Measurement of Fine Silt and Clay Size Distributions

#391

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ABSTRACT

THREE different commercial particle-size analyzers were compared with the conventional pipet technique to measure the size distributions of 15 samples of very fine sediments. These samples were obtained from the deposited sediments in several oxbow lakes in the alluvial plain in west central Mississippi. Approximately 90% of the material in these samples were less than 10μm in size. The basic features, principles and underlying assumptions for each instrument are outlined and compared for all analyzers and techniques used. While the results from the four methods showed drastic differences, reconciliation of these data was achieved by use of simple mathematical descriptions of the particle size distributions and of the measurement processes involved with each instrument.

INTRODUCTION

Knowledge of soil and sediment size distribution is important to several aspects of agricultural soil and water research. Several traditional methods have been used through the years with high success and few problems. For larger particles, sieves have been successfully used. For medium sands in the range 60 to 1000 μm the visual accumulation (VA) tube is convenient and simple to use. There is a considerable overlap region where both sieves and VA tubes have been used.

Below 62 μm the pipet method ordinarily has been used. This method is time consuming and labor intensive. At a 10-cm withdrawal depth, it requires a full 8-h day to determine the distribution down to 2 μm (the upper limit of what is ordinarily considered the medium and fine clay fraction).

This time and labor factor has prompted several soil and sediment analysis laboratories to investigate alternative easier and faster means of analysis in the silt and clay size range. There are several instrument systems available on the market designed to perform this measurement.

The systems vary widely in their individual characteristics, advantages, disadvantages, and principles of operation. Generally, the common factor is that they are all quite costly, and the individual requiring such a system desires as much information as possible before making a decision as to which system to acquire.

While the conventional pipet method is and has been traditionally used in all USDA-ARS soil and water laboratories, the individual laboratories have in recent years purchased and tested some of the modern particle size instrument systems to increase the efficiency of size analysis measurements. It was decided to compare these instrument systems using a set of 15 samples obtained from lake bottoms and consisting of primarily clay-sized particles.

In the past it has usually been sufficient to simply measure a percent finer than 2 μm and designate this as the medium and finer clay fraction. With increased emphasis on the role of sediment in the transport and trapping of agricultural chemicals, such as nutrients and pesticides, it has become necessary to measure the fine silt and clay portions of the size distribution in greater detail. The particles in this portion of the distribution have the most surface area to adsorb agricultural chemicals and have chemical and electronic characteristics such that adsorption will occur.

This paper presents the results of a cooperative study between three USDA-ARS laboratories where these modern analyzers are located. The USDA Sedimentation Laboratory at Oxford, MS, collected and prepared all samples and analyzed them with the standard pipet method and with the Computerized Electrozone System*, manufactured by Particle Data, Inc. of Elmhurst, Illinois.

The USDA Water Quality and Watershed Research Laboratory at Durant/Chicasha, OK, analyzed the samples using the Sedigraph Particle Size Analyzer, manufactured by Micromeritics Corporation of Norcross, GA. The USDA Southwest Rangeland Watershed Research Center at Tucson, AZ, analyzed the samples with the Model 7991-0 Microtrac manufactured by Leeds and Northrup Company of Largo, FL.

It is the purpose of this paper to compare the essentials of each system including their individual advantages and disadvantages for size analysis of fine silts and clays as experienced in the measurement of a set of 15 samples, and to compare the measured results and reconcile the observed differences.

COMPARISON OF SYSTEM CHARACTERISTICS

In order to compare the four systems, Table 1 shows the essential features of each on a common basis. It is difficult, however, to present the complete picture in such a condensed form and several of the items require

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*Trade names are included for information of the reader and do not constitute endorsement by the United State Department of Agriculture.

Table 1

INSTRUMENT SYSTEM AND APPROXIMATE COST	PHYSICAL PRINCIPAL	SIZE RANGE Microns	CONCENTRATION REQUIRED	TIME REQUIRED FOR ANALYSIS to 2 microns	EASE OF OPERATION	OUTPUT FORMAT AND INFORMATION	SPECIAL COMMENTS
Pipet method $500	Gravitation settling in stokes range, withdrawal, evaporation and weighing of samples	1-62	2000-5000 mg/l	9 hrs for a 10 cm withdrawal depth	Easy and straight forward operation, slow and time consuming	Data must be obtained by hand weighing of dishes	
Electrozone $19,000	Electrical conductivity change caused by particles moving through small orifice	.3-300	less than .01 mg/l	5-10 min	Requires a very experienced operator for good results	Detailed data output in graphical or tabular form. Output in either cumulative or differential form for particle numbers, area, or volume.	Particle numbers concentration must be very low to avoid coincidence errors. Lower limit of distribution is artificially truncated at a size under control of operator within instrument capabilities.
Sedigraph $18,000	Gravitational settling in Stokes range, mass determination by x-ray attenuation	.1-100	22,000-46,000 mg/l	15 min	Requires limited experience for good results.	Graphical output in cumulative form	Brownian motion may begin effecting accuracy below .5 microns
Model 7991 Microtrac $25,000	Detection of the intensity and scattering angle of laser light by particles	1.9-176	40-2000 mg/l	6 min	Very simple to use	Tabular form in both differential and cumulative form with additional summary data.	Lower limit of distribution is truncated at 1.9 microns

additional elaboration.

In the Electrozone instrument a quantity of water containing particles is drawn through a small orifice (Berg, 1957; Coulter, 1956). If the number of particles per unit volume is required, the volume of water may be precisely controlled. A constant electrical current is caused to flow through the orifice, which constitutes the principal resistance in the electrical path. As a particle moves through the orifice, a voltage pulse is created whose amplitude is proportional to the volume of the particle. The magnitude of each pulse is measured, classified and the information stored in the proper channel indicating that an equivalent sphere in a specific size range passed through the orifice and was counted. This measuring and classifying was accomplished by an analog to digital converter and a small computer. This allows considerable detail in the measurements and provides the capability of performing considerable analysis of the data using a preloaded program and a set of simple commands. The output data may be presented in a number of different tabular or graphical forms. The smallest particle-size counted is operator adjustable within the limitation of the orifice used. The manufacturer cites a value of about 2% of the orifice diameter as the smallest diameter particle distinguishable from the background noise. As smaller orifices are used, difficulties are encountered with orifice clogging. For the particle distributions measured in this study it was nearly impossible to count particles smaller than about 0.9 μm because of clogging. The system offers a particular advantage when very dilute suspensions must be analyzed. Usually dilution is required to obtain few enough particles to avoid coincidence errors. Coincidence errors occur when two or more particles move through the sensing zone at nearly the same time creating the illusion of a single larger particle. Dilutions may be precisely made and the resulting counts scaled back to the original sample. Experience with the Electrozone system has shown that the operator must have considerable practice before the results become reasonable and repeatable.

The Microtrac laser system measures a well defined range of particles in the silt and fine sand range (Wertheimer et al., 1978; Haverland and Cooper, 1981). The lower particle-size limit measured by this instrument is truncated at 1.9 μm because of the wavelength of the light beam used. Unlike the Electrozone instrument the operator has no control in this case. The Microtrac system assumes the particles to be spherical light scatterers. The instrument is very easy to use in that a sample need only be poured into the instrument and no other adjustments must be made. A built-in microcomputer operates on light-scattering data and quickly produces a convenient printed tabular data set describing the size distribution. The required concentrations for this instrument are quite low and the instrument can easily accommodate samples brought directly from the field. In some cases dilution may be necessary.

The Sedigraph utilizes Stokes gravitational settling of the particles in a small cell (Olivier et al., 1970; Welch et al., 1979). The concentration is detected by a weak x-ray beam 50 μm thick. It requires a concentration in the range of 22,000 to 46,000 mg/L. The system operator requires some experience for good results but the Sedigraph is quite easy to use.

In order to compare the time required to process a sample a size of 2 μm was selected. All three automated systems require only a brief time to accomplish their measurement. The Sedigraph requires about 15 min, the Microtrac about 6 min, and the Electrozone instrument about 10 min. If measurement of smaller sizes is required, longer Sedigraph analysis time is also required. It requires 90 min to measure the distribution down to 0.2 μm and 288 min to 0.1 μm.

Unlike the other two automated systems, the Sedigraph does not discard the portion of the distribution smaller than the lower size limit that the operator selects and the percent smaller than this size may be read from the graphical chart as it can for any other size in the size measured.

The manufacturer states that possible errors due to Brownian motion may begin at 1 μm, and depending on the nature of the distribution, become important at 0.5 μm. Other types of errors are shown to be of lesser magnitudes.

The pipet method (Guy, 1969; Schideler, 1976) is probably the best known for measurement of particle sizes in the silt and clay region. It is also time consuming and labor intensive, which explains the interest in the development of alternative systems.

All four methods require similar sample preparation depending upon the requirements of the data. To examine primary particles the organic material must be removed and the suspension must be dispersed both mechanically and chemically.

SAMPLE DESCRIPTION

Fifteen samples were selected from lakes in the Bear Creek System in the Delta area of west central Mississippi. This system of oxbow lakes, formed by the ancient Ohio River, are connected by Bear Creek and drain into the Yazoo River north of Belzoni, MS. The watershed is almost entirely cropped with cotton, soybeans, and rice. The soils are very fine textured with a large clay component. The runoff carries the fine sediment into the waters of the Bear Creek system, resulting in a constant high turbidity due to the presence of suspended inorganic material throughout the system. Sediment cores were obtained from the centers of Three Mile, Wasp, Macon, and Mossy Lakes.

The cores were taken with a 10-cm (4 in.) plastic coring tube and were divided into 10-cm segments.

With only minor differences, all of the samples had essentially the same size distribution. For the purposes of this paper only one of the samples will be presented as an example. The sample selected was the section of the core 20 to 30 cm (8 to 12 in.) deep in the sediment obtained from the center of Three Mile Lake in Sunflower County, MS.

RESULTS AND DISCUSSION

The size measurements obtained by the four methods are shown in Fig. 1. The effects of truncation of the small sizes are readily apparent for the Electrozone and the Microtrac Instruments. It is also evident that a substantial portion, about 62% indicated by Sedigraph, was smaller than 0.2 μm.

The pipet measured distribution is displaced a constant amount toward the larger sizes, the D_{50} size being 0.26 μm as compared with 0.104 μm measured

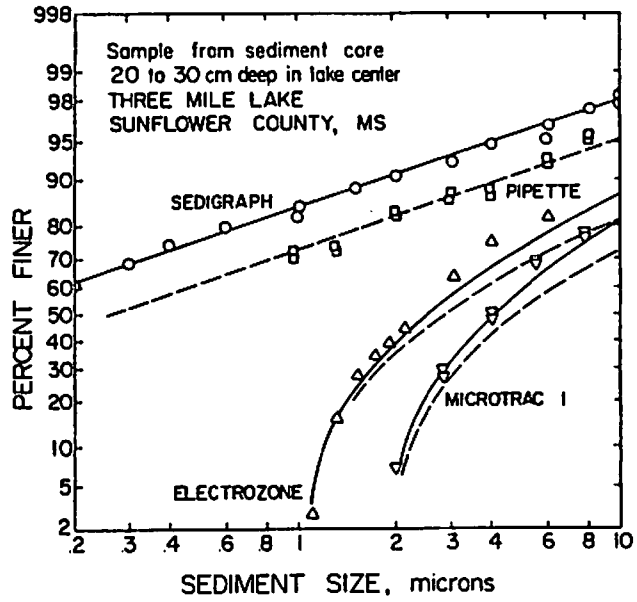

Fig. 1—Typical data set of size distributions obtained by four methods.

with the Sedigraph. The slopes of the two distributions are essentially the same.

Since data from both the Sedigraph and the pipet method plot an essentially straight line on logarithmic-probability graph paper, a reasonable assumption is that the particle distribution can be described by the log-normal density relationship

$$p(d) = \frac{1}{\sqrt{2\pi}\, D\, \sigma_y} e^{-(y-\bar{y})^2/2\sigma_y^2} \quad [1]$$

where

D = particle diameter

y = ln D

$\bar{y}$ = average value of y = ln D_{50}

and

σ_y = standard deviation of y about $\bar{y}$.

Integrating this relationship to any arbitrary particle diameter, D_*,

$$p(D_*) = \int_0^{D_*} p(D)\, dD. \quad [2]$$

allows the computation of the fraction smaller than D_*. This relationship reduces to

$$P(D_*) = 0.5 + \frac{t_*}{|t_*|} \operatorname{erf}|t_*| \quad [3]$$

where

$$t_* = \frac{y_* - \bar{y}}{\sigma_y}$$

$\operatorname{erf}|t_*|$ = error function of $|t_*|$

and

y_* = ln D_*

which defines the solid line through the Sedigraph data in Fig. 1 when

D_{50} = 0.104 μm

$\bar{y}$ = −2.26

and

σ_y = 2.19.

The dashed line through the pipet data is represented by the same relationships where

D_{50} = 0.26 μm

$\bar{y}$ = −1.35

and

$\sigma_v = 2.19.$

The response of an instrument which truncates the lower portion of a particular size distribution can be analytically predicted if the true complete size distribution is known. The mass represented by particles smaller than a critical size, D_c, is not measured by the system. The critical size may be, within the instrument capabilities, under control of the operator or it may be simply pre-set as a characteristic of the machine.

In either case the result is the same, a portion of the distribution, $P(D_c)$, is simply not measured. The total mass seen by such an instrument is $1 - P(D_c)$ and the mass smaller than any measurable particle size, D_*, and larger than the critical size is $P(D_*) - P(D_c)$. The expected smaller than fraction, $F(D_*)$, as measured by such an instrument is the ratio of these two quantities

$$F(D_*) = \frac{P(D_*) - P(D_c)}{1 - P(D_c)} \quad \ldots\ldots\ldots\ldots\ldots\ldots\ldots \quad [4]$$

Using equation [3] and the parameters matching it to the Sedigraph data as the mathematical model of the size distribution, the response of the Electrozone and Microtrac instruments were predicted with equation [4] using the corresponding critical size for each instrument.

The solid lines drawn in Fig. 1 represent these predictions. The close correspondence would tend to indicate that the principal difference between instruments is the amount of mass smaller than the critical size that is neglected. That amount is measured by the Sedigraph as residual mass, but it is not measured by the other two systems because of the nature of the detection system.

The dashed lines are similar predictions using the log-normal distribution model determined by the pipet method as the input size distribution. Discrepancies are apparent at the larger sizes.

The difference between the Sedigraph and the pipet method has been observed in previous research (Welch et al., 1979) and has not been satisfactorily explained. Both methods employ sediment settling but the pipet requires a sample to be withdrawn, evaporated, and weighed while the Sedigraph examines the mass of sediment in a small, well-defined point in the sample cell. The volume withdrawn by the pipet is defined by a sphere centered at the tip of the pipet. This difference may account for some of the discrepancy. Research is still proceeding to resolve this question.

A recent development by Leeds and Northrup has led to Microtrac Model 7991-3 which, compared to previous models, has a lower critical particle size (0.12 μm). The computed response of this instrument is compared with the response of the other automatic analyzers in Fig. 2 for three different distributions of various clays (smaller than 2 μm). Distribution 1 is the verified instrument responses shown in Fig. 1 with the addition of the predicted response of the second Microtrac instrument.

In order to investigate the effect that the medium clay (2 μm and smaller) fraction has on the instrument responses other distributions having the same slope (σ_v held constant) but differing medium clay fractions were numerically investigated using equations [2] and [3]. In Distribution 2 half of the sample is medium clay and smaller and in Distribution 3 a tenth of the sample is medium clay and smaller.

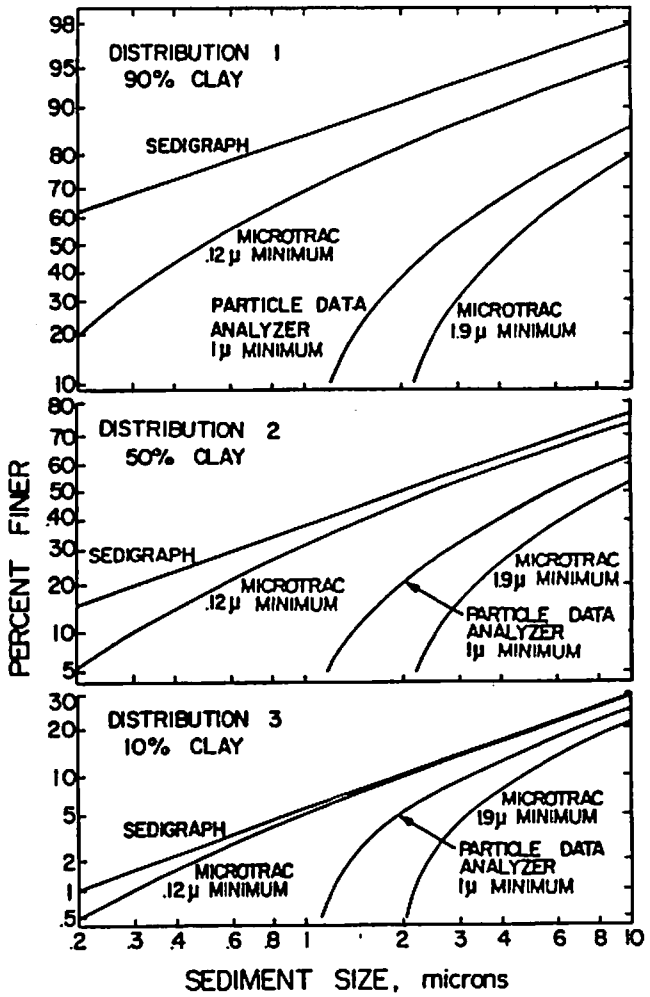

Fig. 2—Prediction of analyzer response for various clay fractions.

These results indicate that as the medium and smaller clay fraction becomes smaller the resulting data can be expected to agree better. However, as indicated in the introduction, recent interest has been in the very fine clay fraction because of the availability of sediment surface area available for chemical adsorption.

SUMMARY

This study has shown characteristics and various advantages and disadvantages of the four instrument systems investigated. The specific type of sediment being investigated with clay contents of over 90% requires analyzers which do not have portions of its distribution which are invisible to the measuring instrument.

While analyzers with critical sizes larger than the sizes required for investigation of clay are quite convenient and useful for many purposes, the clay fraction is partially neglected. The Sedigraph and the pipet methods, while they do not satisfactorily agree, are the two methods which do not ignore mass contained in particles smaller than some critical size. The Microtrac Model 7991-3, while still apparently unverified, appears to be another instrument which may be satisfactory for this purpose.

Research is still required to reconcile the results from the Sedigraph and the pipet method.

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