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MICROTRAC: A RAPID PARTICLE-SIZE ANALYSER OF SEDIMENTS AND SOILS

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INTRODUCTION

Particle-size analysis is basic to laboratory and field studies of soil and fluvial sediment. An improved method of making these measurements is desirable. Weiss and Frock (1976) reported results from an instrument employing the principle of laser light scattering to measure particle-size distribution. The instrument, a Microtrac* particle-size analyser, manufactured by Leeds and Northrup, was reported to be of high precision and yielded reproducible results. We have used such an analyser for soil studies over a two-year period.

Particle-size analysis of soils and fluvial sediment has previously been conducted principally by sieving and sedimentation methods which are tedious and time consuming. The specific surface of a soil is largely dependent on particle size. Conventionally, specific surface of soils is estimated by measuring the retention of a uniform layer of molecules, such as glycerol, over the surface to be measured (Kinter and Diamond, 1958). The Microtrac particle-size analyser estimates specific surface by calculations based on particle size. Our purpose is to report on its performance as an alternative to the conventional pipette and hydrometer methods of soil particle-size analysis and glycerol method of specific surface area.

METHODS AND MATERIALS

General Description and Operation of the Microtrac

The Microtrac measures particle size by low angle forward-scattering of laser light which has passed through a sample cell (Wertheimer et al., 1978). The laser light is produced by a helium neon source of 0.6 microns wavelength (Fig. 1). The nature of scattering is dependent upon the ratio of particle diameter to the wavelength of the laser light. The relationship of particle size to the intensity and angle of scatter of the laser light, after light-particle interaction, is of prime importance in Microtrac theory. Light intensity is directly proportional to the particle diameter squared, the angle of light scatter is inversely proportional to the diameter of the particle (Jenkins and White, 1975).

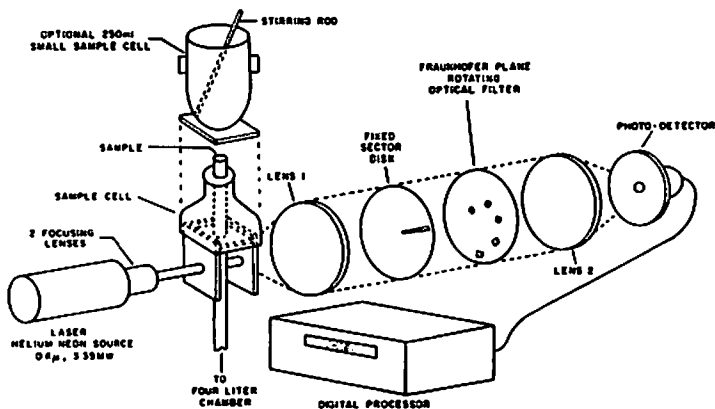


Figure 1.--Exploded view of Microtrac system.

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

Following the interaction of light and particle in the sample cell, the light passes through a rotating Fraunhofer plane optical filter which has openings designed to accommodate light fluxes proportional to respective powers (d^2 , d^3 , and d^4) of the particle diameter. A photodetector and microcomputer convert the scattered light into numerical data describing particle-size distribution. The concentration is displayed visually on an LED readout, and data is recorded by a digital printer.

Samples are put in aqueous suspension, if not already in that form, with two sample cells available for use depending upon sample concentration. A 4-liter chamber is used for an approximate range of 2,000-40 mg/l, and a smaller 250 ml chamber can be used for an approximate range of 400-40 mg/l. Sampling error and background interference increases with lower concentrations, while very high concentrations introduce error due to increasing attenuation and multiple scattering. Thus, the Microtrac method is particularly well suited to particle-size analysis of suspended sediment samples having low concentrations or where sample quantity is limited, since soil or fluvial sediment quantities of 0.01 g may be routinely analysed with the small sample chamber. Using the 4-liter chamber, the sample is continuously pumped from the cell between parallel glass lenses where the laser light beam is intercepted. The 250 ml chamber is filled manually and mixed by a mechanical stirring rod.

Data is provided on 13 channels (size fractions) between 1.9 and 176 microns (μm) yielding both channel percent and cumulative percent less than (or cumulative percent greater than). These size ranges correspond to one-half phi intervals of the Udden-Wentworth scale as expressed by Krumbain (1934) where $\phi = -\log_2(\text{diam.}) (\text{mm})$. This notation is widely used in sediment analysis.

Output includes a cumulative graph, a relative volume graph, cumulative and histogram data, and summary data (Fig. 2). Summary data consists of the values, in microns, at the 10th, 50th, and 90th percentile points, the mean diameter of the volume distribution, the calculated mean specific surface area, and a value (dv) representative of sample concentration (Fig. 2). Different combinations of these outputs are switch-selectable and obtainable in % smaller and % larger modes. Particle scanning time

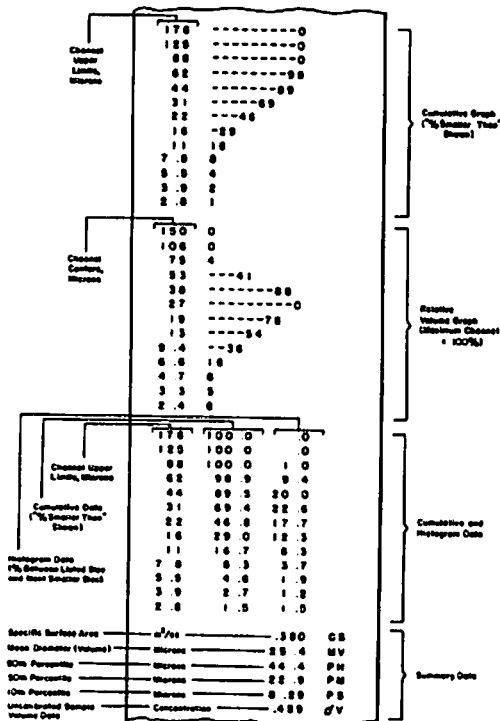


Figure 2.--Typical data printout with selector switch at position 6, sizes % smaller.

can be selected between 3 to 800 seconds. Our work has shown 100 seconds to be ample measurement time for good analysis, although a 50-second analysis produces roughly similar results. The extremes of measurement time are provided to allow for a wide range of applications for industry and research. Samples

may be repeatedly analysed to reflect particle swelling and aggregate breakdown processes. Tap water is commonly used for circulation of the sample with the 4-liter chamber by using a "Set Zero" switch before introduction of the sample. The unit thus provides background compensation for particles in the tap water or stray minute dust particles on the lens system. Deionized water, which has passed through a 0.45 μm millipore filter, is used in conjunction with the 250 ml small sample chamber to insure minimal background interference. If desired, other solvents may be used as a carrier for the sample.

Following background compensation, the sample is put into the chamber for a short period (at least 30 seconds) to insure adequate mixing. Between measurements, the chamber is drained and rinsed, with a total time between samples of approximately one minute. Using our techniques, approximately ten prepared samples may be analysed per hour.

Other Microtrac units are available which are capable of analyses in the ranges as follows: Model 7991-1, 3.3-300 μm , Model 7991-2, 16-1000 μm , and Model 7991-3, 0.12-20.50 μm .

Design of Experiments

In our experiments, tests were conducted using the 4-liter sample chamber. Our experiments were designed to provide insight on the relationship of the printout parameters to changes in concentration, soil texture, and dispersive techniques. In addition, Microtrac specific surface area data is compared with data obtained by Post (1977) who used the glycerol method (Kinter and Diamond, 1958) on subsamples of the same ten soils. Soils for all experiments were selected so as to provide a wide range within which the vast majority of sediments could be texturally classified.

Sample Preparation

Sample preparation and dispersion remain important factors for satisfactory analysis. Our sample dispersion was accomplished by a combination of two treatments, a chemical dispersant, and the subsequent application of ultrasonics immediately prior to analysis. The chemical dispersant consists of 53.52 g Na_2PO_4 and 4.24 g Na_2CO_3 , in one liter of distilled water (C.L. Lameris, 1964), which is added to the soil at a ratio of 1 ml dispersant/1 gram soil. A 350 watt, 20 KHZ ultrasonic probe with a 1/2-inch disruptor horn was submerged into the soil solution, and ultrasonics were applied for 30 seconds. Following this dispersion, each sample was wet-sieved with an ASTM No. 80, 180 μm sieve, 176 μm being the upper size limit of this Microtrac model.

RESULTS AND DISCUSSION

The effects on specific surface area of chemical and ultrasonic energy pretreatments for dispersion are shown in Fig. 3. The dispersed soil consistently has a higher specific surface area (SS) indicating the destruction of aggregates. This phenomenon is more dramatic with finer textured soils such as the Guest clay.

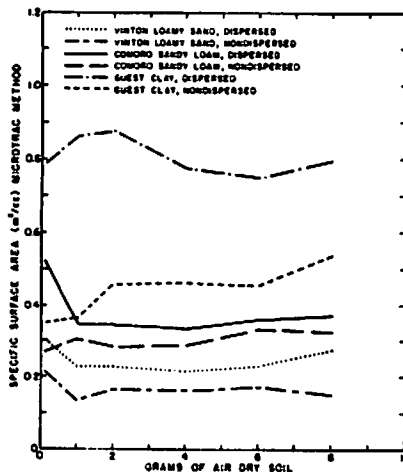


Figure 3.--The effects on measured specific surface area of chemical and ultrasonic energy pretreatments for dispersion.

The sample concentration parameter is a system volume response number (dv) obtained by sensing the light which has passed through an opening in a solid disc. The opening is designed for the transmission function to have a (d³) third power response to the particle diameter. The relationship of dv to sample weight and soil texture is shown in Fig. 4. There is a definite influence of the textural (relative % sand, silt, clay) characteristics of the sample upon the dv response to increased sample weight. This influence can be avoided by removal of the clay fraction prior to Microtrac measurement. The coarser textures produce much larger dv's at heavy concentrations such as 6 or 8 grams. The finer clay rich samples never produce very high dv's such as 1.0, and peak at lower concentration values and then decline. The decrease in slope can be viewed as an "overloading" on the optical analyser; therefore, we generally recommend 3 grams as optimum for the 4-liter chamber regardless of sample texture.

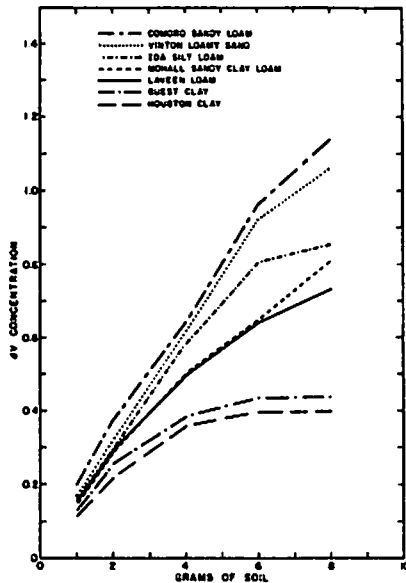


Figure 4.--The relationship of sample concentration (dv) to soil weight and texture.

As long as sample loading is within the suitable range, the dv is directly proportional to the total volume. Variations in particle density (but to a much lesser extent than optical considerations) affect the relationship between Microtrac dv and grams of material in the unit. If particle density can be approximated, the dv number can be used to indicate sample weight. Clays may have densities as low as 1.2 g/cc, while sands may be approximately 1.6 g/cc.

Figure 5 is a comparison of the glycerol method of specific surface area (Post, 1977), with the Microtrac optical determination of specific surface area (CS). Microtrac particle-size determinations are initially computed on the basis of a volume distribution; the surface is then calculated from the thirteen segments of the volume histogram and expressed as m²/cc. Glycerol specific surface area is in units of m²/g. Glycerol values are for laboratory analyses for the less than 2.0 millimeter fraction, whereas Microtrac data is for a range of 1.9-176 microns. The linear regression yielded

$$Y = 166.7X - 23.2 \quad r^2 = 0.93$$

where X = Microtrac data and Y = pipette data. The correlation is viewed to be in excellent agreement, especially when the differences in measurement range and methodology are considered.

SUMMARY

Our study has shown the Microtrac to be a valuable instrument for soil and fluvial sediment research. The Microtrac is capable of showing the results of aggregate destruction, and therefore may be used to infer the relative degree of aggregation of various soils and fluvial sediments. Instrument parameters of concentration and specific surface area operate in a useful and accurate manner. Another major advantage of the Microtrac method is the speed and ease by which the measurement is performed. Approximately ten prepared samples can be processed per hour. The same number of samples would require

approximately 3 1/2 hours by X-ray-sedimentation analysis (Welch, 1979). Electronic analysis by the Coulter counter is approximately twice as fast as the pipette method (Pennington, 1979), and 10 samples by pipette would require a full 8-hour day. We believe the Microtrac represents an attractive method for rapid particle-size analysis.

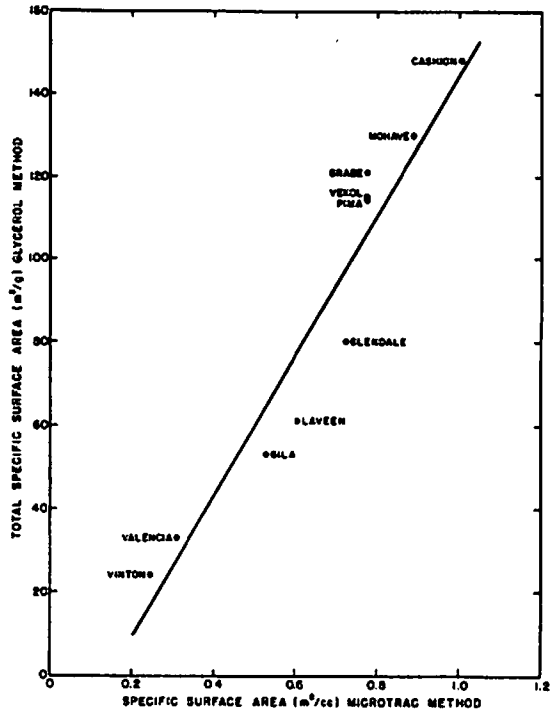


Figure 5.--A comparison of specific surface area values for 10 Arizona soils obtained by glycerol and Microtrac methods.

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