 400 grams 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 from the sieve 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 mm sieve were used for this 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 pipette 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 method, VA-tube method, and pipette method. For such samples, the initial part of the procedure is the same as that 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. The pipette fraction is separated from the VA-tube fraction by means of the same procedure recommended in Section V of this chapter; the splitting procedure is the same whether the sample is of suspended sediment or is of bed material.

Calculation of results.-- The procedure for calculation of results of particle-size analyses are generally well-known and need not be repeated; however, a few general statements concerning the procedure may be helpful.

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 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 method was used for an analysis, only the total weight of 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 0.625 are then read directly from the scale.

If the direct measurement, the sieve, the VA-tube, and the pipette methods are all used for an analysis, then the weight of the entire sample, weight of each large particle, weight of each sieve fraction, weight of split portion which includes the VA-tube and pipette fractions, and weight of pipette fraction 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 pipette 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, classification of sand separates (very fine sand, fine sand, medium sand, etc.) 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, then 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, then the visual-tube method should probably be used for the analysis of the sand. The pipette method should be used for analysis of the silt-clay fraction regardless of the method of sand analysis.

Soil samples will commonly contain considerable amounts of both microscopic and macroscopic organic material. The method for removal or organic matter in soil samples is the same as for sediment samples; the method is discussed in section VIII of this chapter.

Samples collected from some soil horizons 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.

MISCELLANEOUS FACTORS FOR CONSIDERATION IN OPERATION OF A SEDIMENT LABORATORY

(Section VIII)

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, since concentration is defined as the ratio of the weight of dry matter in the sample to the weight of the water-sediment mixture. Exception to this may be found where streams are utilized for washing coal. Organic material does, however, influence average specific weight and greatly influences the particle-size analysis if present in sufficient quantities.

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 material consists of 5 percent or more of organic matter. 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.

In the process of analyzing sediment for particle size gradations, 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 it is removed. Fourfold increases in the percentage of clay were obtained for some samples by treatment with hydrogen peroxide. Other investigators (Baver, 1956) have found that oxidation of organic matter with hydrogen peroxide is essential for the complete dispersion of soil particles.

In close agreement with recommendations of the International Society of Soil Science, the following procedure is set forth for removal of most forms of organic material: Add about 5 milliliters of 6 percent solution of hydrogen peroxide for each gram of (dry) sample which is contained in about 40 milliliters of water. Stir thoroughly and cover. If oxidation is slow, or after it has slowed, the mixture is heated to 200° F 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 2 or 3 times with distilled water.

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 is due 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 this problem, it is recommended that for some samples, particle-size distribution be determined using three different settling media: (1) one portion to be treated with hydrogen peroxide, and chemically and mechanically dispersed, (2) a second portion to be chemically and mechanically dispersed but not treated with hydrogen peroxide, and (3) a third portion to be analyzed in the native water medium and, of course, not treated with hydrogen peroxide. This suggested method 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.

For samples containing significant quantities of coal, it is essential that separation and quantitative determination be made on the basis of difference in specific gravity. This has been accomplished with a mixture of bromoform and acetone adjusted to a specific gravity of 1.95 (White, W. F. and Lindholm, C. F. 1950). The sediment then either floats or settles into portions lighter or heavier, respectively, than a 1.95 specific gravity. In the programming for determination of particle sizes, attention should be given the feasibility of analyzing both the mixture of all sediment and the part heavier than a specific gravity of 1.95 for some samples.

Due to the drastic and unknown effects on the sediment (other than organic matter), 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 one 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.

Dissolved solids.--The term dissolved solids is theoretically 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~~, F. H. 1957~~ and Thatcher, 1959) due to the fact that massive residues give up their waters of crystallization more slowly than their residue films, and due to the possibility of entrapped pockets of water from "sealing 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 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 to determine reliable gradation data; (3) The dissolved solids concentration is published with size analyses determined with native water as a settling media for possible correlation with flocculation tendencies.

The method of determination for sediment laboratories should be 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 one 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 due to 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 principally sand.

Calculation for concentration is as follows:

$$\text{ppm dissolved solids} = \frac{\text{gm. residue} \times 1{,}000{,}000}{\text{ml. of sample}}$$

The answer should be reported to the nearest whole number and to only 3 significant figures above 1,000 ppm.

Other analyses for chemical quality.--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 analysis and made 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. The results of these chemical analyses are then noted as constituents of the native water settling media for the size analysis and probably are not representative of the stream at the time the sediment samples were collected.

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 fair degree of accuracy. The accuracy of the method then depends on the accuracy of the volume measurement. If the sample is fairly large, the volume is determined by noting the difference in volume of a liquid before and after immersion of the sample. This direct method of volume measurement is most suitable for large fragments but may result in considerable error due to air-filled pore space in or on the object or sample. For fine sediment where small samples may be used, 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 cc of the water and insert 1.000 gm 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. The specific gravity is, therefore, $= \frac{1}{(a+1) - b}$. If some other liquid is substituted for water to avoid difficulty with air bubbles adhering to the sand or crushed material, the computation must take into account the specific gravity of the liquid.

Specific weight.--Specific weight is weight per unit volume. The most common English system of dimensions used in connection with soils and sediment deposits or of water-sediment mixtures is that of lb. per cu. ft. 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 sampler volume considering the fact that any sampling technique is likely to disturb the sample in some way.

A LIST OF LABORATORY EQUIPMENT

(Section IX)

(Equipment needed in the operation of a laboratory to determine suspended-sediment concentration and particle-size distribution of fluvial sediment)

Shaw pipette rack

Sieve shaker with automatic timer (for 3 inch diameter sieves)

Sieves, 3-inch diameter, for use with sieve shaker; 2 each of 16.0 mm, 8.0 mm, 4.0 mm, 2.0 mm, 1.2 mm, 1.0 mm, 0.50 mm, 0.25 mm, 0.125 mm, 0.062 mm and 0.053 mm diameter sieve openings.

Soil dispersion mixer with cup

Jones-type dry sediment splitter: 1 large size and 1 small size

BW-tube sediment splitter

Drying ovens, 30-200 degrees C (2)

Vacuum pump

Gram-atic balance, semi-micro (weighing 0 to 100 gm. to nearest 0.0001 gm.)

Rotap sieving machine

Sieves, 8 inch diameter, same size openings as for set of 3-inch diameter sieves, 1 each

Visual-accumulation-tube apparatus

Visual-accumulation tubes, 120 cm length: one tube each having 2.1 mm, 3.4 mm, 5.0 mm, and 7.0 mm diameter accumulation sections

Visual-accumulation tube, 180 cm length, 10 mm diameter accumulation section

Bottom-withdrawal tubes (6)

Graduated cylinders, 500 ml and 1,000 ml (12 each)

Evaporating dishes, pyrex, 80 mm diameter top (200)

Watch glasses, pyrex, diameter 90 mm, 100 mm, and 125 mm (20 each)

Beakers, pyrex: 250 ml (10), 400 ml (10), 600 ml (10), 1,000 ml (4), 2,000 ml (2)

Thermometers, laboratory, 10 to 260 degrees C., (10)

Stop watches (for use during pipette analyses) (8)

Crucibles, Gooch, 40 mm top (300)

Fiber glass filter papers, 2.4 cm diameter

Still, electric, with low-water cutoff

Carboys, pyrex, 5 gallon capacity (4)

Bottles, pyrex, 1 gallon capacity (10)

Bottles, weighing, high form with covers, 40 mm inside diameter (25)

Glass tubing, pyrex brand, various inside diameters from 3 to 10 mm

Rubber tubing, various bores from 1/8 to 1.0 inch diameter

Copper tubing, one-fourth, five-sixteenths, and three-eights inch outside diameter (25 feet each)

Clamps, tubing; push-type, pinchcock, and screw compressor (10 each)

Dessicators, Scheibler, plain, 250 mm inside diameter, with covers and plates (10)

Dessicators, Scheibler, with ground glass stopcock, with covers and inside plates (2)

Dessicant, for use in dessicators; activated alumina with indicator

Funnels, pyrex, variety of sizes and stem lengths

Pycnometers, pyrex brand

Burettes, with straight stopcock, 50 ml capacity (2)

Burettes, with 3-way stopcock, 50 ml capacity (2)

Pipettes, pyrex: 1 ml, 5 ml, 10 ml, 25 ml, 50 ml, and 100 ml (2 each)

Supports, rectangular base with rings (4)

Stopcock lubricant

Burette support with clamps

Burners, Bunsen, with stapilizer top

Burners, Tirrill, with stabilizer top

Crucible holders, rubber

Tripods, iron, with burner support

Triangles, nichrome with fused silica tubes

Beaker tongs

Dish tongs

Crucible tongs

Balance, double-beam, 200 gram

Scale, Toledo, direct reading (weighing 0 to 1,000 gm. to nearest 0.5 gm.)

Other laboratory accessories such as spatulas, rubber gloves, various stopcocks, aspirators, rubber pressure bulbs, test tubes, sieve brushes, dish-washing machines, various types of flasks, hand lens, microscope, ceramic ink, marking pencils, Berkefeld filter tubes, rubber gaskets, and reagent bottles should be available for convenient and efficient operation of a laboratory.

Selected References

1. Baver, L. D., 1956, Soil physics: John Wiley and Sons, New York, Third edition.

2. Grim, Ralph E., 1953, Clay mineralogy: McGraw-Hill Book Co., New York.

3. Krumbein, W. C. and Pettijohn, F. J., 1938, Manual of sedimentary petrography: D. Appleton-Century Co.

4. Rainwater, F. H. and Thatcher, L. L., 1959, Methods for collection and analysis of water samples: U. S. Geol. Survey Water-Supply Paper 1454.

5. Robinson, G. W., 1922, Notes on the mechanical analysis of humus soils: Jour. Agr. Sci., v. 12, p. 287-291.

6. Rouse, H., 1938, Fluid mechanics for hydraulic engineers: McGraw-Hill Book Co., New York, p. 345-350.

7. White, W. F. and Lindholm, C. F., 1950, Water resources investigation relating to the Schuylkill River Restoration Project: Pennsylvania Department of Forests and Waters. p. 18.

8. U. S. Inter-Agency Report 4, 1941, Methods of analyzing sediment samples: U. S. Army Corps of Engineers, Iowa City, Iowa.

9. U. S. Inter-Agency Report 7, 1943, A study of new methods for size analysis of suspended sediment samples: U. S. Army Corps of Engineers, Iowa City, Iowa.

10. U. S. Inter-Agency Report 10, 1953, Accuracy of sediment size analyses made by the bottom-withdrawal-tube method: U. S. Geol. Survey, Minneapolis, Minn.

11. U. S. Inter-Agency Report 11, 1957, The development and calibration of the visual-accumulation tube: U. S. Geol. Survey, Minneapolis, Minn.

12. U. S. Inter-Agency Report 12, 1957, Some fundamentals of particle-size analysis: U. S. Geol. Survey, Minneapolis, Minn.