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Chapter C1

LABORATORY THEORY

AND METHODS

for

SEDIMENT ANALYSIS

By Harold P. Guy

Book 5

LABORATORY ANALYSIS

UNITED STATES DEPARTMENT OF THE INTERIOR

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PREFACE

This series of manuals on techniques describes procedures for planning and executing specialized work in water-resources investigations. The material is grouped under major subject headings called books and further subdivided into sections and chapters; Section C of Book 5 is on sediment analysis.

The unit of publication, the chapter, is limited to a narrow-field of subject matter. This format permits flexibility in revision and publication as the need arises.

Provisional drafts of chapters are distributed to field offices of the U.S. Geological Survey for their use. These drafts are subject to revision because of experience in use or because of advancement in knowledge, techniques, or equipment. After the technique described in a chapter is sufficiently developed, the chapter is published and is sold by the U.S. Geological Survey, 1200 South Eads Street, Arlington, VA 22202 (authorized agent of Superintendent of Documents, Government Printing Office).

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LABORATORY THEORY AND METHODS FOR SEDIMENT ANALYSIS

By Harold P. Guy

Abstract

The diverse character of fluvial sediments makes the choice of laboratory analysis somewhat arbitrary and the processing of sediment samples difficult. This report presents some theories and methods used by the Water Resources Division for analysis of fluvial sediments to determine the concentration of suspended-sediment samples and the particle-size distribution of both suspended-sediment and bed-material samples. Other analyses related to these determinations may include particle shape, mineral content, and specific gravity, the organic matter and dissolved solids of samples, and the specific weight of soils.

The merits and techniques of both the evaporation and filtration methods for concentration analysis are discussed. Methods used for particle-size analysis of suspended-sediment samples may include the sieve-pipet, the VA tube-pipet, or the BW tube-VA tube depending on the equipment available, the concentration and approximate size of sediment in the sample, and the settling medium used. The choice of method for most bed-material samples is usually limited to procedures suitable for sand or to some type of visual analysis for large sizes.

Several tested forms are presented to help insure a well-ordered system in the laboratory to handle the samples, to help determine the kind of analysis required for each, to conduct the required processes, and to assist in the required computations. Use of the manual should further "standardize" methods of fluvial sediment analysis among the many laboratories and thereby help to achieve uniformity and precision of the data.

Introduction

Acknowledgments

The writer has attempted to document the "special" procedures developed and routinely used by many scientists and laboratory technicians for sediment analysis. These procedures are not individually acknowledged because they have usually evolved as a result of the many sediment conditions encountered in the many kinds of streams in the United States and because the scientists and technicians who have developed these highly efficient systems did so in close association with fellow workers. It is regrettable that the writer cannot document all these procedures in great detail because it is difficult to obtain complete information and because there is need for standardization. Sedimentation theories, however, are better acknowledged; but again, many contributions have evolved from individuals or groups and are not fully documented.

Among the many who have contributed to this chapter, as well as the earlier 1960 draft, special appreciation is extended to C. D. Albert, F. C. Ames, B. C. Colby, B. R. Colby, C. R. Collier, R. K. Flint, J. C. Mundorff, G. Porterfield, and R. B. Vice.

Functions of a sediment laboratory

A sediment laboratory in the Water Resources Division 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, streambed material, and reservoir deposits. Other kinds of analyses, usually associated with these two functions, may include quantitative determination and (or) removal of organic matter and dissolved solids, the specific

gravity of sediment particles, the specific weight of soils and sediment deposits, particle shape determinations, and mineral analyses.

Data from the concentration and particle-size 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 exposed and submerged reservoir deposits. These are but a few of the many uses made of concentration and particle-size data of fluvial sediment. The reliability of these computations and the utility of the data are dependent on the accuracy and reliability of the laboratory analyses. This discussion of the functions of a sediment laboratory is given to provide insight into some of the problems encountered in sediment analyses and to help guide the field technician in the collection of samples that are suitable for the desired analysis.

In the determination of suspended-sediment concentration, the quantity and characteristics of the sediment, and the chemical quality of water from the stream (native water) must be considered in the processing of the sample. Too small a quantity of sediment tends to magnify errors inherent in determining the weight of dry sediment, or in the transfer of material from one container to another. On the other hand, too large a quantity of sediment may cause problems with respect to splitting, drying, and weighing the sample. Samples collected with certain types of mineralized water and (or) containing colloidal clay result in difficult separation of sediment from the native water. A weight correction based on the quantity of dissolved solids is often necessary when the evaporation method is used to determine suspended-sediment concentration. These and other aspects of suspended-sediment concentration determinations are discussed later in this chapter.

The determination of the sediment particle-size distribution in a sample of streamflow offers several challenges in addition to those encountered in determining concentration. As discussed later, the sieve-pipet method or the visual-accumulation tube-pipet method is recommended in order to obtain maximum information regarding fall velocity. Since each kind of analysis requires a rather narrow range of sediment quantity, the much larger range normally found in sediment samples is likely to be a problem. Compositing or grouping of the sediment from several samples into one may be necessary when the stream concentration is low; or, a sample from a stream having a high concentration may need to be split so that an optimum smaller quantity of sediment may be analyzed. For example, the quantity of sand to be analyzed in the visual-accumulation tube can range from 0.05 to 0.8 g (gram) for the smallest tube to about 5 to 15 g for the largest tube; or in the case of the pipet, the quantity of silt and clay must be just sufficient to yield a concentration between 2,000 and 5,000 ppm (parts per million) in the sedimentation tube. Each kind of analysis has its own range of sediment quantity necessary to obtain optimum results.

In addition to the problem of determining the kind of analysis to use, it is necessary to keep in mind the need to determine the concentration of sediment in the samples for which particle-size distribution is determined. Provision should, therefore, be made to obtain the weights of all increments of sediment in the analysis as well as portions not used in the analysis. The sample must be kept wet until the particle-size analysis is complete to prevent the formation of aggregates that resist redispersion. Division of a combined sample into separate portions for separate concentration and particle-size analysis should be avoided unless the sample can be divided into two samples each representing the streamflow at the given time. Splitting may introduce quantitative and qualitative errors that will affect the concentration and (or) particle-size results. Procedures for

determining particle-size distribution of fluvial sediment by sieve and sedimentation methods are discussed in sections to follow. The frequency of sampling for analysis and the need for special analytical procedures for both native and dispersed settling media are also discussed.

The determination of particle-size distribution of bed-material samples is different from the determination of particle-size distribution of suspended-sediment samples because the quantity of material is usually large, the samples are usually sand with a considerably larger median size and most samples will be in a room-dry condition. Unlike suspended-sediment samples, which often have very limited quantities of material available for analysis, the larger bed-material samples with proper splitting allow the use of optimum quantities of material for all size ranges.

Sieves are used to determine particle-size distribution of material ≥ 2.0 mm (millimeter), whereas the VA tube (visual-accumulation tube) is used most frequently to determine particle-size distribution of material from 0.062 to 2.0 mm. However, the sieve can be, and sometimes is, used for the entire range of sand and gravel sizes. Particle size determined by the VA tube is referred to as sedimentation diameter and the relationship between sieve size and sedimentation diameter is well known for quartz spheres. It is usually assumed that the particles have a specific gravity of 2.65 unless the material in the sample appears to contain significant quantities of minerals other than quartz. Both specific gravity and particle shape vary depend-

ing on the geologic and hydrologic characteristics of any given stream and drainage basin. It may be useful to check the specific gravity and shape characteristics of sediment for a specific stream in order to convert the fall diameter of sediment determined by the VA tube to standard fall velocity or actual particle size.

Units of measurement and definitions

The methods used for collecting sediment samples are described in a chapter of this series entitled "Field Methods for Fluvial Sediment Measurement." Though it is possible to determine many kinds of sediment concentration such as the temporal mean or the average along a line, over an area, and throughout a specified volume, the sampling and compositing procedures are usually designed to give a time-integrated, velocity-weighted, cross-sectional average value of concentration or size distribution.

The common unit for expressing suspended-sediment concentration is milligrams per liter (mg/l) and is computed as one million times the ratio of the dry weight of sediment in grams to the volume of water-sediment mixture in cubic centimeters. Other units, such as percent or parts per million (ppm), have been used to express suspended-sediment concentration but are not recommended. In the laboratory it is more convenient to obtain the weight of the water-sediment mixture than to obtain its volume. Therefore, the following formula involving parts per million is used

$$\text{mg/l} = C \text{ (ppm)} = C\left[\frac{\text{weight of sediment} \times 1{,}000{,}000}{\text{weight of water-sediment mixture}}\right]$$

where C is given in table 1. 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 variation due to particle characteristics and compaction makes the assumption of a specific weight for the conversion of volume into weight difficult.

Table 1.—Factors C for computation of sediment concentration in milligrams per liter when used with parts per million or the ratio (times 10^6) of the weight of sediment to the weight of the water-sediment mixture

[The factors are based on the assumption that the density of water is 1.000, plus or minus 0.005, the range of temperature is 0°-29°C, the specific gravity of sediment is 2.65, and the dissolved solids concentration is less than 10,000 parts per million]

Ratio	C	Ratio	C	Ratio	C
0– 15, 900	1. 00	234, 000–256, 000	1. 18	417, 000–434, 000	1. 36
16, 000– 47, 000	1. 02	257, 000–279, 000	1. 20	435, 000–451, 000	1. 38
47, 000– 76, 000	1. 04	280, 000–300, 000	1. 22	452, 000–467, 000	1. 40
77, 000–105, 000	1. 06	301, 000–321, 000	1. 24	468, 000–483, 000	1. 42
106, 000–132, 000	1. 08	322, 000–341, 000	1. 26	484, 000–498, 000	1. 44
133, 000–159, 000	1. 10	342, 000–361, 000	1. 28	499, 000–513, 000	1. 46
160, 000–184, 000	1. 12	362, 000–380, 000	1. 30	514, 000–528, 000	1. 48
185, 000–209, 000	1. 14	381, 000–398, 000	1. 32	529, 000–542, 000	1. 50
210, 000–233, 000	1. 16	399, 000–416, 000	1. 34		

Several measures of particle size 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 [1] are quoted essentially as reported in (U.S. Inter-Agency Committee on Water Resources, Subcommittee on Sedimentation, 1957b, p. 11):

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 diameter of a sphere equal to 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 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 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 generally 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 (the nominal diameter as defined above). When consideration of the density or mass of the particle is necessary, 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 in terms of square openings; but in reality, many irregular particles that have nominal diameters much greater than the stated sieve openings will pass through a given 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. Thus, it is assumed that the nominal and sieve diameters of a sphere are always equal. It should be mentioned further that the nominal diameter of a sieved fraction may be larger than the indicated sieve diameter if the sieve is badly worn

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[1] The meaning of the term "particle" is often rather ambiguous. 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 the "ultimate particles" of soil. The size of the ultimate particles also determines many soil properties. These so-called ultimate particles may also be considered as 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 be aware of the kinds of particles to be measured. In sediment investigations, the ultimate particle size should be used unless otherwise specified. Ultimate particle size in fluvial sediment can be defined for practical purposes as the particle size obtained by standard methods of preparation and dispersion of sediment samples. Standard methods of preparation and analysis in native water usually results in floccules of particles, especially in the clay sizes.

or if some openings have been stretched out of their "square" shape.

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 embedded 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 which is 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. It is apparent from the definition previously given that the *standard sedimentation diameter* of a particle depends only on the volume and shape of the particle, and that the relationship 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.

The physical size of particles, especially for gravel and larger particles, may be expressed on the basis of the length of three diameters of the particle (a, b, and c) which are mutually perpendicular (Inter-Agency Report 12, p. 12). 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 on the basis of 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 by various investigators to express size. 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°, 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 studies, for 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.

With respect to methods of particle-size analyses, the following distinction is made between a dispersed system and a stratified system as reported in (U.S. Inter-Agency Committee on Water Resources, Subcommittee on Sedimentation, 1957b, p. 13):

A *dispersed system* is one in which particles begin to settle from an initial uniform dispersion and in which particles of sedimentation sizes settle together. Size distribution may be determined by measuring the
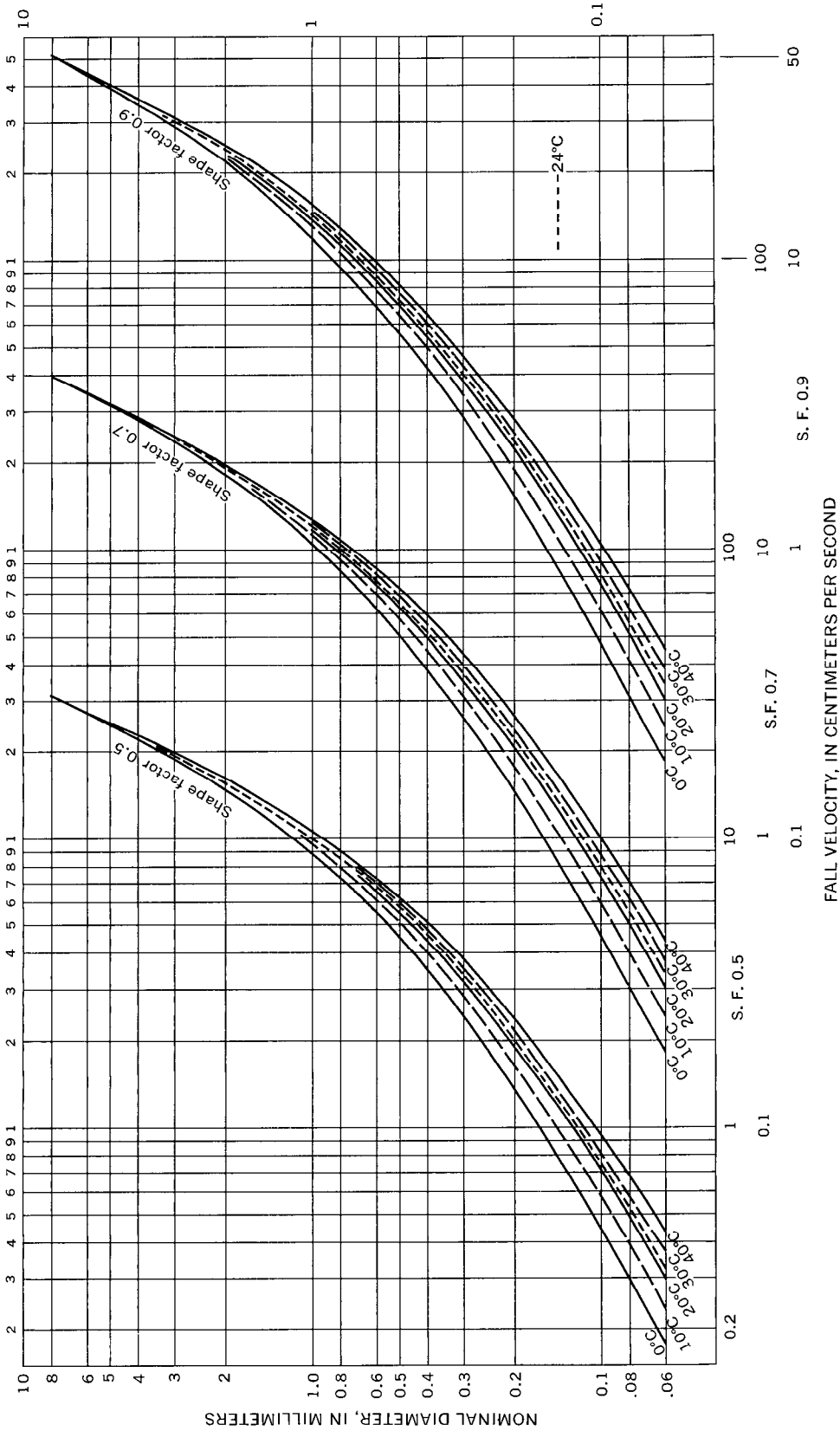
Figure 1.—Relation of nominal diameter and fall velocity for naturally worn quartz particles with shape factors (s.f.) of 0.5, 0.7, and 0.9 (From figure 2 in Inter-Agency Report No. 12 (U.S. Inter-Agency Committee on Water Resources, Subcommittee on Sedimentation, 1957a).)

concentration of sediment at given intervals of depth and settling time, as in the pipet 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 scale 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 as a result of 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 2 gives the number for Tyler and U.S. Standard sieves and the size limits for each class name of the recommended gradation scale.

The phi scale shown in column 6, derived by Krumbein (1934), is a logarithmic transformation that has integers for the class limits that increase with decreasing grain size. This scale was developed to make it easier to directly apply conventional statistical practices to sediment-size data.

Specific gravity is the ratio of the weight of a substance to the weight of an equal volume

Table 2.—Recommended scale of sizes by classes for sediment analysis

[After Lane and others (1947)]

Tyler sieve No. (1)	U.S. standard sieve No.[1] (2)	Class name (3)	Metric units (millimeters) (4)		Metric units (micrometers) (5)		Phi value [1] (φ) (6)	English units (feet) (7)	
----	----	Boulders	>256		----		----	>0. 840	
[2]	[2]	Large cobbles	256	−128	----		−8	0. 840	−0. 420
[2]	[2]	Small cobbles	128	− 64	----		−7	0. 420	−0. 210
[2]	[2]	Very coarse gravel	64	− 32	----		−6	0. 210	−0. 105
[2]	[2]	Coarse gravel	32	− 16	----		−5	0. 105	−0. 0525
[2]	[2]	Medium gravel	16	− 8. 0	----		−4	0. 0525	−0. 0262
2. 5	[2]	Fine gravel	8. 0	− 4. 0	----		−3	0. 0262	−0. 0131
5	5	Very fine gravel	4. 0	− 2. 0	----		−2	0. 0131	−0. 00656
9	10	Very coarse sand	2. 0	− 1. 0	2000	−1000	−1	0. 00656	−0. 00328
16	18	Coarse sand	1. 0	− 0. 50	1000	− 500	0	0. 00328	−0. 00164
32	35	Medium sand	0. 50	− 0. 25	500	− 250	+1	0. 00164	−0. 000820
60	60	Fine sand	0. 25	− 0. 125	250	− 125	+2	0. 000820	−0. 000410
115	120	Very fine sand	0. 125	− 0. 062	125	− 62	+3	0. 000410	−0. 000205
250	230	Coarse silt	0. 062	− 0. 031	62	− 31	+4	0. 000205	−0. 000103
----	----	Medium silt	0. 031	− 0. 016	31	− 16	+5	0. 000103	−0. 0000512
----	----	Fine silt	0. 016	− 0. 008	16	− 8	+6	0. 0000512	−0. 0000256
----	----	Very fine silt	0. 008	− 0. 004	8	− 4	+7	0. 0000256	−0. 0000128
----	----	Coarse clay	0. 004	− 0. 0020	4	− 2	+8	----	
----	----	Medium clay	0. 0020	− 0. 0010	2	− 1	+9	----	
----	----	Fine clay	0. 0010	− 0. 0005	1	− 0. 5	+10	----	
----	----	Very fine clay	0. 0005	− 0. 00024	0. 5	− 0. 24	+11	----	

[1] For maximum size of the given class.
[2] Sieve openings are marked in inches and millimeters.

of water at 4°C. The measurement is then dimensionless. It should not be confused with *specific weight* which is defined as the weight per unit volume; for example, water has a specific weight of 62.4 pounds per cubic foot and a specific gravity of 1.0.

Theory of particles falling in a fluid

Table 3 illustrates the relative position of the finer particles commonly present in sediment samples with respect to the general subdivisions of particulate 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 0.002 mm in the coarse-suspensions category. Further separations would require excessive time and lack practical value in hydraulic engineering work; however, in many scientific fields a knowledge of the size distribution of material finer than 0.002 mm can be useful.

The settling medium

Most methods of determining particle-size distribution gradation are based upon the action, size, and concentration of particles in fluids. The milling, ceramic, cement, abrasive, and paint industries usually accomplish the separation into size classes 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 measures the tendency for particles of similar specific gravity to fall at a rate that is related to the size and shape of the particles. Several laws or mathematical expressions have been developed for expressing the relation of particle size to settling velocity. These laws, especially Stokes law, serve as a basis for many size-separation techniques for particles in the silt and clay classes. Particularly important for the discussion to follow are the pipet and BW tube (bottom-withdrawal-tube) methods. For most particle-size analysis, either sieves or the VA tube are used for 0.062 mm and larger sizes. The latter has the advantage of being a hydraulic method and helps to insure continuity among the size classes representing the range from the fine to the coarse particles. In the following discussion, the settling medium is assumed to be water because it is the universal environment of fluvial sediment and has most of the desirable physical characteristics for analytical use.

Table 3.—The subdivision of matter and the resulting properties

[From table 1, U.S. Inter-Agency Committee on Water Resources, Subcommittee on Sedimentation, 1941]

SIZE CHARACTERISTICS

Millimeters .... mm..	1–0.001	0.001–0.0001	0.0001–0.00001	0.00001–0.000001
Micrometers .... μ..	1,000–1	1–0.1	0.1–0.001	0.001–0.0001
Nanometers .... nm..	1,000,000–1,000	1,000–100	100–1	1–0.1
Relative number of particles	1–10 ^{9}	10 ^{6}–10 ^{12}	10 ^{12}–10 ^{18}	10 ^{18}–10 ^{21}
Relative surface of particles	1–10 ^{3}	10 ^{3}–10 ^{4}	10 ^{4}–10 ^{6}	10 ^{6}–10 ^{7}

QUALITIES STUDIED	COARSE SUSPENSIONS	COLLOIDAL SUSPENSIONS	COLLOIDAL SOLUTIONS	MOLECULAR SOLUTIONS
Appearance in water	Very cloudy	Turbid	Clear	Clear.
Particles observed	With naked eye	With microscope	With ultra-microscope ..	Cannot be observed.
Rate of settling	Quickly or overnight	Slowly or not at all	Do not settle	Do not settle.
Particles separated from water	With filter paper	With clay filters	With ultra-filter	Not by filtration.
Determination of particle sizes	Sieves, microscope, gravity, or centrifugal settling.	Microscope, centrifugal settling, absorption or dispersion of light.	Ultra-microscope, ultra-centrifuge.	Ultra-centrifuge.
Form after evaporation	Loose powders	Powders and gels	Gels	Crystals.
Soil separates	Sand, silt, and (or) clay ..	Suspended clay	Ultra clay	Soil solution.

NOTE.—Limits of size measurements of microscopes and filtering materials:

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

Stokes law for small particles

The nature of particle movement in a settling medium depends on a balance of the accelerating and retarding forces which, in turn, depend 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; U.S. Inter-Agency Committee on Water Resources, Subcommittee on Sedimentation, 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 the relative movement of the fluid extends a considerable distance away from the particle. Stokes law expresses the viscous fall of particles under such conditions, usually for quartz sediment particles finer than 0.062 mm. If the fall velocity of a particle is somewhat faster than that normal for the Stokes law range, then the inertial effects of the fluid reduce the viscous influence to a thin layer around the particle which produces the effect of surface drag. For the larger sizes of particles, the inertial effects of the fluid become increasingly important in the flow around a particle, and therefore the form type of resistance dominates. Such flow causes a wake or low pressure area behind the particle, and eddies are formed; the net result is a force opposing motion.

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. Remembering that the net downward force is the force of gravity on the spherical particle minus the buoyant force of the liquid, or

$$4/3 \ (\pi r^3 \ \rho_s g) - 4/3 \ (\pi r^3 \ \rho_f g),$$

then these forces can be stated as

$$6\pi r \mu v = 4/3 \pi r^3 g (\rho_s - \rho_f),$$

where

$r=$ radius of sphere in centimeters,
$\mu=$ dynamic viscosity of the fluid in dyne-seconds per centimeter,
$v=$ velocity of fall in centimeters per second,

$g=$ acceleration due to gravity (980 centimeters per second,
$\rho_s=$ density of sphere in grams per centimeter,
$\rho_f=$ density of fluid in grams per centimeter.

The fall velocity as defined by Stokes law is

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

Assuming that the specific gravity of the particles is 2.65 and that water is the settling medium, the fall velocity is

$$v=\frac{0.8983 \ d^2}{\mu} \text{ or } d=\frac{\sqrt{\mu v}}{0.9487}$$

where $d=$ diameter of particle in millimeters. Figure 2 gives a graphical solution to the Stokes equation for a water temperature of 20°C. Viscosity correction factors for use with the graph when temperatures other than 20°C are used are tabulated in table 4.

Basic assumptions 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 near 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 (U.S. Inter-Agency Committee on Water Resources, Subcommittee on Sedimentation, 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 group of particles creating a region of high concentration may cause a localized area of higher density than the settling medium and thus settle as a unit at an abnormally high rate. The sphericity of the particle is of no great concern since the particle velocity is compared with 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.
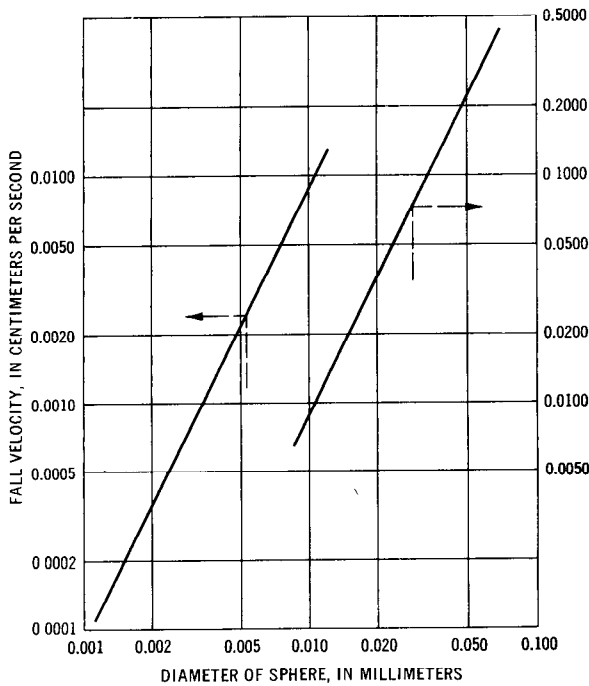

Figure 2.—Relation between fall velocity and diameter (Stokes law) for quartz spheres in water at 20°C.

Table 4.—Fall velocity correction factors for use with figure 2 when temperatures are different from 20°C.

Temperature (°C)	Correction factor $\left(\frac{\mu_T}{\mu_{20}}\right)$	Temperature (°C)	Correction factor $\left(\frac{\mu_T}{\mu_{20}}\right)$
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		

Drag-Reynolds number

Stokes law is not applicable for quartz particles larger than about 0.060 mm falling in water because the inertial effects of the fluid

reduce the viscous influence. The Reynolds number, **R**, indicates the ratio of inertial forces to viscous forces and, therefore, the character of the resistance to fall. For spherical particles falling in a fluid, **R** is a dimensionless quantity defined as $vd\rho_s/\mu$. The resistance to fall of these larger particles is $C_DAv^2\rho_s/2$. where C_D is the coefficient of resistance and A is the projected cross-section area of the particle. This resistance is equated to the net downward force as previously shown in deriving Stokes law.

$$\tfrac{1}{2}C_DAv^2\rho_s=\tfrac{4}{3}\pi r^3g(\rho_s-\rho_f).$$

The relationship between C_D and **R** has been established empirically for spheres and for some other shapes (Inter-Agency Report No. 4, p. 39, and Inter-Agency Report No. 12, p. 21 and 51). The data indicate that Stokes law is applicable up to **R**=0.1 and sometimes 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.

Significant figures for reporting results

The number of significant figures to be used in recording data from analysis in the sediment laboratory is the product of compromises between the need for uniformity in the final tables of data, precision of measurement, and also the degree of precision necessary for the use of the data.

For concentration of sediment in a sample, it is practical and desirable to report to the nearest 1 mg/l up to 999 mg/l and then for higher values to use three significant figures. Often the third significant figure in the series 500 to 999, 5010 to 9990, and so forth, has very weak significance, but is justified on the basis of uniformity. Again, in some instances it may be desirable to use

the nearest 0.1 mg/l up to 9.9 mg/l, especially between 0.1 and 0.9 mg/l. These recommendations are based on the assumption that the net sediment can be weighed to the nearest 0.0001 g and the water-sediment mixture can be weighed to the nearest 1 g.

With respect to size gradation, the goal should be to publish the percentage of material in each fraction to the nearest whole percent. In some analyses, where fractions may contain 2 percent or less, it may be useful to report to the nearest 0.1 percent. Whenever practicable, the net quantity of sediment in a given fraction should be weighed to the nearest three significant figures. With balances weighing to the nearest 0.0001 g, the significant figures must be reduced to two when the quantity is less than 0.0100 g.

Procedure for Determining Suspended-Sediment Concentration

The best methods for making determinations of concentrations and particle-size distributions from suspended-sediment samples must be selected from numerous possible procedures. The samples may contain only minute quantities of sediment of very small particle size or may contain large quantities of sediment with a wide range of particle size. Samples with such extremes usually require the use of special procedures. Samples for particle-size analysis always 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 advantages and disadvantages. The two most commonly used methods are evaporation and filtration. The filtration method may be somewhat faster than the evaporation method for samples of low concentration. However, larger quantities of sediment tend to clog the filters and inhibit the use of this method for the higher concentrations. The evaporation method requires an adjustment for dissolved solids if the dissolved solids content is high, especially for samples having low sedi-

ment concentration. Thus, the filtration method may best be used on samples ranging up to about 10,000 mg/l of sediment that is mostly sand and about 200 mg/l of sediment that is mostly clay. The evaporation method may be best used when the concentration is more than these limits.

Evaporation method

The evaporation method consists of 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. The main advantage of the method over the filtration method is the simplicity of equipment and technique. The method works well if the sediment readily settles to the bottom of the sample container because of its coarseness or because of the natural flocculation of the suspended clay. Conversely, with suspensions of naturally dispersed clay, the settling time may make the method impractical unless special procedures are used.

NOTE A.—Special procedures to handle unsettled samples involve the use of (1) a filter tube, in which case, it may be just as easy to adopt the complete filtration method, or (2) a flocculating additive to reduce the settling time. In the first procedure, the sample is allowed to settle as much as practical and then, by use of a Berkefeld filter tube (or a similar device) and a vacuum apparatus, most of the native water is removed from each bottle. The sediment in suspension adheres to the outside of the filter tube and the water passes through the walls. The material on the outside of the tube is removed by back flushing with native water from the sample and can be treated in the same manner as the dissolved solids content.

A second special procedure is to use a flocculating agent to reduce the settling time of the naturally dispersed clays. This method will require the use of a correction factor to be applied to the weight of dried sediment. Such a factor is likely to be 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 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 one most effective will depend on the nature of the sediment and the dissolved minerals in the water. Extreme care is necessary in

using either dilute HCl or CaCl₂ as a flocculant. 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 supernatant liquid is carefully decanted from the sample so that the sediment in the bottom of the container will not be disturbed nor removed. A small quantity of the supernatant liquid is therefore left with the sediment. A specified amount of such liquid should be left with each sample, usually about 20 to 50±2 ml (milliliters). This specified amount is especially significant if a correction for dissolved solids is necessary. Distilled water is used to wash the sediment and remaining liquid into the evaporating dish. The contents of the dish are then dried in an oven at a temperature that is slightly below the boiling point. If the boiling point is reached, then sediment may be lost from the dish. After all visible moisture has evaporated, the temperature should be raised to 110°C for at least 1 hour.

The weight of natural dissolved solids in the supernatant water is included in the weight of dry material obtained by the evaporation method and must be subtracted to determine the weight of sediment. The following may be used as a guide to determine if a correction for the weight of 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 concentration is equal to or greater than the sediment concentration, assuming that an aliquot of 20 ml of native water is used in the evaporation, and that 400 g of water-sediment mixture is contained in the original sample. For example, a dissolved-solids correction should be made when the dissolved solids concentration is 200 mg/l or higher and the sediment content is 200 mg/l or less. Similarly, a 2 percent improvement will result if the dissolved solids concentration is 40 percent or more of the sediment concentration. The weight of the dissolved-solids correction for different aliquots and a range of concentrations can be obtained from figure 3.
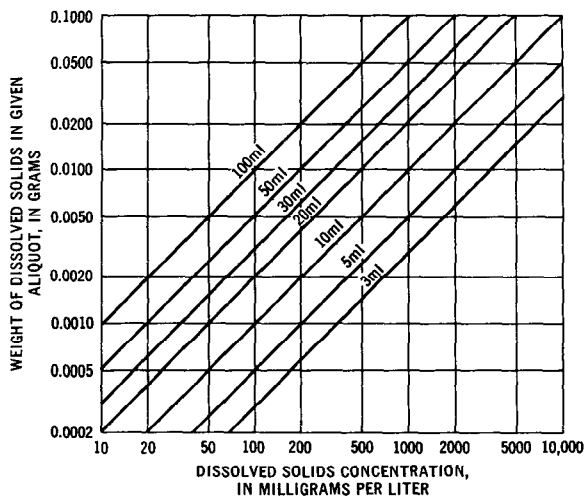

Figure 3.—Weight of dissolved solids for given size of aliquot and given concentration.

Filtration method

The filtration method for the determination of suspended-sediment concentration has obvious advantages over the evaporation method and should be used whenever it is practical to do so. The method usually utilizes a Gooch crucible in conjunction with various types of filter material. The Gooch crucible is a small porcelain cup of approximately 25 ml capacity with a perforated bottom. The crucible is easily adapted to an aspirator system and vacuum filtration; it is lighter in weight and consumes less oven and desiccator space than the evaporating dishes; its tare weight is less likely to change during the weighing because less surface is exposed to sorption by moisture; and dissolved material passes through the crucibles and thus eliminates the need for a dissolved-solids correction.

In most laboratories the Corning #32960 Pyrex fritted glass crucible and glass-fiber filter disks, #934–AH or similar specifications, have proven satisfactory for filtration of most 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 than do other types of filters. 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 a glass-fiber filter disk in conjunction with an asbestos mat. The crucible with this extra asbestos mat is prepared by placing the glass-fiber filter disk in the crucible while vacuum is applied and then pouring an asbestos slurry on top of the disk also while vacuum is applied. (See the next paragraph concerning the preparation of asbestos mats.) During the filtration process, the somewhat coarse asbestos mat retains much of the sediment that would ordinarily clog the glass-fiber disk. The glass-fiber 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 double glass-fiber asbestos-type filter.

If glass-fiber filters are not available, asbestos is considered satisfactory because of its inertness, resistance to heat, and low hygroscopicity. The asbestos filter mats can be prepared in 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 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 asbestos 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.

If the sediment sample contains extremely fine material, an actual loss of fine material through the filter may result in a turbid filtrate. This problem is 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, or (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 rapid clogging of the filter. 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 usually recommended because undesirable reactions with the sediment are likely to occur. As already mentioned, the use of a fiber-glass filter disk in conjunction with an asbestos mat will eliminate most filter problems.

Sometimes it is possible to utilize another advantage of the filtration over the evaporation method when the concentration or the amount of sediment is small. Specifically, preparation time can be saved by using the same mat two or more times. When the crucible and mat are re-used, the gross weight of the prior use becomes the tare weight for the new use.

Laboratory forms

Figures 4, 5, and 6 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 *Short form* for depth integrated samples (fig. 4), space 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 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 later withdrawn or 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, expanded notes can be retained on the front by using space ordinarily used for other bottles.

Form 9.265e (REV. 10-62)

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 _____

Figure 4.—Sample of laboratory form, Sediment Concentration Notes, Depth Integrated Samples (Short form)

Form 9 265f (REV. 10-62)

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								
Discharge								
Temperature								
Sampling Sta.								
WEIGHT OF SAMPLE — Gross								
Tare								
Net								
Container no.								
WEIGHT OF SEDIMENT — Gross								
Tare								
Net								
D.S. Corr.								
Net								
Conc. (ppm)								
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. (mg/l)								

Figure 5.—Sample of laboratory form, Sediment Concentration Notes, Depth Integrated Samples (Comprehensive form)

Form 9.265g (REV. 10-62)

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																			
Discharge																			
Sampling Sta.																			
Temperature																			
Sampling depth, ft																			
Total depth, ft																			
Filling time, sec																			
WEIGHT OF SAMPLE — Gross																			
WEIGHT OF SAMPLE — Tare																			
WEIGHT OF SAMPLE — Net																			
Filling rate, cc/sec																			
Nozzle size																			
Velocity, ft/sec																			
Specific conductance																			
Container no.																			
WEIGHT OF SEDIMENT — Gross																			
WEIGHT OF SEDIMENT — Tare																			
WEIGHT OF SEDIMENT — Net																			
D.S. Corr.																			
Net																			
Concentration (ppm)																			

Figure 6.—Sample of laboratory form, Sediment Concentration Notes, Point Integrated Samples

The entry for *D.S. Corr.* (dissolved solids) is used when the evaporation method is used and then only when the dissolved-solids content is relatively high, when an adsorbing or flocculating agent has been added to the sample, or when an adsorbing agent is used with the filtration method. Although details concering the principles of dissolved-solids determinations are discussed in the last section of this chapter, "Related water-quality analysis," 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 daily samples when the streamflow rate is relatively uniform. The composite determination is made from a sample prepared by withdrawing nearly equal volumes of the sediment-free water from each daily sediment sample. The sample, whether for a single day or a composite for several days, is evaporated to dryness, heated at 110° C for 1 hour, cooled in a desiccator, and weighed to obtain a unit weight. The dissolved solids correction to be applied to the dry weight of sediment solids in each evaporating dish is equal to the product of the unit volume correction and the volume of water evaporated from the dish. Also see page 12 and figure 4.

Procedure for concentration analysis

With the above information it is possible to compile the following step-by-step procedure for concentration analysis:

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 organisms.
3. Arrange the samples for a given location in chronological order while assembling them for transport to the sample weighing station.

4. At the sample weighing station, record the sample information from the sample labels on the appropriate concentration form. (See figs. 4, 5, and 6.)
5. Record the gross and tare weights to the nearest gram for each sample, assuming the net sample to weigh in excess of 200 g. It is often desirable to use a balance with a tare removing attachment and a procedure that will directly yield the net weight of the sample. This avoids copying and computation errors, but puts greater responsibility on the operator.
6. Store the weighed bottles on a convenient rack or table for several hours, or overnight, to assure that the sediment settles from suspension. In the event that the sediment does not settle, follow the procedure outlined in "Note A (p. 11)." Samples to be analyzed for particle-size distribution should be separated from those to be analyzed for concentration only.
7. Some laboratories at this point obtain and record the conductance of the supernatant water for each sample set. This is not published but is used as a control on the person taking the samples and as an aid in finding and correcting errors made in recording the field and lab data. In this case the comprehensive form (fig. 5) would be used.
8. Decant most of the sediment-free water using care not to disturb or remove sediment. If a dissolved-solids correction is necessary when using the evaporation method, then the amount of supernatant liquid left with the sediment should be approximately equal to one of the aliquots indicated in figure 3.
9. Use distilled water to wash the remainder into a previously weighed evaporating dish or filtration crucible, depending on the method used. Check to be certain that the container number on the form *Sediment Concentration Notes* corresponds with the given sample or samples.
10. The sample bottles must be thoroughly cleaned, air dried, recapped and packed in carrying cases for reuse.
11. Dry the evaporating dishes or crucibles and, after loss of all visible water, heat at about 110° C for 1 hour. The evaporating dishes must be dried at a temperature low enough to prevent "spattering" of sediment from the dishes by boiling action.
12. Cool the containers in a desiccator at room temperature.
13. Weigh the containers to the nearest 0.0001 g on an analytical balance. The tare weight of the evaporating dish should be obtained before each use because of the loss of weight caused by small chips, frequent washing, and slight differences in washing or cleaning methods. The tare weight of the crucible must be obtained before each use

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

14. Compute the net weight of sediment in the container and deduct, if necessary, the dissolved-solid correction.

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

Determination of Particle-Size Distribution of Suspended Sediments

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

Particle-size data have many uses. One use is to compute the probable specific weight of sediment as it will deposit in reservoirs. This computation requires plotting the median particle size against sediment load in tons per day. Information concerning the behavior of sediment particles in different environments may be studied and, therefore, requires analyses for particle-size distribution of fine particles in both native and dispersed settling media. The size distribution of both suspended and bed material has an important use in the computations of total (measured plus unmeasured) sediment discharge. The frequency and kind of particle-

size analyses should be adequate to describe the pertinent characteristics of sediment particles so that satisfactory comparisons can be made between sediments collected from different places or from the same place at different times.

Recommended frequency of analyses

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

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

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

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

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

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

Method of analysis

Limitations

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

Dispersed Settling Medium

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

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

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

Table 5.—Guide to selection of VA tube size

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

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

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

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

Pipet analyses are made using either 500 or 1,000 ml of suspension with a suspension range of 2,000 to 5,000 mg/l. However, if sufficient sediment is not available to obtain a concentration of 2,000 mg/l for a 500 ml suspension, less than 500 ml can be used for the suspension. Though not recommended for general use, such abbreviated pipet analyses, in which only the 0.002, 0.004, and 0.016 mm determinations are made, can conveniently be performed using as little as 200 ml of suspension. By decreasing the volume of suspension and the number of

sizes determined, and by using withdrawal depths of 5 or 3 cm (centimeters) or less, sufficiently accurate pipet analyses can be made using as little as 0.4 g of silt and clay. By reducing the volume of suspension in this manner, pipet analyses can be made on samples containing a small amount of sediment which otherwise would have to be analyzed by the more time-consuming and possibly less accurate BW tube method.

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

Native water settling medium

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

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

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

1. The particles in suspension loosely join to form masses in which both size and density are changing with time. Such masses fall faster than the component particles but, because of their loose aggregation, fall slower than a quartz sphere of the same nominal diameter.

2. The floccules may be in the process of accretion while settling and therefore would have an increasing and accelerating velocity rather than the constant terminal fall velocity.

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

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

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

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

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

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

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

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

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

If information concerning the chemical quality of the water is essential to a sediment investigation, standards and methods currently followed in chemical quality laboratories (Rainwater and Thatcher, 1959) should be used to

make determinations for total dissolved solids, specific conductance, pH, calcium, magnesium, sodium, potassium, bicarbonate, or alkalinity. These analyses of the water can supplement knowledge obtained by other kinds of sediment examination.

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

Sample preparation for native water analysis

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

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

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

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

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

1. Allow the samples to settle as much as practical.

2. By use of a Berkefeld filter tube (or a similar filter tube) and a vacuum apparatus, remove as much of the native water from each bottle as possible without disturbing the sediment in the bottom of the container. The fine sediment remaining in suspension adheres to the outside of the filter tube and the water passes through the walls.

3. Back flush the material on the outside of the tube with native water and carefully rinse it into the container to be used for compositing the sediment.

As an alternative procedure for treatment of samples that do not settle, the following method has been used successfully. Allow the samples to settle for several days and then, using extreme care not to agitate the sample, siphon off the top two-thirds to three-fourths of the water and

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

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

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

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

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

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

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

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

Equipment

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

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

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

Attached to the right stem of the stopcock with rubber tubing is an inverted Y-shaped glass tube. A length of rubber tubing, with a pressure bulb at one end, is attached to the top stem of the Y. A length of rubber tubing is attached to the short stem of the Y connecting the apparatus to a distilled water supply with a head of 1–1.5 meters. The flow of distilled water is controlled by a stopcock.
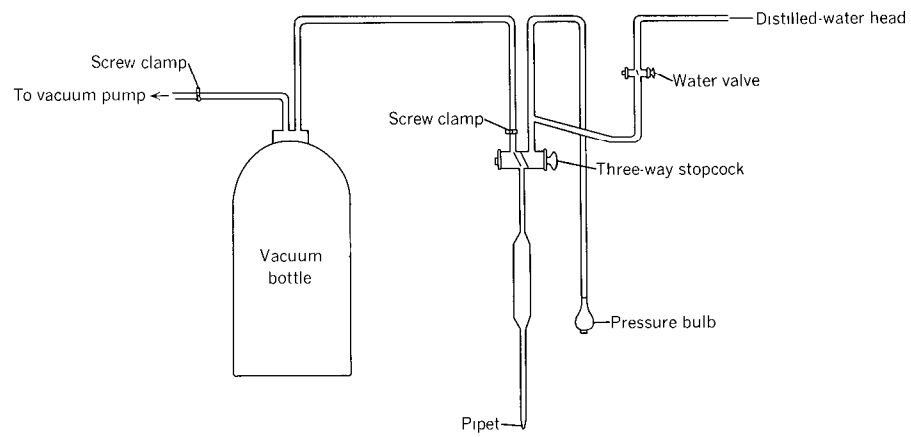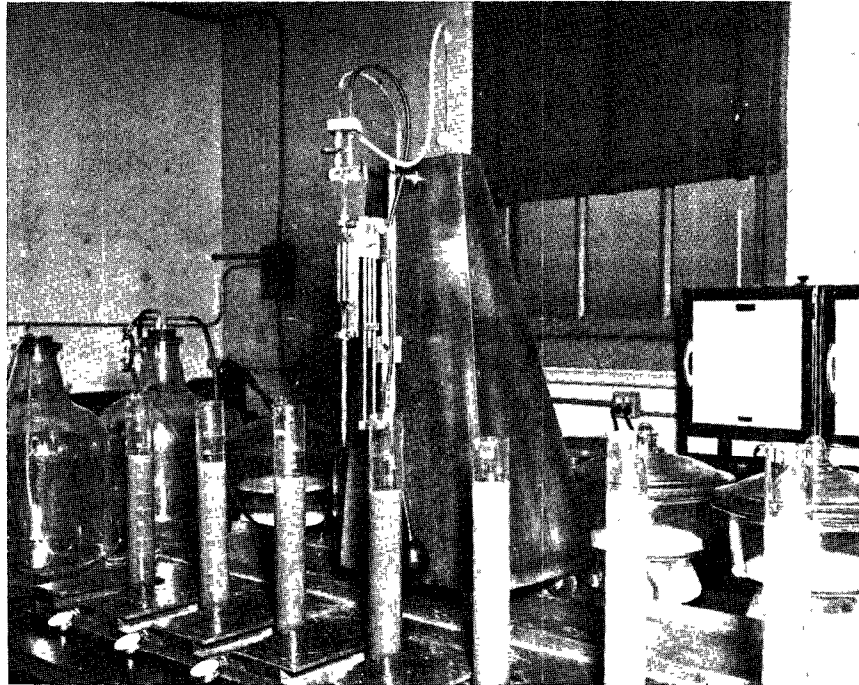
Figure 7.—Apparatus for pipet particle-size analysis.

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

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

Preparation of sample

Recording and decanting

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

stream, the date, and other pertinent data are also recorded in this block.

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

Mechanical dispersion

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

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

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

Form 9.265h(REV. 10-62)

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

File no. _____

ANALYSIS DATA	DISSOLVED SOLIDS	TOTAL SAMPLE DATA

ANALYSIS DATA

Date _____ by _____

Portion used _____

Disp. agent _____ cc.

Pipet suspen.
- Sed. _____ gm.
- Vol. _____ cc.
- Concen. _____ ppm

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

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

DISSOLVED SOLIDS

Volume _____ cc. dispersed / native

Dish no. _____

Gross _____ gm.

Tare _____ gm.

Net _____ gm.

WEIGHT OF PORTION NOT ANALYZED

Portion _____ Dish no. _____

Gross _____ gm.

Tare _____ gm.

Net _____ gm.

Dis. solids _____ gm.

Net _____ gm.

TOTAL SAMPLE DATA

Stream _____

Location _____

Date _____ Time _____

G.H. _____ Sta. _____ Temp. _____

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

Dis. solids _____ ppm

Spec. cond. _____ pH _____

Other chem. qual. _____

Remarks

SIEVE

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

PIPET

Pipet no. _____ Volume: _____ Volume factor: _____

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

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

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

Sieving

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

Separation of sand from fines

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

Dry method

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

Wet method

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

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

Analysis of silt-clay fraction

Splitting

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

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

Dispersing

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

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

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

To obtain the dissolved solids correction factor to compensate for the dispersing agent added, the following procedure is recommended.

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

Pipetting

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

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

Calculation of results

Total weight of sediment in sample

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

1. Evaporate the sample to dryness before analysis and determine the oven-dry weight to the nearest milligram. This method is not recommended because sediment when once dried is difficult to disperse and requires special treatment, especially a sediment that contains considerable quantities of clay minerals.

2. Determine the weight of the silt and clay fractions from the mean concentration and volume of the pipet settling suspension. The average concentration of the suspension is determined by making a "concentration withdrawal" immediately after mixing. The weight of sediment in the suspension cylinder is then added to the weight of the sand which was determined separately.

3. Determine the dry weight of sediment remaining in the suspension after all pipetting has been completed. To this add the dry weight of sediment in each pipet withdrawal and the dry weight of the sand fraction if it was separated.

4. Split the sample in two portions by means of a suitable splitting device. Evaporate one-half to dryness to determine the weight of sediment, and use the other half for the sieve-pipet analysis. The accuracy of this method depends on the quantitative and qualitative accuracy of the splitting operation which is subject to considerable error; therefore, the method is not recommended.

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

Calculations

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

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

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

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

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

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

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

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

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

Equipment

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

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

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

Preparation of sample

The VA tube method is suitable for samples whose particles are mainly sand. As mentioned previously, if particles larger than about 1.2 mm are present in the sample, they should be removed by sieving. If clay or silt is contained in the sample, it is removed before analysis by wet sieving with a fine sieve, by settling the sample through a sedimentation tube, or by other means such as the ultrasonic treatment mentioned on
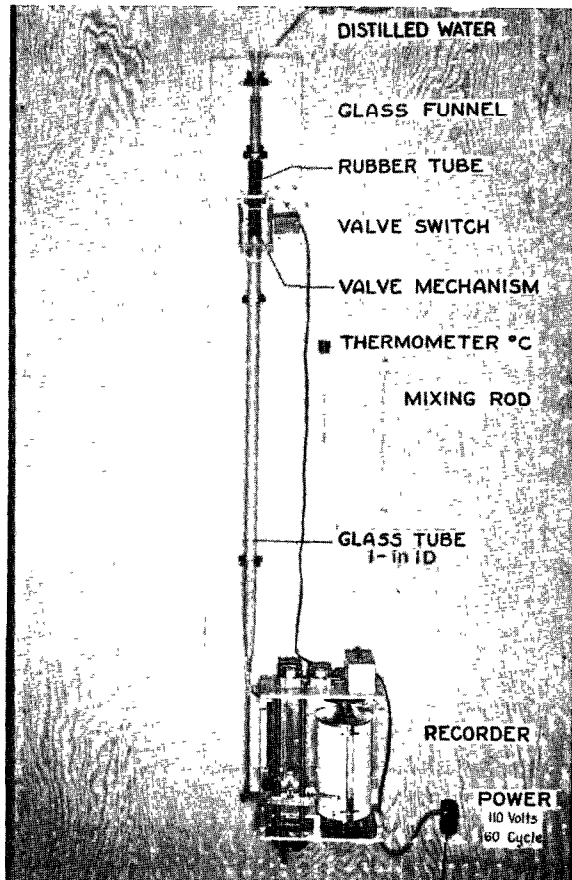
Figure 9.—VA tube and recording apparatus for analysis of sand-sized materials in terms of fall diameter.

page 20. The more thoroughly the clays and silts are removed from the sand, the simpler and faster will be the VA tube analysis although some coarse silt in the VA tube does not affect the accuracy of the analysis.

Separation of sand from fines

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

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

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

Splitting and dispersing

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

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

The VA tube analysis

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

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

Figure 10.—Sample of laboratory form, Particle-Size Analysis, VA Tube Method for use with a 120-cm tube. The reverse side is designed for sieve and pipet analysis of the same sample. See figure 12.
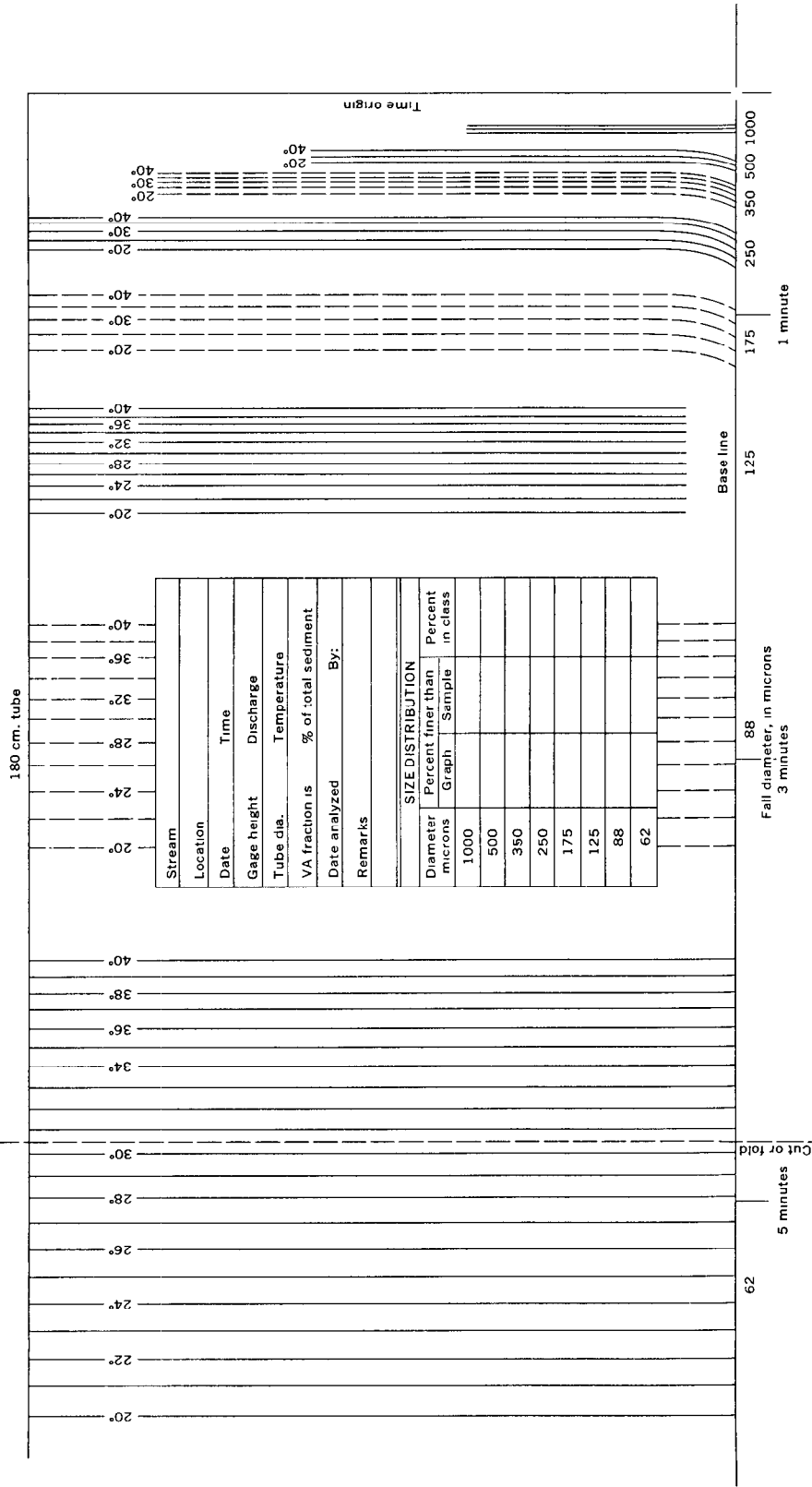
Figure 11.—Sample of laboratory form, Particle-Size Analysis, VA Tube Method for use with the 180-cm VA tube (size reduced about 35 percent). The reverse side is designed for sieve and pipet analysis of the same sample. See figure 12.

SIEVE ANALYSIS

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

Cut or fold

Stream _____

Location _____

Sampling stations _____

Method of sampling-_____

Remarks _____

Date		Lab No.	
		Time	Gage height
Start			
Finish			
Mean			
Temp.		Mean vel.	
Stream width		Depth	
Discharge			

SAMPLE FRACTIONS

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

CONCENTRATION DETERMINATIONS

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

Analysis remarks

PIPET ANALYSIS

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

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

Tube selection

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

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

Procedure

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

1. The chart is chosen for the length of tube; after notes to identify the sample, operator, and analysis are recorded, the chart is placed on the cylinder. The base line of the chart should be parallel to the bottom of the cylinder so that the pen trace will be parallel to the base line except when sediment is accumulating. (If the chart is used for two or more samples, a different base line, somewhat above the original, may be used by drawing it with the recorder pen. The 180 cm and the 120 cm tubes require different charts because of the unequal distances through which the sample must settle.)

2. The recorder pen is oriented on the zero-accumulation and zero-time lines of the chart. The pen should be started to the right of the zero-time line and brought to the line by the motor-driven rotation of the cylinder.

3. The recorder is adjusted to bring the horizontal hair in the eyepiece level with the top of the tube plug where the accumulation of sediment begins.

4. When the apparatus, including the proper sedimentation section, is assembled, the tube is filled with distilled water to just above the valve. The temperature of the water in the tube is determined and recorded, and the valve is closed. Normally the water need not be changed after each analysis.

5. The electrical tapping mechanism is started; this operation also closes the electrical circuit to a switch at the valve so that rotation of the cylinder will start when the valve is opened.

6. The sand sample is washed into the funnel above the closed valve; the funnel is filled to the reference mark; then the sample is stirred briskly for 10 seconds with a special stirring rod.

7. The valve is immediately and fully opened. Because opening the valve automatically starts the cylinder, the chart time and settling of the particles in the tube begin simultaneously.

8. The operator watches through the eyepiece and, as soon as the first particles reach the bottom of the tube, he starts moving the carriage vertically at a rate that keeps the horizontal hair level with the top of the accumulation of sediment. This procedure continues until the pen has passed the 0.062 mm size on the chart; then rotation of the cylinder automatically stops. If material is 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 a horizontal line of maximum accumulation back across the chart to the time of zero accumulation.

10. After the valve is closed (and the tube plug is removed), the sample is (extruded from the tube into a beaker by gravity or by squeezing the rubber tube between the valve and the VA tube) * * * The valve (may then be) opened slightly to drain out excess water and to wash out the lower end of the tube more completely.

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

11. The chart is removed from the recorder.

Chart interpretation

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

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

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

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

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

The Oden theory

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

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

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

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

If tangents are drawn to the curve at any two points corresponding to times t_1 and t_2, and the tangents allowed to intersect the ordinate axis at W_1 and W_2, then the difference between the percentage W_2 and W_1 will represent the amount of material in a size range with limits determined by the settling times t_1 and t_2.
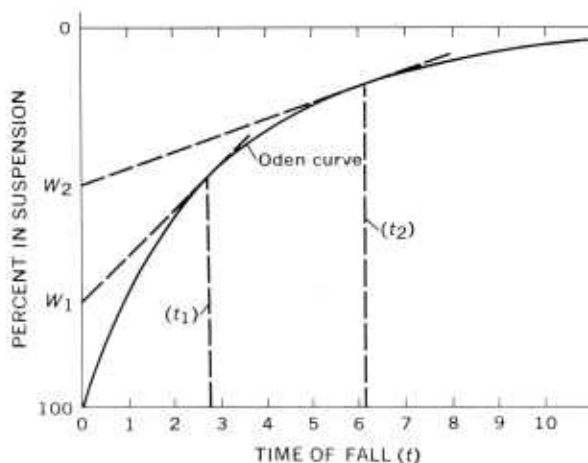
Figure 13.—Oden curve showing the relative amount of sediment remaining in suspension with time. The intercept of a tangent to the curve with the ordinate represents the percentage of sediment in suspension at a specific time of fall.

Equipment

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

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

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

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

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

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

Preparation of sample

The net weight of the water-sediment mixture for the entire sample is recorded on the form *Sediment Concentration Notes*, (figs. 4 or 5) before samples are selected for particle-size analysis. Pertinent sample information should be recorded on the *BW Tube form* (fig. 15) in the *Total Sample Data* block. After the sediment

settles to the bottom of the sample bottles, as much sediment-free supernatant native water as possible should be decanted. If the sediment is slow in settling, "Note A, p. 11."

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

Form 9.265k (REV. 10-62)

UNITED STATES DEPARTMENT OF THE INTERIOR
Geological Survey-Water Resources Division
PARTICLE SIZE ANALYSIS, BOTTOM WITHDRAWAL TUBE METHOD

Tube no. _____

ANALYSIS DATA

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

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.

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

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

TUBE WASH

TOTAL SAMPLE DATA
File no. _____

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

Remarks:

WITHDRAWAL NO.		0	1	2	3	4	5	6	7	8	9	10
a	Clock time											
b	Fall distance-cm	100	90	80	70	60	50	40	30	20	10	
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	1.67	2.00	2.50	3.33	5.00	10.0	
m	Sed. in susp. (k × l)											
n	% sed. in suspension											
o	Time for 100 cm(c × l)											

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

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

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

The BW tube analysis

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

Dispersion

To insure complete dispersion of the sediment for the dispersed settling medium, it is necessary to add 1 ml of dispersing agent (see

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

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

Withdrawals

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

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

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

Samples are withdrawn into a 100 ml graduate in order to eliminate the possibility of losing any of the sample by splashing and to permit accurate measurement of the amount withdrawn. The withdrawals are carefully transferred to evaporating dishes by washing with a stream of distilled water. The evaporat-

ing dishes are placed in the oven to dry at a temperature near the boiling point, but not so hot as to cause splattering by boiling. A small flask instead of an evaporating dish may be used for drying the sediment, if feasible to weigh and if cleaning is not too difficult. When the evaporating dishes or flasks are visibly dry, raise the temperature to 110°C for 1 hour, after which transfer the containers from the oven to a desiccator and allow them to cool. The weighing procedure is the same as that for sediment concentration determinations.

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

Recording of data

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

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

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

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

Experimental Form, May 1967

UNITED STATES DEPARTMENT OF THE INTERIOR
Geological Survey-Water Resources Division
PARTICLE SIZE ANALYSIS, BOTTOM WITHDRAWAL TUBE METHOD

Tube no. _____   File no. _____

ANALYSIS DATA

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

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

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

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

DISSOLVED SOLIDS
Volume _____ cc. dispersed / native
Dish no. _____

Weight
Sand fract. _____ gm.
Gross _____ gm.
Tare _____ gm.
BW fract. _____ gm.
Net _____ gm.
Total sed. _____ gm.
Concentration _____ mg/l

TOTAL SAMPLE DATA

Stream _____
Location _____
Date _____ Time _____ G.H. _____
Station _____ Temperature _____ °C
Spec. cond. _____ pH _____
Remarks:

WITHDRAWAL NO. 100/80

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

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

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

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

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

The Oden curve

The Oden curve is plotted on a form having rectangular coordinates such as shown on experimental form figure 17. The complete plotting of the data from entries "n" and "o" to an enlarged scale (0–7,000 min) results in a complete upper curve. Lower curves represent ex-

panded scales such as 0–350 and 0–70 min for better definition of the coarser fractions. If only silt and clay sizes are analyzed, then it may not be necessary to use a 0–70 min scale. Other horizontal (time) scales may be used on other kinds of rectangular coordinate paper so long as it is convenient to draw smooth curves through the plotted points.

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

The intercept of the tangent from the time point indicated by the given size to the ordinate at zero time (percentage in suspension) can be read as the percent finer than the indicated size. Care should be exercised in the construction of the Oden curve and in the drawing of tangents because the shape of the curve will greatly affect the intercept of the tangent with the percentage scale. From most samples, the slope of the curve does not approach zero over the period of time covered by the analysis because many fine particles are still settling at the time of the last scheduled withdrawal. Obviously, the curve should never have a reverse or increase in slope. If an increase in slope is noted, it may be the result of the flocculation of silt and clay particles during the sedimentation process. This flocculation will most likely occur in the native water settling medium and may occur in an improperly dispersed settling medium. It also should be apparent that a tangent from a curve with too steep a slope or too sharp a curvature will not result in the desired accuracy. Proper use of the expanded time scales will alleviate some of this difficulty. Comparison of intercepts for a given particle size from two curves at different scales is desirable to insure consistent construction of the curves. It should be noted that if sand is separated before analysis, the "curve" will be a straight line up to the "0.062 time."
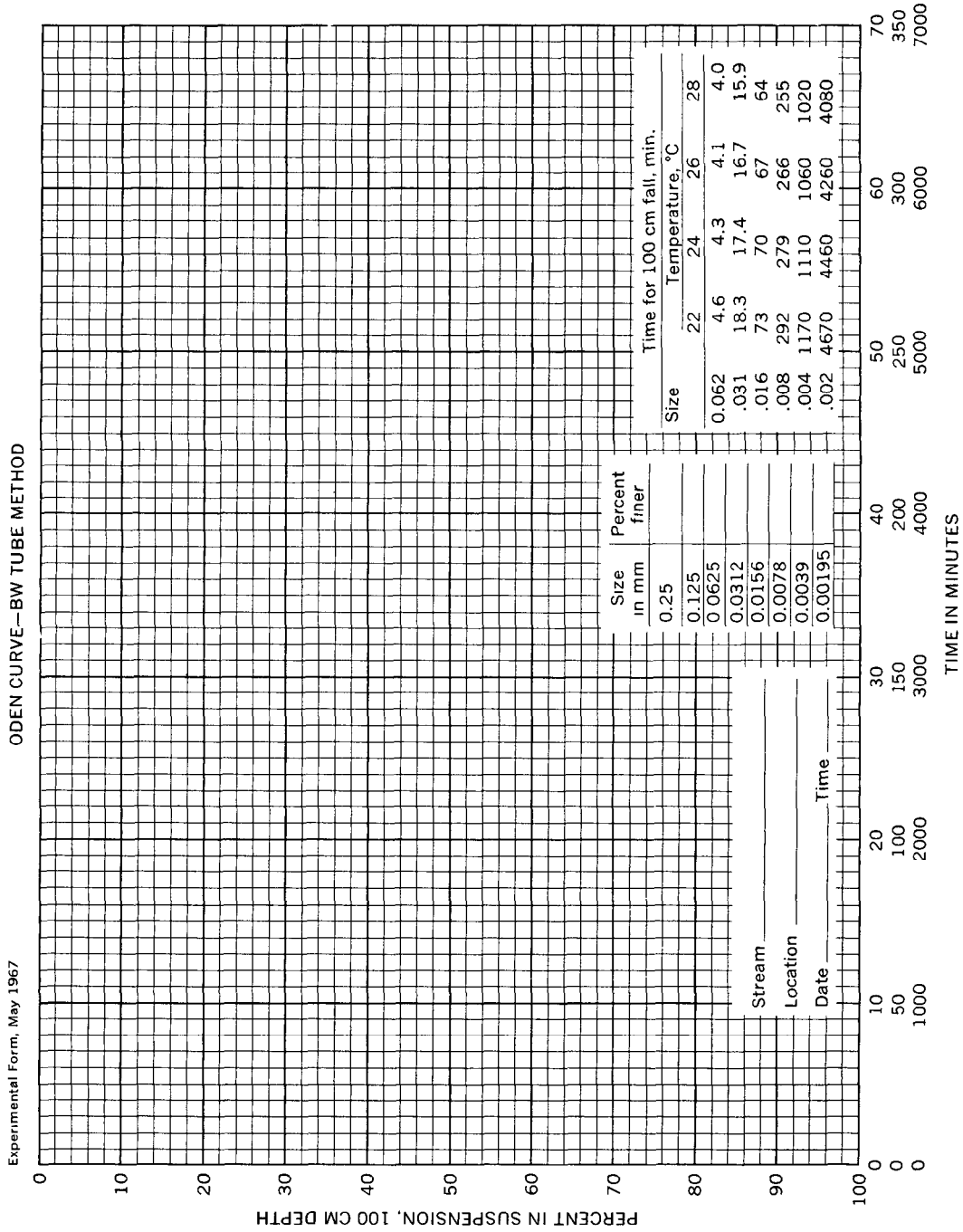
Figure 17.—Sample form for ploting Oden curve.

Limitations of BW tube for sand

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

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

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

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

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

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

Equipment and method of handling

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

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

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

In situ measurement

Large particles must be measured in situ because it is impractical to take an adequate or representative sample to the laboratory. Roughly 100 particles should be measured to represent a sample (more if the size range is

from gravel to boulder and less if the material is quite uniform in size). A grid pattern locating the sampling points can be paced, outlined by surveys, or designated by floating bobbins. The "particle" underlying the toe or the one at the intersection of the grid point is retrieved (Wolman, M. G., 1954) and a measurement made of the long, intermediate, or short diameters, or all three.

Nominal diameter by immersion

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

The Zeiss analyzer

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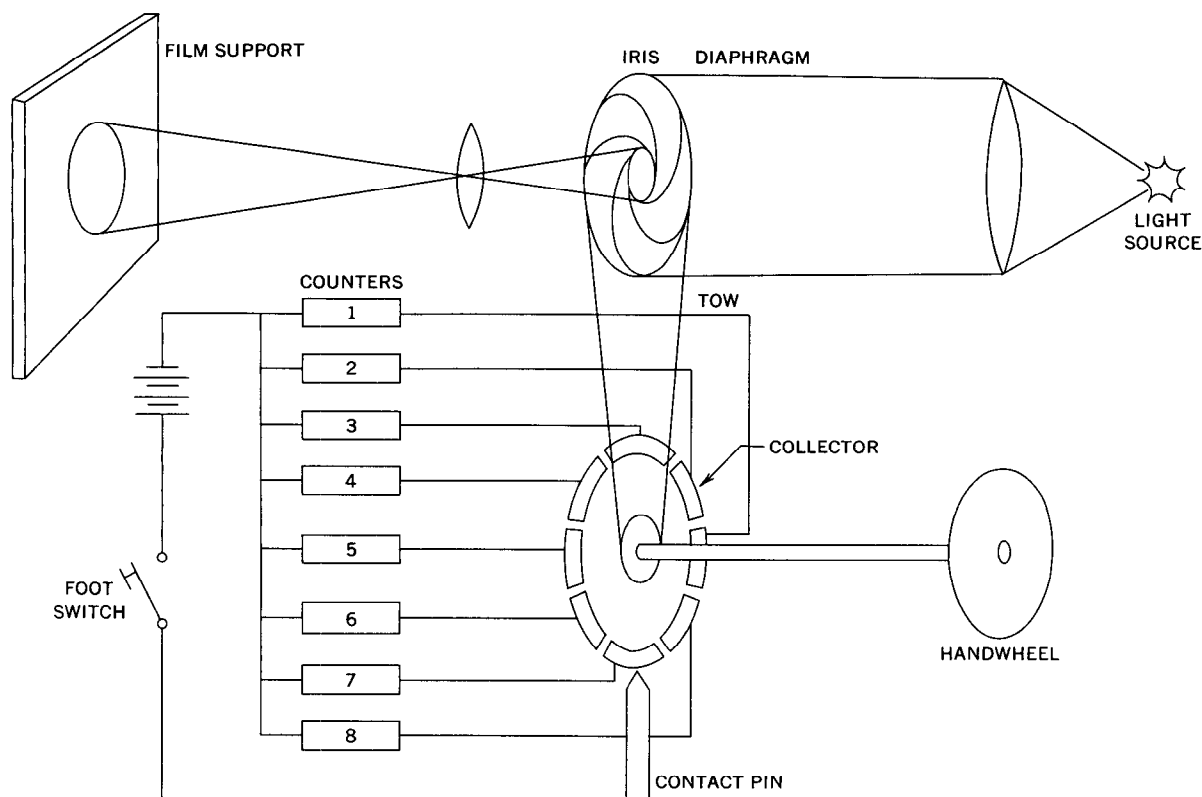
Figure 18.—Diagrammatic Sketch of Zeiss Particle-Size Analyzer.

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

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

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

Laboratory analysis

Preparation and procedures

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

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

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

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

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

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

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

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

If the sample is composed of loose sand and if the sieve method is to be used for analysis of very coarse sand and other coarse particles and the VA tube method is to be used for analysis of the coarse through very fine sand, the following procedure is recommended. Record the weight of the entire sample on the analysis

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

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

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

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

Calculation of results

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

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

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

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

Mechanical analyses of soil samples

The methods for the determination of particle-size distribution of soil samples are essentially the same as for bed material or other deposited sediment. The purpose of a mechanical analysis of soil determines the best method for its analysis. In soil science, the classification of

sand separates (very fine sand, fine sand, medium sand, and so forth) is based on sieve diameters of the sand particles. Therefore, if the purpose of a mechanical analysis is to determine the soil texture and percentage composition by soil separates, and if the results are to be compared with available soils data obtained by other investigators, the sieve method should be used to determine the particle-size distribution of the sand fraction. However, if the purpose of a soil analysis is to determine the hydraulic or transport characteristics of the sand particles, the VA tube method should probably be used for the sand analysis regardless of the method used for analysis of the silt-clay fraction.

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

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

Other Determinations Related to Sediment Analysis

Organic material

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

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

Relationship to particle-size settling media

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

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

Procedure of removal

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

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

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

Aggregate destruction

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

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

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

Dissolved solids

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

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

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

The recommended calculation for concentration is

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

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

Related water-quality analysis

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

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

Specific gravity

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

ments, but may result in considerable error because of air-filled pore space in or on the object or sample.

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

Specific weight

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

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[Italic page numbers indicate major references]

BED-MATERIAL SAMPLE

Wet storage
Dry storage

| Weigh |

| Hand remove | (Coarse particles)
|---|
| Coarse sieve |

Sieve
Immersion

(Unused) ——— | Split |

| Weigh |

| Weigh |

| Organic removal |

| Fine sieve | (Medium particles)

| Split | (Unused)

(Fine particles)

| Weigh |

(Unused) ——— | Split |

| Organic removal |

| Weigh |

| Organic removal |

Dry sieve
Wet sieve
VA tube

Pipet
BW tube

| Weigh |

| Weigh |

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