A STUDY OF METHODS USED IN

MEASUREMENT AND ANALYSIS OF SEDIMENT LOADS IN STREAMS

COLLABORATING AGENCIES
BUREAU OF RECLAMATION
CORPS OF ENGINEERS
IOWA INSTITUTE OF HYDRAULIC RESEARCH
INDIAN SERVICE
GEOLOGICAL SURVEY
DEPARTMENT OF AGRICULTURE
TENNESSEE VALLEY AUTHORITY

REPORT NO. 4

METHODS OF ANALYZING SEDIMENT SAMPLES

NOVEMBER 1941

A Study of Methods Used in

MEASUREMENT AND ANALYSIS OF SEDIMENT LOADS IN STREAMS

Planned and conducted jointly by

Tennessee Valley Authority, Corps of Engineers,
Department of Agriculture, Geological Survey,
Bureau of Reclamation, Indian Service, and
Iowa Institute of Hydraulic Research

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Published at

St. Paul U. S. Engineer District Sub-Office
Hydraulic Laboratory, University of Iowa
Iowa City, Iowa

November 1941

The cooperative study of methods used in
MEASUREMENT AND ANALYSIS OF SEDIMENT LOADS IN STREAMS
covers phases indicated by the following report titles.

<u>Report No. 1</u>

FIELD PRACTICE AND EQUIPMENT USED IN SAMPLING SUSPENDED SEDIMENT

<u>Report No. 2</u>

EQUIPMENT USED FOR SAMPLING BED-LOAD AND BED MATERIAL

<u>Report No. 3</u>

ANALYTICAL STUDY OF METHODS OF SAMPLING SUSPENDED SEDIMENT

<u>Report No. 4</u>

METHODS OF ANALYZING SEDIMENT SAMPLES

<u>Report No. 5</u>

LABORATORY INVESTIGATIONS OF SUSPENDED SEDIMENT SAMPLERS

SYNOPSIS

A description and discussion of methods of determining particle size, particularly with reference to their adaption to suspended sediment samples, occupies the major portion of this report.

The size determination of fine-grained materials, i.e., particles smaller than about 1/16 mm., is based almost entirely upon fall velocities in a fluid, the relation of fall velocities and fluid properties being interpreted in terms of particle diameters by Stokes' law. Convenient graphical methods for calculating particle diameters from the observed fall velocities and fluid properties are presented for conditions where Stokes' law does and does not apply. The review reveals that the greater number of the present methods are not sufficiently sensitive and accurate for analyzing suspended sediment samples of very low concentrations. The need for development of rapid, accurate, and reliable methods adaptable to mass analysis of suspended sediment samples is apparent.

Although sieving has been the common method for analyzing coarse sediments, a number of hydraulic separation methods have been devised that appear promising. Further development of these settling rate methods is needed in this country, for, as emphasized in the report, a diameter calculated from a fall velocity is generally more valuable in sediment studies than a sieve diameter or a direct physical measurement.

The methods of analyzing sediment samples for total solids concentration are also described and discussed; first, as separate independent procedures and then in relation to size analysis methods. Several relatively new methods, advantageous to use with certain types of sediment samples, are presented.

Procedures for a number of the more common methods of particle size and total concentration analysis, as used by various agencies, are described in detail in the appendix. Applicable standard procedures of the American Society for Testing Materials are indicated.

An extensive bibliography is given, classified by subjects as well as by authors, in alphabetical order.

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I. INTRODUCTION

1. Purpose of report--A critical inspection of the present status of sediment analysis in the United States reveals a lack of standardization of laboratory methods and technique, particularly in the determination of particle sizes. This condition has prompted a study in an effort to promote or aid in a standardization of procedure and technique of the various Government agencies engaged in sediment work so that more analogous data will be secured.

More explicitly, the purpose of this report is to describe and discuss methods of determining total sediment concentration and particle sizes in samples of suspended sediment, bed-load, and deposited bed material. As a review it has the following objectives:

> a. To present information from a large number of scattered references so as to be readily available to other workers.
>
> b. To analyze existing methods as to their applicability to sediment samples, a subject that is not complete in existing literature.
>
> c. To eliminate possible repetition of effort, particularly in methods of particle size analysis, by presenting the great number of ideas that have been developed.
>
> d. To be the foundation for the development of a satisfactory, rapid laboratory procedure for analyzing the low concentrations usually existing in suspended sediment samples.

This report tends in no way to detract from the excellent reviews of particle size analysis methods presented by Gessner (37), Krumbein (60), Heywood (46), Rubey (94), Oden (66), and Keen (53). These reviews were presented largely for their value in the fields of soils, geology, and mining with no specific reference to their adaptation to the problems of sediment analysis, particularly not to the analysis of the low concentrations of suspended sediment samples. A certain repetition of review will be apparent, but this is necessary to make this study complete and to substantiate the evaluation of methods for sediment analysis.

2. Purpose and scope of present investigation--The study of laboratory methods presented herein is one phase of a joint project conducted by a number of Government agencies to study the equipment, the field technique, and the laboratory procedures involved in sediment investigations. Particular emphasis is given to the measurement of suspended sediment. Other reports of this cooperative project are:

> Report No. 1--"Field Practice and Equipment Used in Sampling Suspended Sediment;" a review of sediment investigations which presents and discusses the various methods of selecting the verticals to be sampled in a stream section, the methods of selecting the sampling points in a vertical, and the considerations in determining the frequency of sampling the particular stream section; and which describes, classifies, and discusses the numerous suspended sediment samplers.
>
> Report No. 2--"Equipment Used for Sampling Bed-Load and Bed Material;" a review and classification of the various types of equipment.
>
> Report No. 3--"Analytical Study of Methods of Sampling Suspended Sediment;" an analysis of the various methods of sampling a stream vertical, based on sediment distribution as indicated by the theory of turbulence.
>
> Report No. 5--"Laboratory Investigations of Suspended Sediment Samplers;" presents the results of an experimental study of the effect of sampling action upon sediment concentration, of deposition in instantaneous trap samplers, of loss in transferring samples from samplers with non-removable containers, and of the filling characteristics of a number of slow-filling samplers.

3. Authority and personnel--The comprehensive program of which this

report is a part, is conducted by the informal cooperation of the U. S. Geological Survey, Indian Service, Bureau of Reclamation, Department of Agriculture, Corps of Engineers, Tennessee Valley Authority, and the Iowa Institute of Hydraulic Research. The investigation is being conducted at the Hydraulic Laboratory of the Iowa Institute of Hydraulic Research, State University of Iowa, Iowa City, Iowa, under the supervision of Professor E. W. Lane. Engaged on the project as representatives of their respective cooperating Government agencies are: Vernon J. Palmer, Soil Conservation Service; Cleveland R. Horne, Jr., Engineer Department; Victor A. Koelzer, Geological Survey; Clarence A. Boyll, Tennessee Valley Authority; and Philip M. Noble, and Donald E. Rhinehart, who succeeded Mr. Noble, Bureau of Reclamation. This report was edited by Martin E. Nelson, Engineer in charge of the U. S. Engineer Sub-Office, Iowa City, Iowa.

The personnel of the U. S. Engineer Sub-Office, Iowa City, and of the Hydraulic Laboratory of the University of Iowa, assisted in the administrative details, the testing program, and in the preparation of reports. Professor Hunter Rouse reviewed critically the part of this report concerning the fall of particles in fluids. A number of the illustrations, particularly of the older size-analysis apparatus, were secured from W. C. Krumbein's and F. J. Pettijohn's "Manual of Sedimentary Petrography," published by D. Appleton-Century Co., New York in 1938, and from H. Gessner's "Die Schlammanalyse," Leipzig, 1931. Appreciation is expressed for their use in this report. The cooperation of a large number of agencies in supplying material for this study is gratefully acknowledged. Dr. Gordon Rittenhouse of the Sedimentation Division of the Soil Conservation Service under the Department of Agriculture furnished the supplementary bibliography which is included in the appendix of this report.

II. NATURE OF PROBLEM

4. Character of samples--In a discussion of methods of analyzing sediment samples it is necessary to consider the three general types of samples that, because of their individual nature, will affect the choice of laboratory methods for determining total concentration and particle sizes. These types are: suspended load, bed-load, and deposited material samples.

Suspended load samples, obtained primarily to secure a measure of the solids being transported in suspension by flowing water, consist necessarily of a given volume of water and sediment. The laboratory problem is to separate the sediment from the water, before or after a particle size determination has been made, generally by an indirect, sedimentation method, in order to secure a figure representing the total solids being transported per unit volume of water, and, if a size analysis has been made, the amount of sediment of various sizes being carried. The solids concentration of this type of sample is inherently low and the sediment is fine-grained because only material carried in suspension is secured.

Bed-load samples are obtained for the purpose of determining the amount of material being transported per unit time along the bed of a stream. Sand and gravel are collected in relatively large quantities that permit easy drying and weighing for a total solids determination and the application of rapid methods, such as sieving, for particle size analysis.

Deposited material samples are secured for a number of reasons. Compactness or volume-weight relationship and the character of the material, which includes particle size, shape, and composition, are particularly associate with sediment investigations. The former requires the collection of an undisturbed sample of known volume. The practical size of such samples provides an abundance of material for particle size analysis and permits, if desired, a repetition of the analysis. From the standpoint of both field and laboratory methods, deposited sediment is logically divided into two types: (1) material such as sand and gravel that is not coherent, and (2) material that is coherent such as silts and clays. Field sampling equipment necessarily varies for these two types of material as do the laboratory methods for determining total quantity of dry solids and particle sizes. As with other general types of sediment samples the size analysis becomes more difficult as the particles decrease in size but for deposited material the problem is less acute than for suspended sediment because larger samples are obtained.

Of the three types of samples described, the suspended sediment sample presents the greatest problem due to the very low concentrations that generally exist; ordinarily below 0.2 per cent or 2000 p.p.m. and often as low as 0.02 per cent or 200 p.p.m. With these concentrations, a liter sample, which is larger than that ordinarily secured with present suspended sediment samplers, contains only from 2 to 0.2 gm., respectively, of solids. The difficulty of accurately separating these small amounts into particle size ranges without excessively laborious procedures is obvious. This may be overcome to some extent by combining a number of samples or by securing a large sample by pumping. However, the volume to handle becomes excessive, particularly for the latter, and the procedure is complicated further by the very low settling rates of fine material requiring long settling periods before the clear liquid can be removed.

With suspended sediment samples and fine-grained deposited material samples the control of flocculation is a fundamental consideration in the size analysis procedure because sedimentation methods are generally used. The question arises with either of these types of fine-grained sediment as to whether the sample should be dried for a total solids determination and then redispersed for the size analysis or whether the size determination should be the first consideration. To return the sediment to individual grains after drying, by chemical and mechanical dispersion, is often a difficult procedure.

5. Character of particles--It is impossible to state the limits of particle size for either bed-load or deposited sediment samples, particularly the latter, as deposited material may include clay or gravel or any intermediate gradation. The methods of analysis must be somewhat variable and flexible to handle such ranges in size. Generally, the bed-load will be sand and coarse material which lends itself to sieve analysis. The suspended load particles generally do not exceed about 0.5 mm. diameter with the minimum extending down below 1 micron (0.001 mm.) and into colloidal sizes.

Table 1 is presented to illustrate the relative position of the particles dealt with in sediment samples to the general subdivisions of matter. This table, a slightly modified form of one presented by Truog (118), contains a list of the properties of the various grades of material and related information such as limits of microscopes and diameter of pores in various types of filtering materials. Ordinary sediment size

TABLE 1

THE SUBDIVISION OF MATTER AND THE RESULTING PROPERTIES(118)

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

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

NOTES

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

analysis deals only with the coarse suspensions down to about 2 microns in that division. Further separations are prohibitive due to the excessive time required for separation and, fortunately, they lack practical value in ordinary hydraulic engineering work.

It is not the purpose to elaborate here on the composition and shape of particles but to point out that there is more or less variation in both of these particle features, which are dependent upon geologic location, type of watershed, type of stream, and a great number of other factors. Since variations do exist, which are uncontrollable and of unknown magnitude, except in certain streams where extensive study has been made, it is reasonable to assume that total concentrations should be expressed in terms of weight and not volume, and that sub-sieve particle size should be expressed in terms of a common specific gravity and diameter based on the fall velocity in water. These latter items will be discussed more fully in following sections.

6. Grade scales and expression of particle size--In discussing sediments it is very desirable to have standard terms which convey definite ideas regarding particle size. Many groups of such terms which have been given the name "grade scales" have been proposed (60), together with the corresponding size limits. However, only a few of these grade scales have come into general use. In Table 2 is given one which was developed and has been extensively used by the U. S. Bureau of Soils in the study of soils from the agricultural standpoint. This classification has also been widely used in the engineering field in various phases of soil mechanics. Beginning in 1938 the division between silt and clay was

changed by the U. S. Bureau of Soils from 0.005 to 0.002 mm., but it is doubtful if this change has been accepted widely in engineering practice.

Another grade scale which is generally used is that of Wentworth which is also given in Table 2. It has been extensively used by geologists in this country for the study of sediments, and is now nearly universally used by them. It has also been extensively used by engineers in the sediment field. Two important advantages of Wentworth's classification are that it is based upon a definite class interval, $\sqrt{2}$ mm., and its division points conform to a standard system of sieves. Thus, its subdivisions fit in readily with the analysis of sizes and size distribution characteristics, as developed by Krumbein (60) in accordance with the principles of statistical analysis, and which are rapidly coming into use.

One disadvantage of Wentworth's classification is that some of its subdivisions cover too wide a range of particle size. Prior to Wentworth's work, Udden (119) proposed subdivisions of the silt and clay sizes which also follow intervals of $\sqrt{2}$ mm. using the same outside limits for silt and clay as did Wentworth. In his studies of the Missouri River, Straub (121) independently subdivided Wentworth's silt class, using the same terms and limits as Udden. A modified Wentworth-Udden classification is suggested in Table 2, which preserves the Wentworth classification throughout but subdivides the silt classification as suggested by Udden and Straub and the clay classification as suggested by Udden. The pebbles and cobbles are also subdivided into classes, the upper size limit of each subclass being $\sqrt{2}$ times the lower limit, as is the case in the subdivision of the other classes.

Since sediment particles are not spheres but are actually of various

TABLE 2

CLASSIFICATION OF SEDIMENTS ON THE BASIS OF PARTICLE SIZE

U. S. Bureau of Soils		Size Limits		Udden (119) 1898 and 1914	Wentworth (135) 1922	(Modified Wentworth - Udden)	
Size Limits							
Millimeters	Designation	Millimeters	Inches(approx.)	Designation	Designation	Major Division	Subdivision
		Above 256	Above 10		Boulders	Boulders	-----
		256 - 128 128 - 64	10 - 5 5 - 2 1/2	Large boulders Medium boulders	Cobbles	Cobbles	Large Small
		64 - 32 32 - 16 16 - 8 8 - 4	2 1/2 - 1 1/4 1 1/4 - 5/8 5/8 - 5/16 5/16 - 5/32	Small boulders Very small boulders Very coarse gravel Coarse gravel	Pebbles	Pebbles	Large Medium Small Very small
		4 - 2	5/32 - 5/64	Gravel	Granules	Granules	-----
2 - 1	Gravel	2 - 1	5/64 - 5/128	Fine gravel	Very coarse sand	Sand	Very coarse
		Millimeters					
		Fractions	Decimals				
1 - 1/2 1/2 - 1/4 1/4 - 1/10 1/10 - 1/20	Coarse sand Medium sand Fine sand Very fine sand	1 - 1/2 1/2 - 1/4 1/4 - 1/8 1/8 - 1/16	1.0 - .5 0.5 - .25 0.25 - .125 0.125 - .0625	Coarse sand Medium sand Fine sand Very fine sand	Coarse sand Medium sand Fine sand Very fine sand		Coarse Medium Fine Very fine
1/20 - 1/500	Silt	1/16 - 1/32 1/32 - 1/64 1/64 - 1/128 1/128 - 1/256	0.0625 - .0312 0.0312 - .0156 0.0156 - .0078 0.0078 - .0039	Coarse silt or dust Medium silt or dust Fine silt or dust Very fine silt or dust	Silt	Silt	Coarse Medium Fine Very fine
Below 1/500	Clay	1/256 - 1/512 1/512 - 1/1024 Below 1/1024	0.0039 - .002 0.002 - .001 Below 0.001	Coarse clay Medium clay Fine clay	Clay	Clay	Coarse Medium Fine

shapes and various degrees of angularity, a somewhat arbitrary definition of size has been necessary. A common objection to sieves with square openings of uniform dimensions is that they determine or express a size value that obviously becomes more and more of a misconception of the true volume as the particle deviates from sphericity. For materials which lend themselves more readily to size analysis by a sedimentation method, these deviations from sphericity become of less importance because the determination of the actual settling rate of a particle is generally of more importance in sediment studies than an exact measure of volume or diameter. The common method is to express the settling rate in terms of a diameter of sphere of the same specific gravity which will fall at that rate. Some workers have even suggested expressing sizes of particles directly as fall velocities rather than converting into hypothetical spheres.

7. Organization of material--The material to be presented in this report divides itself readily into two main subjects: (1) The determination of particle size, and (2) the determination of total concentration. Actually, they are related, as pointed out previously, because in the final choice of methods they are dependent each upon the other.

The study of size analysis methods, occupying by far the greatest portion of this report, will be presented first. Since the analyses are based largely upon the sedimentation rates of the particles, a presentation of the laws governing particle fall will comprise the introductory part. An effort is made to present this information in readily usable graphs and forms with a minimum of theory. The analysis methods adapted to fine-grained sediments (below about 1/16 mm.) are discussed separately from those used for coarser material. This is logical because of the difference in methods and technique applicable to each.

The later portion of this report is devoted to descriptions and discussions of methods for determining total sediment concentration.

Two important items in sediment analysis that, in themselves, require extensive discussion, are omitted. They are the problems of flocculation and dispersion with methods for their control, and the actual presentation and interpretation of size analysis data. Pertinent references to these subjects are presented in the classified bibliography.

The appendix includes detailed laboratory procedures for the more common methods as used by various agencies for both particle size and total concentration determinations.

The bibliography, classified by subject as well as alphabetically by author, contains, not only references referred to directly in the report, but also others pertinent to various phases of the report.

PART ONE

FALL OF PARTICLES IN FLUIDS

III. RELATION TO PARTICLE SIZE ANALYSIS

8. Application of fall velocities to size analysis--The methods of determining sizes of particles below about 1/16 mm. (or approximately the 200 mesh sieve) are based largely upon the action of the particles in fluids. In the milling, ceramic, cement, abrasive, and paint industries, the separation into various size grades is frequently accomplished in a rising column of air. In the fields of geology, soils, and river hydraulics, water has been the most common medium, with the separation accomplished in either a stationary or ascending column. Whatever the medium and whatever the direction of flow, the principle is the same; namely, that particles of similar density tend to fall in a given fluid at a rate that is in relation to the size and shape of the particle. Since this is such a widely used basic principle of size analysis, there is need in presenting a resume of the theory and a number of the formulas that have been developed, including graphical methods of solution which permit rapid determination of either fall velocity or particle size, depending upon which is unknown.

Although, at present, sieve analyses largely predominate among the methods of determining sizes of sediment particles greater than about 1/16 mm. diameter, there are as yet relatively unexplored possibilities toward the development of methods for larger sizes, based upon fall velocities in water or in some liquid of higher viscosity. There would be distinct advantages in having an entire sample analyzed hydraulically rather than by a combination of hydraulic and sieving methods, as the former eliminates the rather abrupt break in the size curve that not infrequently results when the two methods are combined. Thus, it is felt important to present, also, the laws that have been developed for the fall of particles greater than those considered sub-sieve size.

The various laws or mathematical expressions which have been developed for expressing the relation of particle size to its settling velocity, serve as a basis for a large number of size separation methods. Since water, with its obvious advantages of abundant supply, low cost, ease of handling, and desirable qualities at ordinary temperatures, is, and probably will continue to be, the most widely used fluid, the simplified expressions and graphical methods of calculation to be presented will be limited largely to this medium.

9. Nature of particle movement--The resistance to motion of a particle immersed in a fluid and settling under its own weight can be any one of three distinctly different types of drag (93): (1) "deformation," (2) "surface," or (3) "form," depending upon the interrelation of the characteristics of particle and fluid. Resistance to motion that is due entirely to the viscosity of the fluid, the viscous effect extending far into the surrounding fluid, is called "deformation" drag. Stokes' law expresses the fall of particles under such conditions and is applicable to relatively small particles falling at low velocities.

As the fall velocity of a particle increases, inertial effects begin to become important and the zone of viscous influence is confined to a progressively thinner layer surrounding the particle. The viscous shear within this boundary layer produces the effect termed "surface" drag. The law governing such fall must necessarily consider inertial effects in addition to viscous effects.

The third type of resistance, "form" drag, occurs when the relative fall velocity of the particle, the size and form of the particle, and the conditions of the fluid are such that eddies are developed in the wake of the particle. A low pressure area behind the particle results in a force that opposes the motion.

The Reynolds number has been advantageously used to determine the character of the resistance to motion which exists in a given case and so aids in the selection of the proper equation of fall velocity. This relation is presented in Section 14.

A type of motion not previously mentioned is the so-called Brownian movement, which applies to minute particles that never settle but rather exist in a state of continual molecular agitation. No further mention will be made of this type of action because it concerns such small sized particles that it is well beyond the range of separation possible in ordinary size analyses.

10. Factors affecting particle fall and definitions of particle size--Size, density, and shape affect the rate of fall of a particle in a given fluid. Of these, density is the simplest to qualify, while expressions or definitions of size and shape tend to be difficult to develop accurately and reliably, especially for the smaller particles. Obviously, the simplest solution would be to assume that each particle is a sphere. This has been the trend as evidenced in definitions set forth (130) by Schone in 1868, Oden in 1915, and others. Wadell (130) in 1932 summed it

up rather completely with the definition, "The sedimentation radius is the radius of a sphere of the same specific gravity and of the same terminal uniform settling velocity as a given particle in the sedimentation fluid." Fortunately, this assumption adapts itself very readily to the size analysis of sediments because the sedimentation diameter, calculated from a settling rate, is more important in most sediment investigations than a mean particle diameter. It is a function not only of the particle volume and density but also the shape which includes surface properties. It is a much less difficult value to secure.

However, the influence of particle shape on fall velocities is important and can not be entirely neglected in certain types of analysis. There has been considerable study (45, 86, 95, 107, 131) of the shapes of particles and the types, origin, and processes producing non-spherical forms. Also, fairly extensive experimental and analytical studies on the effect of shape of particles on fall velocities have been made (46, 93, 130, 145). This subject is too extensive to do more than indicate the sources of information and to present several conceptions for particle definition.

Wadell (128) defined the true sphericity as the ratio of the surface of a sphere of the same volume as the particle, to the actual surface area of the particle, and analyzed previous experimental data from this standpoint, showing significant effect of shape upon settling velocities. He developed a practical expression of fall velocity that assumes a hypothetical particle between a sphere and disk, which, in effect, gives a 36 per cent reduction in settling velocity over that of a sphere. This is discussed in a following section.

Heywood (45), in a rather detailed study of particle shape, considered a volume constant, $\underline{k}$, and a surface constant, $\underline{f}$, defined as follows:

$$k = \frac{\text{volume of particle}}{d^3} \quad \text{and} \quad f = \frac{\text{surface of particle}}{d^2}$$

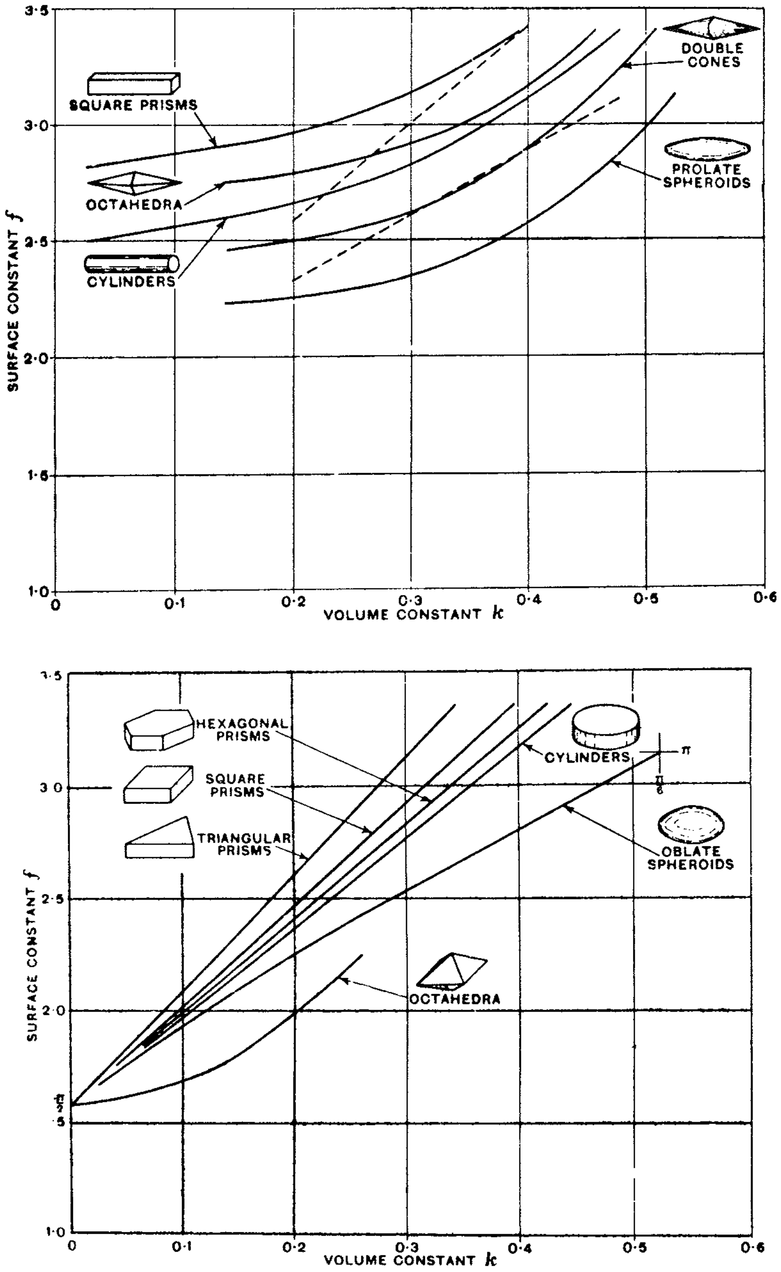
where $\underline{d}$ is defined as the diameter of a circle of area equal to the projected area of the particle when placed in its most stable position. The volume constant, $\underline{k}$, varies from $\pi/6 = 0.524$ for a sphere, to values of less than 0.1. Values of $\underline{k}$ and $\underline{f}$ for particles of various shapes are given in Fig. 1. Heywood has included this volume coefficient, $\underline{k}$, with expressions of fall velocity so that, if an approximate value of $\underline{k}$ can be determined or estimated from observation, a fall velocity can be calculated that tends to be more nearly the true value than if the particle was assumed to be spherical. This method will be presented later.

The following two chapters are devoted to a presentation and discussion of the laws of particle fall within and outside the range of Stokes' law. Graphical methods for determination of either fall velocity in water or particle diameter are presented largely for quartz spheres, with specific gravity 2.65, a value commonly assigned to sediment particles. The sizes of the particles considered range from approximately 1.00 mm. down to 0.001 mm.

Figure 1

SHAPE CONSTANTS*

*Calculated values by H. Heywood, Proc.
Inst. Mech. Engineers, vol. 125, 1933



$$\text{Volume constant } k = \frac{\text{Volume of particle}}{(\text{Projected diameter of particle})^3}$$

$$\text{Surface constant } f = \frac{\text{Surface of particle}}{(\text{Projected diameter of particle})^2}$$

FIG. 1

IV. FALL OF PARTICLES WITHIN THE RANGE OF STOKES' LAW

11. Development of Stokes' law--After an initial acceleration due to gravity, a particle, if of sufficient size to overcome Brownian movement, falling in a fluid of sufficient extent, will acquire a constant rate of fall. G. G. Stokes in 1851 expressed this terminal fall velocity of a sphere whose fall is dependent solely upon viscous effects as:

$$v = \frac{2}{9}\frac{(\rho_1 - \rho_2)\ gr^2}{\mu} \quad \dots\dots\dots\dots\dots\dots\dots\dots \quad 1$$

where v = velocity of fall in cm./sec.

ρ_1 = density of sphere in gm./cm.^{3}

ρ_2 = density of fluid in gm./cm.^{3}

g = acceleration due to gravity (981 cm./sec./sec.)

r = radius of sphere in cm.

μ = dynamic (absolute) viscosity of the fluid in dyne-sec./cm.^{2}

The development of this expression was as follows: Net downward force = (force of gravity on sphere) - (buoyant force of liquid),

$$= \frac{4}{3}(\pi r^3 \rho_1 g) - \frac{4}{3}(\pi r^3 \rho_2 g).$$

Viscous resistance to fall of a sphere in a fluid = $6\pi r\mu v$; when the net downward force equals the viscous resistance the velocity of fall becomes constant. Therefore,

$$6\pi r\mu v = \frac{4}{3}(\pi r^3 \rho_1 g) - \frac{4}{3}(\pi r^3 \rho_2 g)$$

and

$$v = \frac{2}{9}\frac{(\rho_1 - \rho_2)\ gr^2}{\mu} \quad \dots\dots\dots\dots \quad 1 \text{ Stokes' law}$$

It will be developed later that there is a maximum size of sphere, dependent upon the fluid and type of particle, beyond which this law gives values of velocity that are too high or, if velocities are known, diameters that are too low, due to inertial effects neglected by Stokes.

12. Simplified forms and graphical solutions--Since water is the fluid most commonly used in sediment analyses and 2.65 is the most widely used value for specific gravity of the particle, Stokes' law can be reduced to simplified forms.

$$v = \frac{89.83\, d^2}{\mu} \quad \ldots\ldots\ldots\ldots \quad 2$$

or

$$d = \frac{\sqrt{\mu v}}{9.478} \quad \ldots\ldots\ldots\ldots \quad 3$$

where v = velocity in cm./sec.

d ' = diameter of particle in cm.

μ = dynamic (absolute) coefficient of viscosity in dyne-sec./cm.^{2}

This simplified form is made possible because the specific gravity of water does not vary significantly in the ordinary temperature range and can be assumed constant.

Equation 2 can be changed to

$$t = \frac{0.01113\, H\mu}{d^2} \quad \ldots\ldots\ldots\ldots \quad 4$$

where t = time in sec.

H = distance of fall in cm.

A graphical solution of Stokes' law, presented in Fig. 2, allows a

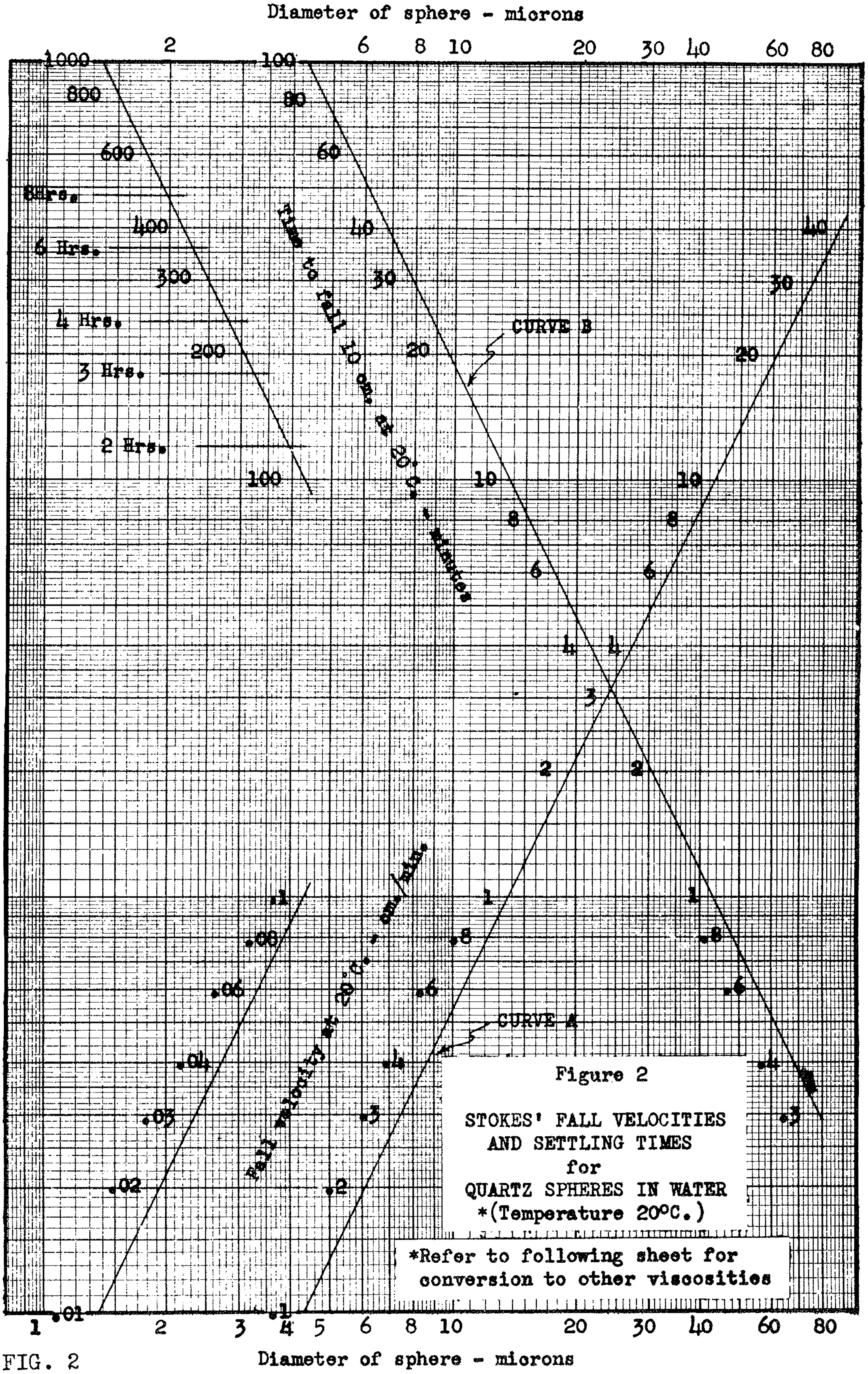


Figure 2

STOKES' FALL VELOCITIES
AND SETTLING TIMES
for
QUARTZ SPHERES IN WATER
*(Temperature 20°C.)

*Refer to following sheet for
conversion to other viscosities

FIG. 2

METHOD OF CONVERSION TO OTHER VISCOSITIES FOR APPLICATION TO FIGURE 2

Nomenclature:

$D =$ diameter in microns

v_{20} and $v_T =$ fall velocities in cm./min. at 20° C. and T° C., respectively

μ_{20} and $\mu_T =$ coefficients of viscosity of water at 20° C. and T° C., respectively

t_{20} and $t_T =$ time in minutes to fall 10 cm. at 20° C. and T° C., respectively

Case I: D known, determine v_T

Obtain $\underline{v}_{20}$ from curve A and $\left(\frac{\mu_T}{\mu_{20}}\right)$ from table

Then $v_T = v_{20} \div \left(\frac{\mu_T}{\mu_{20}}\right)$

Case II: $\underline{v}_T$ known, determine D

Obtain $\left(\frac{\mu_T}{\mu_{20}}\right)$ from table. Calculate $\underline{v}_{20}$ as

$v_{20} = v_T \cdot \left(\frac{\mu_T}{\mu_{20}}\right)$ Then obtain D from curve A

Case III: D known, determine time to fall 10 cm. at temp. T

Obtain $\underline{t}_{20}$ from curve B and $\left(\frac{\mu_T}{\mu_{20}}\right)$ from table

Then $t_T = t_{20} \cdot \left(\frac{\mu_T}{\mu_{20}}\right)$

Viscosity Correction Factors

For use with 20° C. curves of velocity, time, and diameter determined by Stokes' law.

T deg. C	$\frac{\mu_T}{\mu_{20}}$	T deg. C.	$\frac{\mu_T}{\mu_{20}}$	T deg. C.	$\frac{\mu_T}{\mu_{20}}$	T deg. C.	$\frac{\mu_T}{\mu_{20}}$
0	1.79	11	1.28	22	.960	33	.752
1	1.74	12	1.24	23	.940	34	.737
2	1.68	13	1.21	24	.917	35	.723
3	1.62	14	1.17	25	.894	36	.708
4	1.56	15	1.14	26	.875	37	.694
5	1.52	16	1.11	27	.856	38	.681
6	1.48	17	1.08	28	.838	39	.669
7	1.43	18	1.06	29	.820	40	.656
8	1.39	19	1.03	30	.801	41	.643
9	1.35	20	1.00	31	.785	42	.631
10	1.31	21	.982	32	.769	43	.601

Note: Since μ_{20} equals 0.010, values of coefficient of viscosity equal (μ_T/μ_{20}) divided by 100.

rapid and fairly accurate determination of time for a particle to fall 10 cm. or the determination of settling velocity in water at various temperatures and viscosities.

A rather flexible nomograph of settling velocities, Fig. 3, developed by Ostwald (37), allows a rapid determination of fall velocities or particle diameters. It is a convenient graph adapted to a range of viscosities, and to a range of specific gravities and therefore can be applied to fluids and materials other than water and quartz. It is only useful, however, for determination in the region where the resistance is due only to viscosity.

13. Assumptions and their significance--The basic assumptions in Stokes' law are:

a. That the particle be spherical in shape.

b. That the particle be smooth and rigid, and that there be no slipping between the fluid and surface of the particle.

c. That the particle be falling at a constant velocity as it would in a fluid of unlimited extent.

d. That the particle be sufficiently large for the fluid to be considered homogeneous in relation to the size of the particle.

e. That the resistance to fall be due only to the viscosity of the fluid.

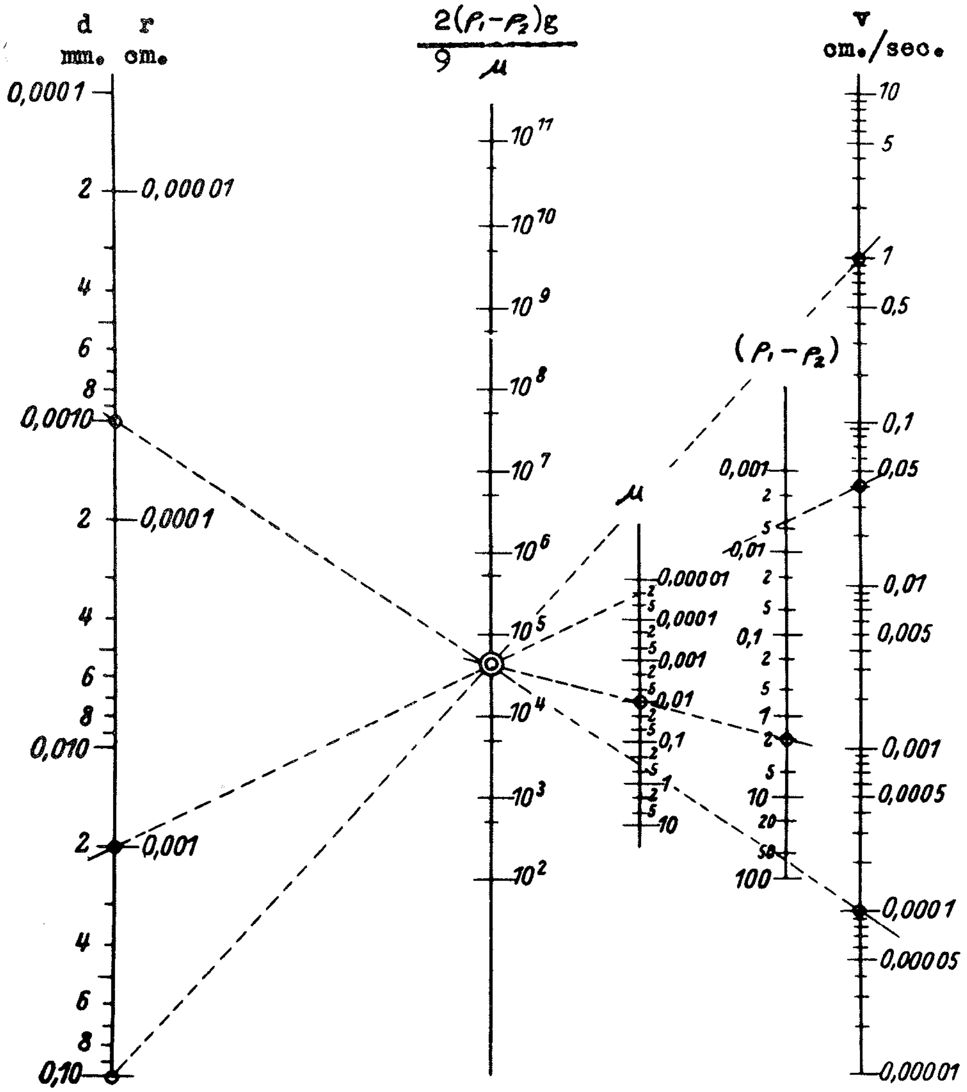
It has been commonly appreciated that sediment particles are not spherical in shape and that particle diameters calculated by Stokes' law from a certain velocity of fall are not exact. However, as pointed out previously, the diameters obtained are functions of the settling rates and, for this reason, are probably more important parameters for studies of sedimentation, than the exact diameters. Wadell (60), using the same

Figure 3

NOMOGRAPH FOR FALL VELOCITIES OF PARTICLES WITHIN RANGE OF STOKES' LAW

Nomenclature:

d = diameter of particle in mm.
r = radius of particle in cm.
ρ_1 = density of particle in gm./cc.
ρ_2 = density of fluid in gm./cc.
g = acceleration due to gravity, equals 981 cm./sec.^{2}
μ = absolute viscosity of fluid in dynes-sec./cm.^{2}
v = fall velocity in cm./sec.



(by W. Ostwald)

FIG. 3

approach as Stokes in his original derivation, developed a practical expression which considers the effect of particle shape. Instead of arbitrarily choosing a spherical particle he assumed one having a shape between that of a disk and a sphere and having the viscous resistance to fall of 9.44 r v instead of 6 r v. Equating this to the net downward force he obtained the expression

$$v_p = \frac{1}{7} \frac{(\rho_1 - \rho_2)\, g r_p^2}{\mu} \; . \; . \; . \; . \; . \; . \; . \; . \; . \; . \; . \; . \; . \; . \; . \quad 5$$

which is similar to Stokes' law except for the coefficient, 1/7. The ratio of Stokes' velocity to v_p equals 0.64 or the effect of the non-spherical shape is to reduce the settling velocity 36 per cent. Wadell termed v_p and r_p the "practical sedimentation velocity" and "sedimentation radius," respectively.

Rigidity of form is a property of sediment particles because of the very nature and type of the material. Smoothness of surface is accounted for in shape and in the "sedimentation diameter" conception. Arnold (60) demonstrated that with a small particle the effect of a pitted surface on fall velocities is negligible. As far as is known, no fluid permits slipping between particle and fluid. Thus, assumption *b* is among those least difficult to satisfy.

The time for a small particle to attain terminal or constant fall velocity is negligible in water. Weyssenhoff (60) determined that a sphere of 0.05 mm. diameter requires only about 0.003 sec. to attain this state. Of more consideration are the effects of the nearness of a wall on the settling velocities and the proximity of other particles. For practical considerations the effect of the nearness of a wall is negligible in ordinary sediment analysis work if the diameter of the settling tube is not less than 2 cm.

The proximity of particles to each other offers the greatest consideration in assumption c. Deformation effects extending out from each particle may influence the fall of adjacent particles within the zone of influence. When a graded material exists in suspension an increase in fluid density occurs toward the base of the sedimentation tube that tends to reduce settling rates of large particles. It is also conceivable that with high concentrations, a group of small particles, producing a localized area of higher density than the settling medium, may tend to settle as a unit at an abnormally high rate. The actual displacement of fluid as the particles settle may produce vertical currents that influence settling rates. Eddying currents set up by the rapid fall of large particles may seriously interfere with the fall of finer ones. High concentration may also tend to promote flocculation. Krumbein (60) estimates 2.5 per cent to be about the maximum allowable concentration without serious interference between particles. Other workers have indicated that concentrations below 1 and 2 per cent show no appreciable effect. The maximum concentration producing negligible error, in addition to varying with the type and viscosity of fluid, will vary with the size, shape, and range of particle sizes present.

The requirement that the particles be sufficiently large so that the medium may be considered homogeneous in relation to the size of the particles offers no practical difficulties. All particles that will settle in water under the influence of gravity are sufficiently large so that this assumption is fulfilled.

Assumption e , namely, that the resistance to fall is due only to the viscosity of the fluid, defines the maximum size of particle, of given density and in a given fluid, to which Stokes' law applies. Above this maximum size inertial effects develop and the resistance to fall is greater than that considered in Stokes' law.

V. FALL OF PARTICLES OUTSIDE THE RANGE OF STOKES' LAW

14. Relation to the Reynolds number--The resistance to fall of a particle immersed in a fluid has been correlated (93) with the Reynolds number to serve as an indication of the character of the resistance. An expression for a coefficient of resistance, C_D, was first developed from Newton's general equation of resisting force:

$$\text{Resisting force} = \frac{1}{2} C_D A v^2 \rho_2$$

$$\text{Gravity force} = \frac{4}{3}\pi r^3 (\rho_1 - \rho_2) g$$

where: C_D = coefficient of resistance dependent upon the interrelation of the properties of particle and fluid

A = cross-section area of the particle

v = fall velocity of the particle

ρ_1 and ρ_2 = density of particle and fluid, respectively:

g = acceleration due to gravity

For a constant rate of fall:

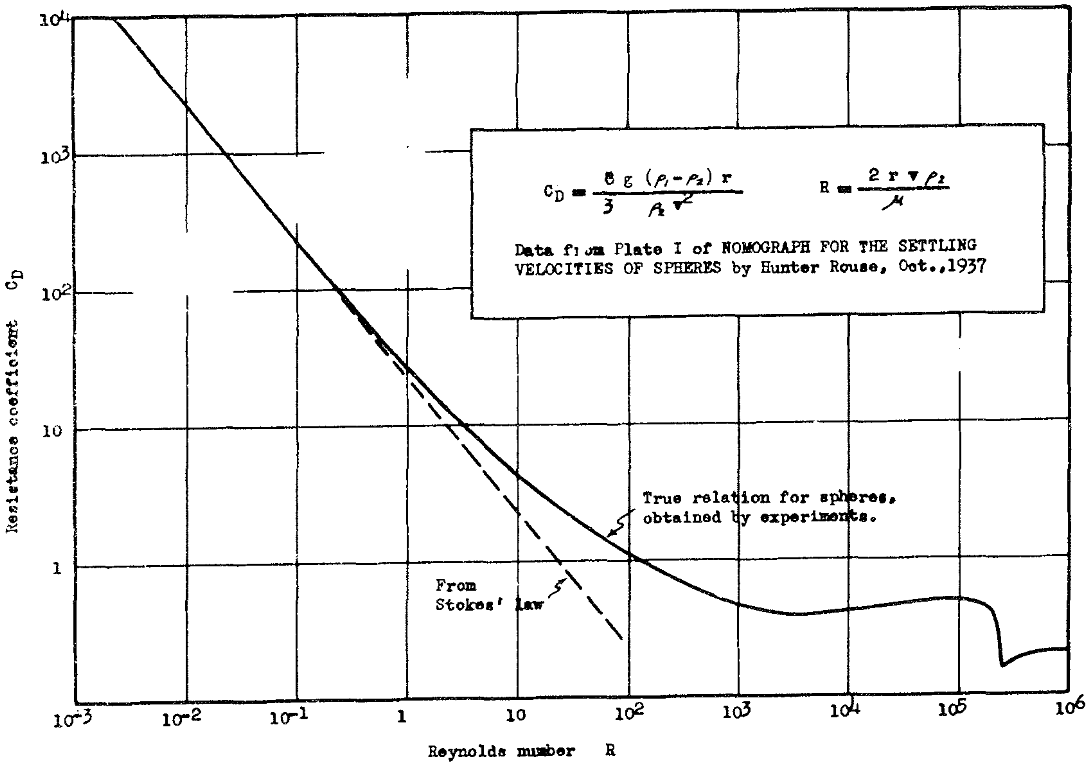
$$\frac{1}{2} C_D A v^2 \rho_2 = \frac{4}{3}\pi r^3 (\rho_1 - \rho_2) g$$

$$C_D = \frac{8g}{3} \frac{(\rho_1 - \rho_2) r}{\rho_2 \quad v^2} \quad \ldots \ldots \ldots \ldots \quad 6$$

$$\text{The Reynolds number, } R = 2rv\rho_2/\mu \quad \ldots \ldots \quad 7$$

Corresponding values of C_D and R have been calculated for given conditions of particle and fluid, where fall velocities have been determined experimentally in air as well as in a variety of liquids, and are shown plotted on logarithmic scales in Fig. 4.

Substituting for v in the equation for C_D , the velocity expressed in Stokes' law results in $C_D = 24/R$. The point of deviation of the actual curve as shown in Fig. 4 from the straight line that this equation represents, indicates the development of resistance to fall greater than that expressed by Stokes' law. Stokes' law appears in excellent agreement up to R = 0.1 and even fair agreement up to 0.4. For these values the diameters of quartz spheres in water at 20° C. will be about 0.05 and 0.08 mm. diameter, respectively.



$$C_D = \frac{8\,g\,(\rho_1 - \rho_2)\,r}{3\,\rho_2\,v^2} \qquad R = \frac{2\,r\,v\,\rho_2}{\mu}$$

Data from Plate I of NOMOGRAPH FOR THE SETTLING VELOCITIES OF SPHERES by Hunter Rouse, Oct.,1937

Fig. 4--Relation of the Reynolds number to resistance coefficient.

Heywood (46) introduces into the expression of C_D the volume constant k , described previously in Section 10, with values given in Fig. 1. His formula for C_D is

$$C_D = \frac{8k}{\pi} \frac{(\rho_1 - \rho_2)gD}{\rho_2 v^2} \quad \ldots\ldots\ldots\ldots\ldots\ldots\ldots \quad 8$$

where D = the mean projected diameter of a particle defined as the diameter of a circle having an area equal to the projected area of the particle when placed in the most stable position.

For spherical particles $k = \pi/6$ and this reduces to the same formula as originally developed. The practical application of Heywood's volume or shape factor, k , in determination of velocities or particle diameters is described in Section 15 and outlined in Fig. 6.

From the relation of C_D to R for spheres the cooperative project conducted by the U. S. Soil Conservation Service and California Institute of Technology, Pasadena, California, has determined the correct fall velocities for quartz spheres, ranging in size from 0.01 mm. up to 1.0 cm., in water at temperatures of 0, 10, 20, 30, and 40° C. These values are presented in Fig. 5. The curves lend themselves to ready, convenient use. The limitations are that they give correct values only for quartz spheres in water and do not consider irregularity of shape, do not allow the calculation for a different particle density, or permit use with other fluids. However, as mentioned before, for many investigations this expression of size in terms of sphere diameter determined hydraulically is very satisfactory. Heywood (46) developed a comparatively simple method of calculating fall velocities or particle diameters in any fluid that does allow an estimate of the effect of shape. His method will be presented in detail.

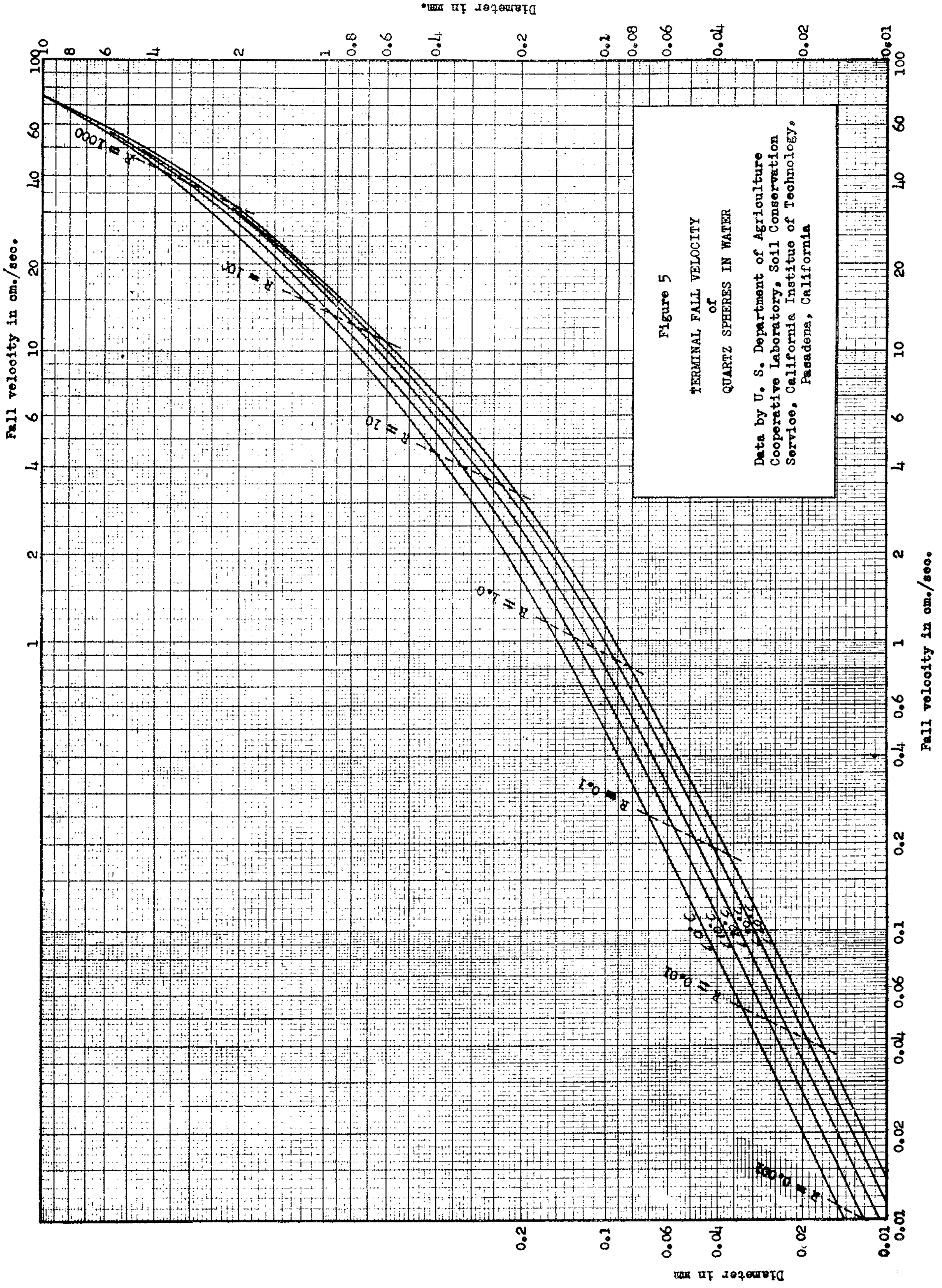


Figure 5

TERMINAL FALL VELOCITY
of
QUARTZ SPHERES IN WATER

Data by U. S. Department of Agriculture
Cooperative Laboratory, Soil Conservation
Service, California Institue of Technology,
Pasadena, California

FIG. 5

15. Heywood's graphical solution considering particle shape--Heywood (46) appreciated that the curve demonstrating the relation of C_D to R did not lend itself to a direct determination of velocity or diameter because of its dimensionless nature. To make this relation more readily usable he developed a practical method of calculating the velocity or diameter. His method allows for a consideration of particle shape and is adaptable to any fluid and particle where the value of the Reynolds number does not exceed 1000. The method is based on the relationship of R_e, the true Reynolds number, and R_s, the Reynolds number calculated from values of velocity or diameter determined by Stokes' law.

$$R_e = vD\rho 2/\mu \quad \ldots\ldots\ldots\ldots\ldots 9$$

and

$$R_s = v_sD_s\rho 2/\mu \quad \ldots\ldots\ldots\ldots\ldots 10$$

where v and D = actual values of fall velocity and particle diameter, respectively, and v_s and D_s = values used in Stokes' law.

From these relationships and the expression for C_D developed previously, the following basic relationships are developed:

a. When D is known and v is to be determined,

$$R_s/R_e = C_DR_e/24 \,.\, \pi/6k \quad \ldots\ldots\ldots\ldots\ldots 11$$

for a sphere the shape factor, k, equals $\pi/6$ and then

$$R_s/R_e = C_DR_e/24 \quad \ldots\ldots\ldots\ldots\ldots 12$$

b. When v is known and D is to be determined,

$$R_s/R_e = (C_DR_e/24 \,.\, \pi/6k)^{-0.5} \quad \ldots\ldots\ldots\ldots 13$$

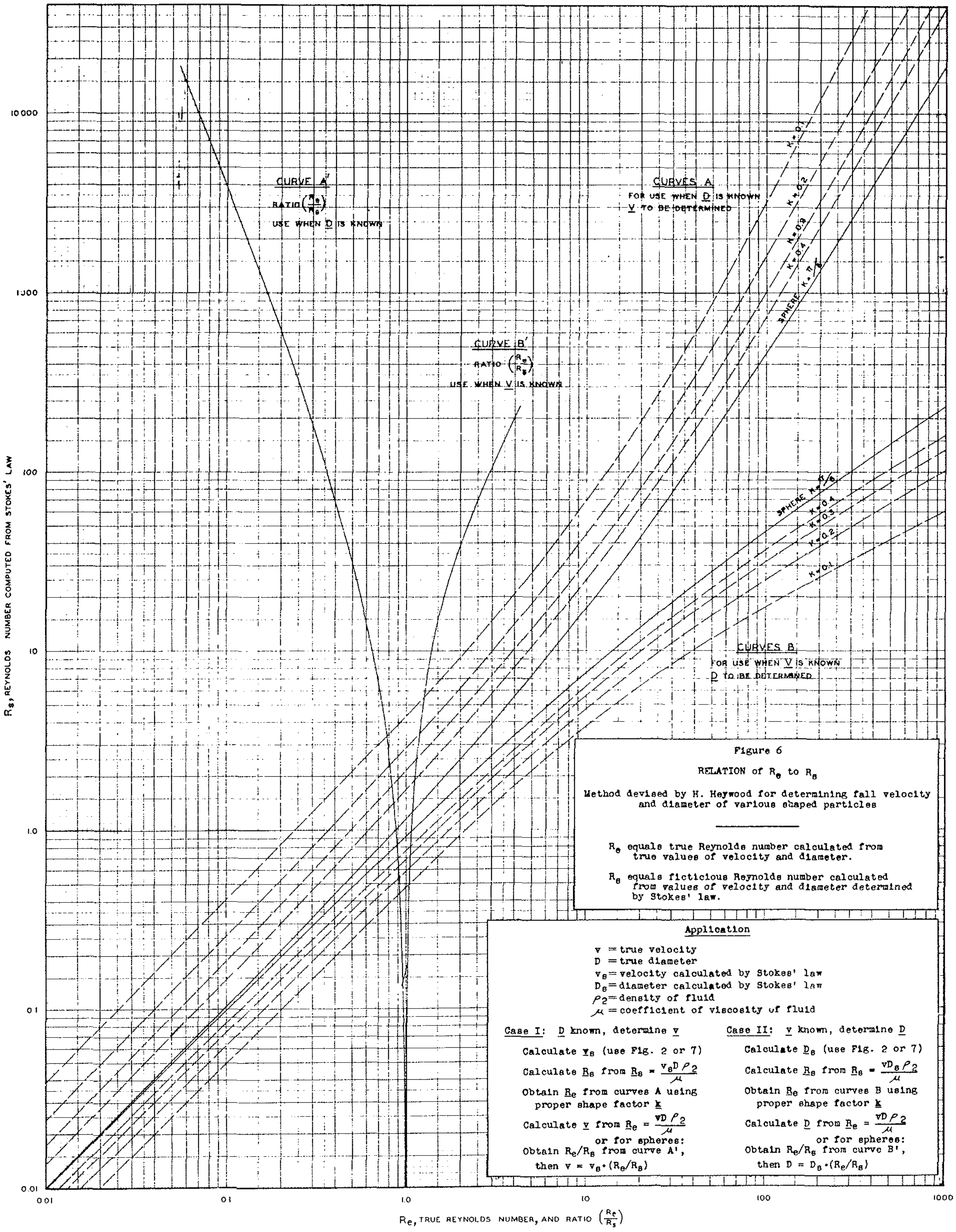


Figure 6

RELATION of R_e to R_s

Method devised by H. Heywood for determining fall velocity and diameter of various shaped particles

---

R_e equals true Reynolds number calculated from true values of velocity and diameter.

R_s equals ficticious Reynolds number calculated from values of velocity and diameter determined by Stokes' law.

Application

v = true velocity
D = true diameter
v_s = velocity calculated by Stokes' law
D_s = diameter calculated by Stokes' law
ρ_2 = density of fluid
μ = coefficient of viscosity of fluid

Case I: D known, determine v

Calculate v_s (use Fig. 2 or 7)

Calculate R_s from $R_s = \frac{v_s D \rho_2}{\mu}$

Obtain R_e from curves A using proper shape factor k

Calculate v from $R_e = \frac{vD\rho_2}{\mu}$

or for spheres:

Obtain R_e/R_s from curve A', then $v = v_s \cdot (R_e/R_s)$

Case II: v known, determine D

Calculate D_s (use Fig. 2 or 7)

Calculate R_s from $R_s = \frac{vD_s\rho_2}{\mu}$

Obtain R_e from curves B using proper shape factor k

Calculate D from $R_e = \frac{vD\rho_2}{\mu}$

or for spheres:

Obtain R_e/R_s from curve B', then $D = D_s \cdot (R_e/R_s)$

FIG. 6

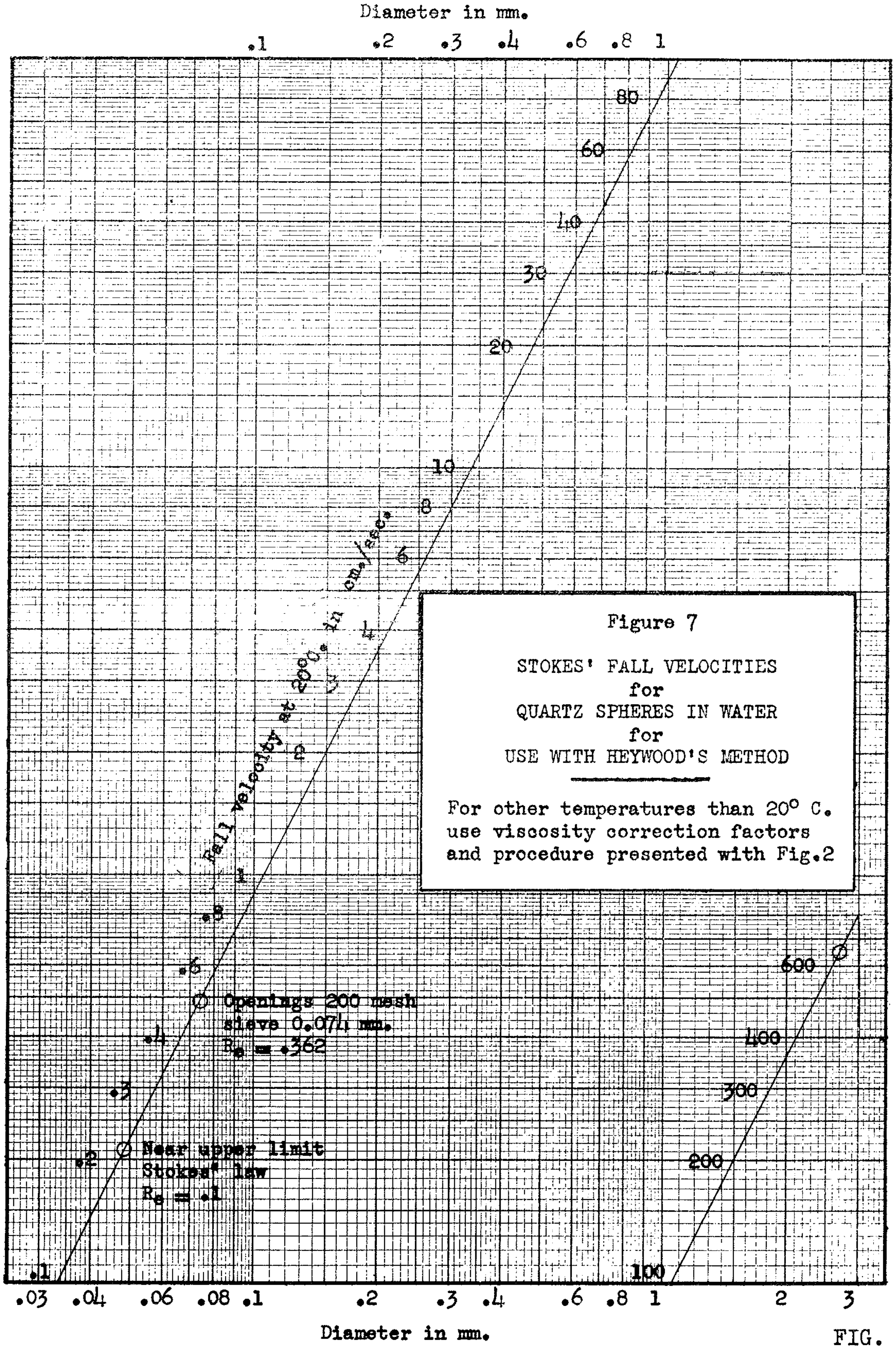


Figure 7

STOKES' FALL VELOCITIES
for
QUARTZ SPHERES IN WATER
for
USE WITH HEYWOOD'S METHOD

For other temperatures than 20° C. use viscosity correction factors and procedure presented with Fig.2

FIG. 7

For a sphere,

$$R_s/R_e = (C_D R_e/24)^{-0.5} \quad \ldots\ldots\ldots\ldots\ldots\ldots \quad 14$$

Values of $C_D R_e/24$ were determined from basic experimental data for irregularly shaped particles in seven different mixtures of water and glycerine. Values of $\underline{R}_s$ corresponding to values of $\underline{R}_e$ were determined from equations 11 and 13 and their relationship is shown graphically in Fig. 6 by two sets of curves, one for use when $\underline{D}$ is known, the other when $\underline{v}$ is known.

In addition to the shape constants given in Fig. 1, Heywood (46) gives values of $\underline{k}$ for particles having equal length, breadth, and thickness, but of various angularities as:

Rounded	0.54
Sub-angular	0.52
Angular & prismoidal	0.47
Angular, tetrahedral	0.38

VI. SUMMARY OF PART ONE

16. Summary--The laws expressing the fall velocity of small particles under the influence of gravity have been presented, because of the almost exclusive adaptation of settling rate methods to particle size analysis of sub-sieve size material. With the recent tendency to adapt settling rate methods to the size analysis of larger particles, fall velocity information is also presented for particle sizes up to 1 cm. diameter.

In addition to the brief summation of the laws of particle fall, graphical methods for determination of fall velocities and particle diameters are given. The graphs apply largely to the fall of quartz spheres in water as most sediments have a specific gravity close to that of quartz, and water is the most widely used fluid in sediment analysis.

Most useful are the curves in Fig. 5 showing the relation of diameter and fall velocity for quartz spheres varying in size from 0.01 mm. to 10 mm. at various temperatures, and the curves in Fig. 2 showing the relation of diameter to Stokes' velocity and time of fall with a method for accurate conversion to various viscosities. Most versatile is the method devised by Heywood, which is shown in Fig. 6, applicable to any fluid, and any type and shape of particle, and the nomograph developed by Ostwald adaptable to different types of fluids and particles, which is shown in Fig. 3.

Although the shape of a particle affects its fall velocity, it has been pointed out that the sedimentation diameter is generally more important in sediment analysis than a mean diameter. Consequently, the diameter of a hypothetical sphere that would fall at the same rate as the particle, has proved to be the most significant parameter to use.

When the shape of particle is an important consideration the method devised by Heywood is applicable when the Reynolds number does not exceed 1000. The basis of the method is a relationship between a Reynolds number calculated by using values of diameter and velocity from Stokes' law to the true Reynolds number. The only direct expression available for application to non-spherical particles is one by Wadell that differs from Stokes' law only by the coefficient, giving values of velocity for similar particles 36 per cent lower. The basic assumption was a hypothetical particle with a shape between that of a disk and a sphere.

The diameter of the sedimentation cylinder and the concentration of sediment have been pointed out to be important considerations when the sedimentation method of size analysis is used. However, if greater than 2 or 3 cm. and if less than 1 or 2 per cent, respectively, they produce no serious effect on fall velocities.

The greatest restriction placed upon the application of Stokes' law is that the resistance to fall must be due only to the viscosity of the fluid. This condition is fulfilled only so long as the Reynolds number is less than about 0.1, since beyond that limit inertial effects begin to influence the resistance.

PART TWO

METHODS OF PARTICLE SIZE ANALYSIS FOR FINE-GRAINED SEDIMENTS

VII. STATEMENT OF PROBLEM

17. Coarse and fine sediments--Although there is no exact size which can be logically selected as a division point between coarse and fine sediments, there is a natural division point at about the 1/16 mm. size, which is very important in many phases of sediment transportation and deposition. For example, approximately this size; forms the division point between sand and silt, the upper size limit of particles which settle according to Stokes' law, the lower size limit of particles which can be determined by sieves, the upper size limit of particles usually found relatively uniformly distributed in suspension in flowing water, the lower size limit of particles usually found in large quantities on the bed of flowing streams, and the upper size limit of particles which may be readily sampled with simple apparatus when flowing in suspension.

In the past routine size analyses of sediments smaller than 1/16 mm. have been made by settling in water and larger sizes by some other method, usually by sieves. The high settling rates of the larger particles cannot be measured with sufficient accuracy in most forms of sedimentation apparatus. In fact, even for sizes of about 1/16 mm. the fall velocity is too fast for accurate work in some of the standard methods; therefore, sieves are usually preferred.

Since many sediments contain a wide range of particle sizes and no one method can be used conveniently for the analysis of the whole range, it is customary to separate the sample into coarse and fine fractions and analyze them separately. The division point which is most convenient for such a separation will depend upon the nature of the data desired and the apparatus available. In many cases in the past a separation at the 200-mesh sieve (0.074 mm.) has been made because this was the finest sieve available. If the material is to be divided into fractions composed of the classes in some grade scale, the screen representing the division point between sand and silt can conveniently be used. It will generally be found desirable to make this separation by sieving rather than by sedimentation methods.

Successful performance of any sedimentation method depends upon the existence of the sediment as individual particles and in accordance with their original natural condition. Aggregation, which tends to occur if the sample is predried, and flocculation, which may occur during a sedimentation period with only slight changes in settling conditions, are actions that must be avoided or the analysis may be worthless. The difficulties in accomplishing and maintaining the sediment in a properly deflocculated condition increases with decrease in particle size and becomes a serious problem in the size analysis of fine material.

18. Outline of methods--Since sediment material is generally in a suspension medium, a size separation method based upon the fall of the particles in that medium will be, not only the most readily adapted separation principle, but will also maintain the particles in more or less their natural, individual state. Of practical engineering importance will be the resulting expression of size in relation to actual fall

velocity. Consequently, routine size analyses of fine-grained sediments are accomplished practically entirely by methods based on settling rates in water. Stokes' law, stating that the fall velocity due to gravity is proportional to the square of the diameter, has been used almost exclusively for conversion of fall velocities to diameters, regardless of the type of sedimentation method. The exception is the centrifugal method where the settling force is a function of the rotor speed and distance from the axis of rotation.

Direct measurement of the size of fine-grained material through the aid of microscopes has been largely replaced by more rapid sedimentation methods. Its present usage is confined, for the most part, either to checking the more rapid but indirect methods, or determining accurately particle sizes in special experimental studies. The discussion of this method in the report will be brief; the references indicated in the classified bibliography will give more detailed information.

The sedimentation methods and apparatus are numerous, and many are ingeniously designed. The more important are presented as follows in an order which is in no sense their order of importance, use, or adaptability to the analysis of fine sediments, but represents a grouping based somewhat on the principle of measurement:

a. Decantation
b. Elutriation with water
c. Elutriation with air
d. Accumulative sedimentation
e. Fractional sedimentation
f. Pipette
g. Hydrometer
h. Manometer
i. Plummet
j. Centrifugal
k. Optical

Less common in their application to the size analysis of sediments are the methods:

l. Filtration
m. Tyndall effect
n. X-Ray
o. Alternating electric fields

VIII. PRESENTATION OF METHODS

19. Microscopic--Direct measurement of sizes of particles by microscopic methods is the most widely used check of particle sizes obtained through indirect sedimentation methods. Although preferred by geologists because it is less subject to uncertainties of analysis than sedimentation methods, the microscopic method has had several objectionable features that limit its application to sediment samples. It requires the measurement of a relatively large number of particles, often a laborious procedure, if accurate results are to be obtained. A value of diameter, secured by measurements of exposed diameters or exposed area, may be significantly different from the sedimentation diameter which has been emphasized as being, generally, the most important parameter in ordinary sediment investigations.

The common methods of microscopic measurement are:

a. Measurement of diameter or diameters through micrometer eyepieces having an engraved scale.

b. Measurement of the exposed area through micrometer eyepieces having an engraved grid.

c. Measurement of the particle image, i.e., diameter or area, by projection on a screen.

d. Measurement of the particles on a photomicrograph.

A representative diameter determined by a microscopic method, whether direct or from measurement of the projected image, or from a photographic plate, is usually determined by one of the following methods of measurement and calculation:

a. Arithmetic mean of exposed diameters.

b. Arithmetic mean of exposed diameters and an estimated third diameter.

c. Intermediate diameter only.

d. Maximum horizontal intercept through a grain.

e. Area of projected image and calculation of a diameter considering the particle a sphere.

f. Harmonic mean, $3abc \div (ab + ac + bc)$, where a = longest diameter, b = intermediate diameter, and c = shortest diameter.

g. Geometric mean of longest and shortest diameter, $\sqrt{ac}$.

h Geometric mean of two exposed diameters and an estimated third diameter, $\sqrt[3]{abc}$.

Of these, the measurement of horizontal intercepts is the most rapid. When particle surface is important the harmonic mean is the most logical to use because the diameter secured is related to the surface per unit weight of the material (72).

The data from microscopic measurements are in the form of numbers of grains of certain size rather than a weight or volume figure for a large number of grains as results from the usual sedimentation methods. The analysis of the microscopic data and interpretation in terms of comparable size grades is most satisfactorily accomplished by statistical methods.

The development of a sufficiently rapid microscopic method to be practicable for the analysis of large numbers of suspended sediment samples is being studied by the Sedimentation Division of the Soil Conservation Service. Promising results have been obtained, but the work is not sufficiently complete to justify publication.

20. Decantation--In the decantation method the sediment is dispersed uniformly throughout the sedimentation medium and then, after a

certain period of time, the liquid is decanted to a certain depth. All particles with a fall velocity exceeding a certain value will have settled past the elevation of decantation and will remain in the settling tube. However, in addition to these larger particles, many particles that did not have to settle from the top of the column, have also settled past the point of decantation. Therefore, repeated decantations are required, the number depending upon the accuracy of separation desired to reduce the amount of finer material to a negligible quantity. Table 3 shows the percentages of smaller sizes remaining in the sedimentation cylinder after one to nine decantations.

TABLE 3

PERCENTAGE OF SMALLER PARTICLES REMAINING IN SEDIMENTATION CYLINDER AFTER A CERTAIN NUMBER OF DECANTATIONS

Number of decantations	Percentage of smaller particles remaining after a certain number of decantations for particle sizes expressed as the ratios of the settling rates of the smaller particles to the settling rate of the size desired.								
	Ratio of settling rates								
	0.9	0.8	0.7	0.6	0.5	0.4	0.3	0.2	0.1
1	90	80	70	60	50	40	30	20	10
2	81	64	49	36	25	16	9	4	1
3	73	51	34	22	12	6	3	0.8	0.1
4	66	41	24	13	6	2.6	0.8	0.2	0.01
5	59	33	17	8	3	1.0	0.2	0.03	----
6	53	26	12	5	1.6	0.4	0.07	----	----
7	48	21	8	3	0.8	0.2	0.02	----	----
8	43	17	6	1.7	0.4	0.07	----	----	----
9	39	13	4	1.0	0.2	0.03	----	----	----

The decantation method has a number of features that make it especially favorable in the analysis of the fine-grained sediments, particularly in the analysis of suspended sediment samples. The more important features are:

> a. The simplicity of equipment; no special equipment other than that available in an ordinary laboratory is required.
>
> b. The accuracy and reliability; the accuracy can be controlled by the number of decantations and, if reasonably careful technique is used to prevent flocculation, the results will be reliable.
>
> c. Satisfactory with low concentrations; the separation, as conducted, results in the total fraction for each grade being available for a weight determination.

Although it has these advantageous features, the decantation method is laborious unless special equipment is devised to carry on the decantations more or less automatically. The increased opportunity for flocculation because of the numerous changes in sedimentation medium is also objectionable.

Kuhn's and Appiani's decantation cylinders (60), shown in Figs. 8 and 9, respectively, were among the first devices used. Kuhn's device, with the side outlet, has been subjected to severe criticism because of the extreme unevenness of the withdrawal and the excessive currents that may be set up. Appiani's tube merits consideration, for its only apparent disadvantage is the introduction of sediment prematurely into the withdrawal tube during the initial dispersion procedure. Among these very early devices should be mentioned the ordinary siphon which probably has been the most widely used withdrawal device.

About 1912 Atterberg (60) devised a side-outlet tube, which is shown in Fig. 10. Fig. 11 shows the streamlines for this type of withdrawal, which, as discussed in the preceding paragraph, is not satisfactory.

Sometime before 1921, the U. S. Bureau of Mines devised the

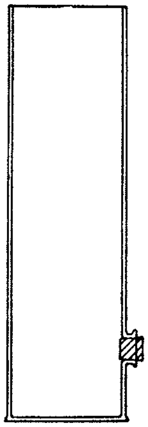


Fig. 8--Kuhn's decantation cylinder (before 1890).

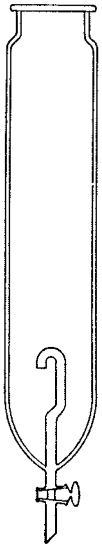


Fig. 9--Appiani's decantation cylinder (1894).

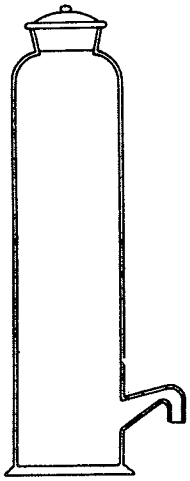


Fig. 10--Atterberg's decantation cylinder (1912).

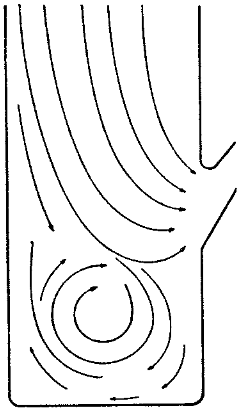


Fig. 11--Streamlines of withdrawal from Atterberg's decantation cylinder.

decantation apparatus shown in Fig. 12 for use chiefly in size analysis of clays. As with Appiani's tube the withdrawal is through the bottom. In addition to a withdrawal tube through the bottom of the cylinder the device included a plunger equipped with a rubber stopper so that the outlet tube in the cylinder could be closed. This feature eliminated premature introduction of sediment into the withdrawal tube.

To facilitate the removal of sediment, Trask (60) devised a sedimentation cylinder, which is shown in Fig. 13, with a removable cup at the bottom. A ground glass joint served as the connecting device. This appears to be an advantageous feature if the dispersion technique does not require the cylinder to be inverted.

The decantation apparatus devised by Emery (35) for accomplishing one grade separation will be discussed in Section 42 under the methods applicable to coarse sediments. With some modifications, such as raising the withdrawal tube so as to be further from the base of the column, this device could be applied to the analysis of the finer-grained sediments.

The decantation method was adopted for certain rather extensive investigations carried on at the Institute of Hydraulic Research, Iowa City, Iowa. The simple, siphon-type apparatus used is shown in Fig. 14, while the complete procedure is presented in detail in Appendix A. The short sedimentation cylinder was used for the finest separation so that the time of fall would not be excessive. The separation into fractions proceeded from fine to coarse with three decantations usually found sufficient to produce a clear liquid to be siphoned off.

An automatic decantation apparatus has been developed in Soviet Russia. This apparatus, shown in Fig. 15, described by Archangelsky and

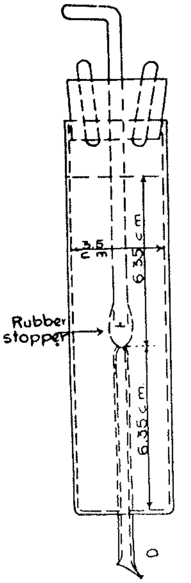


Fig. 12--U. S. Bureau of Mines decantation apparatus (before 1921)

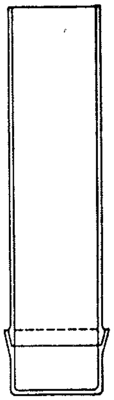


Fig. 13--Trask's decantation cylinder with removable base (1930).

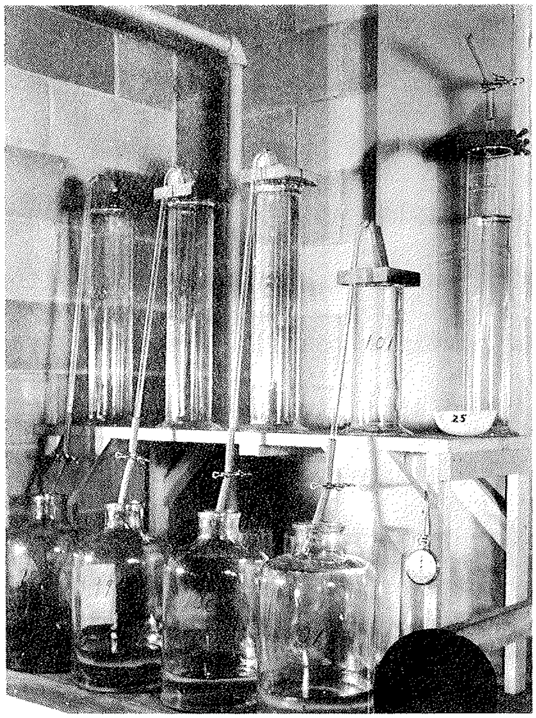


Fig. 14--Decantation apparatus used at the Institute of Hydraulic Research, Iowa City, Iowa, (1938).

FIGS. 12, 13 & 14

Michailov (7), is apparently one of the better and more popular methods for size analysis of soils. It allows simultaneous separation in six cylinders with an automatic, electric, time control for filling, agitating, sedimentation, and emptying. Electromagnets are utilized to actuate closing mechanisms for the inlet and siphon tubes. The total time for one complete cycle of operation is stated to be only 200 sec. Apparently the sedimentation column was quite short. The material separated varied from 0.25 to 0.01 mm. in size.

21. Elutriation methods--The Webster standard dictionary defines elutriation as the process of purifying by washing, straining, or decanting.

The common usage in size analysis refers practically entirely to separation by rising currents and it will be used in this sense throughout this report. The principle upon which the elutriation method is based, is that, in rising currents of various magnitudes, a grading is accomplished because the particle settling velocity varies with the size of the particle. Water and air have been the fluids most widely used; the former for analysis of sediments or particles initially immersed in water, and the latter for cement and other material in dry powder form.

22. Elutriation with water--Water elutriators have not been extremely popular for making routine size separations of small quantities of fine-grained materials. Due to certain limitations that will be discussed, their chief value appears to be in the separation of the coarser material where somewhat crude separation is satisfactory and temperature

and velocity control are not so important. Andrews (4), in discussing their use for continuous separation of ground material in the mining industry, states that there is no limit to the size of material or separation for which they can be used.

In separation by elutriation, the establishment and maintenance of a uniform velocity over the entire cross section of the tube is the most important factor to consider and the most difficult to control. Goldman (38) states that the separation of particles less than 0.25 mm. cannot be satisfactorily accomplished by this method because of the difficulty in controlling the very low inflow rates. Heywood (46), on the other hand, states that the process of elutriation is suitable for grading of sand and other heavy particles of sizes 0.20 to 0.01 mm.

Baker (15) showed that, with steady, irrotational type of flow, the velocity varies over the cross section of a narrow tube, being twice the average at the center.

Boswell (21) sums up the limitations and experimental difficulties of the elutriation method in the following factors that may also be limitations in other methods of separation that will be presented subsequently:

<u>a</u>. It is difficult to check the separations microscopically, not only because of the labor involved, but also because the particles tend to lie in their flattest position, resulting in values for diameters tending to be too large.

<u>b</u>. The density of the material varies to a considerable extent.

<u>c</u>. Temperature control is necessary.

<u>d</u>. The use of tap water is risky because it tends to induce flocculation. Distilled water should be used, but the amount needed becomes a serious problem if the material is larger than clay size.

> _e_. If tap water is used the degree of aeration is often such that large air bubbles collect in the tubing or vessel or attach themselves to the individual grains, and with the subsequent rise carry off large particles, ruining the analysis.
>
> _f_. Bacterial and algal growth in the tubes sometimes upsets the analysis by changing the inflow velocity or by streaming filaments up into the settling tube. Frequent cleaning, of course, will eliminate this factor.

Water elutriation apparatus generally consists either of a single tube with the lower portion tapered or of a series of tubes of increasing diameter. With the former, only one fraction at a time is obtained and it is necessary to make a number of runs using different inflow rates. The separation with the single tube elutriator proceeds more efficiently from fine to coarse than vice versa. The multiple tube devices are operated with constant inflow rates and with decreasing velocities from tube to tube, resulting in separations into size fractions from coarse to fine.

Shone's and Schulze's devices, shown in Figs. 16 and 17, respectively, represent the simplest elutriators. Hilgard's churn elutriator, shown in Fig. 18, is somewhat more complicated being equipped with a small paddle wheel device to break up floccules and to produce a more uniform velocity distribution. Kopecky's multiple tube elutriator, illustrated in Fig. 19, accomplished separation of material into four size grades with the sample placed in the base of the smallest tube and a constant inflow rate maintained.

Among the earlier devices employing the principle of elutriation was Yoder's centrifugal elutriator, shown in Fig. 20, developed in 1904, (25), for size separation of fine soil particles below 0.030 mm. in size. During the rotation of the centrifuge a constant flow of suspension enters at _F_ and a suspension of finer material flows out at _T_. The size of

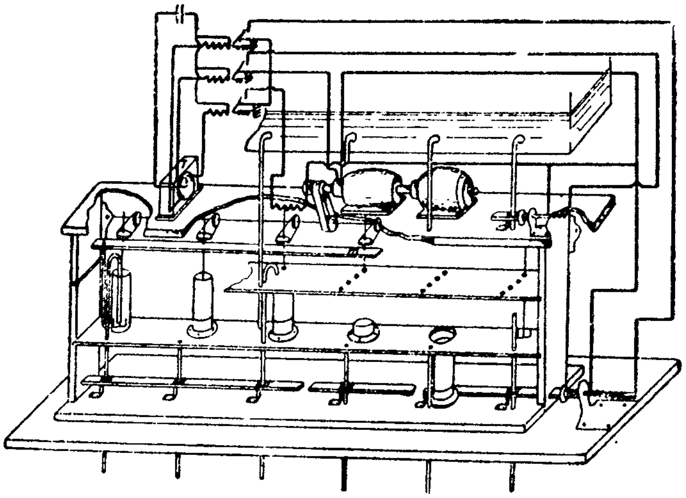


Fig. 15--Russian automatic decantation apparatus.

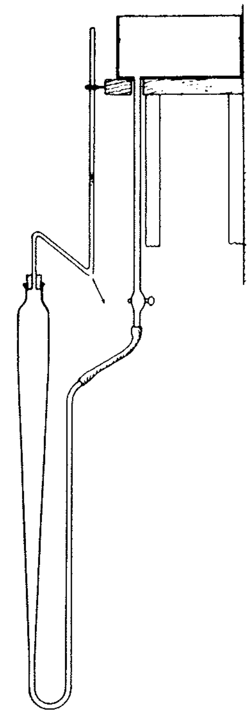


Fig. 16--Schone's water elutriator (1867).

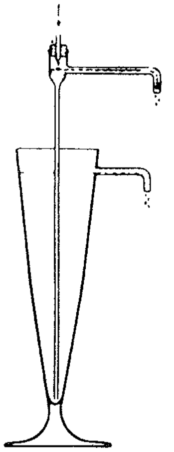


Fig. 17--Schultze's water elutriator.

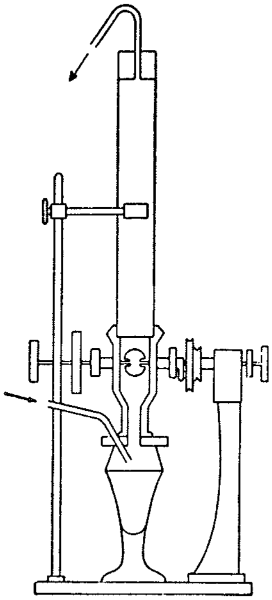


Fig. 18--Hilgard's "churn" water elutriator (1873).

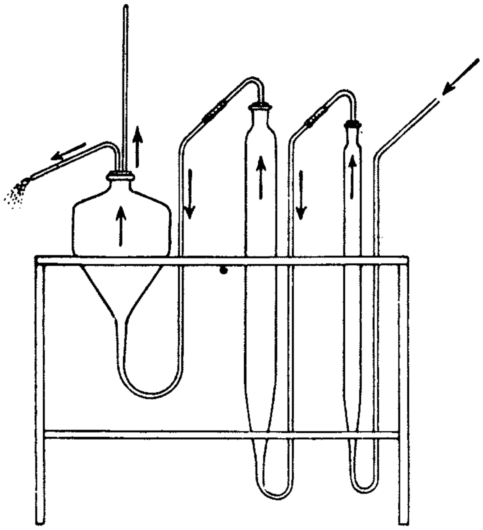
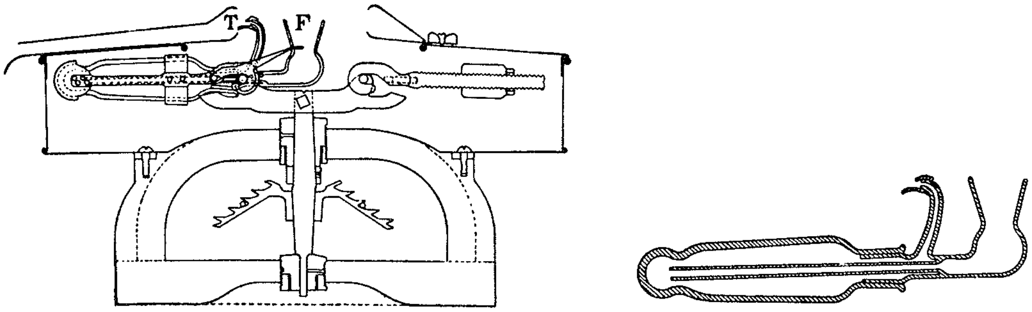


Fig. 19--Kopecky's multiple tube water elutriator (1901).



a. Cross-sectional view of entire apparatus.

b. Cross-sectional view of elutriator bottle.

Fig. 20--Yoder's centrifugal water elutriator (1904).

FIGS. 18, 19 & 20

material remaining in the sedimentation tube is dependent upon the relative effects of the settling force controlled by centrifuge speed, and the velocity of the outflowing water acting in direct opposition to that force. The sedimentation-elutriation tube, shown in Fig. 20b, is tapered so that the water velocity and centrifugal force decrease toward the axis of rotation at the same ratio, thus subjecting each particle to a uniform force. The reason for employing centrifugal force as the settling agent rather than gravity was to permit the use of relatively high water velocities, thereby overcoming one of the limiting features of the elutriation method for size separation of fine material.

With a departure from the conventional tube elutriators, Thompson (109) in 1910 devised the apparatus shown in Fig. 21, for size analysis of cement using kerosene as the liquid. The apparatus consisted of five cones of decreasing size, suspended one above the other, a circulatory system with electric motor and pump, and manometer and equipment for controlling the flow. The flow progressed downward from the top cone with decreasing velocity. Two hours were required for the analysis of a 10-gm. sample of cement.

In 1919, Gardner (36) devised the elutriator shown in Fig. 22, which consists of nineteen vessels of increasing size. The sample was placed in tank A where a constant level of water was maintained. An electrically operated stirrer kept the particles in suspension, allowing a continuous inflow of water-sediment into the first vessel. Separation continued until all the sediment was removed from A. Then, to insure against the overflow of larger particles into the next vessel and the inclusion with that fraction, the material in each vessel was removed and poured into the funnel of the preceding vessel. The flow at the same constant rate, about 0.7 liter per minute, was started again and continued until the separation was considered complete.

The separator, shown in Fig. 23, was designed by Andreason (60) and represents a recent form of the simplest type of elutriator. The material to be separated is placed in the tube of smallest diameter and a constant flow of water from the reservoir is maintained. Different vertical velocities of flow are produced in the three consecutive tubes effecting a separation of particles into four size grades, the fourth being the very fine material carried out in the overflow. As operated by Andreason, material of the sizes 0.060, 0.040, and 0.020 mm. were secured in the three tubes.

A recent water elutriator, developed by Andrews (60) is illustrated in Fig. 24. It is so constructed that the material, entering at the top of the elutriator tube, is thrown against a rigid cone by an upward current of water which breaks up aggregates and flocculated material. The continued upward current of water carries off the fine material while the coarser settles to the base of the largest chamber where it is again picked up by the water and directed against the cone. This procedure may be continued until all the material has been dispersed. Then the separation of the coarse material is accomplished in the lower tubes.

The elutriation device, shown in Fig. 25, which was used for tests of the core material of the Cobble Mountain Dam (43), was found accurate and reliable in controlling the nature of core material. For expediency, four units were used, allowing four samples to be analyzed simultaneously.

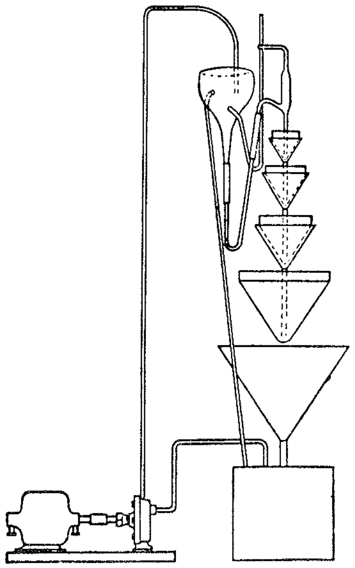


Fig. 21--Thompson's kerosene elutriator (1910).

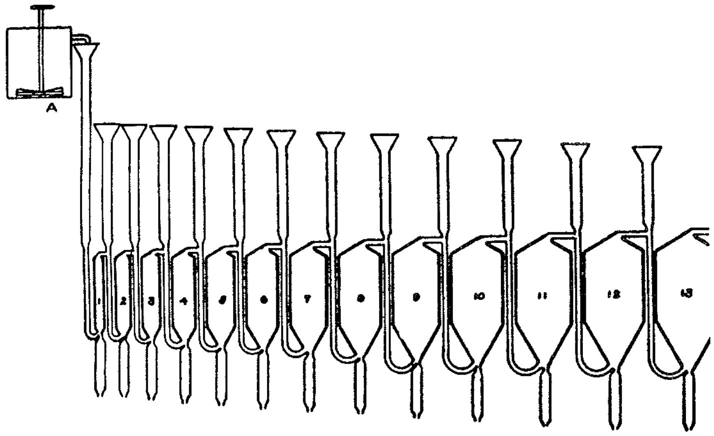


Fig. 22--Gardner's water elutriator (1919).

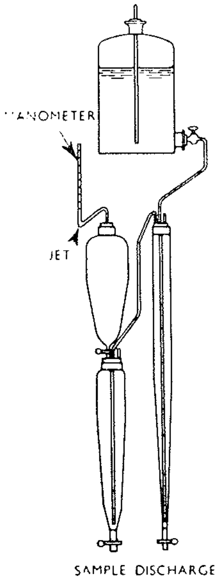


Fig. 23--Andreason's water elutriator (1927).

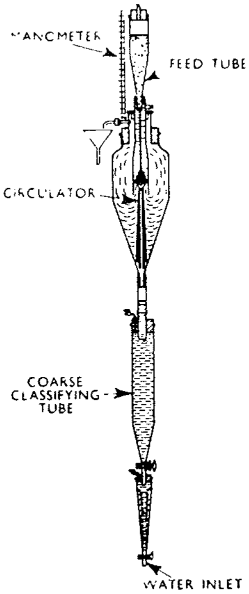


Fig. 24--Andrews' "kinetic" water elutriator (1927).

FIGS. 21, 22, 23 & 24

Temperature control was maintained by submerging the units in water in a large tank under thermostatic control.

23. Elutriation with air--In general, size separation of fine material by a rising column of air is not practical for analysis of sediment because the material must be in a dry powder form and available in relatively large quantities. It can be used advantageously only with deposited material and where an abundant sample is available. The objectionable feature is the necessity of predrying the sample and redispersion into powder form. It is usually more favorable to proceed with an analysis based on the fall velocities in water than to subject the sediment to predrying and to risk the uncertainties involved in securing the proper dispersion. However, to make this study of existing size analysis methods complete a brief review and discussion of the existing equipment for accomplishing separation by air is presented.

There are three major items or problems involved in the design and operation of air analyzers, namely:

a. The control of velocities.

b. The prevention of adherence of fine particles to various surfaces in the apparatus.

c. The elimination of aggregates or the reduction of aggregates into individual particles.

The first two can be overcome to some extent by proper design; the third is dependent upon the character of the material to be analyzed. Air analyzers can be used only to a limited extent for analyzing sediment samples, since the material that is small enough to be adapted to air elutriation generally cannot be dried out without the formation of aggregates. The result is that the air analyzer has been limited largely

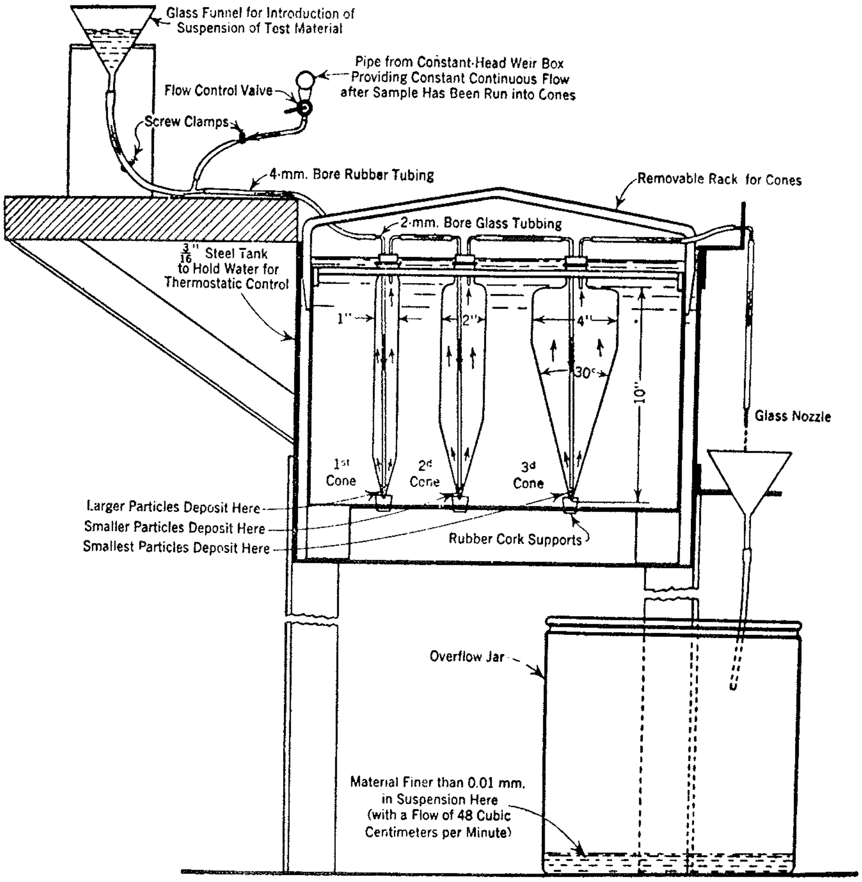


Fig. 25--Cobble Mountain Dam water elutriator (1932).

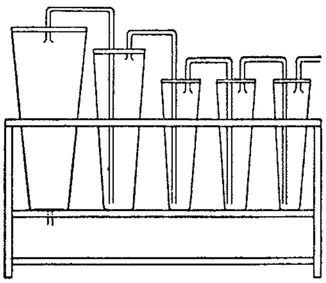


Fig. 26--Cushman's and Hubbard's air elutriator (1907).

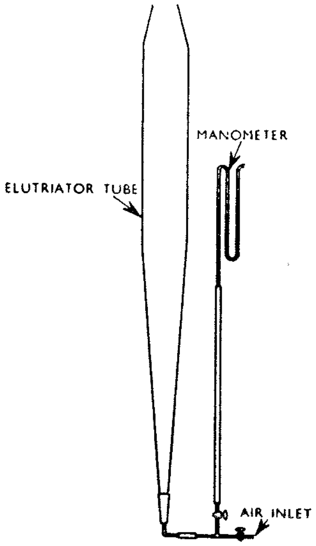


Fig. 27--Petersen's air elutriator (1909).

to analysis of cement, pigment, and ceramic materials in the dry powder form.

Early designs of apparatus which accomplished separations with various degrees of accuracy are shown in Figs. 26 and 27. Cushman's and Hubbard's (71) elutriator was constructed in 1907 for separation of rock powders. A constant stream of air entered at the base of the largest vessel where the sample was placed. Petersen's apparatus, a modification of Schone's water elutriator, was not satisfactory because, with the introduction of air from the bottom, there were decided tendencies for choking or plugging. Apparently neither of these devices was very satisfactory. Mackey (71) developed the cement analyzer shown in Fig. 28 which gave fair separations but the control and refinement were not as accurate as would be desired.

The Gary-Linder apparatus (71), shown in Fig. 29, was developed about 1906 for analyzing cement and has been used widely. A 20-gm. sample is placed in chamber I and the air flow in chambers I, II, and III is adjusted so that certain sizes of particles will deposit in each. This results in four grades of particles with somewhat overlapping sizes but with significant differences between them.

The most comprehensive study of air analyzers was conducted at the Bureau of Standards, U. S. Department of Commerce, in 1915 (71) with the purpose of developing a reliable apparatus for size analysis of cement. After making an exhaustive review and testing existing equipment without finding an apparatus conforming to the desired specifications, a new design, shown in Fig. 30, was evolved. It had the following characteristic features:

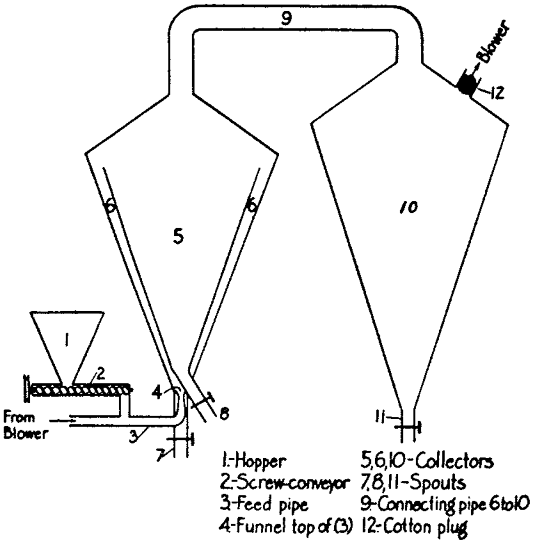


Fig. 28--Mackey air elutriator.

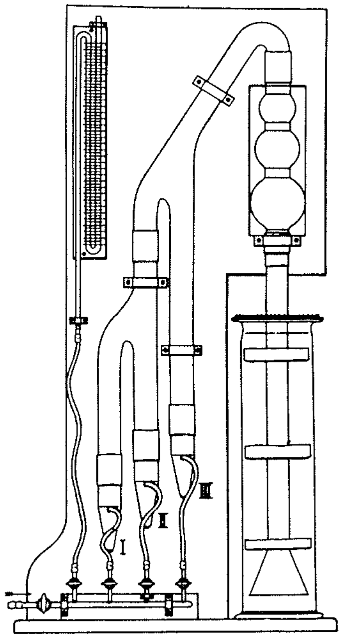


Fig. 29--Gary-Linder air elutriator (1906).

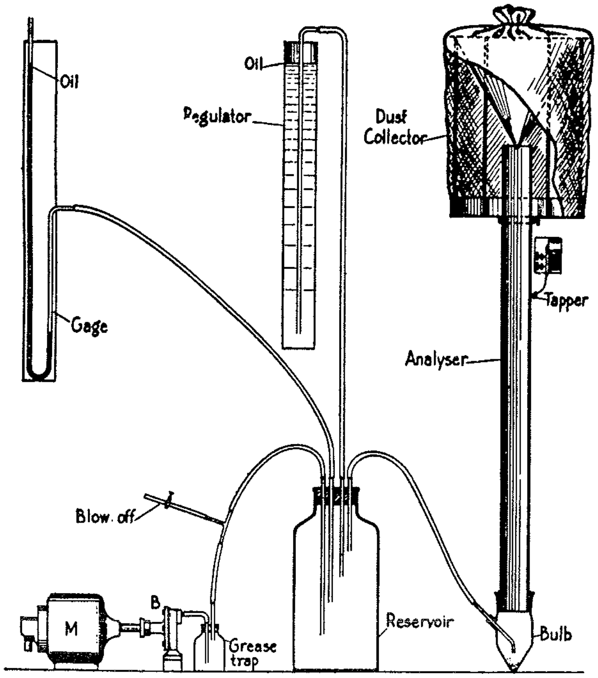


Fig. 30--Bureau of Standards air analyzer (1915)

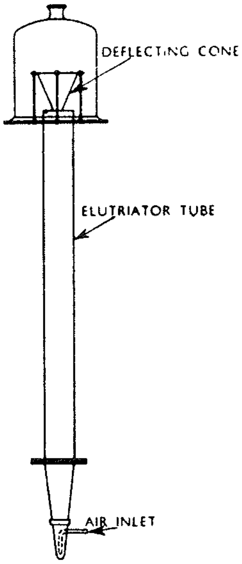
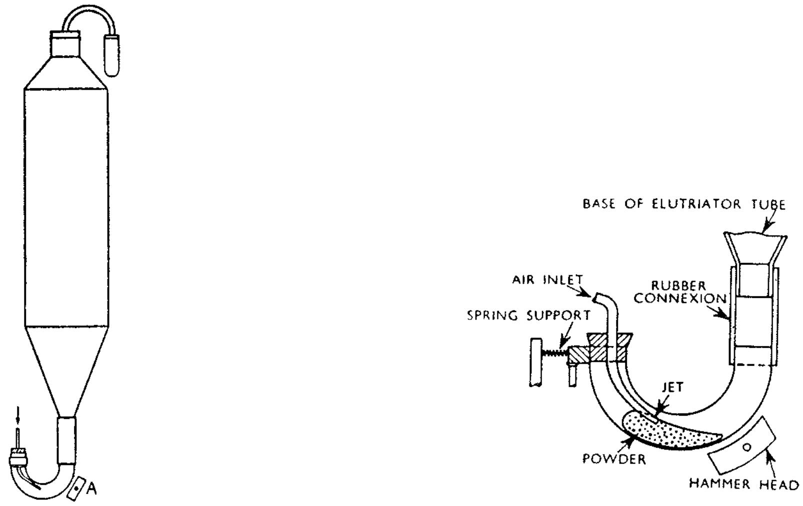


Fig. 31--Gonell's air elutriator (1929).



a. Diagrammatic sketch of separation column.

b. Detail of sample location and air inlet.

Fig. 32--Roller's air elutriator (1931).

a. The air jet pointed downward and impinged on the granular material in a cone-shaped bulb instead of being forced through the material.

b. The separating column was a tall smooth tube without rough surface where particles might lodge.

c. An automatic tapping device minimized adherence of particles to surfaces.

d. A collecting chamber was provided at the top to retain all the very fine material.

The apparatus designed by Gonell (60) in 1931 and Roller (91), shown in Figs. 31 and 32, respectively, embody the main principles stated above. In addition, Roller found that electrically grounding the metal tubes and bends completely eliminated the tendencies for adherence of particles to metal surfaces. His apparatus is one of the most popular at present.

24. Accumulative sedimentation--Particles settling in a fluid will accumulate at the base of a sedimentation column at a rate dependent upon their fall velocities. Methods of measuring the accumulation, either by weight or by volume, at designated times during an analysis, are termed "accumulative sedimentation." Sedimentation methods where individual fractions are secured and measured separately are termed "fractional sedimentation" and are discussed separately.

Although a direct volume measurement of accumulated material is far simpler than a weight measurement, it has been subjected to severe criticism, especially in its use with the fine-grained sediments. Factors which affect the volume of sediment deposited are:

a. Gradation and size of particles.

b. Shape of particles.

c. Time of standing.

d. Weight of overlying material.

Because these factors are variable and cannot be controlled, weight measurements are generally used.

Accumulation methods are divided into two groups: first, those where all the material is added at the top of the column in such a manner that all particles have to settle the entire length of the column, and second, those where the material is dispersed uniformly throughout the sedimentation column. This classification is necessary because there is a basic difference in the character of the data secured.

When sedimentation of all particles proceeds from the top of the column, periodic measurements of the accumulated material at the base represent, directly, size grades with limits which are easily determined from the velocity of fall. A graphical plot of the weight of accumulated material with respect to time provides a curve commonly referred to as the "cumulative curve." Since an ordinate at any time $\underline{t}$, represents the weight of particles having a greater fall velocity than is represented by that settling time $\underline{t}$, direct readings of size grades can be made, the selection of which is flexible and dependent upon the refinement desired.

In a "disperse sedimentation system," where the sediment is dispersed throughout the sedimentation column at the start of the settling period, the accumulation will consist not only of particles of sufficient fall velocity to settle from the top of the column by the time of reading, but also of smaller particles that had a shorter distance of fall. A curve showing the amount of accumulated material with respect to time does not allow a direct determination of size because the finer particles are included. It is referred to as an "accumulative curve" and must be considered as distinct from the "cumulative curve" previously described. In

1915, Oden set forth the theory of the disperse sedimentation system and developed both an analytical and a graphical method of interpreting the data in terms of particle size. Krumbein (60) presents an excellent summation of Oden's original theory and methods of analysis. Since the analytical interpretation is complicated and awkward to apply, the graphical solution has generally been used. The graphical interpretation is as follows:

> The accumulative curve is plotted with time as abscissa and percentage by weight of total material settled out as ordinates, as shown in Fig. 33. Tangents to the curve at any points, $\underline{T_1}$ and $\underline{T_2}$, intersect the ordinate axis at $\underline{w_1}$ and $\underline{w_2}$, respectively. The difference between the percentages $\underline{w_2}$ and $\underline{w_1}$ represents the amount of material in a size grade with limits determined by the settling times $\underline{T_2}$ and $\underline{T_1}$ which are representative of fall velocity which, in turn, is a function of particle size.

The main objection to the graphical interpretation is the difficulty of drawing tangents accurately. Weinig (132), concerned with the analysis of ore grinds, has developed an analytical method which is apparently simpler and a graphical method which is more accurate than the so-called "tangent method" just described. The methods are based on an arithmetic series of numbers, termed "ordinal numbers," each representing a particle diameter. The system is so devised that diameters of the particles of adjacent numbers vary as $\sqrt{2}$, which, in the range of Stokes' law, means that their fall velocities vary as 1/2. In the graphical method the "ordinal numbers" are plotted as abscissae and cumulative weights as ordinates on semi-logarithmic paper to provide an accumulation curve that may be interpreted using chords rather than tangents. Greater accuracy and less influence of personal judgment is obtained.

A number of devices have been developed where the material starts its fall from near the top of the sedimentation column. Popular among

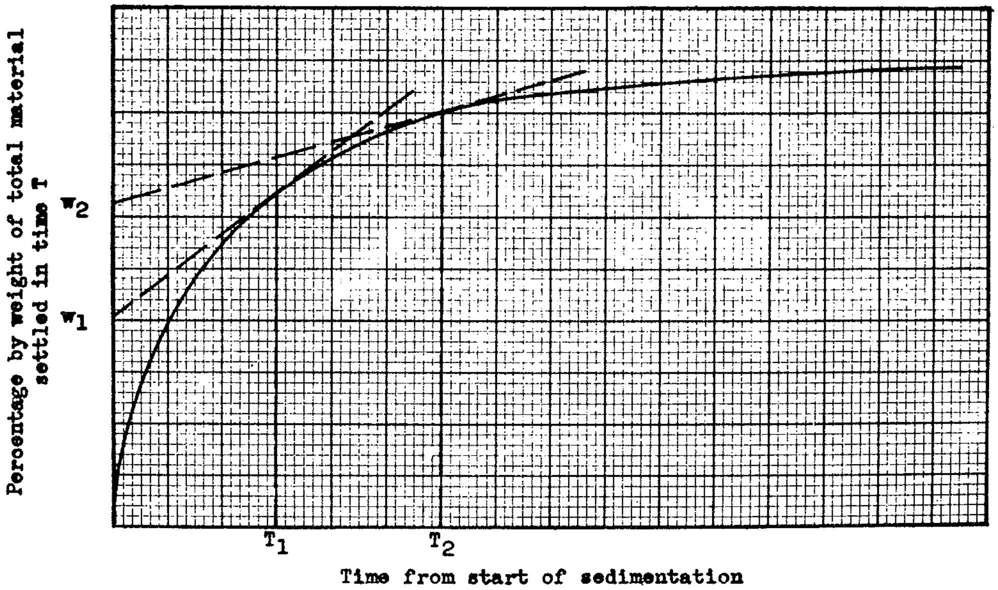


Fig. 33--Graphical solution of accumulative sedimentation curve.

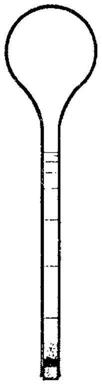


Fig. 34--Bennigsen's silt flask (1860).

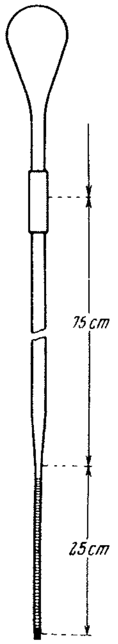


Fig. 35--Clausen's modification of Bennigsen's silt flask (1899).

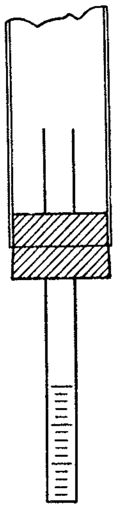


Fig. 36--Werner's volumetric accumulative device (1925).

FIGS. 33, 34, 35 & 36

the earlier devices (60) of this type was Bennigsen's silt flask, which is shown in Fig. 34. The water-sediment mixture was agitated in the flask and then after inversion the depth of the material that settled in the stem in certain time intervals, was observed. Improvements were effected by Clausen, as shown in Fig. 35, by making the stem removable from the mixing or dispersion bulb, and by using a smaller bulb and a longer sedimentation column. A more recent apparatus was devised by Werner (139) consisting of a small tube, 1.5 cm. in diameter, 1 to 1.5 m. in length, with a smaller, graduated measuring tube for volumetric measurements. A magnifying glass was provided for accurate readings. The apparatus is illustrated in Fig. 36. The first analyses were made with the sediment dispersed throughout the tube, but the apparatus was later modified so as to produce a cumulative rather than an accumulative curve by placing a "mixing room" at the top of the sedimentation column, probably similar to Clausen's apparatus.

Although the apparatus of Bennigsen, Clausen, and Werner provide data which allow direct interpretation into size grades the procedure of introducing the material is difficult and questionable and the feature of volumetric measurement is objectionable. They have had very limited application to size analysis of fine materials.

Following the development of the settling theory of a disperse system, Oden (66) devised the apparatus, illustrated in Fig. 37, which consists of an analytical balance with one pan suspended near the base of a sedimentation cylinder, the other so arranged that, as sediment settled upon the immersed pan, small spheres would be deposited upon it, returning the balance to equilibrium. The rate of deposition of the small weights

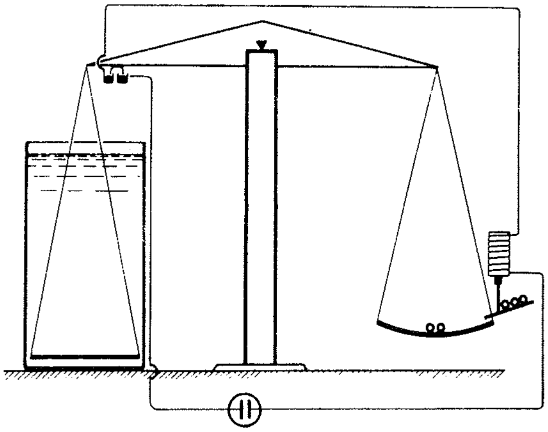


Fig. 37--Oden's original sedimentation balance (schematic) (1915).

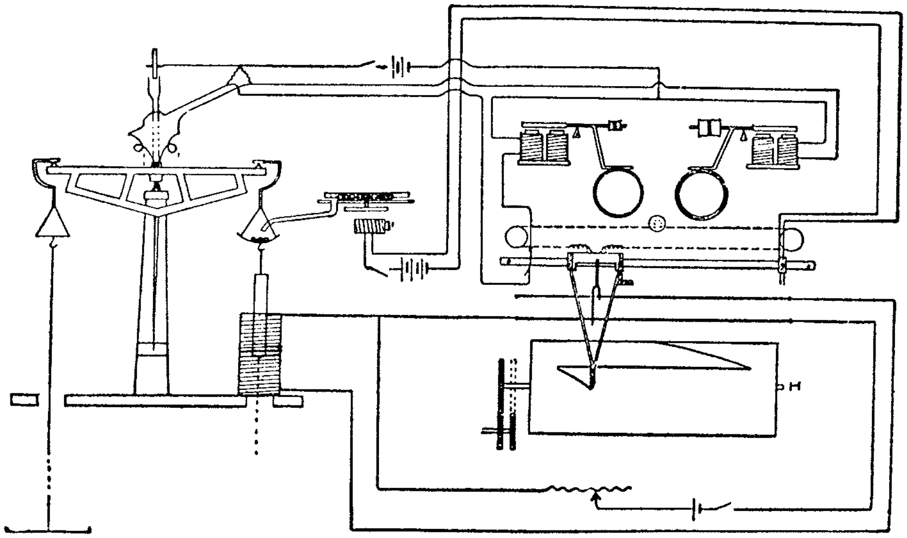


Fig. 38--Oden-Keen's improved sedimentation balance with automatic weighing and recording equipment (1924).

determined the accumulative curve. Fig. 38 shows a modification of the original apparatus with an automatic recording mechanism consisting of a pen moving on a revolving drum H that recorded the addition of the small weights.

Johnson (66) devised a somewhat simpler recording device, which is shown in Fig. 39, using a controlled spark between the pointer on the balance arm and the drum to burn a series of small holes in the recorder paper. This produced an accumulative curve directly.

Weinig (134) describes the use of an ordinary analytical balance with a special noncorrodible sedimentation pan. Using a regular 800 cc. beaker for the sedimentation cylinder, the analysis is conducted with manual determination of the weights necessary to maintain the balance at equilibrium.

An automatic weighing apparatus, with a concave mirror mounted at the fulcrum of the analytical balance perpendicular to the balance arm, is described by Bishop (20). When the balance is not in equilibrium, light from a constant source is reflected by the mirror into a photoelectric cell. This augments an electric current that, in turn, operates a relay and starts a motor connected to a resistance. A decrease in resistance increases the current in the solenoid sufficiently to restore the balance to equilibrium and deflect the light away from the photoelectric cell. This action occurs repeatedly during the sedimentation period and from the current consumption record an accumulative sedimentation curve can be drawn.

A much simpler apparatus for securing an accumulative curve was used by Schramm and Scripture (97), consisting of twelve test tubes, 30 cm. in height and about 3 cm. in diameter, held vertically in a special rack. Clay suspension was poured into each tube to a predetermined level, care being taken to secure the same concentration in all tubes. At the start of the analysis the rack of tubes was shaken sufficiently to obtain a uniform distribution of clay throughout the suspension in each tube. At different selected times during a sedimentation period, the suspensions above common marks were siphoned off from each tube and each residue was analyzed for total weight. From the values obtained an accumulative sedimentation curve was constructed that lent itself to Oden's graphical analysis.

Coutts and Crowther (30) probably have made the most exhaustive study of the accuracy and reliability of the sedimentation balance, conducting experiments on the effects of size and rim of pan and height of pan above the base of the sedimentation cylinder. In their summary they state:

> "............The low density of the suspension immediately below the pan after the sedimentation has proceeded a few minutes inevitably sets up a flow of liquid which interferes with the free vertical fall of the particles. With the large narrow-rimmed pans hitherto used the observed yields are appreciably below the theoretical values. With the pan close to the base there is a very rapid change in yield with very small changes in position of the pan. The extent of the disturbance varies with the size of particle and thus produces a distortion of the distribution curve."

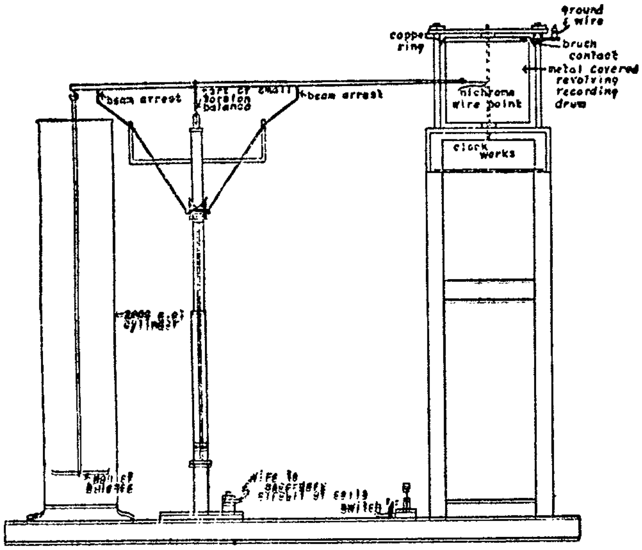
Keen (53) investigated the accuracy of the sedimentation balance and concluded that the method fails to give an accurate curve of particle size distribution.

25. <u>Fractional sedimentation</u>--Fractional sedimentation applies to methods where the sediment is measured in individual fractions. Although

such methods have been fairly popular for analysis of coarse sediments, their use with fine particles has been very limited due to several major difficulties. As the size of particles decrease it becomes increasingly more difficult to add the sediment at the top of the sedimentation column without introducing serious disturbances and tendencies for the material to settle as a mass rather than as individual particles. If the material is dispersed throughout the entire sediment column it necessitates a complicated and awkward analysis, even more so than that of the accumulative sedimentation curves secured with the sedimentation balance. Even though a satisfactory method of calculation is devised, there still exists the disadvantage that only a portion of each desired gradation is secured and the errors in the corrections that must be applied are magnified.

With the presence of small particles slight disturbances that might be produced during the removal of the fraction become more serious, tending to invalidate the data. Because of these limitations, an apparatus has not yet been developed that can be considered satisfactory for routine analysis of sediment samples by this method. However, the advantage of a method whereby each grade can be collected as an individual fraction is obvious.

One of the first apparatus to embody the principle of fractional sedimentation was that constructed by Schloesing (66) in 1903. His experimental apparatus, shown in Fig. 40, consisted of a rather complicated arrangement for adding drops of clear water at a constant rate, forcing out drops of the settled sediment and water at the base of the sedimentation column <u>A</u>. At the start of an analysis, the water-sediment mixture was merely poured into the top of the column <u>A</u>. A series of small boxes,



WIRING DIAGRAM OF BALANCE.

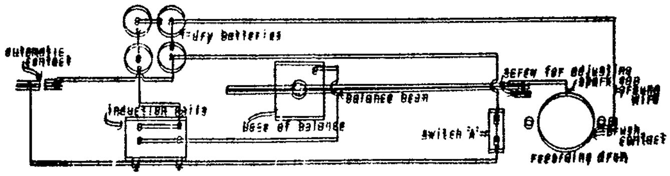


Fig. 39--Johnson's automatic recording device for Oden's sedimentation balance (1923).

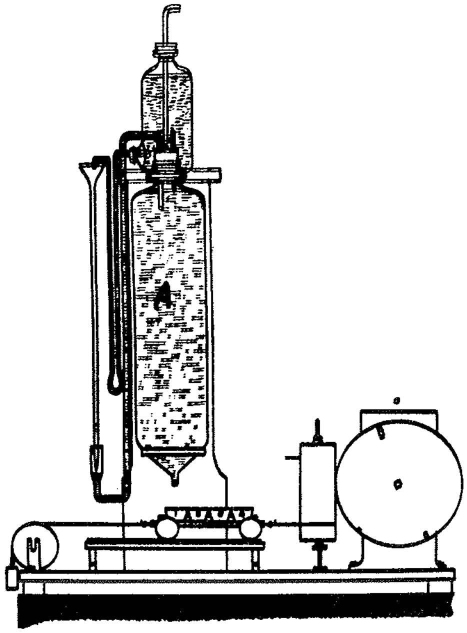


Fig. 40--Schloesing's fractional sedimentation apparatus (1903).

actuated by a clock-work and drum arrangement, passed under the outlet at the base of the column. Particles of a certain size range were collected in each box, the gradation being dependent upon the height of the sedimentation column and upon the length of time each box was collecting drops. The objectionable points in this device are the method of adding sediment and the tendency for particles to lodge on the sloping surfaces at the base of the column.

A fractional sedimentation apparatus, developed by Kuhl (37), which is similar but much simpler in design, is illustrated in Fig. 41. The device for introducing the sediment at the top of the column is shown. During the analysis, water is added very slowly to the stoppered sedimentation tube forcing out an equal volume of water-sediment which is collected over given time periods in a number of containers. The residue in each container represents a certain size grade of the material.

With a flask similar to that used by Bennigsen, illustrated in Fig. 34, but with the neck separate as used by Clausen, Lober (60) obtained fractions by holding a finger over the opening, immersing in water, and then removing the finger a short time so that the collected sediment was deposited in a container. Repeating this process resulted in a series of fractions each representing a certain size grade. Vierhiller's (37) sedimentation apparatus, shown in Fig. 42, functioned in a similar manner.

In 1935, Cole and Edlefsen (29) developed a somewhat unique sedimentation tube, which is shown in Fig. 43. Segments of brass tubing, 2 in. long, fit snugly inside of an outer brass tube. Each end of the

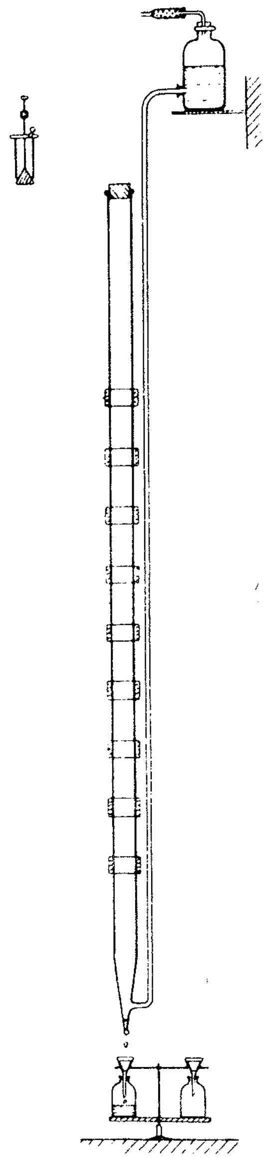


Fig. 41--Kuhl's fractional sedimentation apparatus (before 1926).

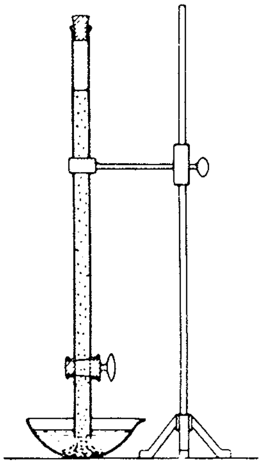


Fig. 42--Vierhiller's fractional sedimentation apparatus (1926).

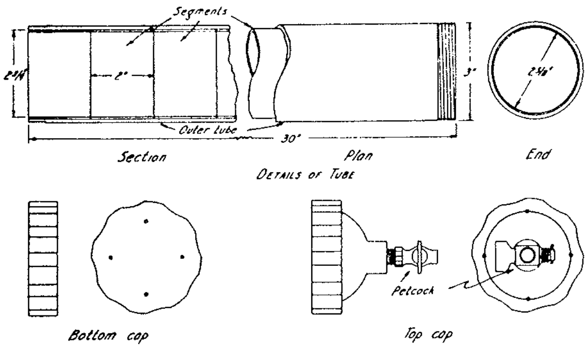


Fig. 43--Cole's and Edlefsen's fractional sedimentation tube (1935).

cylinder is equipped with a screw cap to force the 2 in. segments closely together. With the bottom cap in position, the water-sediment mixture is poured into the cylinder and the top cap screwed in place. Water is added through the pet cock until the remaining space above the cap is filled. The device is then mounted on a wheel and rotated until complete dispersion is effected after which it is stopped and maintained in a vertical position for a predetermined period of time. Then it is rotated into a horizontal position and left undisturbed for 10 to 12 hours. Small plugs in each cap are removed and the water is drained very slowly from above the deposited sediment, a procedure that requires another 10 to 12 hours. Then each segment is removed carefully and the contents washed into a drying dish. Being initially a disperse system, each fraction obtained is not of one size range of material but includes smaller sizes, a condition that makes the interpretation difficult.

26. Pipette- The pipette method, developed independently in 1922-23 by Krauss (60) in Germany, Robinson (89) in England, and Jennings, Thomas, and Gardner (50) in the United States, appears to be used more universally at the present time than any other method for size analysis of fine material. It consists essentially of the withdrawal of a small sample of suspension at a fixed point in a sedimentation cylinder after a certain period of time.

The principle of action or theory is very simple. After thoroughly dispersing the material throughout the suspending medium the particles are allowed to settle. If H represents the distance from the top of the column to the point or elevation where the pipette sample is to be withdrawn, then, after a time, $\underline{t}_1$, all particles with a settling velocity greater than H/t_1 , will have settled past the withdrawal elevation. If a pipette sample is secured at time $\underline{t}$, at depth $\underline{H}$, its concentration will represent the original concentration of all particles with settling velocities less than H/t_1 . When another period of settlement has elapsed and at time $\underline{t}_2$ another sample is secured its concentration will represent, similarly, the original concentration of particles with settling velocity less than H/t_2 . The difference between the concentrations in two consecutive samples is a proportional part of the gradation in the original suspension with the size limits determined by the corresponding fall velocities. The ratio of concentration of the grade in the pipette sample to that in the original is the same as the ratio of the volume of the pipette sample to the total volume of the sedimentation column.

The popularity of the pipette method for routine analysis, where the amount of material in suspension can be controlled, as in soil analyses, is due to:

<u>a</u>. Its accuracy and reliability.
<u>b</u>. The ease of interpreting the data.
<u>c</u>. The relatively simple technique.
<u>d</u>. The simplicity of the equipment.

Theoretically, to function correctly the pipette should withdraw its sample instantaneously, removing a layer of infinitesimal thickness at the desired depth. Actually, the withdrawal is made over a finite period of time in such a manner that a minimum of disturbance is introduced into the sedimentation cylinder. Obviously, it is impossible to sample a layer of infinitesimal thickness and what actually may occur is illustrated in

Fig. 44, showing the tendencies towards spherical effects (60). Except for a single pipette of ordinary design used by Robinson (89), the earliest withdrawal devices had multiple tube intakes as illustrated in Figs. 45 and 46, that more nearly approached the ideal withdrawal condition. It has been generally conceded that the withdrawal effects are not serious even with a single pipette if the withdrawal is at a reasonable rate and the sampling is not too close to the surface.

Multiple inlets have been practically abandoned in favor of single stem pipettes and more emphasis is being placed upon external arrangements to facilitate withdrawal of the sample, and emptying, and rinsing the pipette. A number of these pipettes are illustrated in Figs. 47 to 51, incl. The volume of the pipette bulb is generally 10 or 20 cc., the smaller size being the more common.

An advantageous feature of the pipette method is its flexibility. By using a shallow withdrawal depth the size determination of the very fine material can be conducted without an excessively long settling time and with less difficulty in maintaining a constant temperature. Although the ordinary, and probably the practical withdrawal depth lies between 10 and 20 cm. below the surface, a special pipette sampling at only 2 cm. below the surface has been introduced by Vinther and Lasson (40). A 4-min. withdrawal time with a constant suction is used as compared with about 40 sec. for the ordinary type of pipette. Vinther and Lasson claim to have obtained accurate results with this method and to have effected separation of particles as small as 0.16 microns, in a 4-day settling time with a fall distance of only 2 cm.

To separate material below 0.5 microns in size, Steele and Bradfield

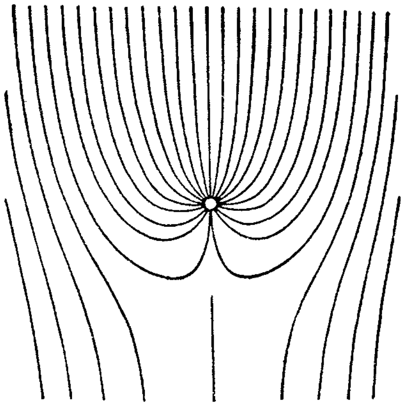


Fig. 44--Streamlines of pipette withdrawal (by Kuhn).

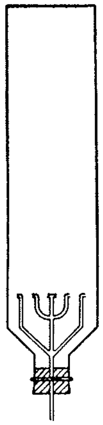


Fig. 45--Multiple inlet pipette used by Jennings, Thomas, and Gardner (1922).

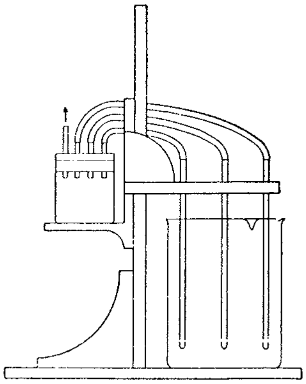


Fig. 46--Krauss's multiple inlet pipette (1922).

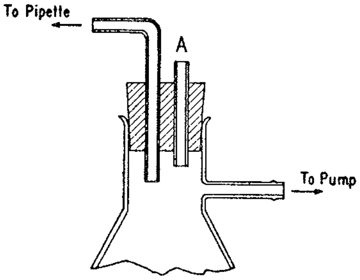
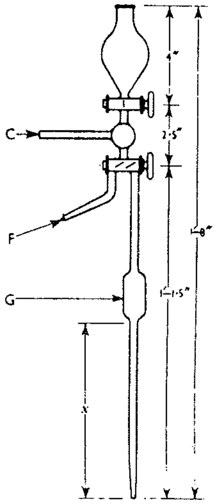


Fig. 47--Whittle's withdrawal device.



Detail

C Safety bulb pressure inlet tube

F Outlet tube

G Standard 10 cm. pipette, dimension x to be as large as possible

Bore of tubing to be 4 m.m. where possible

Fig. 48--Constant volume pipette.

FIGS. 44, 45, 46, 47 & 48

(101) used centrifugal force to decrease the sedimentation time to within practical limits. Separations down to 0.0625 microns were accomplished.

A consideration which is more important in the pipette than in the decantation method, where it may merely increase the number of decantations, is that complete, uniform dispersion is necessary in order to obtain accurate results. When single pipettes are used this becomes increasingly important. In the modified Andreason apparatus, shown in Fig. 49, compressed air is used to effect the dispersion.

Disturbances during the sedimentation period should be minimized. The pipette should be attached to a rigid stand and, if withdrawal is necessary, it should be equipped with a suitable lowering and raising mechanism, such as Shaw's pipette rack, illustrated in Fig. 50.

As in other sedimentation methods, temperature control is a primary consideration. The apparatus shown in Fig. 51a, is one of the latest developments in England for making routine size analyses. Four of Andreason's pipette units, a cross-sectional view of which is shown in Fig. 51b, are housed in a thermally insulated cabinet with automatic temperature control.

The concentrations for which the pipette method generally is suitable vary from 1 to 2 per cent (10 000 to 20 000 p.p.m.). Although somewhat lower concentrations could probably be analyzed with sufficient accuracy, the pipette method is definitely limited because the sample is a "sample of a sample," and as the concentration decreases the technique must be refined accordingly to provide a satisfactory degree of accuracy. The difficulty of applying this method to suspended sediment samples with concentrations below 2 000 p.p.m. or 0.2 per cent, is obvious.

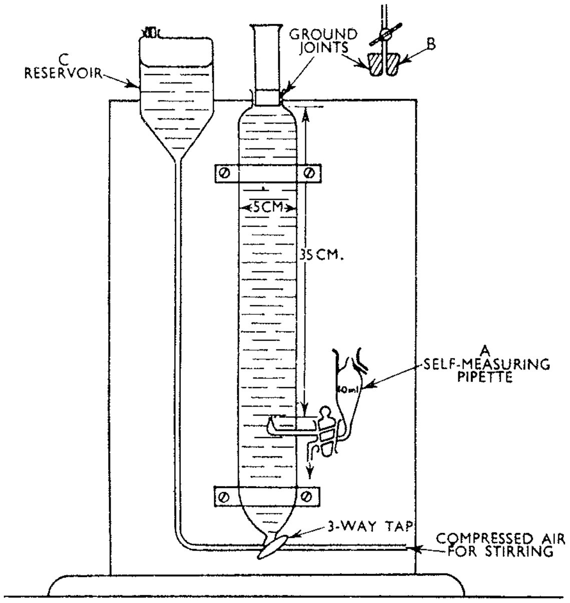


Fig. 49--Modification of Andreason's pipette apparatus.

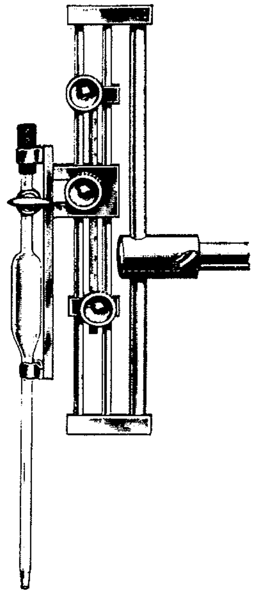
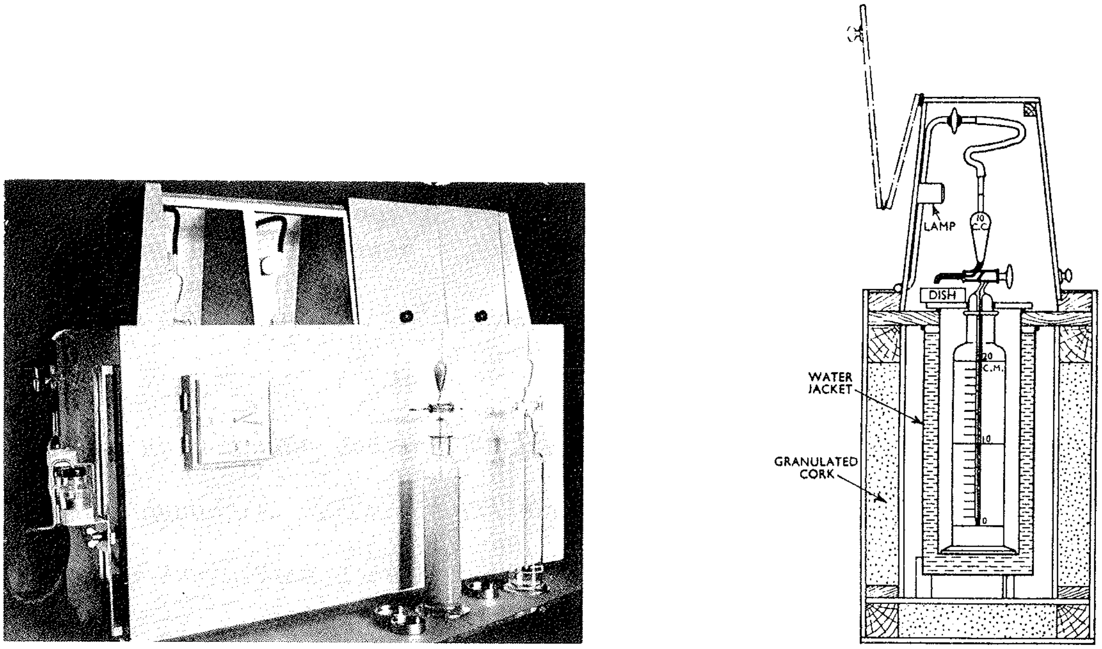


Fig. 50--Shaw's pipette rack.



a. Complete apparatus in insulated cabinet.

b. Cross-sectional view.

Fig. 51--Andreason's pipette apparatus (1928).

FIGS. 49, 50 & 51

Some pipette procedures require that the suspension be redispersed after each sample has been taken. Rittenhouse (87) conducted a series of tests and found that redispersion does not result in greater accuracy and that a tremendous saving in time is effected by allowing the sedimentation process to proceed continuously from the start. In additional efforts to reduce the unit time and cost of the analyses, Rittenhouse (88) has devised what can be termed "mass production methods." He has set up a procedure where 48 samples may be analyzed simultaneously and introduces minor modifications for saving time. His paper is complete even to the extent of providing data forms.

Krumbein (60) presents in detail a procedure for the pipette method as commonly used in this country.

27. Hydrometer--The progressive decrease in density that occurs at any given elevation in a disperse sedimentation system can be correlated with the fall velocities of the particles. The measurement of this change in density is the basis of a number of size analysis methods. The common method of determining density is to measure the buoyant effect of the suspension on a floating or on an immersed object, ordinarily referred to as the hydrometer and plummet methods, respectively. A less common method is the adaptation of a manometer to measure the relative height to which a column of liquid will be supported by the sedimentation column. The plummet and manometer methods will be discussed in later sections.

Since Bouyoucos introduced the hydrometer in 1927 (60) it has found widespread use for size analysis, particularly in the study of soils. The hydrometer developed by Bouyoucos, shown in Fig. 52, measures the density at some level below the surface in terms of solids remaining in suspension, per liter of the total suspension. The Casagrande hydrometer, shown in Fig. 53, measures the density of the suspension relatively in terms of specific gravity. Since the calibrations for both of these instruments apply to water at 67° F., corresponding to a certain density, their use with other temperatures necessitates a correction factor. As the density of the suspension decreases the center of gravity of the hydrometer rises, necessitating the application of another correction factor. These correction factors are included in the A.S.T.M. standard hydrometer procedure (11).

Conducting the actual analysis with the Bouyoucos or Casagrande hydrometer is comparatively simple. Before the sedimentation process is allowed to start the material is thoroughly dispersed throughout the sedimentation column. Then, at predetermined increasing time intervals, hydrometer and temperature readings are taken. The frequency and number of readings taken depend largely upon the character of the material and the type of investigation.

The particularly advantageous features of the hydrometer method are the simplicity of equipment, the rapidity of the analysis, and the easy interpretation of the data. However, there has been considerable discussion, despite the claims made by Bouyoucos (24), as to its accuracy, sensitiveness, and theoretical soundness. The features considered most objectionable are:

> a. The bulb extends over a portion of the sedimentation column and therefore indicates an average value of density rather than the density at a certain elevation.
>
> b. The common type of hydrometer sinks deeper as the density decreases and therefore measures the density over a changing height of column.

c. The hydrometer tends to increase in weight because of particles settling on its shoulders. On the other hand, if removed after each reading and then immersed again, some disturbance is introduced into the sedimentation cylinder.

d. Rising currents, due to the fall of large particles at the start of the analysis, may produce abnormally high density readings until the currents subside.

e. As with other types of sedimentation methods, there is a decrease in relative sensitivity as the suspensions decrease in concentration.

To minimize these objections Bouyoucos conducted numerous experiments and developed a more sensitive instrument (23) reading from 0 to 10 gm. and graduated to 0.2 gm.

Thoreen (111) studied the hydrometer method in connection with highway materials investigations and concluded, "The errors involved in the use of a sensitive hydrometer are subordinate to those due either to partial deflocculation or to the assumptions involved in Stokes' law."

Biddle and Klein (19) studied the application of the hydrometer to size determination of Portland-puzzolan cements and found it entirely satisfactory for routine analyses.

Hatch (43) found the hydrometer sufficiently accurate for analysis of hydraulic fill dam material and recommended its use because of the rapidity of analysis and the simplicity of apparatus.

The objection to the hydrometer, in that it secures an average density over a certain column height, was studied by A. N. and B. R. Puri (81) who concluded, after a laboratory investigation by pipette sampling of 150 soil samples, that the average density of a column 10 to 12 cm., located at least 5 cm. below the surface, can be assumed to be its density at its midpoint. They also concluded that this assumption becomes more valid as the particles decrease in size.

Regardless of the value and established accuracy of the ordinary hydrometer for many types of studies, it has, from the standpoint of application to suspended sediment samples, the objectionable feature of requiring a relatively high concentration. Even the Bouyoucos sensitive hydrometer reading from 1 to 10 gm./liter lacks the sensitivity necessary for size analysis of this type of sample.

Appreciating the simplicity of the hydrometer method, but not satisfied with existing types, particularly because the manner of reading the density was somewhat crude and because errors of parallax were introduced in observing the height of the meniscus on the stem, Puri developed a new type of hydrometer (78). His hydrometer, shown in Fig. 54, had essentially the same bulb form as the former types, but was radically different in the manner of reading. Instead of a graduated paper inserted in the stem, a pin was mounted at the top of the hydrometer stem. This stem could move within a short glass tube, graduated completely around the circumference so as to avoid parallax. The graduate glass tube was adjustable in elevation and, by means of an attached pointer, it could be referenced to the actual water surface. Readings accurate to a fraction of a millimeter are claimed for this device.

A less refined instrument, but more rugged and better adapted to field use, shown in Fig. 55, also was developed by Puri (77). It has the unique feature of allowing an adjustment of the hydrometer weight to compensate for a difference between the actual temperature and the temperature at which the initial calibration was conducted. The calibration was made at 95° F., the highest temperature at which the instrument would probably be used, and with each degree drop in temperature a small weight was added to compensate for the increased density of the suspension. This was accomplished by having a hollow stem with a cap into which could be screwed a thin brass wire of such a weight that the increased density was balanced. Wires of different weights were prepared during the initial calibration. When the temperature change was other than a full degree a small correction was applied to the hydrometer reading. The advantages of this feature were that the hydrometer could be held at approximately the same level in the suspension, some of the corrections that are necessary with changes in center of gravity were eliminated, and other calculations were simplified.

Probably the most sensitive and accurate hydrometer yet devised was that introduced by A. N. and B. R. Puri (81) in 1939 which is illustrated in Fig. 56. The apparatus, which was termed "chaino-hydrometer" by the inventors, allows a very accurate weight measurement instead of a volume determination as in former hydrometers. Its development embodies the accuracy and precision of the analytical balance without sacrificing rapidity in the analysis. It has been found very sensitive in the analysis of soil suspensions.

The chaino-hydrometer has a number of unique features in its design and operation. A rectangular wire loop with a needle point at one lower corner is attached to the stem of the hydrometer to gage its immersion to the same depth for all readings. At the top of the loop is a short aluminum tube through which a fine chain passes. One end of this chain is attached to a hook on the rectangular wire loop while the other end is fastened to a slide that is moved up and down by an adjusting screw. Small increments of weight can be added or subtracted, by adjusting the

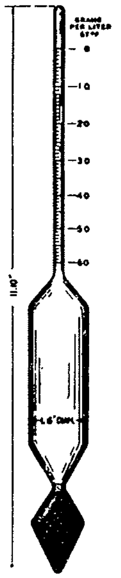


Fig. 52--Bouyoucos hydrometer.

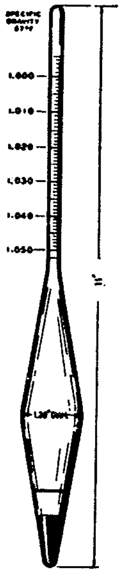


Fig. 53--Casagrande hydrometer.

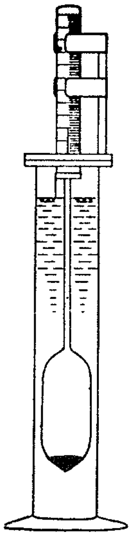


Fig. 54--Puri hydrometer (1933).

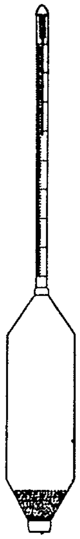


Fig. 55--Puri field hydrometer (1933).

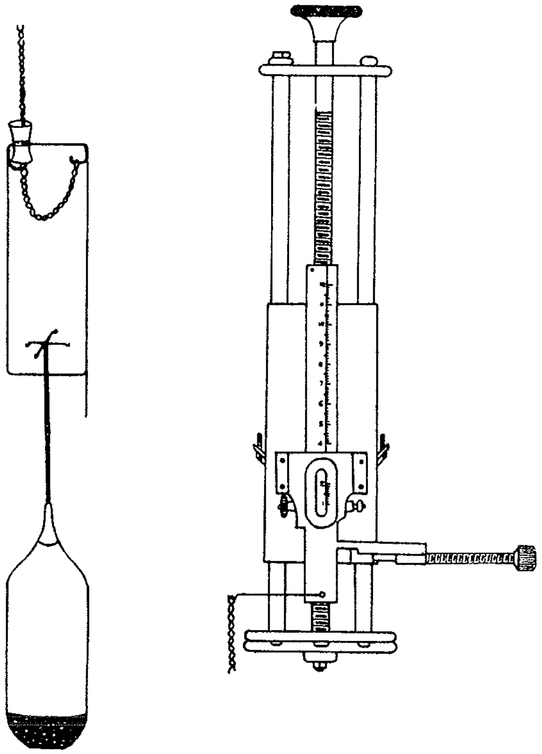


Fig. 56--Puri "chaino-matic" hydrometer (1937).

FIGS. 52, 53, 54, 55 & 56

length of the loop of chain, thus changing the weight of the hydrometer. The weight added is indicated by a vernier on the slide and a fixed scale. If more weights are needed than can be applied by the chain they can be added to the special hook at the top of the stem. The vernier can be set to zero at the start by moving the entire scale. The correction for changes in buoyancy due to changes in density of the water with respect to its temperature is accomplished by means of a correction chart developed for each instrument.

The degree of sensitivity claimed for this new type of hydrometer is remarkable. With the aid of the vernier, weight readings can be made accurately to 0.001 gm. A 1.0 per cent soil suspension gives an increase in density of about 0.006 gm./cc. An immersed body of only 1.0 cc. would therefore allow an accurate reading of density 1/6th of 1.0 per cent, or 0.17 per cent. Since hydrometer bulbs are 100 cc. in volume and greater, the chainomatic hydrometer could be made sensitive to 0.0017 per cent, or 17 p.p.m. Apparently the accuracy in reading is not the factor which controls the sensitivity. The actual accuracy of the instrument is more dependent upon other factors, such as settling of particles upon the shoulders of the hydrometer bulb, disturbances caused by removal after each reading, if this is the procedure, and the fact that an average density over the length of the hydrometer bulb is measured.

28. Plummet--The plummet differs from the hydrometer in that it is not a floating object and, unless suspended externally, it would sink to the bottom. The plummet is completely immersed and the buoyancy afforded by the suspension is determined solely by weighing, a feature that

presents probably the greatest advantage, from the standpoint of accuracy, of the plummet over the ordinary hydrometer with its volume readings.

The plummet, being necessarily an object of some length and diameter, is subject to all of the objectionable features listed for the hydrometer in Section 27, except b, for the plummet is entirely submerged at a fixed depth and the length of column over which the density is measured remains unchanged. This feature simplifies the calculations over those of the common hydrometer, the only correction necessary being for changes in density due to changes in temperature. The plummet, on the other hand, requires much more elaborate measuring equipment. Various studies (34, 67, 125) of the accuracy of the plummet method have revealed sources of error that do not permit extreme sensitivity; yet, a fair degree of accuracy is claimed for certain types of equipment.

In 1921, the U. S. Bureau of Mines developed one of the first plummet devices (98) which, as shown in Fig. 57, consisted of a glass plummet, partially filled with mercury, suspended from a chemical balance. It was used for size determination of clay particles in analyses extending over a 30-day period. The plummet was left immersed at a fixed depth for the first 7 hr., after which it was removed and was inserted again only when readings were desired. Temperature control was maintained by storing the apparatus in a well insulated box.

Schurecht (98) states that the plummet method is accurate to 0.0001 gm./cc., corresponding to 100 p.p.m. for a plummet of 100 cc. volume, and points out the following advantages over the decantation method:

<u>a</u>. It allows separation of extremely fine particles (less than 0.003 mm.) more readily than the decantation method.

b. Temperature control is easier because a small, constant volume of water is used.

c. With no change in liquid, flocculation tendencies are reduced to a minimum.

d. More analyses can be conducted in the same period of time.

A more complicated plummet apparatus, shown in Fig. 58, was developed in 1928 by Van Nieuwenburg and Schoutens (125). It was devised to eliminate the tedious work involved in the decantation method, and the disturbance due to the drawing off the sample in the pipette method. They criticized the length of Schurecht's plummet (6.35 cm.) as extending over too long a section of the sedimentation column, and used instead, one that was only 3 to 4 cm. long. This plummet, c in Fig. 58, weighing only 2 to 3 gm. when submerged, measured the density between 20 and 25 cm. from the top of the column. The exact weight of the plummet was obtained with a delicate balance, H . The apparatus was so constructed that it could be converted into a Schone water elutriator by merely removing the plummet and inserting the tube B at the top of the cylinder.

An error inherent in both the plummet and hydrometer methods, discussed by Van Nieuwenburg and Schoutens (125), is that there will be an increased concentration in the region of the immersed body where the cross-sectional area is reduced if the plummet is left immersed between readings. This tends to give higher density readings and, in the final interpretation of the data, may indicate a greater percentage of fine particles than is correct. Attempts to apply an arbitrary correction were not very successful and the conclusion was that a correction determined from the experimental data for each analysis is more satisfactory.

Olmstead, Alexander, and Laken (67) desiring a mechanical analysis method more rapid than the pipette method and more dependable than the ordinary hydrometer method devised a plummet apparatus incorporating a chainomatic analytical balance. A pear-shaped 10 cc. volume plummet, designed to minimize disturbance to the suspension, was suspended by a fine platinum wire from a rack and pinion arrangement. Although the accuracy of the pipette method was not obtained the method appeared promising.

The plummet method was investigated further by Edlefsen and Cole (34) using a similar analytical balance apparatus with rack and pinion to allow immersion and removal of the plummet with a minimum of disturbance and effort. The entire apparatus is illustrated in Fig. 59. An ellipsoidal plummet, 15 cm. in length, was lowered to 15 cm. below the surface at a constant rate of only 1.0 cm./sec. The balance was brought to equilibrium with special care so as to cause minimum movement of the plummet. In removing the plummet after each reading it was raised at the same rate.

Edlefsen and Cole (34) conducted a series of tests with plummets of different shapes using the pipette method as a check. Their conclusions were:

> _a_. Data from ellipsoidal plummets of different sizes, all 10 cm. in length but with volumes of about 46, 14, and 4.5 cc., were in close agreement mutually and with pipette data.
>
> _b_. A spherical plummet was not in agreement at the start with either the ellipsoidal plummets or the pipette due to settling of material on top, when withdrawal between readings was not possible. In later tests, when the plummet was withdrawn between readings, there was good agreement.
>
> _c_. Plummet data on duplicate samples agreed closely.

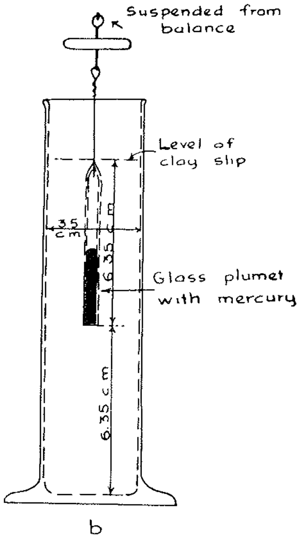


Fig. 57--Plummet apparatus U. S. Bureau of Mines (1921).

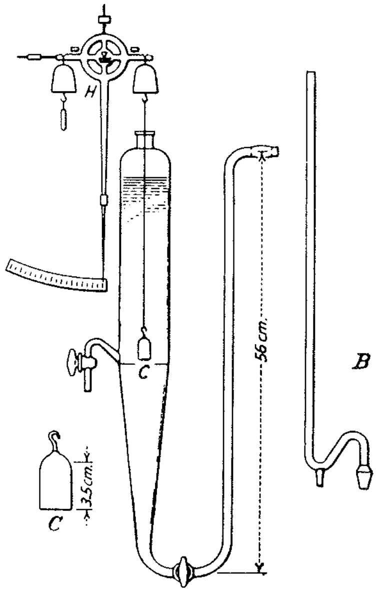


Fig. 58--Nieuwenburg and Schoutens plummet apparatus (1928).

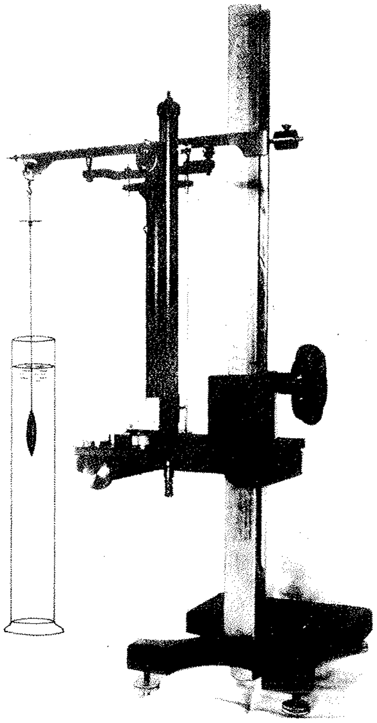


Fig. 59--Edlefsen and Cole plummet apparatus (1936).

d. When compared with the pipette method the plummet method is more rapid and can be used over about the same range of sizes to which the pipette is adaptable.

e. Because it requires about a minute to take the first reading it is not possible to use the Edlefsen-Cole plummet apparatus for particles with fall velocities greater than 0.25 cm./sec.

29. Manometer--If a manometer is connected near the base of a sedimentation column, the height to which the pure liquid will rise above the level in the sedimentation column is directly proportional to the relative density of the two liquids. The rate of decrease of the difference between the two column heights can be correlated with fall velocities of particles so that size determinations can be made.

The numerous devices that have been developed on this principle since its first use by Wiegner (60) in 1918, evidence its widespread popularity and apparently successful adaptation to particle size determination in a variety of fields. Wiegner's apparatus, shown in Fig. 60, represents the manometric device in its simplest form. With the stop cock closed to prevent premixing, the level of the suspension in the sedimentation column and the water level in the manometer tube are adjusted to the same level. After the particles are uniformly dispersed throughout the sedimentation column the stop cock is opened at the instant the settling action begins. At the start, and at predetermined times from the start, direct readings of the water level in the manometer tube are made. With complete dispersion at the start, the data determined in this manner can be plotted with time as abscissae to obtain a curve of a form similar to that of the Oden curve but different in that one is a depletive and the other an accumulative curve. The interpretation of the depletive curve produced by

Wiegner's tube, and all others that consist of only one manometer opening and where initial dispersion is used, is identical with that for the Oden type accumulative curve which, as described and discussed in Section 24, requires a graphical solution in routine analysis because of a complex analytical relationship.

There are a number of inherent features, present in all manometric devices applied to measurement of density, that may or may not be serious, depending upon the design and operation. Since the densities in the two connected columns are different, particularly so at the start, at the moment of opening or the moment the liquids come in contact, there is a decided tendency for mixing to take place that may set up currents which will affect the fall of particles. As the water level in the manometer tube falls, water flows into the sedimentation tube thereby producing objectionable cross currents. The inherent lag in the fall of the meniscus in the manometer, due mostly to adherence to the sides of the tube is particularly serious when the particles are large and have high fall velocities. Consideration must also be given to evaporation from both tubes when the analysis continues over a long period of time.

The small liquid level differential in the two columns demonstrates the need for sensitive manometers or precise observation equipment and general refinement in technique as illustrated in the following example. Consider a sedimentation column with 100 cm. distance from the top to the manometer opening. At the start of an analysis with a 1.0 per cent concentration of solids of specific gravity 2.65, completely dispersed through the column, the manometer liquid level will be only about 6 mm. higher than the sedimentation column. Since the ordinary type of size gradation produces a curvilinear accumulation or depletion curve, a large portion of this 6 mm. difference will be depleted early in the analysis. The difficulty in securing accurate visual readings for an entire size analysis with a simple Wiegner tube is obvious.

Modifications and improvements to Wiegner's tube appeared very shortly. Stiny (37) placed a small float at the top of the manometer tube, as illustrated in Fig. 61, that actuated a pointer moving on a circular scale. In addition, to eliminate the somewhat objectionable graphical interpretation of the Oden-type curve, he introduced the material at the top of the sedimentation column through a trap door arrangement. As discussed previously, this has not been demonstrated as being reliable for fine material although it has been apparently successful with silts and sands.

Gessner (37) devised a photographic device for Wiegner's tube that automatically recorded the water column height on a revolving drum with somewhat increased sensitivity and accuracy. Barnes (16) modified Wiegner's apparatus by using a smaller diameter manometer tube equipped with a sensitive measuring device. A needle point was adjusted to the surface of the water with a ratchet-gear arrangement. The exact point of contact was determined with a galvanometer in an electrical circuit between the water column and needle point. A movement of this needle point of only 0.005 mm. could be observed on a dial. An advantageous feature of this arrangement was that both tubes could be covered at the top to prevent evaporation. A small tube connected the air chambers above the columns so that pressures would be equalized.

Knapp (56) describes a rather recent (1934) apparatus, shown in

Fig. 62, which is adapted to mass analyses. The pressure is transmitted from the sedimentation column to a bellows arrangement B . The material is dispersed at the start through use of the stirring rod SR , the bellows being compressed to its limit prior to opening valve V . At the moment of opening valve V the mirror M , mounted so that one side may move with movements of the bellows, is tipped a certain degree depending upon the initial concentration. An intermittent beam of light from a source E impinges upon the mirror, is reflected through lens L_2 , into a rectangular slit, and on to a lantern slide plate LS . The lens brings the reflected light beam into sharp focus on the slide so that movements of the mirror produced by changes in pressure or column density are recorded as points. The sensitized slide rests in a carriage that moves at a known rate of speed. This device has been developed for mass production to the extent that one arrangement houses six complete sedimentation units with unified control of time, temperature, and removal of material.

Kelley (55) modified Wiegner's tube by inclining the manometer tube to greatly magnify the changes in density and thereby permit more accurate, direct readings. His device is illustrated in Fig. 63.

The Fort Peck manometer, shown in Fig. 64, which is similar in principle, has a small water reservoir at the top of the sedimentation column to reduce evaporation. The Tennessee Valley Authority and the U. S. Engineer Department, Omaha District, have also developed and used to some extent similar manometer devices for size analysis in their sediment investigations.

The "siphonic sedimentometer" (129) shown in Fig. 65, also utilizes

Fig. 60--Wiegner's manometer (1918).

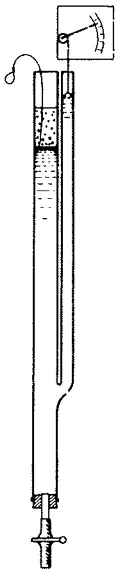


Fig. 61--Stiny's modification of Wiegner's manometer (1927).

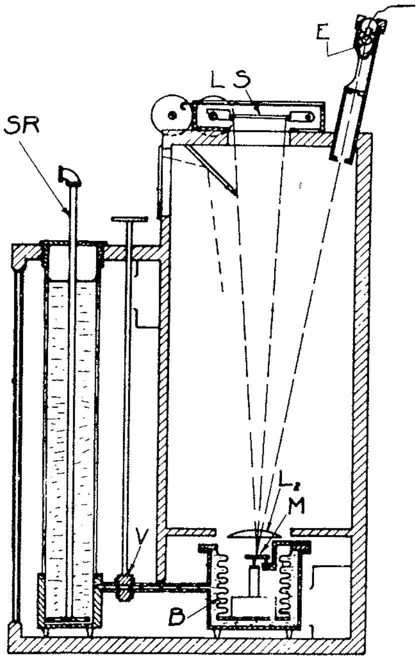


Fig. 62--Knapp's bellows manometer with automatic recording apparatus (1934).

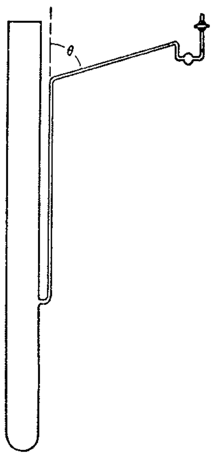


Fig. 63--Kelley's inclined manometer tube (1924).

FIGS. 60, 61, 62 & 63

the inclined tube for greater accuracy and, in addition, has the advantage of not requiring special equipment. The siphon type manometer, with a pointer attached for initial referencing to the surface of the sedimentation column, operates on the principle of hydrostatic equilibrium in communicating vessels.

Lambert and Wightman (61) developed a sensitive manometric apparatus with an automatic recording device but, instead of a tube inclined at some angle with the horizontal, they used a capillary tube exactly horizontal at approximately the top of the suspension column. The rate of movement of the meniscus in this capillary tube can be related to the change in density. The automatic recording device consists of a camera and moving cylinders with sensitized paper. A light beam above the manometer tube flashes periodically and the image of the water column is recorded upon the photographic paper, producing a depletion curve, like FCE in Fig. 66, that requires graphical interpretation as demonstrated previously. Recordings A and A' are initial and final calibration curves, respectively. Although the volume of water entering the sedimentation cylinder is reduced through the use of a capillary tube, Lambert and Wightman illustrate how it may be compensated for by initial calibrations or by certain corrective equations given in their article. They also recognize the lag in the capillary tube which is due to viscosity and apply a correction to the depletion curve during the initial settling time where this effect is of significant magnitude.

There have been a number of other adaptations of the manometric principle, mainly in the use of additional manometer openings and radically different types of pressure measuring devices. Zunker's apparatus (37), shown in Fig. 67, was an improvement over Wiegner's, chiefly in that the liquid level in the sedimentation column was measured at a point slightly below the surface because during initial dispersion, foam tended to form on the surface. The manometer could be disconnected from the sediment column at points A , an advantageous feature in that no disturbance to the manometer occurred during the initial dispersion.

Crowther's U-tube manometer (37), shown in Fig. 68, measured the change in density over a short distance near the base of the sedimentation column and provided a density-time curve whose ordinates could be interpreted directly in terms of fall velocities and corresponding particle sizes. Obviously, an objectional feature is the difficulty of measuring accurately the small differences in the height of the liquid columns.

The apparatus shown in Fig. 69a, which was developed by Lubirsky and Kosman (37), is similar to Crowther's in that changes in density are measured over a short column height. Fr is the sedimentation column and S_1 and S_2 manometer columns extended to about the same height as Fr so that water could be used rather than a liquid of greater density as is necessary with Crowther's apparatus. In operation the density difference between two adjacent connecting tubes, for example H_1 - H_2 , is secured by having only the proper connecting tubes open into S_1 and S_2 . The object of having five sets of connecting tubes is to maintain a somewhat constant and reasonable settling time as the material to be analyzed decreases in size. This is possible because the water-sediment is poured into the sedimentation column at the top rather than being initially dispersed. The automatic recording device, illustrated in Fig. 69b, consists essentially of a mirror, mounted on a platform supported

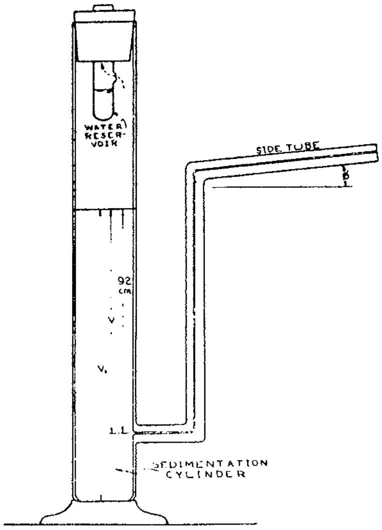


Fig. 64--Fort Peck manometer (1939).

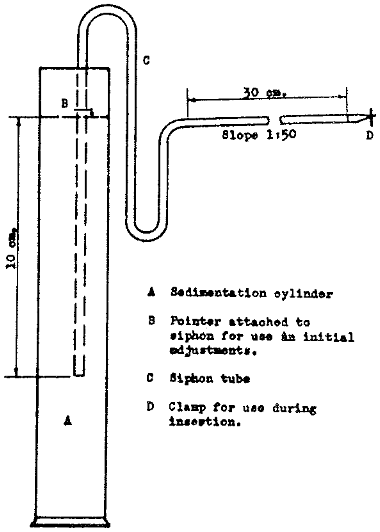


Fig. 65--Russian "siphonic sedimentometer" (1935).

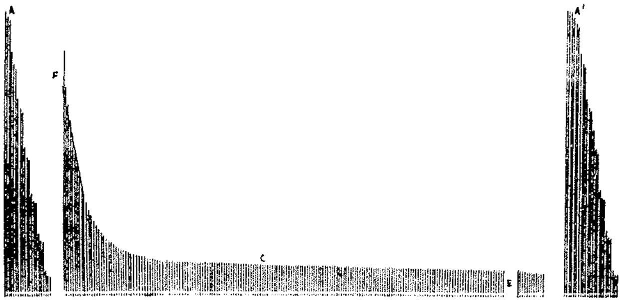


Fig. 66--Graphical data from Lambert's and Wightman's photographic recording manometer.

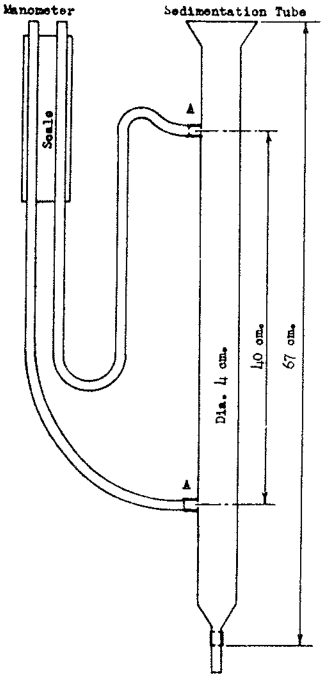


Fig. 67--Zunker's manometer (1922).

Fig. 68--Crowther's manometer (1927).

by floats $\underline{S_1}$ and $\underline{S_2}$, that reflects light from a fixed source on to a revolving drum covered with sensitized photographic paper. The most questionable point about this apparatus and method is the manner of introducing the material.

In 1925, Oden (66) investigated existing equipment and methods that employed the change in density principle and was not satisfied with the sensitivity and accuracy which they afforded. As a result he developed the apparatus shown in Fig. 70, in which greater differences in column height were obtained by using pentane, a liquid of low specific gravity, for a balancing liquid. To avoid effects of temperature changes he immersed the manometer in the sedimentation column. He states that the instrument proved successful in laboratory work. Details of the apparatus and its operation are:

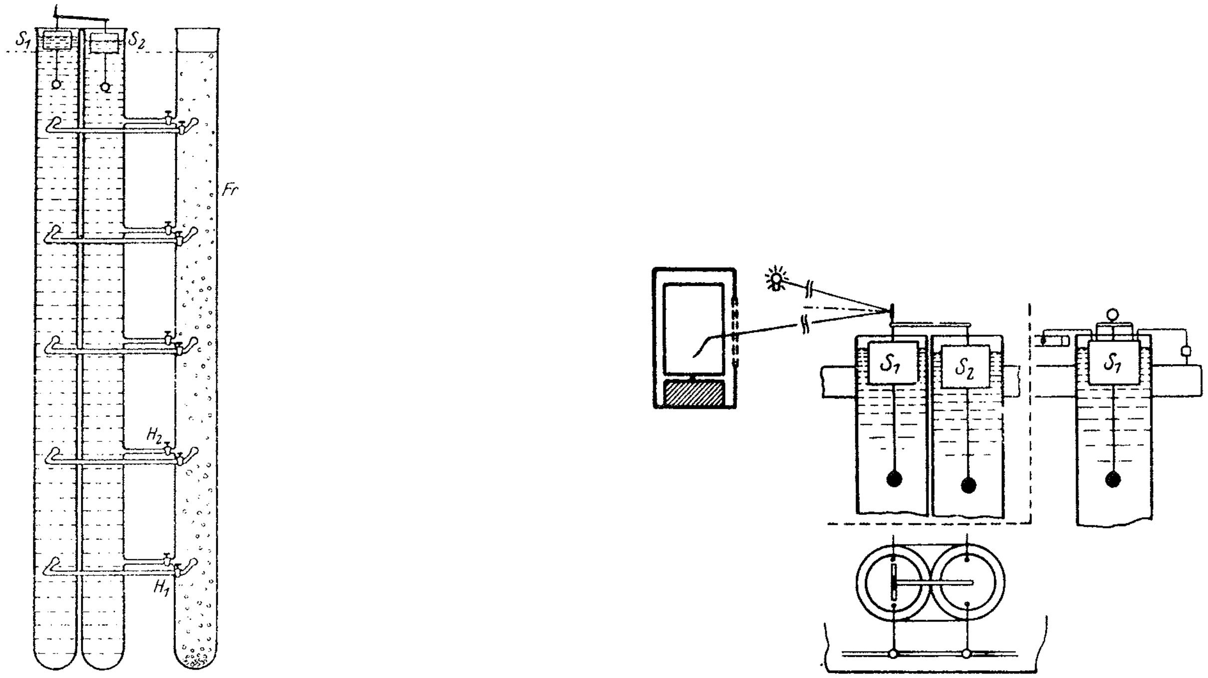
> The manometer _A_ , graduated at the top in millimeters, enlarges into a bulb _C_ which has a small communicating opening at the bottom. At the start, a very small amount of water is drawn up into _C_ so that, as the density decreases at level _D_ in the sedimentation column, water rather than pentane bubbles will be emitted. The suspension is initially in _S_ where it is shaken vigorously before the air pressure from supply _P_ is turned on, whence it is forced into the sedimentation column in a dispersed condition. When the column is full, as evidenced at an outlet _T_ , the valve _N_ is closed and the actual analysis proceeds after proper referencing of the column height to the pentane height in the manometer.

The data requires graphical interpretation, as do other methods with single point density readings.

A radically different type of pressure or density measuring device, illustrated in Fig. 71, was devised at an early date by Ostwald (37). The suspension was poured into an open container _S_ . The air was evacuated in chamber _E_ , to a predetermined pressure. Then, by means of a three-way stop cock the evacuated chamber was connected to the sedimentation column that extended into the suspension in *S* . The suspension was drawn to a certain height and, maintaining the same vacuum in *E* , the height of the sedimentation column was observed at stated intervals as it raised as the material settled into the container below. The accumulation type of curve was obtained. Obviously, difficulties would be encountered from variations in atmospheric pressure if the analysis was of long duration and from variations in pressure in *E* due to slight temperature changes.

A simple apparatus developed by von Hahn is illustrated in Fig. 72. It consisted of an inverted U-tube with the open ends immersed in separate vessels one of which contained the suspension, the other a pure liquid. A vacuum pump lifted the liquids to relative heights proportional to their densities. Then the valve in the upper stem was closed, sedimentation in the suspension column proceeded and the column heights were observed periodically. It is apparent that this device is simpler to operate than Ostwald's, which is designed on the same principle.

30. *Centrifugal methods*--The centrifuge has been employed advantageously for size separation of extremely fine particles. It has been used most extensively in the study of colloidal suspension, and, more recently, in the actual separation of molecules for molecular weight determinations. Ultra-centrifuges with rotors traveling at 135 000 to 160 000 r.p.m. producing intense centrifugal fields exceeding the force of gravity by a million times have been developed for determining weights of molecules.



a. Sedimentation and manometer tubes.

b. Details of automatic recording mechanism.

Fig. 69--Lubirsky's and Kosman's manometer (1927).

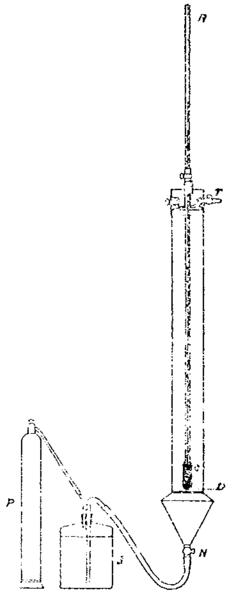


Fig. 70--Oden's internal manometer (1925).

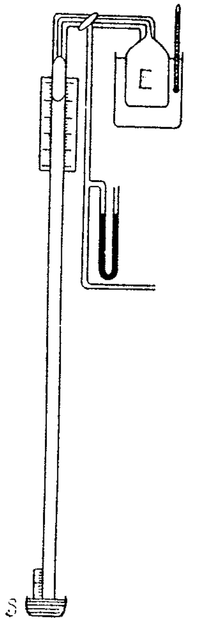


Fig. 71--Ostwald's manometer (1922).

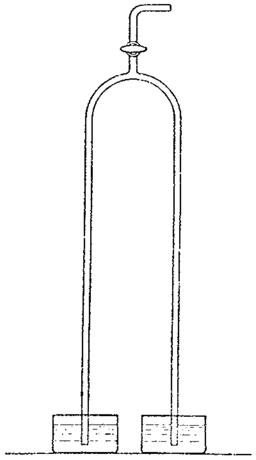


Fig. 72--von Hahn's manometer.

FIGS. 69, 70, 71 & 72

The centrifuge appeared among the early devices used in mechanical analysis of soils. Yoder's apparatus, described previously in Section 22, combining the methods of elutriation and centrifuging, was devised in 1904. About the same time the simple centrifuge, shown in Fig. 73, was developed by the U. S. Department of Agriculture (25) for analysis of soils. A more recent apparatus, shown in Fig. 74, developed primarily for analysis of suspensions with particles smaller than 1.0 micron diameter, was developed by Svedberg and Nichols (103). Its essential parts consisted of a rotar A, directly connected to a special 20 000 r.p.m. motor, and two sedimentation tubes L. A disk N, with a slot directly beneath the sedimentation tubes, allows a beam of light reflected from an inclined mirror P to pass through the suspension and on to a sensitized photographic plate. The density of the suspension is determined qualitatively from the relative density of the negative.

Trask (116) developed a centrifuge especially for analysis of sediments, that operated fairly satisfactorily with silt-clay suspensions. Four graduated sedimentation tubes of 100 cc. volume were whirled at rotor speeds varying from 360 to 2 520 r.p.m. Measurements of the volume of the deposited sediment were made after controlled rotation periods. A rapid technique was developed whereby one man could analyze four samples completely, including the calculation of data, in 2 hours.

At the present time the centrifuge method is not in common use for routine size analysis of soils or sediments. Although it is adapted to rapid analysis of fine material, and, if the equipment is properly designed, allows a number of analyses to be conducted simultaneously, there are several reasons for its not being used more generally.

a. For the ordinary analysis of soils and sediments, separations below 2 microns have not been required and often not below 5 microns. Since there are several other methods more or less satisfactory as to accuracy and time, which require only ordinary laboratory equipment for separations of particles larger than these sizes, there has been no real urgent need for a centrifugal method.

b. The centrifuge method has a number of objectionable inherent features such as the swirling currents in the sedimentation tubes, after rotation has stopped, and the necessity for uniform accelerating and decelerating rates to validate the corrective factor.

c. It has the practical difficulty of obtaining a completely dispersed suspension at the start.

d. Because the sedimentation tube is in rapid motion rather than stationary, the measurement of particle settlement becomes difficult. The methods of accomplishing this will be discussed in the following paragraph.

In conducting an analysis with a centrifuge, the general practice is to start with the sediment completely dispersed in the sedimentation tube. Five different methods have been used for obtaining data which can be expressed in size fractions:

a. Accumulation curve of the Oden type, subject to graphical analysis. Data is secured by stopping the centrifuge at various times and noting the depth of accumulated sediment.

b. Pipette samples. The centrifuge is stopped at certain times during the analysis and a pipette sample is secured from each sedimentation tube.

c. Removal of finer material by elutriation. The entire fraction of a certain size range is secured for weight measurement.

d. Use of special tubes. The tube shown in Fig. 75, devised for heavy mineral separations, allows two fractions to be obtained. During initial dispersion the stopper A closes the opening into D ; during rotation the entrance into D is open allowing the heavy material to settle there. The most important feature of this special tube is that the upper fraction can be poured out without intermixing with that below.

e. Measurement of the turbidity by means of the light absorbing or reflecting properties of the suspensions.

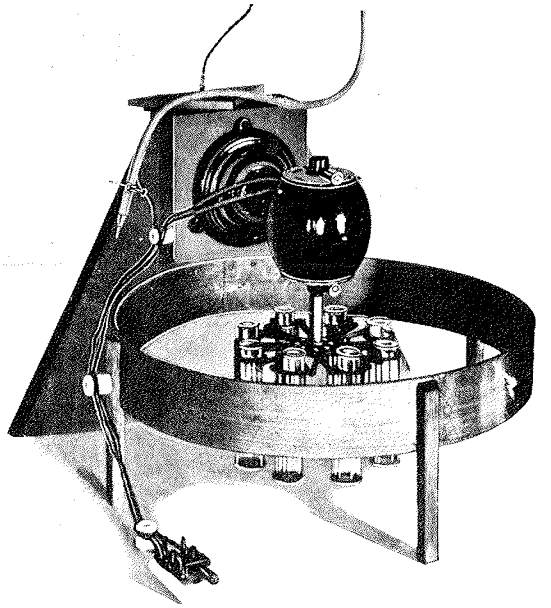


Fig. 73--Early U. S. Department of Agriculture centrifuge (about 1904).

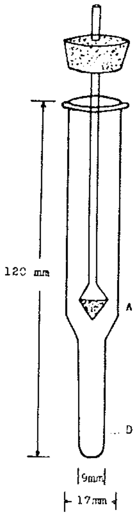


Fig. 75--Taylor's centrifuge tube (1933).

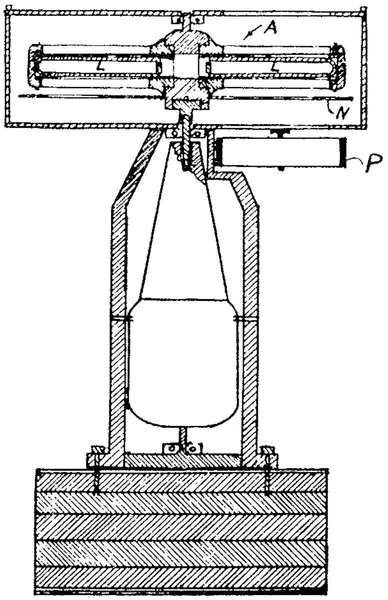


Fig. 74--Svedberg's and Nichols' centrifuge (1923).

A different application of the centrifuge was employed by Westgren (104) in 1915. He placed suspensions of gold sols, a very fine material, in small sedimentation cylinders and centrifuged them until all the solids were deposited at the bottom. Then he placed the cylinders bottom up and allowed the sedimentation to proceed a given length of time after which he turned them 90° to a horizontal position. The material was deposited along the side of the sedimentation cylinder in some order of size corresponding to the fall velocities which provided data for a distribution curve. The errors due to opportunities for diffusion and intermixing were recognized.

In a centrifugal sedimentation system the force inducing sedimentation of a particle is a function of the angular velocity and the mass of the particle. This force, and consequently the settling rate of the particle, is not constant because the angular velocity varies with distance from the axis of rotation. The development of the theory and equations for interpretation of centrifuge data in terms of particle size are presented by Svedberg and Nichols (103).

31. *Optical methods*--The volume of light transmitted or reflected by a given suspension is a function of the concentration, the number, and the properties of the particles suspended in it. Optical measurement of turbidity has generally been done either by passing the light beam directly through the suspension into a photo-electric cell to secure a measure of the amount of light absorbed, or by measuring at right angles to the light beam, the amount of light reflected.

The particularly advantageous features of an optical method are the elimination of disturbances in the sedimentation cylinder and the rapidity and ease of securing the data because there are no samples to dry and weigh. Favorable, also, is the ease of incorporating automatic recording devices without the equipment becoming too complex and too sensitive to normal laboratory disturbances. Kalitin (52) points out the excellent sensitivity of photometric equipment to changes in concentration, and shows that in certain tests he conducted the galvanometer satisfactorily reacted to changes in concentration at the rate of 0.1 gm. per 4500 cc. of water or 22 p.p.m.

Unfortunately, a large number of factors (96) affect the light transmission or reflection in a suspension and the application of this easily executed method has been limited. The more important of these factors are:

<u>a</u>. Refractive index, absorption coefficient, and thickness of the suspending medium.

<u>b</u>. Wave length and condition of the light beam.

<u>c</u>. Number, size, size distribution, and other characteristics, such as the color, transparency, composition, shape, angularity, and nature of the surface of the suspended particles.

With present equipment and technique it is possible to evaluate and control the properties of the suspending medium and light source within practical limits. The effect of the wave length of light becomes important only when the particle diameter approaches it in size. A wave length greater than about 2 microns has no apparent effect. The greatest difficulty in the interpretation of light intensity data ~~lies~~ in relating the effect of number, size, distribution, and inherent characteristics of the particles themselves. In the cement industry, where the properties of the particles can be more or less controlled, the Wagner turbidimeter (132) has been used advantageously for the determination of surface area which, with a given concentration, can be related to particle size. In a similar manner it has been adapted to analysis of paint pigments (102).

Traxler and Baum (117) conducted tests to compare the Wagner turbidimeter and Roller air analyzer, using a wide variety of dispersed material, such as cement, trap rock, silica, limestone, and green, red, and black slate powders. Their conclusion was that there is no direct relation between surface area and turbidity which is common to all these materials, because of the wide differences in the optical properties of the various particles.

Among the first investigators to apply the method to the mechanical analysis of soils was Morison (64) who used it in 1925. The results appeared promising but application of the method to routine studies was impossible because absorption coefficients for different types of particles were not available.

A beam of parallel light which is projected and measured after being reflected through some angle, generally 90°, is commonly known as the Tyndall beam. Special apparatus to accomplish this is known as the Tyndallmeter. Tolman, Ryerson, and others (114), using a suspension of silica ground fine enough to pass a 200-mesh sieve, obtained Tyndall readings in direct proportion to concentration up to nearly 0.2 per cent concentration. With greater concentration the increased opaqueness caused appreciable absorption of light. The Tyndallmeter was sensitive to concentrations of less than 0.01 per cent, suspensions which appeared clear to the naked eye. However, this excellent correlation was obtained with material identical in gradation and optical properties. The investigators appreciated that particle size would have an effect and promoted further studies to determine its importance.

The study of the effect of particle size on the intensity of the Tyndall beam showed a definite relationship. With a given concentration, the intensity of the beam or reflected light increased as particle size decreased. The test material used was ground silica in suspensions of 0.15 to 0.01 per cent concentration, and particle size ranging from about 1 to 20 microns. For a single type of material very satisfactory correlation of particle size and concentration was secured.

Stamm and Svedberg (100) devised equipment and technique for measurement of the light scattering property of suspensions. Their equipment consisted of a small tubular container of suspension located between two opposing sources of light. Opposite the sedimentation tube, and at right angles to the light beams, was a narrow slit of the same height as the sedimentation tube through which the reflected light could pass onto a photographic plate. At the start and at selected times during the sedimentation period exposures were made on individual photographic plates, securing a record of the change in density with time. A photometer was utilized in determining density from the negatives.

Suspensions of 1.0 per cent concentration were ordinarily used by Stamm and Svedberg with particles varying in size from about 0.5 to 10 microns. The settling of the particles was considered to follow Stokes' law. For the size range of material used, the tests showed that the light scattered to a degree proportional to the surface area and the number of particles. Microscopic measurements appeared to verify this conclusion

However, Cheveneau and Audubert had previously shown (84) that for fine material, ranging in size from 1 to 12 microns, the absorption is proportional to the diameters rather than to surface area, and independent of color. For still finer material, colloidal in nature, Zeorell (84) had found that the light absorption was independent of the size but strongly dependent upon the color.

Richardson developed a photo-electric apparatus (84) for recording change in light absorption of particles greater than colloidal size and determined that for particles between 1 and 12 microns diameter the absorption of light varied as the summation of the diameters of the particles, and that for particles greater than 12 microns the absorption was proportional to the total surface area. The unique feature of his device was a specially shaped drum to obtain directly a logarithmic record of the light intensity. Greater sensitivity was obtained during the initial period where the rate of change of turbidity is most rapid.

The U. S. Engineer Office, Kansas City, Missouri, incorporated a turbidity determination in the laboratory procedure for sediment investigations in the Missouri River (121). The sample was poured into a graduate, where its volume was determined, and then it was shaken thoroughly. When the sediment was completely dispersed a turbidity determination was made. The true concentration, expressed in parts per million, obtained by the ordinary sedimentation-decantation and evaporation method, was divided by this turbidity value to give a figure that was termed the "coefficient of fineness."

Since turbidity varies approximately as the square of the diameter for particles greater than 0.01 mm. a difference in concentration, indicated by the sedimentation and evaporation method, in samples of equal turbidity necessarily means that the particles in the sample of lower concentration are of a smaller mean size. Conversely, in two samples of equal concentration but with different turbidity readings, the sample of greater turbidity must contain the finer particles.

The main purpose in obtaining turbidity readings of all suspended sediment samples in the Kansas City Engineer District and in determining a fineness factor was to allow interpretation of old municipal waterworks records where only turbidity values were available. Nevertheless, this instance indicates that this method could be incorporated into standard laboratory procedure to check the results of regular analyses and possibly to eliminate a portion of them if a reliable correlation is obtained.

32. Miscellaneous--Prof. E. W. Lane has suggested a sedimentation apparatus consisting of a sedimentation column and a series of slides, spaced at unequal vertical intervals, dependent upon the size limits desired for individual fractions, that can be inserted at will across the column. The slides would be inserted at specified times, starting with the lowest slide. Finally the sedimentation column would be divided into a series of compartments, each having a slide at the base to catch a certain size fraction. The slides could be screens or very shallow porous crucibles, depending on the size of particles, which would allow the water to be drawn from the column if necessary with the aid of an aspirator. When the column was entirely drained the individual screens or crucibles could be removed and placed in an oven for drying. The principle of this device is similar to that of Apollov (Section 43) but it was originated independently.

The advantages of the apparatus and the opportunities for automatic control are apparent. A possible difficulty is the development of a satisfactory method for introducing the water-sediment mixture at the top of the sedimentation column. A complete dispersion throughout the column would probably not be satisfactory because of the difficulties in calculation and because of the greater significance of errors in technique since only portions of each size fraction would be secured. A study of the practicability of this device is proposed.

In addition to the methods previously presented that have had varying degrees of success and popularity in size analysis of particles in the microscopic range, there are a few other methods which are more elaborate but less commonly used. They have been developed largely for analysis of particles of colloidal size and are probably of little practical value at the present for size analysis of sediment samples.

Svedberg (104) discusses the X-ray method with respect to colloidal size analysis and states that it appears to be one of the most promising optical methods for studying the structure and size of particles. Aborn and Davidson (1) investigated the application of this method to particles of microscopic size. Studying silica powders with grades of mean particle size varying from 0.0017 to 0.036 mm. they concluded that:

> _a_. The size distribution had an appreciable effect upon the X-ray pattern.
>
> _b_. An estimate of average particle size would be erroneous unless the distribution is known. Also they could find no quantitative relationship between the X-ray pattern and the size distribution.

The use of filters of very fine pores to determine the size of colloidal particles is described by Svedberg (104). Since ordinary sized colloids will pass through porcelain filters with openings of 0.16 microns diameter, it is necessary to impregnate the filter with collodian or a gelatin solution of different concentration. Pore sizes were determined by means of air flow. The method has not been generally satisfactory because there are a number of factors difficult to control.

Another method for size separation of extremely fine material is to use an alternating electric field such as developed by Burton and Ried (26). Under certain conditions particles that are extremely small will not settle in a given fluid by gravity, but move continuously to and from with Brownian movement. When an electric field is set up the colloidal particles will move toward the cathode or anode, depending upon their charge. If the electrodes are located in a vertical plane, one above the other, and the current is flowing first in one direction and then in the other for the same duration of time, the difference between up and down movement of the particles will be due to the force of gravity, thus allowing an application of Stokes' law.

Since the hydrometer and pipette size analysis methods have become so popular in the last few years, it is of interest to learn of the recent development by Hellman and McKelvey (145) of an apparatus which combines both methods in an instrument similar to the ordinary storage battery hydrometer. It was designed to provide a method comparing in speed with the hydrometer analysis and yet incorporating the theoretical soundness of the pipette.

The apparatus consists of a hydrometer inclosed within the barrel of a pipette. The hydrometer is graduated in grams per liter (67° F., sp. gr. 2.65) in half gram divisions from -2 to 30 grams per liter. It is approximately 19 cm. in over-all length; the bulb is 1.4 cm. in diameter. The barrel of the pipette is approximately 28 cm. in length and has an inside diameter of 1.9 cm. to allow complete freedom of movement of the hydrometer. The stem of the pipette is 15 cm. in length and is graduated in half centimeters linear distance measured from the tip. Both the hydrometer and the pipette are made of pyrex glass. The volume of sample necessary to float the hydrometer at its maximum immersion is approximately 35 cu. cm.

This design permits the taking of a sample according to the accepted pipette method, the only difference being that the sediment content is read directly in grams on the inclosed hydrometer instead of drying and weighing the sample.

It is claimed that this method has certain advantages over the straight hydrometer method, namely:

> _a_. The density of the suspension is measured at a point to which particles of a given size will settle in a given time.
>
> _b_. Little or no disturbance is created by the entrance of the stem of the pipette into the suspension.
>
> _c_. The sampling time for any grade size may be computed directly from Stokes' law and does not need to be determined empirically as in the hydrometer method.

One disadvantage as compared with the hydrometer method is the fact that the time required is greater. As an example, size analysis can be made down to two microns by the hydrometer in two hours. A similar analysis by the hydrometer-pipette method requires eight hours when sampled at a depth of 10 cm. This disadvantage is offset somewhat by the fact that it does not require any more of the analyst's time.

In addition, certain advantages are cited over the straight pipette method as follows:

a. The time-consuming weighing and handling of beakers and aliquots is eliminated.

b. The results may be computed while the analysis is in progress. It is thus possible to determine how far it is necessary to carry the analysis to attain a good coverage of the size distribution of the particles.

c. Less steps are involved in handling the aliquots, therefore there is less opportunity for personal errors.

d. Less equipment is necessary (assuming that many analyses are to be run at once).

The quantity of sediment used by the authors in testing the apparatus was from 20 to 30 gm. dispersed as in the straight hydrometer method. The material coarser than silts was decanted, dry or wet sieved, and weighed.

This hydrometer-pipette method probably is a valuable addition to methods of size analysis. It is simple, fast, susceptible to the application of quantity production methods, and apparently accurate at a concentration of 30 grams per liter (3 per cent). The application to lower concentrations of suspended sediments has not been investigated.

The use of sieves in the analysis of suspended sediment is not yet entirely satisfactory. For example, a definite correlation between the grain size determined by means of sieves and the sizes indicated by settling rates has not been developed. Such data as available indicate that sizes determined by the two methods are in close agreement but additional substantiating information is desirable.

The sieving standards of the American Society for Testing Materials were developed principally for purposes other than sediment studies and are therefore probably not the best which could be developed for this field. Until better standards are developed, however, it is believed advisable to follow them, except in cases where there are definite reasons for doing otherwise. Experienced technicians will no doubt find cases where improvements can be made, but those with less experience will probably secure the best results if they are followed closely.

IX. SUMMARY OF PART TWO

33. Summary--An extensive review of methods of particle size analysis of fine-grained sediments has been presented, including a discussion of their individual features. In routine analyses, sedimentation in water has been used almost exclusively as the means of separating particles into size fractions, interpretating the fall velocities in terms of particle size by means of Stokes' law.

In the design of sedimentation equipment for size analysis of fine-grained sediments the following general limits should be considered:

a.	Minimum diameter of column	2 cm.
b.	Maximum concentration of suspended sediment	2 per cent
c.	Temperature control to within	0.5°
d.	Native or distilled water for suspending medium.	

The advantages and disadvantages of each main size analysis method are summarized in Table 4. In addition there have been a number of developments in individual techniques, procedure, and equipment that deserve emphasis:

a. Decantation method--The Russian development of a multiple automatic decantation apparatus, Fig. 15.

b. Pipette method--(1) Andreason's apparatus with a thermally insulated cabinet for conducting four analyses simultaneously, Fig. 51, and, (2) Rittenhouse's "mass production" methods.

c. Hydrometer--(1) Puri's adaptation of the chainomatic analytical balance to the hydrometer to secure accurate weight measurements, Fig. 56, (2) Puri's laboratory hydrometer, Fig. 54, permitting more accurate readings of volume, (3) Puri's field hydrometer, Fig. 55, permitting change in weight of hydrometer so that, with different temperatures, depth of immersion can be maintained approximately constant, (4) Bouyoucos' sensitive hydrometer for suspensions of 0 to 10 gm./liter.

d. Manometer--Oden's internal manometer, Fig. 70, using pentane, a liquid of lower specific gravity than water.

TABLE 4

SUMMARY DISCUSSION OF PRESENT METHODS OF SIZE ANALYSIS FOR FINE-GRAINED SEDIMENTS

Method	Advantageous features	Disadvantageous features
Decantation	1. Simple equipment; available in most laboratories. 2. Laboratory technique relatively simple. 3. Total amount of each size fraction secured making this method advantageous to low concentrations. 4. Analysis of data simple because of total fraction being secured. 5. Accuracy can be controlled by the number of decantations, making this method adaptable to various types of investigations.	1. Laborious unless automatic equipment is used to accomplish the decantations, (minimum 3 or 4), that are necessary. 2. With numerous changes of suspending medium the opportunities for flocculation are greater. 3. Requires considerable native or distilled water. 4. Temperature control difficult with ordinary equipment - requires frequent temperature measurements and a flexible time scale.
Elutriation (water)	1. With multi-cylinder elutriators separators proceed more or less automatically. 2. Analysis of data simple because of total fractions being secured. 3. Does not require an initial uniform dispersion throughout the sedimentation tube. 4. Adaptable to separation of large quantities of material.	1. Difficult to obtain and maintain uniform velocity over cross section. 2. Separation of very fine particles difficult due to the difficulties of controlling low inflow rates. 3. Opportunity for flocculation due to a continually changing suspending medium. 4. Requires a relatively large amount of native or distilled water which may be costly or difficult to acquire. 5. Requires the maintenance of a constant temperature of system and of inflowing water.
Elutriation (air)	1. Rapid separation of very fine material. 2. Fractions secured directly in dry form and weight can be determined directly. 3. No flocculation problems.	1. Requires very careful control of velocities, inflow rates, and distribution over cross section. 2. Material must be in a dry, powdered form, existing as individual particles. 3. Requires special equipment.
Accumulative sedimentation	1. Analysis proceeds automatically. 2. Utilizes the sensitivity of the analytical balance. 3. Adaptable to automatic recording equipment. 4. Adaptable to relatively low concentrations. 5. Adaptable to a relatively wide range of particle size.	1. Present technique results in an accumulative weight curve that requires a graphical interpretation by tangents which is not very accurate. 2. Does not give an accurate size distribution curve because of effects of pan. 3. Requires elaborate, costly weighing and recording equipment that cannot be used to conduct more than one analysis at a time.
Fractional sedimentation	1. Total size fraction secured making method favorable to low concentrations. 2. Eliminates the objectionable graphical analysis necessary with accumulative sedimentation method. 3. Adaptable to a wide range of particle sizes. 4. Adaptable to relatively simple automatic apparatus. 5. Flexible in allowing shortening of sedimentation column and therefore securing advantageous settling times with very fine material.	1. Requires the introduction of the material at the top of the sedimentation column, a technique that has not been satisfactorily perfected for fine material. 2. Practical difficulties in collection of fractions for weight measurements so as not to disturb the sedimentation column.
Pipette	1. Laboratory technique rapid and relatively simple. 2. Simple equipment may be used; available in most laboratories. 3. Accuracy and reliability have been definitely established for use with moderate concentrations (1-2%). 4. Character of data is such that direct interpretation into size grades is possible. 5. Less disturbance to sedimentation column than either hydrometer or plummet. 6. Adaptable to mass analyses and the use of automatic equipment. 7. Flexible in allowing shortening of sedimentation column and therefore securing advantageous settling times with very fine material.	1. Small sample, (10-20 cc generally), requires careful and accurate laboratory technique because of a magnification of errors to the ratio of the volume of the sedimentation column to the volume of the pipette sample. 2. Its use with low concentrations is questionable because of the very small sample secured. 3. Requires a very uniform initial dispersion because of the sampling occurring more or less at a point. 4. It withdraws a sample from a somewhat spherical area rather than a very thin layer over the entire cross section.

Hydrometer	1. Laboratory technique rapid and simple. 2. Simple equipment, available in most laboratories. 3. Accuracy and reliability have been definitely established for certain types of analyses. 4. Character of data is such that direct interpretation into size grades is possible. 5. Adaptable to mass analyses. 6. If accurate weight measurements are used there are possibilities for development for use with low concentrations.	1. Ordinary hydrometer not adapted to very accurate work with low concentrations largely because of difficulty in reading immersed volume accurately. 2. Change in weight of hydrometer due to particles settling on shoulders of bulb introduces errors. If the procedure is to have hydrometer in suspension only during reading, the disturbance in removing and immersing is objectionable. 3. If hydrometer is left immersed the decrease in cross-sectional area results in an increased density, tending to give higher density readings and therefore an abnormally high percentage of small particles. 4. Bulb extends over a portion of the column and therefore secures an average value of density rather than density at a certain elevation. 5. Ordinary hydrometer sinks as density decreases resulting in a measurement of density over a changing height of column.
Plummet	1. It lends itself readily to weight determinations by analytical balance; more so than the hydrometer. 2. Laboratory technique rapid and relatively simple. 3. Equipment fairly simple unless automatic weighing and recording devices are incorporated. 4. Sensitive and appears adaptable to low concentrations. 5. Character of data such that direct interpretation into size grades is possible. 6. Adaptable to mass analyses. 7. Has advantage over hydrometer in being of smaller volume and introducing less disturbance. 8. Calculations simpler than with hydrometer because of no change in immersed volume or significant change of position in column.	1. Change in weight of plummet due to particles settling on shoulders must be considered. If procedure is to have plummet in suspension only during reading, the disturbance in removing and immersing is objectionable. 2. If plummet is left immersed the decrease in cross-sectional area results in an increased density, tending to give higher density readings and therefore an abnormally high percentage of small particles. 3. Bulb extends over a portion of the column and therefore secures an average value of density rather than density at a certain elevation.
Manometer	1. Laboratory technique relatively simple. 2. Adaptable to either simple or complex equipment depending upon the accuracy and extent of automatic control desired. 3. Readily adaptable to automatic recording equipment. 4. Adaptable to mass analyses.	1. Not accurate for direct visual readings of vertical tube. Accuracy increased when an inclined tube is used. 2. Errors due to parallax when direct visual readings are made. 3. Tendency for intermixing and currents in sedimentation columns at start due to difference in densities in column and manometer. 4. As the density in the sedimentation column decreases a flow of liquid from manometer into column occurs, producing cross currents tending to affect particle fall. 5. Single manometer opening produces Oden type curve, (a depletion rather than an accumulative curve, however), that necessitates a graphical interpretation. 6. Two interconnected manometer openings near base of column produces data for direct interpretation but this decreases sensitivity. 7. Lag in meniscus when rapid changes occur in density.
Centrifugal	1. Allows rapid separation of very fine material. 2. Allows a number of separations to be conducted simultaneously.	1. Practical difficulties in securing weight measurements of the settled material during the analysis since volume measurements are not considered satisfactory with fine particles. 2. Swirling currents in the sedimentation tube after centrifuging has stopped. 3. Requires special equipment.
Optical	1. No disturbance to sedimentation column. 2. Minimum of laboratory work because of no samples to dry and weigh. 3. Extremely sensitive. 4. Readily adaptable to automatic recording equipment. 5. Adaptable to mass analyses.	1. Light transmission and reflection are functions of concentration as well as particle size, complicating the interpretation. 2. Light transmission and reflection are affected by the optical properties of the suspended material - such as color, shape, surface condition. 3. Requires special equipment.

The concentration of the suspension as well as the range of particle sizes present, will influence the selection of the type of sedimentation method that should be used. When a relatively unlimited supply of the material to be analyzed is available, the concentration may be adjusted at will so as to permit the use of a variety of methods. Much more difficult is the adaption of the present sedimentation methods to analysis of suspensions of low concentration that cannot be controlled as, for example, suspended sediment samples. Based upon this study the individual existing methods most applicable to analysis of low and high concentrations, listed in the approximate order of their extensiveness of use, are as follows:

<u>a</u>. <u>Low concentrations</u> (less than 0.5 per cent by weight)

(1) Microscopic.
(2) Decantation.
(3) Pipette; has been used considerably but doubt is expressed as to reliability with very low concentration samples.
(4) Hydrometer; if weight measurements rather than volume readings are determined.
(5) Plummet; if weights are secured with the accuracy of the analytical balance.
(6) Optical; reliability with sediment samples not determined.
(7) Fractional sedimentation; if a satisfactory introduction of fine material could be developed.

<u>b</u>. <u>High concentrations</u> (greater than 0.5 per cent by weight)

(1) Pipette.
(2) Hydrometer.
(3) Manometer.
(4) Plummet.
(5) Elutriation.
(6) Accumulative sedimentation.
(7) Decantation.
(8) Centrifugal.

The general conclusion is that, although there are several satisfactory methods available for high concentrations, there is no rapid, reliable method available for "mass production" analysis of suspended sediment samples of low concentration. Microscopic methods may be reliable and accurate but are generally too laborious. The pipette method is rapid but becomes of doubtful reliability as the concentration becomes very low. Probably the most satisfactory method available, at present, for low concentrations is the decantation method, but it has the disadvantages of being laborious and requiring large quantities of native or distilled water.

The present problem in sediment analysis is to refine existing methods and to develop technique and equipment especially suited to suspensions of low concentration. It appears that the most promising lines to pursue are:

> _a_. Development of equipment and technique for the hydrometer or plummet methods to permit weight measurements with the accuracy of the analytical balance.
>
> _b_. Development of the decantation technique and equipment to allow mass analysis.
>
> _c_. Development of equipment and technique allowing all the material to fall from the top of the sedimentation column. Not only would this eliminate the objectionable Oden type accumulative curve, but it would permit also the development of apparatus and technique for obtaining size groups by individual fractions. Such an apparatus has been described in Section 32.
>
> _d_. Determination of the range of variation in optical properties of sediment particles and a study of the possibilities for adapting optical methods to routine analysis.

PART THREE

METHODS OF PARTICLE SIZE ANALYSIS FOR COARSE-GRAINED SEDIMENTS

X. DIRECT MEASUREMENT

34. General--The division between fine and coarse-grained sediments comes at about 1/16 mm. size. Such a division has proved advantageous in a discussion of size determination methods because a different type is applicable to each. Whereas, for the fine-grained material, sedimentation methods are utilized practically entirely, the coarse material lends itself more readily to either direct physical measurement or to separation by sieves. Recently, however, attempts have been made to utilize sedimentation methods for the finer of the coarse-grained sediments.

35. Direct methods--Krumbein (60) makes a division of coarse material at 16 mm.; the particles smaller being pebbles and sands that can be analyzed readily on standard sieves, the particles larger being of sufficient size to allow a direct measurement of diameter and volume.

The direct measurement of the material coarser than 16 mm., and often somewhat below that size, although sieving in that range is more common, consists of the determination of one or more of three diameters:

> a. Nominal diameter--The volume of each individual particle is obtained by its displacement in water. The nominal diameter, then, is the diameter of a sphere of that volume.
>
> b. Mean diameter--The longest, intermediate, and shortest diameters are determined by means of some type of caliper. The mean diameter is the average of these three dimensions.
>
> c. Intermediate diameter--The mean of the longest and shortest diameters is termed the intermediate diameter.

For particles somewhat smaller than 16 mm., measurement of diameters or projected area may be accomplished through magnification, either by direct readings through an eyepiece, or by measurement of projections on a screen or photomicrographs. When the quantity of material in a sample is not large enough to make advisable a sieve analysis and the particles too small to allow direct measurement, microscopic magnification and measurement is the most direct method available.

XI. SIEVE ANALYSIS

36. Standard sieves--Advancements in manufacturing methods, in conjunction with rigid American Society for Testing Materials (A.S.T.M.) specifications (13), have resulted in accurate, reliable sieves down to the 200-mesh size with openings of 0.074 mm. Some doubt has been expressed as to the accuracy and reliability of smaller openings, especially after considerable use.

The advantages to the analysis and graphical presentation of data, of varying the sieve sizes in a geometric series has been recognized, and in the A.S.T.M. standard sieve series they are varied as $\sqrt[4]{2}$. Table 5

TABLE 5

U. S. STANDARD SIEVE SERIES

From A.S.T.M. Designation E11-38T, Specifications for Sieves (13)

Sieve number (mesh)	Opening		Sieve number (mesh)	Opening	
	mm.	in.		mm.	in.
3 1/2	5.66	0.223	40	0.42	0.0165
4	4.76	0.187	45	0.35	0.0138
5	4.00	0.157	50	0.297	0.0117
6	3.36	0.132	60	0.250	0.0098
7	2.83	0.111	70	0.210	0.0083
8	2.38	0.0937	80	0.177	0.0070
10	2.00	0.0787	100	0.149	0.0059
12	1.68	0.0661	120	0.125	0.0049
14	1.41	0.0555	140	0.105	0.0041
16	1.19	0.0469	170	0.088	0.0035
18	1.00	0.0394	200	0.074	0.0029
20	0.84	0.0331	230	0.062	0.0024
25	0.71	0.0280	270	0.053	0.0021
30	0.59	0.0232	325	0.044	0.0017
35	0.50	0.0197	400	0.037	0.0015

presents the size of openings in the A.S.T.M. standard series. Although there is a trend toward the use of round apertures in preference to the square openings formed by woven wire, the latter type of sieve generally is considered standard.

37. Technique--In order to insure comparable data by sieve analysis, it is necessary that certain conditions or standards be determined, such as the following:

a. Size of test sample.
b. Type of sieving, wet or dry.
c. Order of sieves.
d. Method of shaking.
e. Duration of sieving or end point.

The size of sample, according to Weinig (134), often will depend upon the diameter of the coarsest particles; the larger the particle the larger the sample must be. On the other hand, if the material is relatively fine, smaller samples may be taken. A greater depth of sand necessarily requires a longer sieving time but it also increases the efficiency of sieving because the elongated grains have a better chance to become oriented with the aperture of the sieve. Concerning the total time of sieving, Weinig states that 30 or 40 min. generally is required, exclusive of additional time to check the efficiency.

Material may be sieved either wet or dry. Wet sieving has the advantage of cleaning the material if it contains silt and clay particles. Often it eliminates the need for a shaking device. When used with the finer sands, however, any advantage secured through the cleaning action is overbalanced by the disadvantage that the water film on the screen and particles does not allow a complete separation. The procedure has often been to wet sieve for cleaning and then resieve after drying.

The procedure of dry sieving as set forth by A.S.T.M. (10) is:

> "The sieving operation shall be conducted by means of a lateral and vertical motion of the sieve, accompanied by a jarring action so as to keep the sample moving continuously over the surface of the sieve. In no case shall fragments in the sample be turned or manipulated through the sieve by hand. Sieving shall be continued until not more than 1 per cent by weight of the residue passes any sieve during 1 min. . . . .
> When sieving machines are used, their thoroughness of sieving shall be tested by using the hand method of sieving as described above."

There are three methods used to determine the end point in a sieving process:

a. By a standard sieving time.

b. By specifying that the weight of material passing in a given time must be less than a certain per cent of the gross initial sample.

c. By specifying that the weight of material passing in a given time must be less than a certain per cent of the weight of the residue on the sieve considered.

The first mentioned, which obviously is the simplest, is usually considered satisfactory if the material does not vary too greatly in fineness. A sieving time of 15 min. has been somewhat common. Methods b and c are more difficult to apply in most routine work as they tend to make the procedure laborious. Their value probably lies in serving as standards in the establishment of a standard sieving time for a certain range and type of material that is subject to routine tests.

38. Accuracy--As the elongation and angularity of the particles become greater the diameters secured from a sieve analysis become more of a misconception. This condition is a definite consideration in sedimentation studies because a sedimentation diameter is nearly always more

important than a physical measurement secured directly by calipers or indirectly by sieve apertures of known size.

In addition to the shape of particle affecting the validity of sieve analyses, there are the following sources of error that may or may not be serious, depending upon the equipment available and the refinement of the technique:

a. Incomplete sieving.
b. Faulty manipulation.
c. Worn or damaged screens.
d. Inherent errors in mesh.
e. Weighing.

The first is generally the most serious because it is not practical to sieve a sufficient length of time to accomplish a complete grade separation. With the numerous factors affecting the efficiency of sieving, such as size of particles, depth on sieve, and shape of particles, it becomes difficult to secure comparable data of more than fair accuracy.

The errors entering a sieve analysis through faulty manipulation are introduced largely in the removal of the material from a sieve. Angular particles always have the tendency to become wedged in the mesh. A brush can be used advantageously to facilitate the removal of a large portion of this wedged material. Regardless of how the retained material is removed, extreme care should be exercised to avoid damage to the mesh.

Screens should be checked occasionally against new standard sieves to determine whether they have been worn or damaged to the extent that incomparable data are being secured. The necessity for checking undoubtedly becomes more important with the finer mesh sieves. The finer meshes are often checked for wear or damage by microscopic examination.

Rigid A.S.T.M. specifications have resulted in excellent U. S.

Standard sieves of 200 mesh and larger and inherent errors in the mesh of these sizes are seldom important enough to consider.

Errors in weighing, other than accidental, can be controlled by using a balance of the desired sensitivity.

XII. METHODS BASED ON SETTLING RATES

39. Adaptability to coarse material--Methods for the size determination of coarse granular material based on settling rates are not common. The sieve analysis method, being effective, convenient, and generally reliable, has dominated this field to such an extent that there has been no real incentive for development of new methods. In addition, the development of settling rate methods has been discouraged because the fall velocities of the large particles are high and necessitate rather tall columns, if water is used as the settling medium. Also, the separation occurs in a very short time period, which restricts very greatly the number of settling rate methods that can be used because of the rapidity necessary in making observations.

The relation between the fluid properties, the particle dimensions, and the fall velocity under gravity have been presented in Part One of this report. Graphical solutions expressing the fall velocities for corresponding diameters of coarse particles are given in Fig. 5. A solution taking into account particle shape is presented in Fig. 6.

40. Optical lever siltometer--Probably the first device employing the settling rate principle for separation of coarse material into size grades was that developed in India, in 1933, by Vaidhianathan (124). The apparatus, shown in Fig. 76, embodies the principle of the manometer and includes an automatic recording device. Vaidhianathan termed it an "optical lever siltometer." Mercury was used as the manometer fluid, probably because it has the property of not wetting glass and thus provides a convex meniscus with a definite, measurable height. A small

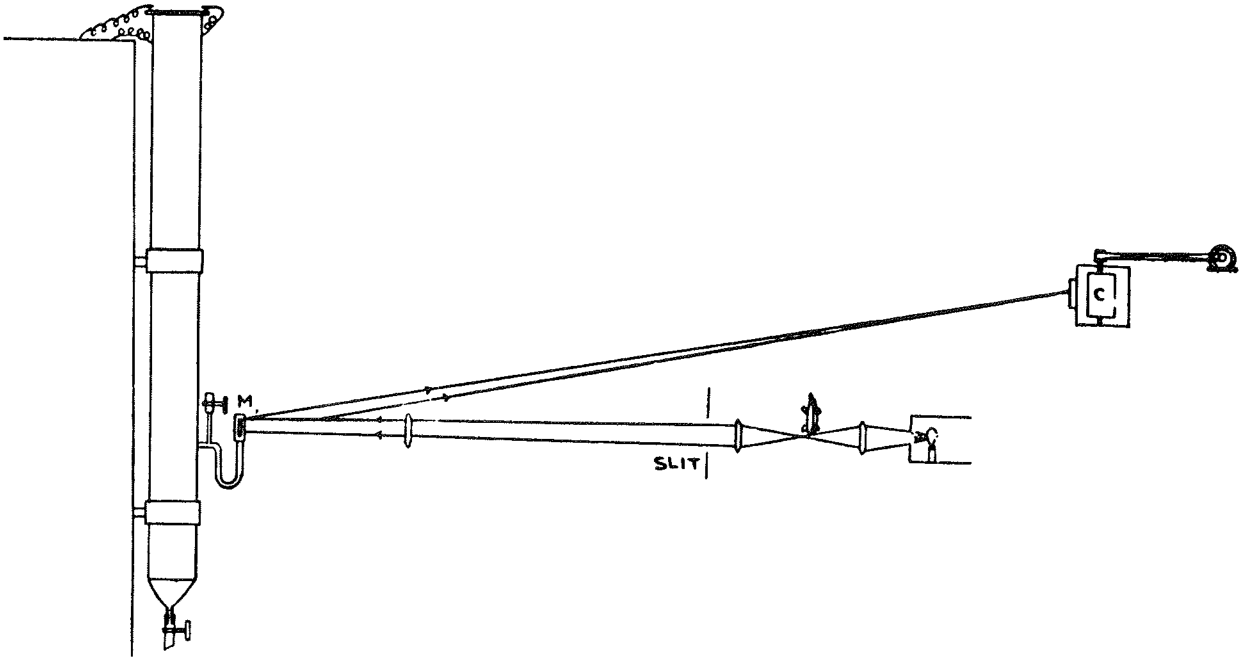


Fig. 76--Vaidhianathan's "optical lever siltometer" for coarse sediments (1933).

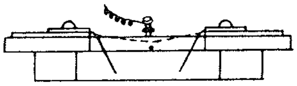


Fig. 76a--Details of sediment dropping mechanism.

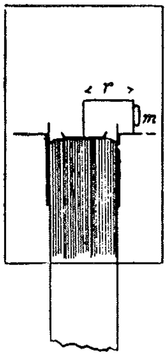


Fig. 76b--Details of float-mirror device actuated with change in manometer column height.

FIGS 76, 76a & 76b

mirror m, shown in Fig. 76b, was attached to a small frame r, one leg of which would follow the movement of the manometer column. A beam from a constant source of light was reflected by the mirror through a lens and upon photographic paper on a revolving drum c, shown in Fig. 76. Movement of the mirror, caused by a decrease in density of the settling medium and, consequently, a fall of the mercury column, would be recorded on the sensitized paper on a greatly enlarged scale. The sediment was introduced as a unit by the door-like device shown in Fig. 76a. The apparatus was designed for a size range from about 0.07 to 0.30 mm. Although the device appeared reasonably satisfactory it has objectionable features relative to its use in routine sediment analysis. The use of mercury in the manometer instead of water decreases the relative change in density over thirteen times; a seemingly unnecessary decrease in sensitivity that must be regained through the delicate mirror-float arrangement. This necessitates a mechanism so delicate that special mountings for the apparatus are necessary to eliminate effects of vibrations produced in the ordinary laboratory.

41. Siltometer--A year later Puri (79) developed a more rugged, but less delicate apparatus, termed a "siltometer." Fig. 77 shows the essential features which consist of four main parts:

> a. A sedimentation tube made of brass tubing, about 6 cm. inside diameter, 200 cm. long, is attached to lever B so that by actuating this lever the entire tube can be raised a distance of about 1 cm.
>
> b. The sediment dropping device A, made of two funnels, is used to introduce the sediment under water. The funnels fit tightly together allowing the water-sediment to be introduced without entering the sedimentation tube. At the desired time the inside funnel is lifted and the sediment enters the main tube.

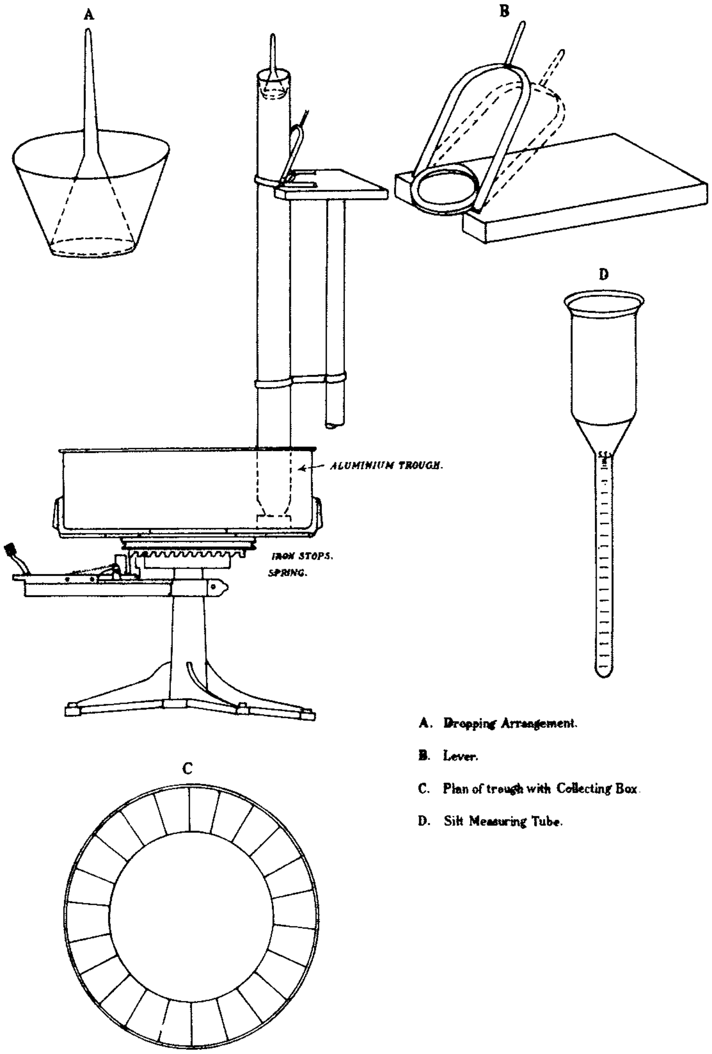


Fig. 77--Puri's "siltometer" for coarse sediments (1934).

FIG. 77

c. The sediment collecting device consists of a circular aluminum trough C, divided at the circumference into twenty individual pockets. The apparatus is so constructed that this collection trough can be moved 1/20th of its circumference by operating a lever manually at any desired time.

d. The container D, with a slender, graduated stem, is used when a volumetric measurement of the sediment collected in each pocket is desired.

The procedure in conducting an analysis is as follows: The circular trough is filled with water to a depth of 3 to 4 in. A rubber stopper is placed in the pocket immediately below the sedimentation tube and the tube is lowered by lever B, so that the stopper fits tightly in the end of the tube. The sedimentation tube is then filled with water and the inner funnel is inserted snugly. A sample of about 10 gm. of sediment is placed in the dropping device and covered with water. The actual separation into fractions by size is ready to proceed. The inverted funnel is removed quickly and a stop watch is started at the same instant. The remaining funnel section is removed as rapidly as possible and a rubber stopper inserted tightly into the top of the sedimentation tube. By actuating lever B, the tube is raised sufficiently to remove the lower stopper. The sedimentation into this first cup is allowed to continue until a period of 26 sec. has elapsed; at this time the trough is rotated one division, placing the second cup in position. Rotation of the trough continues at 4-sec. intervals up to 66 sec., then at 10-sec. intervals up to 106 sec., and then after 136, 166, 196, 376, and 556 sec. At the completion of the separation, which consumes less than 10 min., the material in each compartment is either filtered, dried, and weighed, or washed into the measurement container D, for a volumetric determination.

When operated as described above this apparatus produces an extremely large number of fractions which would have the following limits at 20° C.:

Fractions No.	Size range of particles mm.	Duration of each sample sec.
1 to 10	0.562 - 0.249	4
11 to 15	0.249 - 0.174	10
16 to 18	0.174 - 0.116	30
19	0.116 - 0.080	180
20	0.080 - 0.065	180

The standard technique is adaptable only to sediments with particle sizes between about 0.6 and 0.06 mm. It has been used principally in analyzing bed sediments. No adaptation to finer material has been mentioned although an application appears practically possible down to at least 20 microns, with a settling time of 1.5 hr. for a fall of 200 cm. at 20° C., and probably even to 10 microns, with a settling time of 6 hrs. However, the practical difficulty of introducing the sample in such a manner that it does not fall as a mass, a tendency that increases with decreased particle size, and the limited number of samples that could be analyzed per day, place certain restrictions upon the adaptation of the "siltometer" to the smaller sizes of sediment. It is possible to use it with somewhat larger material, but errors will increase because initial disturbances become of relatively greater importance. It probably could be used with particles up to 1.0 mm. diameter, with a settling time of about 12 sec., and even up to 2.0 mm., settling time of 7 sec., with some degree of separation resulting.

42. <u>Accumulative sedimentation tubes</u>--Similar apparatus allowing

rapid size analysis of sands were devised independently by Van Veen (127) and Emery (35) during the period 1936 to 1938. Emery's apparatus is illustrated in Fig. 78. The objective of both investigators was to secure a method which would be more rapid than sieving, and one that could be used on board ship where balances can not be used. The simple apparatus devised by Emery consisted of a long settling tube, tapered at one end and connected to a tube of small diameter which was either graduated or had a scale behind it. Distilled water was the settling medium. The technique and operation of the apparatus as used by Emery will be developed further.

An initial calibration for the development of true fall velocity-diameter curves was accomplished as follows:

> "Numerous samples of quartz sand were analyzed by sieving to find the size distribution (Wentworth system) in per cent of total weight. The various size fractions were then recombined and the total sample allowed to settle through distilled water in the tube described above. All grains were started essentially together. . . . . The sample was allowed to settle twice. The first time, notation was made only of the total height in millimeters reached by the sample after all the sand has accumulated at the bottom. Knowing this height, it was possible to calculate the cumulative height of each size fraction, because for any size fraction the per cent of total volume (expressed here as per cent of total height) is practically the same as per cent of total weight. The time required for each size fraction to fall was found by allowing the sample to settle in the tube a second time. In this case notation is made of the time since introduction of sample required for the sand column to reach the cumulative height of each size fraction. From timings obtained in this way the velocities of all the size fractions may be calculated."

The fall velocities secured in that manner were related to sieve diameters and were influenced by particle shape. This method of calibration and expression of particle size in terms of fall velocity, although probably advantageous when comparisons are to be made with sieve analysis data, is not used as commonly as the conversion of fall velocities to sedimentation diameters of quartz spheres.

The standard procedure with the Emery apparatus is as follows: A sample of sand weighing about 3 gm. was placed first in a 10-cm. length of glass tubing, keeping the lower end closed with a finger tip. Water was added and then the tube was closed at both ends and shaken thoroughly to dispel air bubbles. The mixture was allowed to flow rapidly into the water at the top of the sedimentation tube and a stop watch was started at the moment the pouring began. The height of sand accumulated was read from the start of fall at times corresponding to the grades of Wentworth's scale. During the settling period the graduated tube was tapped lightly to insure even and gradual compaction, and to level off the top of the sand column so that more accurate readings could be made.

The coarsest and finest grades obtained by this technique were greater than 1 mm. and 0.12 to 0.06 mm., respectively. In all, five grades were obtained. To define the 1 mm. limit, a reading at about 13 sec. from the start was necessary, which is probably the shortest time period that can be used because of the increasing effect of initial disturbance caused by larger particles. The limitations of this apparatus for separation into sizes less than about 0.06 mm., due to the decided tendencies for the material to settle as a mass, was recognized.

When a measurable amount of silt and clay was present in a sample which was predominately composed of sand, Emery used a decantation apparatus, shown in Fig. 79, to accomplish a single separation. After the sample was poured into the decantation cylinder, water was added to a depth of 11 cm. Air was then blown in for 5 to 20 min. to disperse the sediment. After allowing the particles to settle for 2 min., the upper 10 cm. of the column was decanted. This process was repeated four or five times until a clean residue of particles greater than about 0.03 mm. diameter remained in the decantation cylinder. This residue was introduced into the tall sedimentation tube described previously, and the weight of the particles in the range 0.03 to 0.06 mm. was determined separately after separation of the coarser grades had been made.

43. Fractional sedimentation tube--A somewhat unique settling method was developed in Russia by Apollov (6). The apparatus is illustrated in Fig. 80. The size separation would proceed as follows: Pipe A was disconnected at C so that the lower portion of the sedimentation tube could be filled with water. All the clamps P , except the lower one, were open. The suspension or sample was poured into A and the remainder of the pipe was filled with water to within about 1 cm. of the top. After a sufficient time had been allowed for all the particles to settle in the bulb, tube A , still held upright, was connected to the flexible tube C . Then A was inverted quickly. The small volume of air left purposely in A would rise immediately into the bulb disuniting the material that otherwise might tend to fall as a unit. At desired times the clamps P were closed, enclosing a fraction between them. When the last or upper clamp was closed, the four fractions obtained could be drained out separately from the bottom.

The range of sizes to which Apollov's apparatus has been used is not given in the reference reviewed. Its limitations are readily perceived, however, from an inspection of the apparatus. The exceedingly

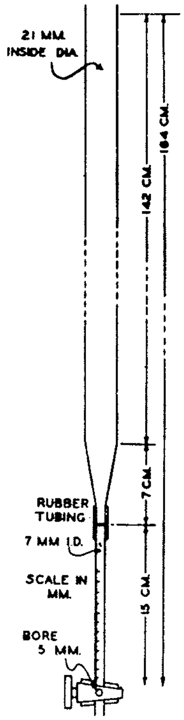


Fig. 78--Emery's accumulative sedimentation apparatus for coarse sediments (1938).

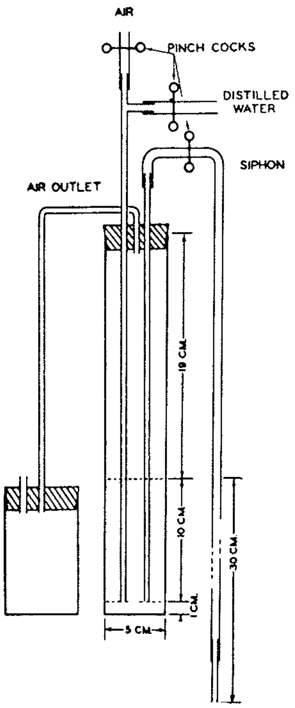
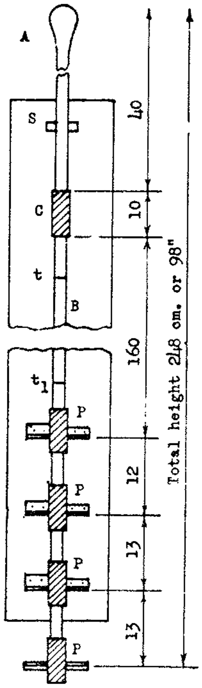


Fig. 79--Emery's decantation apparatus for effecting initial separation between fine and coarse particles (1938).



A Removable pipe with small bulb at upper end. Inside diameter 1.1 cm.

B Glass sedimentation tube.

C Flexible rubber connecting tube.

P Clamps to close tube at desired times.

S Clamp to hold pipe A after inversion.

t and t_1 Marks exactly one meter apart to allow timing of fall of large particles.

Fig. 80--Apollov's fractional sedimentation tube for coarse sediments (1934).

small diameter of the sedimentation tube, only 1.1 cm., has an appreciable effect on the fall velocities of all sand particles. Apparently this has been recognized, for the marks $\underline{t}_1$ and $\underline{t}_2$ were used to time the actual fall velocity of the larger particles. With graded material the reliability and accuracy of such measurements are open to question. The maximum size of particle was probably about 1 mm. The minimum size of particle would depend more upon the completeness of dispersion in the bulb of tube $\underline{A}$, than on the time for an analysis, as separations down to 0.010 mm. could be obtained in somewhat over 6 hr. The efficiency of this unique method of dispersing or introducing the sample to the main sedimentation tube merits further investigation.

XIII. SUMMARY OF PART THREE

44. Summary--A review of size analysis methods applicable to coarse-grained sediments, particles larger than about 1/16 mm. diameter, has been presented. There are three distinct types of methods:

> a. Measurement of the volume of the particles and conversion to a hypothetical sphere of equivalent diameter or direct measurement by calipers of one or more diameters.
>
> b. Sieving with screens of standard apertures.
>
> c. Correlation of observed settling rates of the particles in water to diameters of spheres falling at those rates.

The study reveals that sieving is used more than other methods and has reached a higher degree of development in this country. This no doubt is due to the ease, reliability, and general success of sieving methods as well as to the fact that excellent sieves are available.

Direct measurement of the volume or the diameters of the coarser material has been used. Krumbein has made a division between coarse and fine particles at 16 mm. Direct measurement can be made readily of particles which are larger, and sieving is a generally suitable method of separating the smaller sizes.

However, in sediment studies, a value of particle size determined by methods which allow a direct correlation with settling velocities is more satisfactory than a value determined by a sieve. In a size analysis method based on fall velocities, the important properties of the particles, such as, volume, shape, and specific gravity, are taken into account. In size data secured from a sieve analysis the specific gravity is entirely neglected and the data are affected appreciably by the particle shape, with an increasing misconception of particle size as the particle departs more and more from a spherical shape.

Since 1930, a number of settling rate methods for separating coarse material have been developed. India has led in this field with the development of a manometric, automatic recording device, and a sedimentation apparatus allowing direct weight measurements of individual size fractions. A number of settling tubes employing volumetric accumulation and measurement have been used principally on board ship where balances cannot be used for weight measurements.

The most promising avenue for future development, in this country, of size analysis equipment for coarse sediments, appears to be along the lines of a fractional sedimentation apparatus patterned after Puri's siltometer, shown in Fig. 77. Volumetric measurements have always been open to criticism and, if possible, methods utilizing weight measurements should be used.

PART FOUR

METHODS OF DETERMINING TOTAL CONCENTRATION

XIV. GENERAL

45. Influence of type of sample--Three general types of sediment samples; namely, suspended load, bed-load, and deposited material, have been considered in the discussion of methods of particle size determination. Similar consideration must be given to the effect of the character of these types of samples in the choice of methods for determining total solids concentration. The influential factors, which are largely dependent upon the type of sample, are particle size, concentration, and whether the sample consists, as does a suspended sediment sample, of a given volume of water and sediment, or of a volume or weight of solids, secured over a given period of time.

A bed-load sample, generally, consists of relatively coarse material that arrives in the laboratory in a moist or dry condition. Its total oven dry weight may be secured immediately or a size analysis by sieves or by some hydraulic settling rate method may be made first. No particular difficulties in technique are involved.

Deposited material samples may include material ranging in size from fine to coarse. They may be moist or relatively dry. The laboratory analysis may consist of securing a volume-weight relationship and a particle size determination. If the latter is required, the question arises in the analysis of fine-grained sediment as to whether the material should be dispersed and the size analysis made before oven drying, or whether it should be dried first to secure total weight, and then redispersed. If the size and initial nature of the sample, the particle sizes present, and the laboratory facilities permit, the sample should be divided and a portion used for each analysis.

Suspended load samples offer the greatest problem in the laboratory because the sediment generally present is in only minute quantities and of small size particles, necessitating extremely careful technique to obtain accurate results. The problem is particularly difficult if a size analysis is desired, since a predrying for determination of total solids favors the formation of aggregates that resist redispersion and a return of the particles to their original state. Division of the sample into two portions, one to be used for each analysis, is not advisable because of the very small amount of solids generally present. Furthermore, it is very difficult to divide a sample into two portions of identical size particles.

46. Units of measurement and methods of calculation--Volume rather than weight measurements have sometimes been used in the determination of total sediment concentration, especially for sands and coarse material. Although apparently satisfactory in certain types of investigations, the general unreliability of volume measurements, due to variables of particle shape, size, and compaction, and the necessity of assuming a density for conversion into weights, should discourage such technique.

The common units for expressing total sediment concentration in this country are percentage dry material by weight for high concentrations and parts per million (p.p.m.) for low concentrations. Such practice is advantageous for it eliminates large, awkward figures for high concentration, and decimals for low concentrations. In addition, it is in accord with common usage in sanitary and waterworks engineering.

Three different methods have been used for calculating sediment concentration by weight:

a. Dividing the weight of dry solids by the weight of the entire water-sediment sample.

b. Dividing the weight of dry solids by the weight of the water in the water-sediment sample.

c. Dividing the weight of dry solids by a weight figure for the entire sample calculated from its initial volume by considering the specific gravity of the entire volume equal to that for water.

Although the first is the method or basis generally used and recommended, there are sometimes situations where the calculations are expedited by using one of the other two methods; particularly the third for a volume measurement in the field. Sometimes a partial decantation is advantageous. If either of the latter two methods are used, a conversion to the basis of total sample weight is recommended where the differences between the two results is significant. Fig. 81 shows the percentage difference if the total concentration values have not been based on the total weight of the sample. The difference is definitely negligible below 1000 p.p.m. and even at 10 000 p.p.m. it is only 1 per cent. Fig. 81 also presents a graphical determination of the correction to be subtracted when either method b or c is used.

47. Dissolved material--In certain types of water investigations dissolved solids may be equally as important as the suspended material. Generally, however, in routine sediment studies it is a secondary item and its determination is made only if the procedure necessitates. It is

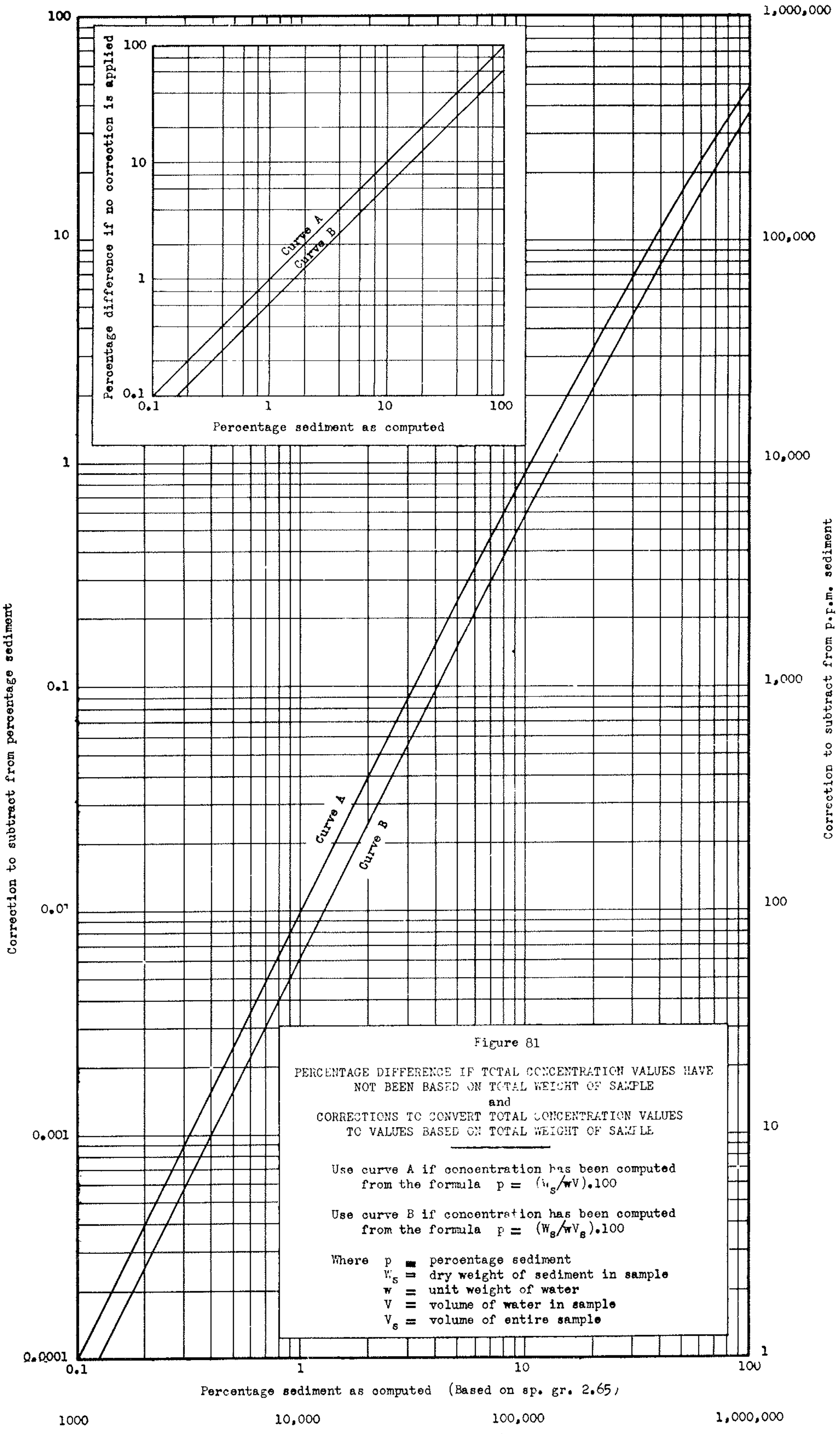


Figure 81

PERCENTAGE DIFFERENCE IF TOTAL CONCENTRATION VALUES HAVE NOT BEEN BASED ON TOTAL WEIGHT OF SAMPLE
and
CORRECTIONS TO CONVERT TOTAL CONCENTRATION VALUES TO VALUES BASED ON TOTAL WEIGHT OF SAMPLE

Use curve A if concentration has been computed from the formula $p = (W_s/wV).100$

Use curve B if concentration has been computed from the formula $p = (W_s/wV_s).100$

Where p = percentage sediment
W_s = dry weight of sediment in sample
w = unit weight of water
V = volume of water in sample
V_s = volume of entire sample

FIG. 81

important in the evaporation method when a rather large volume of water needs to be evaporated and the dissolved solids are left as a residue. Whether or not a dissolved solids determination, for correction purposes, is incorporated into a standard procedure, is dependent upon the method being used, the character of the study, and the ratio of suspended to dissolved matter. The common procedure is to evaporate a measured volume of clear, supernatant liquid and obtain a value of dissolved material that can be applied to the volume of water in the sample being evaporated.

48. <u>Manner of presentation</u>--The methods of determining total concentration to be presented will apply directly to suspended sediment samples and more or less indirectly to bed-load and deposited material samples because of the far greater difficulties and problems involved with the former.

A presentation of the material is advantageously divided into a discussion of so-called "basic methods" and the application of these methods to the methods of size analysis presented in Chapter VIII. The basic methods, each more or less applicable to the solids determination of any type of water-sediment sample, whether it be the total sample or a portion resulting from a size analysis method, are:

a. Sedimentation, decantation, and evaporation.
b. Filter paper.
c. Gooch crucible filter.
d. Alundum crucible filter.
e. Filter candles.
f. Centrifuge.

g. Optical.

h. Displacement.

Detailed descriptions of procedures used by various Government agencies are presented in the appendix.

XV. BASIC METHODS OF DETERMINING TOTAL CONCENTRATION

49. Sedimentation, decantation, and evaporation--Among the simplest methods used in determining the total concentration of suspended sediment samples is that of allowing the sediment to settle to the bottom of a container, decanting the supernatant liquid, washing the sediment into an evaporating dish, and drying it in an oven. Its advantages are simplicity of equipment and technique. In dealing with larger size sediment particles the method becomes more satisfactory. Conversely, as the sediment becomes finer grained the settling time required increases until a point is reached at which it becomes impractical to incorporate it into an efficient laboratory procedure. Also it becomes undesirable to decant liquid from a point close to the top of the deposited material because of the danger of withdrawing some of the entremely fine particles. The transfer of sedimenting to an evaporating dish becomes more awkward because of the relatively large volume of wash water which may necessitate another settling period. The use of flocculating agents will reduce markedly the settling time but this introduces additional material so that a correction factor must be introduced. This correction factor is necessarily, in routine investigations, more of an estimate than an exact value because of the difficulty in determining the exact portion of the flocculent that is incorporated into the final weight and also the amount of dissolved material which is precipitated by the action of the flocculent. As the samples decrease in concentration the opportunities for making serious errors may increase due to this estimated correction. Errors also occur in this method since any dissolved solids in the water evaporated is included in the weight of the sediment obtained.

Because of the above objections to the sedimentation and decantation method when used with finer grained sediment, vacuum filter methods which make for better laboratory procedure have come into prominence.

50. Filter paper--The common practice when using filter paper is to fit the filter paper into a funnel, as is done in ordinary chemistry laboratory practice, then to pour the water-sediment mixture into the funnel allowing the water to percolate through under the influence of gravity. The oven dry weight, at temperatures between 100° and 110° C., of the residue obtained on the filter is used directly in determining the total solids by weight of the original sample. When the water-sediment mixture is of such a large volume that all of it cannot be added at the start of the filtration, it is necessary either to add it intermittently or to arrange the apparatus so that it filters automatically. This may be accomplished by having the bottle which contains the sample inverted with its opening at the desired water elevation in the filter.

The filter paper method of separating sediment particles from water has been and still is used extensively. Like all other methods it has certain advantages and disadvantages. One objection is the possibility of losing material through the filter. Pore diameters of hardened filter paper vary between 0.0015 and 0.0022 mm. which are not unusually small in terms of suspended sediment samples. However, this loss of sediment has been prevented in some cases by the addition of predetermined weights of Kaolin or Fullers Earth. Coagulants may be added to incorporate the minute particles into flocs but as was the case for the sedimentation and decantation procedure a correction factor must be applied for the floculent retained on the filter.

Some laboratory technicians have experienced difficulty in keeping the filter paper at the same tare weight before and after a filtration. Such a variation in weight is largely a matter of moisture retention. Experiments by Archangelsky and Mortinov (6) with air dry filter paper under favorable conditions showed a moisture content of 3.5 per cent. However, a method for handling the filter paper which prevents any appreciable change in moisture content has been developed by the Tennessee Valley Authority. Detailed instructions for making a total solids concentration analysis by this method are given in Appendix B of this report.

For analyzing only a few samples the filter paper method might require a longer time than some other methods since the common filter paper is not adapted to a vacuum system of filtration. However, by the TVA system of handling suspended sediment samples in groups, one man can analyze 500 samples in a 5-day week or 100 samples per 8-hour day.

When using filter paper, ordinarily, it is not possible to reclaim the residue sufficiently for a grain size analysis unless it is relatively coarse and in considerable volume. Experiments by Archangelsky and Mortinov with fine-grained material indicated a loss in sample of from 9 to 25 per cent for samples of 1 and 0.25 gm., respectively. To overcome this difficulty the grain size analysis could be made prior to using the filtration method, or if the sample were large enough, it could be split into two fractions.

51. Gooch and alundum crucibles--The Gooch crucible generally used in sedimentation studies consists of a small porcelain cup of approximately

25 cc. capacity which lends itself to attachment to an aspirator system and subsequent vacuum filtration. Although customarily used for ordinary drying temperatures of 105 to 110° C., Gooch crucibles are used for temperatures as high as 1000° C. Other types, such as platinum, are used also.

The flat circular base of the common Gooch crucible is perforated with openings about 0.7 mm. diameter. Prior to the filtration of a sample a small portion of loose, uniform asbestos, which may be purchased especially for this purpose, is seated firmly on the bottom of the crucible, thus forming a complete cover over the base. This layer of asbestos serves as the filtering medium. Asbestos has proved very satisfactory for this use because of its inertness, resistance to heat, and low hygroscopicity. Gooch crucibles with asbestos mats are oven dried and cooled in a desiccator prior to securing a tare weight for each time used. A detailed explanation of this procedure is given in the appendix.

The alundum crucible, similar in size and shape to the Gooch crucible, is made of fused aluminum oxide and may be purchased in a plain or an ignition type. These crucibles are available in three porosities, porous, medium porosity, and dense; therefore, the selection depends upon the nature of the samples. Fig. 82 shows alundum crucibles installed with flasks in an aspirator system.

Small crucibles of these types have a number of advantages which have made them popular in analyzing minute samples of fine-grained material. With very fine pores, produced through the use of asbestos or inherent in the crucible, the loss of material through them is generally negligible. They are light in weight which tends toward greater balance sensitivity. Their tare weights are less subject to change with change in atmospheric conditions because less surface is exposed. The vacuum filtration is generally fairly rapid and under some degree of control. The U. S. Engineer Office at Vicksburg, Mississippi, has developed equipment and technique to such a point that thirty samples per day can be analyzed by one man. As is the case with the filter paper method, dissolved material passes through the crucibles which eliminates the need of correcting for it.

In certain instances the material may be so fine that even under vacuum an unreasonably long filtering time is necessary. Under these conditions it is sometimes more advantageous to resort to the addition of a flocculating or adsorbing agent and use the ordinary sedimentation and decantation procedure.

If the sediment is extremely fine, an actual loss through the Gooch or alundum crucibles may occur, evidence of which is a turbid filtrate. Through the use of adsorbing agents this colloidal material may be retained in the filter for inclusion in the final weight. This is a relatively common practice in industrial chemistry, water purification, and sewage treatment. The principle of action is largely a mutual attraction between the adsorbing agent and the colloidal particles due to difference in electrical charges. Activated carbon has probably been the most widely used agent. Hjulstrom (47) adopted this method for analyzing sediment samples containing a significant percentage of colloidal sized particles, using successfully, finely ground asbestos. A predetermined weight was added to the sediment sample prior to filtration and the entire sample shaken very thoroughly. In addition to preventing loss through the filter, the use of adsorbing agents should reduce the time of settling and should prevent the filter from clogging as rapidly. Accomplishing the same purpose as a flocculating agent, though probably less positive in action and less effective, inert adsorbing agents of low hygroscopicity may be more advantageously used with certain types of sediment samples because the proper correction for the amount retained in the filter is known.

52. Filter candles--Chamberlain-Pasteur filter candles have been successfully used at the Soil Conservation Experiment Station, Statesville, North Carolina, for the removal of water from water-sediment samples varying in concentration from a fraction of a per cent to more than 10 per cent.

Filters of this type are approximately 1.5 cm. in diameter and 15 cm. long and are manufactured of unglazed porcelain in a number of grades of porosity. A suction line is attached to the open end of the filter so that when the filter is immersed in the water-sediment sample the water will be drawn through the filter and into a receptacle placed in the suction line. The suspended sediment is retained on the surface of the filter. A constant suction is maintained until the water is removed down to a volume that can be conveniently handled in an evaporating dish. The sediment collected on the filter is removed by reversing the pressure momentarily; however, in some instances it has been necessary to use a brush.

The method is readily adapted to the mass reduction of samples, being limited only by the equipment available. From 2 to 4 hr. is necessary to reduce quart samples down to a 100 cc. volume.

For samples of small volume and low concentration the use of the filter candle method would be less advantageous than the Gooch or alundum crucible methods. In these latter methods the solids are retained in the crucible, thereby eliminating the transfer into evaporation dishes and the need for evaporating the relatively large volume of water necessarily used in the filter candle method. For samples of high concentration where clogging of the crucibles might retard the filtration seriously, for samples of large volume, or for conditions where a number of samples are to be combined, the use of filter candles may be advantageous because of the large filtering surface and the ease of cleaning this surface.

53. Centrifugal method--The U. S. Waterways Experiment Station, Vicksburg, Mississippi, has developed and used with success in its model studies a centrifuge which is shown in Fig. 83, for separation of relatively coarse solids from a large volume of water (123). In operation the water-sediment is pumped into a steel bowl which has around its circumference, at equal intervals, pyrex tubes in which the sediment collects as the apparatus is rotated at 1800 r.p.m. The water-sediment mixture is pumped continuously into the bowl with the sediment collecting in the tubes and the water overflowing into a waste container. If the material is not too fine an inflow rate of 2 gal./min. can be maintained and a satisfactory separation accomplished with a water-sediment mixture of 1 per cent concentration.

The centrifuge may be used in the field with certain types of pump

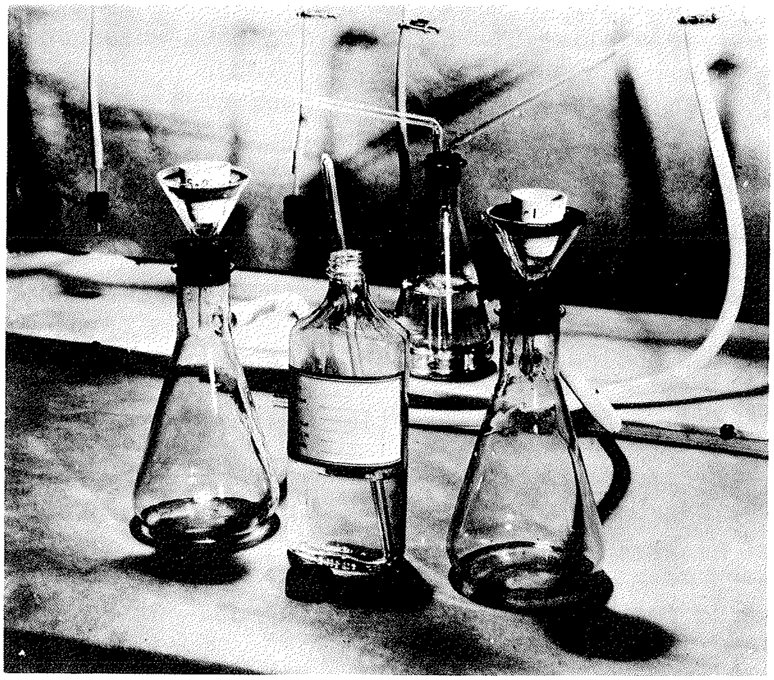


Fig. 82--Alundum crucibles installed in aspirator system.

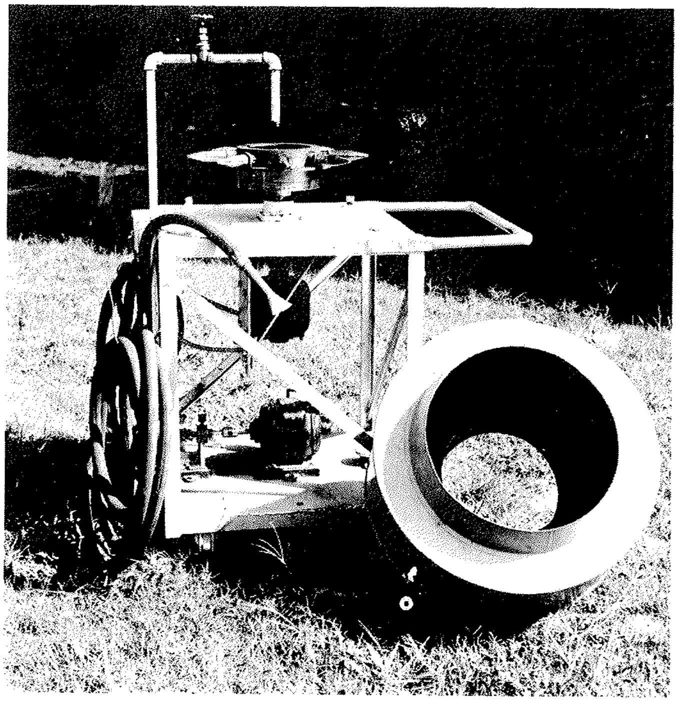


Fig. 83--Centrifugal "silt separator" of the U. S. Waterways Experiment Station, Vicksburg, Miss.

samples, thereby separating the solids from the water as the sample is collected. Italian engineers (128) have used such an arrangement on the Po River. A serious disadvantage is the amount and size of the necessary equipment.

When the sample is composed of very fine material centrifugal force may be used in the ordinary sedimentation and decantation procedure to decrease the necessary sedimentation time. In addition to promoting more rapid separation of solids from the water, such a procedure would eliminate the necessity for adding a flocculent, and consequently eliminate the need for adding an adsorbing agent. Generally the ordinary laboratory which handles suspended sediment samples lacks the necessary amount of centrifugal equipment.

54. Optical methods--A rather complete presentation of the progress made in optical measurements, i.e., measurements of absorption or reflection of light, in particle size analysis has been presented in Part Two, Section 31. In the discussion it was pointed out that a light absorption or reflection value obtained with the photo-electric cell, Tyndallmeter, or any other device is a function of both particle size and concentration, as well as a number of other factors. Thus, unless the particle size and distribution remain practically constant, it is not possible to use an optical method in determining solids concentration of sediment samples.

The continuous measurement of suspended sediment concentration in the field by means of hollow tube samplers with a photo-electric cell opposite a source of light has been presented and discussed in Report

No. 1, "Field Practice and Equipment Used in Sampling Suspended Sediment." Its unreliability due to changes in particle size and distribution has been pointed out. However, this equipment may be used to indicate changes in either or both the concentration or particle size of the sediment flow and thus serve as a guide as to the proper time to collect a representative sample.

55. Displacement method--An indirect method of determining the weight of solids in a water-sediment sample without oven drying is described here as the displacement method. It embodies the same general procedure as that of determining specific gravity of solids, the major difference is in the method of calculation. The procedure is to obtain the difference in weight between a volume of water and an equal volume of water plus solids. To determine the specific gravity, the weight of solids is known which allows the difference in weight to be interpreted in terms of weight per unit volume. The displacement method for determining weight of solids requires that the specific gravity of the material be known and that it be quite uniform. The difference in weight of equal volumes, one with water alone and one with water plus solids, allows a calculation of weight of dry solids. The easily developed expressions are:

$$V_s = \frac{w}{(S_p - S_w)}$$

where V_s = actual volume occupied by solids in cc.

w = difference in weight in gms.

S_p = specific gravity of solids.

S_w = specific gravity of water.

$$\text{Weight of solids} = S_p V_s = S_p \frac{w}{(S_p - S_w)}$$

Weight of solids equals 1.606w for quartz particles (S_p equals 2.65) in water (S_w equals 1.0 with no significant change at ordinary temperatures).

The obvious advantage of this method is the rapidity with which the weight of total immersed solids can be determined since oven drying is not necessary. It was used very successfully to determine total solids in the experimental laboratory study of sampler intakes, presented in Report No. 5, "Laboratory Investigations of Suspended Sediment Samplers."

The accuracy of the method depends largely upon the sensitivity of the balance available, the total weight of solids in the sample, and the correctness of the specific gravity value used for the solids. In using this method care must be exercised to maintain the same temperature in both weighing bottles.

XVI. RELATION OF BASIC METHODS TO METHODS OF SIZE ANALYSIS

56. Methods whereby individual size fractions are obtained--The size analysis methods presented in Chapter VIII can be advantageously separated into two divisions:

> a. Methods in which individual size fractions are obtained for individual oven drying and weight determination; these are the decantation, elutriation, fractional sedimentation, and centrifugal methods.
>
> b. Methods in which the sediment accumulates at the base of the sedimentation column and indirect observation for determination of particle size is made; these are the accumulative sedimentation balance, pipette, hydrometer, plummet, and manometer methods.

In this section will be discussed the application of basic methods of determining total concentration to those size analysis methods whereby individual fractions are obtained.

The dry weight of material in a fraction obtained in a size analysis method is obtained generally by the basic methods of sedimentation and decantation or crucible filtration. In the former a flocculating agent is sometimes added. The total concentration is obtained by summing up the dry weights of these individual fractions.

The magnitude of the error in the weight of an individual fraction and in the final total weight obviously will depend upon the type of size analysis method used and the laboratory technicians care in conducting the analysis.

57. Methods whereby sediment accumulates--Size analysis methods in which the sediment accumulates at the base of the sedimentation column offer fewer difficulties in accurately determining total concentration

than do other methods, because the total volume of sediment is available rather than a fraction. After all material, except the extremely fine particles which may be flocculated or neglected, deposits at the base of the container, the next step is to decant off the supernatant liquid. Whether that basic method or a filtering method is used to complete the analysis will depend upon the character of the material and other specific conditions.

In the pipette method, the weight of the dry material in each aliquot obtained during the size analysis must be included in the final answer.

From the standpoint of a total solids determination, the size analysis methods which allow an accumulation of the sediment embody more advantages than do the methods in which fractions are obtained, because in using such methods there is no opportunity for accumulative errors.

58. Indirect methods--In addition to allowing a determination of total solids using the entire amount of accumulated sediment, the pipette, hydrometer, plummet, and manometer offer indirect methods. The procedure is to withdraw a pipette sample or secure a hydrometer or manometer reading immediately after thoroughly and completely dispersing the sediment throughout the sedimentation column. The purpose of such a procedure is either to check a total solids value obtained by analyzing the entire sample at the completion of the size analysis or to eliminate entirely, the final analysis for total solids. The errors in this method increase with the size of the particles and with the time required to make the observation.

Since the volume of the pipette sample is extremely small in relation to the entire sample, a serious magnification of errors is possible; however, it appears to be the best of these indirect methods for the determination of total concentration. The hydrometer and plummet are affected by currents in the sedimentation column and take some time to steady sufficiently; this is particularly true of the ordinary hydrometer. The hydrometer is also difficult to read quickly and accurately because foam collects on the water surface and on the stem of the bulb. The manometer cannot be adjusted very rapidly nor is it easy to read accurately.

The method used by the U. S. Engineer Office, First New Orleans District, and U. S. Waterways Experiment Station, Vicksburg, Mississippi, was to obtain a pipette sample immediately after a complete dispersion (122) in order to determine the total concentration before making a size analysis. The samples analyzed were usually composites of four or five original samples varying in concentration from about 600 to 2000 p.p.m. The amounts of solids analyzed as one composite sample varied from 1.7 to 5.7 gm. Generally, more than 95 per cent of the particles were smaller than 0.074 mm. in size and 60 per cent finer than 0.008 mm.

XVII. SUMMARY OF PART FOUR

59. Summary--The presentation and discussion of methods of determining total concentration has applied primarily to suspended sediment samples because, with their low concentrations and small particles, they usually offer far greater laboratory problems than either samples of bed-load or deposited material.

The greater accuracy and dependability of weight over volume measurements has been stressed, especially as the sediment becomes finer grained. Oven dry solids by weight expressed in per cent or in parts per million (p.p.m.) based on the total weight of the sample is the recommended procedure. Recognizing that, in certain instances, it is more advantageous to determine, initially, the concentration based on only the weight of water in the sample, or on a weight of the entire sample calculated from its total volume by considering a specific gravity of the entire sample equal to 1.0, correction curves are given, as shown in Fig. 81, to allow conversion to a true weight basis.

The addition of a flocculating agent to a suspension to precipitate extremely fine material may be objectionable in that a portion of the flocculating agent may be incorporated in the final dry weight, and that some of the dissolved solids may be precipitated. The problem lies in the difficulty of determining an accurate correction.

Inert adsorbing mater als rather than flocculating agents have been used in certain investigations to clarify suspensions of extremely fine sediment particles. In addition to promoting a rapid removal of the particles from the suspension, this procedure prevents a loss that may occur even through Gooch and alundum crucibles. Finely ground asbestos, thoroughly mixed with the sample, has proved extremely efficient in Finnish investigations in adsorbing colloidal size material.

Methods of determining total concentration, adaptable either to the total sample as it arrives in the laboratory, to the accumulated material at the base of a sedimentation column, or to a grade fraction obtained during the course of the size analysis, are described as basic methods. The most common methods employ sedimentation, decantation, and evaporation, filter paper, and Gooch or alundum crucibles.

Chamberlain-Pasteur filter candles, although not used extensively for sediment samples, appear to have, with certain types of samples, advantages over the Gooch or alundum crucibles. Larger volumes and higher concentrations may be handled because of the greater filtering surface and the ease of cleaning.

Optical methods have not proven satisfactory because the absorbed or reflected light varies with the concentration and with the particle size of the sediment.

The centrifugal method has been used when a large water-sediment volume has to be handled. It is especially suitable to a continuous inflow such as is obtained with pumping samplers.

A rapid indirect method has been presented for determining total dry weight without oven drying which is based on the ordinary displacement procedure of determining specific gravity. In this method the specific gravity of the material must be known or estimated. In a laboratory investigation of samplers, which is a phase of this project, this method was successfully used.

In addition to the direct methods of determining total solids in a sediment sample by the application of one of the so-called basic methods to the total accumulated material at the base of a sedimentation column or to a size fraction obtained during a particle size analysis, there are several indirect methods available. With the pipette, hydrometer, plummet, and manometer methods of size analysis, a sample, or readings obtained immediately after a complete dispersion, provides an index of total concentration. Of these, the pipette sample has been the most widely used.

APPENDIX A

DETAILED SIZE ANALYSIS PROCEDURES

APPENDIX A

DETAILED SIZE ANALYSIS PROCEDURES

60. Sieving--Standard procedures for sieve analysis have been developed for use in the size determination of concrete aggregates and other construction materials. The American Society for Testing Materials, *(A.S.T.M.), has set forth standards that are generally accepted in engineering work. Until some more satisfactory standards are devised, it seems advisable, as far as practicable, to adopt the A.S.T.M. standard sieve procedures in the study of sediments.

In order to obtain uniformity it is advisable, also, to use sieves of the U. S. Standard Series, the openings of which have been given in Table 6. These sieves are the standard for the U. S. Government work and are rapidly being adopted in other fields as well. They conform to the A.S.T.M. specification, Designation: E 11-38T, "Tentative Specifications for Sieves for Testing Purposes (wire cloth sieves, round-hole and square-hole screens or sieves)" (13).

The following are details of the A.S.T.M. standard procedure:

For the size analysis of all material larger than the No. 200 sieve (opening 0.074 mm.) the procedure given in A.S.T.M. Designation: C 136 38T, "Tentative Method of Test for Sieve Analysis of Fine and Coarse Aggregates," (10) applies.

When a sample to be analyzed contains material finer than a 200 mesh sieve opening, that finer material is first washed from the coarser sieve by the standard procedure described in A.S.T.M. Designation: C 117-37,

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*Address - 250 S. Broad St., Philadelphia, Pa.

"Standard Method of Test for Determination of Amount of Material Finer Than No. 200 Sieve in Aggregates" (9). When it is desired to make separations according to some grade scale, which does not exactly match the No. 200 sieve, it will probably be desirable to depart somewhat from the A.S.T.M. specifications and use the nearest sieve conforming to the grade scale.

The amount of sample increases with the coarseness of the material. For material which is at least 95 per cent finer than a No. 10 sieve (opening 2.0 mm.) a 100 gm. sample is specified. For material which is at least 90 per cent finer than a No. 4 sieve (opening 4.76 mm.) and more than 5 per cent coarser than a No. 10 sieve a 500 gm. sample is used. These weights are given for 8 in. diameter sieves. For larger sized material the minimum weights are tabulated below:

Nominal maximum size of particle in.	Minimum weight of sample kg.	Nominal maximum size of particle in.	Minimum weight of sample kg.
3/8	1	2	20
1/2	2.5	2-1/2	25
3/4	5	3	30
1	10	3-1/2	35
1-1/2	15		

When the material contains particles both smaller and larger than a No. 4 sieve (opening 4.76 mm.) a separation is first made with that sieve and then proper amounts of samples taken as are specified for those particle sizes.

Other analysis requirements are:

a. The balances should be sensitive to within 0.1 per cent of the initial weight of the sample.

b. When drying the sample the temperature should not exceed 110° C.

c. The sieving specifications have been presented in Section 37.

61. Decantation--A great many decantation procedures have been used. One procedure which has been found quite satisfactory for the size analysis of suspended sediment samples was developed at the Iowa Institute of Hydraulic Research and is described as follows:

a. General features--The distance from the top of the sedimentation column to the point of withdrawal or decantation was chosen for convenience at 16 cm. for the smallest fraction and 32 cm. for all other fractions. The total heights of cylinders were 20 cm. and 37 cm., respectively, with diameters 7 cm. Stokes' fall velocity law was used to determine the time required for particles of the limiting sizes to reach the withdrawal elevation and a chart was developed for determining the time values to use for various temperatures of the sedimentation medium.

Two 1/4 in. glass tube siphons were constructed so that the withdrawal elevation was either 16 cm. or 32 cm. from the top of the sedimentation column. The intakes of the siphons were funnel-shaped in order to withdraw the liquid from as wide an area as possible. Over the outlet ends of the siphons were fitted short pieces of rubber tubing to which were affixed pinch clamps. A short time before using a siphon it was filled with water and left in an inverted position until placed in the sedimentation tube. This practice facilitated withdrawal because releasing the pinch clamp started the siphoning and eliminated any serious disturbance in the sedimentation column. In Fig. 14 are shown the sedimentation cylinders, siphons and other equipment used.

Temperatures of the suspended medium were taken during each sedimentation period in order to determine the proper settling time so that any desired size separation was obtained.

To inhibit flocculation the water used was either siphoned from the original settled sample (referred to as native water) or was distilled water, preferably the former. Tap water was found to be unsatisfactory. No pre-drying of the samples was made.

In the procedure described the separation into size fractions proceeds from fine to coarse particles. This procedure is more advantageous than in the opposite order for it eliminates the necessary delay which results from lengthy settling period of fine material.

b. Preparation of sample--The suspended sediment samples were allowed to stand undisturbed in their original field containers until complete settlement of the sediment was attained. The entire sample and container were weighed and then clear water (termed native water) siphoned off without disturbing the settled material and saved for the analysis. The sediment was next washed into a dispersion cup, native water added until 2/3 full, and the suspension mixed for 2 min. The dispersion equipment consisted of an ordinary malted milk mixer and a container fitted with four baffles as specified in the A.S.T.M. Designation: D 422-38T (11).

c. Separation into fractions--Immediately after the dispersion in the mixer the suspension was poured into the short sedimentation cylinder and native or distilled water added to fill the cylinder within 1/2 in. of the top. This 1/2 in. allowed for the rise when the siphon was inserted. A smooth rubber pad, backed by a small block was held firmly against the top of the cylinder and the cylinder inverted and righted a number of times to distribute the sediment throughout the column.

When the distribution was satisfactory the column was placed upright upon a firm base simultaneously with the starting of a stop-watch. The rubber pad was removed and the short siphon, filled with water, was inserted in the cylinder. The settling was allowed to proceed undisturbed for the length of time required for all particles larger than the smallest size desired to have settled past the decantation level (at 20° C. a 2 hr. settling period for particles 0.005 mm. diameter). Then the clamp on the siphon was released and the water and fine particles decanted into a gallon jar.

The cylinder was refilled with native or distilled water and the same dispersion and settling procedure repeated. As previously explained in Section 20, the sediment remaining in a sedimentation tube includes finer material that did not have to fall the entire height of column so that repeated decantations are needed to obtain a satisfactory separation into the desired size fractions. In Table 3 are presented percentages of smaller size particles which still remain after a certain number of decantations. As used at the Iowa Institute of Hydraulic Research the decantations were repeated until the siphonings were clear. Three decantations were usually sufficient with the type of sediment handled.

The entire siphonings from this first separation were retained in a gallon jar in which settling, sometimes aided by a coagulant, was allowed to proceed. When the top water was clear it was siphoned off and the remaining sediment and water washed into a porcelain evaporating dish, dried at 110° C. in an electric oven, cooled in a desiccator, and weighed on an analytical balance.

Immediately after the last decantation of the fine material the remaining water-sediment was transferred into the taller (36 cm.) sedimentation column. The same procedure as described for the first fraction of filling with native or distilled, dispersion, settling, decanting, and retention of the siphonings for final analysis was repeated three times for each remaining size fraction; the only variation being decreased in settling time.

As outlined the procedure requires over 8 hr. actual settling time for a single analysis when five fractions are obtained with the smallest at 0.005 mm., not including the time to prepare the initial sample and redisperse it in the sedimentation column before each decantation, siphoning, and so forth.

62. Pipette--The general pipette procedure as applied to soils is presented in the U. S. Department of Agriculture Technical Bulletin No. 170, 1930 (68). A basic problem in the textural analysis of soils, which may also be present in the analysis of deposited sediment, is the dispersion of the material into individual grains or extremely small groups of colloidal sized particles. As defined in the bulletin proper dispersion of soils is obtained when the colloidal coatings are removed from silt and sand grains and the aggregates are separated into individual grains or into a group of grains not greater in size than 0.002 mm. A complete discussion of dispersion by chemical means is presented in the bulletin.

After a preliminary preparation of a 10 gm. sample which includes, prior to final dispersion, a determination of organic matter and the exact weight of dry material, the sample is washed upon a 300 mesh Tyler sieve. The material passing is retained in a 1000 cc. graduate acting as the sedimentation cylinder. The exact weight of solids added is determined indirectly from the amount retained on the sieve. After the suspension is stirred with a motor driven propeller and the temperature determined, the sedimentation is allowed to proceed.

A special 25 cc. pipette operated by a rack and pinion is used to collect a sample at a point exactly 10 cm. below the water surface. The time of sampling is determined by Stokes' law considering the temperature during the sedimentation period. The time required to fill the pipette under even suction is approximately 40 sec. The contents of the sedimentation column were stirred after each sample; a procedure that was investigated by Rittenhouse (87) and found to be unnecessary in routine pipette analysis.

Detailed instructions and procedure for the pipette method of particle size analysis of sediment samples are presented by the U. S. Waterways Experiment Station, Vicksburg, Mississippi, in typewritten instructions* titled, "Mechanical Analysis of Sediment Samples by Pipette Method as Used in the Soil Mechanics Laboratory." The following information has been extracted from this article:

> a. Apparatus--"The sedimentation chamber consists of a plain glass cylinder 2 inches in diameter and 12 inches high, with pour-out at top. Capacity approximately 500 cc."
>
> "The pipette consists of a length of 12 mm. glass tubing drawn out at the bottom to the proper length to contain about 10 cc. of suspension when submerged to a depth of 15 cm. The top is drawn out to allow connection of a short length of rubber tubing to which is attached a spring pinch clamp. The

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* Unpublished.

pipette is surrounded by an adjustable wooden collar which grips the pipette slightly; a small strip of waterproof tape is placed on the pipette at a distance of 15 cm. from the end. In taking an aliquot specimen this collar rests on the top of the glass cylinder, and the pipette is pushed downward through the collar until the tape mark coincides with the surface of the suspension. When the spring pinch-clamp is released at the proper time, the suspension fills the pipette to the tape mark; the rubber tubing is pinched shut, the pipette and collar removed and the contents emptied into the specimen container."

"The containers used for weighing and evaporating the aliquot specimens are weighed glass specimen vials of 4 gram capacity. Identification numbers are etched into the sides of the vials by means of an abrasive marking material; this type of container has a small, fairly constant weight which allows weighing the sediment residue after evaporation of water to 0.0001 gm."

Three different balances were used so that a consistent accuracy of three significant figures was obtained regardless of the weight. A trip balance (sensitivity 1 gm.), a torsion balance (sensitivity 0.05 gm.), and an analytical balance (sensitivity 0.0001 gm.) were used, respectively, to weigh the total sample, the pipette aliquot, and the sediment residue from individual aliquots.

b. Procedure--"The sample is allowed to settle and the supernatant liquid siphoned off as completely as possible into a 500 ml. graduated cylinder and its volume noted. The sediment residue and small amount of water left therewith is mixed, poured into a 100 cc. graduate and its volume noted. The sum of these two volumes is taken as the weight or volume of the sediment sample. The material in the small graduate is transferred to the weighed glass sedimentation cylinder and washings from the graduate and original sample container added thereto, so that all sediment is now contained in the latter. Sufficient distilled water is added to bring the volume to approximately 400 cc., the cylinder and contents weighed, mixed thoroughly and set at rest on a firm table. An aliquot specimen is taken immediately before any appreciable settling occurs to determine the total sediment content, and at intervals of time afterward sufficient to give the desired number and spacing of points on the grain-size distribution curve. The following schedule is used for routine tests at the Experiment Station: 10 sec., 1 min., 4 min., 30 min., 2 hr., 5 hrs., 24 hr., 48 hr. The amount of sediment in suspension at each time of sampling is calculated from the weight of suspension in the cylinder and in each aliquot specimen, and the weight of sediment residue in each aliquot specimen. These are referred to the amount obtained from the first aliquot to obtain percentages of the sample finer than the corresponding grain sizes calculated from the depth of sampling, specific gravity of sample and observed temperatures using Stokes' Law. The original concentration of sediment in the sample as received may be obtained, if desired, by referring the weight of sediment at the beginning of test to the original volume or weight of sample. The total weight of sediment in the sample may be checked, if desired, by evaporating the suspension left after the test and adding to the weight of residue obtained the weights of residue removed in the aliquot specimens."

"The procedure as outlined above is applicable to sediment samples containing moderate amounts of dissolved matter and small amounts of sand. If large amounts of dissolved matter are present, the sample will be flocculated; the original water siphoned off is tested to determine dissolved content and its volume replaced by distilled water. The sample is mixed thoroughly and allowed to settle again. The supernatant water is again siphoned off, and the process repeated until the sample is deflocculated. Correction may be made for dissolved matter by applying to the original dissolved content appropriate dilution factors (ratio of volume of sample after dilution to volume left before dilution)."

"In the case of samples containing an amount of sediment in excess of about two grams, it is best to analyze a representative portion of the sample. If the amount of sediment is less than 0.5 gm., two or more samples should be combined for analysis."

c. Limitations--"It (the pipette method) has been found applicable to sediment samples containing as little as 0.5 gm. of sediment, corresponding to a sediment concentration of approximately 1000 p.p.m. for a pint sample; analyses of samples containing much less than this amount do not yield accurate results. The optimum amount is 1 to 2 grams per analysis, corresponding to a sediment concentration of approximately 2000 to 4000 p.p.m. for a pint sample. Amounts of sediment in excess of this are liable to flocculation in the volume of water used in the analysis."

63. Hydrometer--The standard hydrometer method of particle size analysis for soils is set forth in detail in A.S.T.M. Designation: D 422-38T, "Tentative Method of Mechanical Analysis of Soils" (11). The procedure applies to the common type of hydrometer as illustrated in Fig. 52 and 53 and individual correction curves, to facilitate interpretation

of hydrometer readings into particle size, are given for both hydrometer scales; that is, specific gravity or grams per liter. A detailed procedure for chemical and mechanical dispersion is given for soils but obviously this will vary considerably with types of sediment samples.

A constant temperature bath is specified in the standard procedure. However, this is often eliminated by obtaining temperature readings immediately before each hydrometer reading. Standard readings are taken at 1, 2, 5, 15, 30, 60, 250, and 1440 min. after the start of the sedimentation. Depending upon the character of the material and type of investigation these have been varied somewhat in sediment studies.

Corrections due to temperature variations from 67° F. at which the hydrometers were calibrated, depth of immersion (center of volume changes as density decreases), specific gravity variations from 2.65, the specific gravity of material for which the hydrometers were calibrated, and viscosity of the suspending medium which varies with temperature, are easily applied through the use of correction curves and tabular forms.

APPENDIX B

DETAILED TOTAL CONCENTRATION ANALYSIS METHODS

APPENDIX B

DETAILED TOTAL CONCENTRATION ANALYSIS METHODS

64. Gooch crucible--The U. S. Geological Survey has used rather extensively the Gooch crucible filtration method as a means of separating solids from water in suspended sediment samples. The general procedure* is as follows:

> The first step upon receiving a sample in the laboratory is to determine its total weight, after which the sample is allowed to remain undisturbed for a reasonable period of time. If not clear at the end of that period a known amount of coagulant is added (1 to 3 cc. of 10% HCl solution), and another sedimentation period allowed. At the end of either the first or second settling period the clear water is decanted off and the sediment washed into a beaker. After a short settling time in the beaker another decantation is made, if possible. The contents in the beaker are then ready for transfer to the Gooch crucible.
>
> After the asbestos mat is prepared, the crucible is heated at 110° C. for one hour, cooled in a desiccator, and weighed for the determination of the tare weight. After the crucible is in place in the suction system the sediment is washed into it and the filtering action proceeds. When completed the crucible and contents are heated at 110° C. for one hour, cooled in a desiccator, and weighed.
>
> If the filtration is determined to be too slow with the Gooch crucible the ordinary sedimentation, decantation, and evaporation procedure is used.

The Gooch crucible procedure as used in Finland, incorporating the use of an adsorbing agent is described by Hjulstrom (47) as follows:

> a. Preparation of crucible--"On the bottom has been placed a rather thick and hard compressed layer of loose and uniform asbestos, through which at least one liter of water has been passed in order to wash away all the loose particles in the asbestos layer. In order that the crucible itself should show the same weight at the various weighings it had already, before the first weighing (before the filtering), to undergo the same treatment which it should undergo before the following

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*Typewritten (unpublished) instructions by S.K.Love, dated Nov. 27, 1940.

weighings. Thus it was heated to red-heat for 15 minutes after the filter had been placed and washed off. After cooling off in the open air about 300 cc. of distilled water were filtered, this partly in order to cause the asbestos to adsorb water, partly for the removal of particles and loose matter made free during the heating. After this the crucible was left to dry in a drying closet at a temperature of 110° C. for 2-1/2 hours, after which it was allowed to cool in a desiccator for 45 minutes; finally it was weighed."

b. Preparation and addition of adsorbing agent--"After being heated to a red-heat and cooled off in a desiccator, 0.3 gm. asbestos is weighed in a platinum crucible; when the water is very muddy a little more asbestos is added.* * * * It has, beforehand, been cooked with strong hydrochloric acid and then carefully washed, and all the hard lumps have been removed and the asbestos loosed with a pair of pincers so that it is uniform and loose. After the asbestos has been transferred into the water-sample, this is shaken energetically for 20 minutes and then filtered through a weighed Gooch crucible."

c. Filtering--Vacuum filtration was conducted through the use of a water suction pump connected to a suction bottle having the Gooch crucible mounted above with air-tight rubber connections. A continuous, automatic feed into the crucible was secured by having an air-tight connection between the top of the crucible and a glass tube extending into the sample bottle. After the automatic feed no longer functioned because of too shallow a depth in the sample bottle that equipment was removed and a large portion of the remaining water-sediment transferred by pipette. Then the bottle was washed three or more times with distilled water until all the sediment was transferred to the filter.

d. Drying and weighing--At the completion of the filtering the crucible was dried for 2-1/2 hr. at 110° C. in the drying closet, cooled in the desiccator for 45 min., and weighed on a balance sensitive to 0.001 gm. In the final calculation the amount of asbestos added as an adsorbing agent is, of course, subtracted from the weight of material retained in the filter.

65. Alundum crucible--Alundum crucibles with vacuum filtration are used extensively in the analysis of suspended sediment samples by the U. S. Engineer Department, Vicksburg, Mississippi. The procedure is very similar to that described for the Gooch crucible with the exception that no asbestos mat is used.

66. Filter paper--After trying a number of various methods the Tennessee Valley Authority has found the filter paper method of making a total silt concentration analysis to be the most satisfactory. The procedure is as follows:

> a. Preparation of samples--Suspended silt samples are delivered to the laboratory in pint milk bottles. Each bottle is wiped clean of all mud and dirt and the bottle cap removed. The pint bottle of silty water is weighed on a balance which is accurate to one gram. This weight is recorded as the gross sample weight.
>
> b. Preparation of filter papers--For separation of silt from the water in the sample, folded filter papers 18.5 cm. in diameter are used. To retain colloidal silt each filter paper is covered with Kaolin and placed in a permanently numbered ointment can. Twelve of these flat tin cans, left partly uncovered, are placed on a metal shelf in an electric drying oven where they are allowed to remain for one and one-half hours at a constant temperature of 110° C. The cans are then removed, closed and placed in a desiccator where they are allowed to cool for one-half hour. Then each can, with the enclosed filter paper, is weighed on a Chainomatic balance which is accurate to 0.0001 gm. The resulting weight is recorded as the tare filter weight.
>
> c. Separation of silt by filtration--The samples are placed on a bench in front of a filtering rack holding glass funnels, and the cans are placed on a shelf above and behind the rack. Each filter paper is then removed from its can, placed in an 8-ounce ribbed glass funnel, and unfolded. A rubber stopper with a 6-in. glass tube through the middle is placed in the neck of each sample bottle. The pint bottles are then inverted on the rack so that the end of each glass tube is at a point below the top of the filter paper within the glass funnel, after which the samples are allowed to filter by gravity.
>
> When all of the water from the sample has drained into the filter paper, the bottle is removed from the rack. The stopper is removed and the silt adhering to it washed into the filter paper by a stream of tap water from a wash bottle. After adding 20 cc. of tap water, the silt adhering to the inside of the sample bottle is loosened with a glass rod to which is attached a "rubber policeman" which consists of two pieces of rubber tire patching. The silt is washed into the filter paper by means of the wash bottle. The empty bottles are weighed in the same manner as were the samples and the weights recorded as tare weights.

d. Drying and weighing papers and silt--After all the water has run through the filter, the papers are folded and returned to their respective cans. Twelve cans, partly uncovered, are placed again on a metal shelf in a pre-drying oven set at 70° C. After two hours in the pre-dryer, the cans and papers are removed and transferred to the constant temperature oven where they remain for an hour and a half at 110° C. The drying is done in two stages in order to avoid transferring moisture from wet to dry samples. In this way individual samples can be placed in the final dryer at any time and withdrawn after the prescribed drying period. They are then removed to a desiccator and cooled for 30 min. The cans and papers containing the silt are weighed on the analytical balance and the results recorded as gross filter weights.

e. Calculating and reporting results--On the work sheet the net sample weights and net filter weights are obtained by subtraction. To calculate the concentration of silt in parts per million, the net filter weight is divided by the net sample weight and the decimal point moved six places to the right.

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