
A STUDY OF METHODS USED IN
MEASUREMENT AND ANALYSIS OF SEDIMENT
LOADS IN STREAMS



REPORT R
PROGRESS REPORT
ELECTRONIC SENSING OF SEDIMENT

DECEMBER 1964

A Study of Methods Used in
MEASUREMENT AND ANALYSIS OF SEDIMENT LOADS IN STREAMS

A Cooperative Project
Sponsored by the
Subcommittee on Sedimentation
Inter-Agency Committee on Water Resources

Participating Agencies

Corps of Engineers	**	Geological Survey
Soil Conservation Service	**	Bureau of Reclamation
Agricultural Research Service	**	Coast and Geodetic Survey
Tennessee Valley Authority	**	Federal Power Commission
Bureau of Public Roads	**	Department of Labor
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Public Health Service		

REPORT R

Progress Report

ELECTRONIC SENSING OF SEDIMENT

Prepared for Publication by the
Federal Inter-Agency Sedimentation Project
at St. Anthony Falls Hydraulic Laboratory
Minneapolis, Minnesota

December 1964

PREFACE

This investigation is part of "A Study of Methods Used in Measurement and Analysis of Sediment Loads in Streams." The study is being made by the Federal Inter-Agency Sedimentation Project at the St. Anthony Falls Hydraulic Laboratory of the University of Minnesota. The project is under the sponsorship of the Subcommittee on Sedimentation of the Inter-Agency Committee on Water Resources and under the executive direction of a Technical Committee composed of: P. C. Benedict, Geological Survey; D. C. Bondurant, Army Corps of Engineers; W. M. Borland, Bureau of Reclamation; H. G. Heinemann, Agricultural Research Service; M. D. Hoover, Forest Service; E. H. Lesesne, Tennessee Valley Authority; E. M. Thorp, Soil Conservation Service. The purposes of the project are the development and improvement of equipment and methods for obtaining and analyzing sediment samples.

Thomas F. Beckers made most of the tests and prepared the original draft of the report. Byron C. Colby, project leader, supervised the test program and assisted in the preparation of the report. Howard A. Jongedyk performed the microscopic analyses. Frederick S. Witzigman supplied data on comparative analyses made with the visual-accumulation tube and sieves. Bruce R. Colby, John V. Skinner, Salvatore La Bella, and Wayne Johnson assisted in some phases of the test program and report preparation.

The Coulter Counter (registered trademark) Model A developed by the Coulter Electronics Company of Chicago, Illinois was the basic instrument on which the tests in this report were made. More advanced models now available (December 1964) are reported to be free from temperature effect, unaffected by moderate changes in electrolyte, free from the need for variable F factors, and supplied with a simplified set of accurate gain ratios. These improvements greatly simplify the computation of the data. An accessory is under development to count particles in size range steps and record volume of particles within the steps and total volume of particles. This report pertains only to the Model A instrument. Permission of the Coulter Company to use material from the manual of instructions and from brochures accompanying the instrument is greatly appreciated.

Robert Berg of Particle Data Laboratories, Inc., and K. T. Whitby, consultant for the American Association of Cereal Chemists supplied valuable material and data.

George W. Whetstone, District Chemist, Branch of Quality of Water, U. S. Geological Survey, Columbus, Ohio, furnished samples and the analyses of these samples by the bottom-withdrawal-tube method for comparison with analyses by the electronic-sensing method.

SYNOPSIS

A commercial electronic-sensing device (counter) was purchased and tested for determination of concentration and particle size distribution of sediments. In operation a known volume is metered from a dilute suspension in which the sediment particles are uniformly dispersed. As the individual particles pass through a small aperture they produce an electrical pulse that is proportional to particle volume. The numbers of pulses larger than several successive reference sizes are counted. By calibration the electrical-pulse sizes are related to particle sizes. The size distribution and concentration can then be determined.

Particles with diameters from 2 to 40 percent of the aperture diameter can be analyzed. Apertures with diameters of 50, 140, and 400 microns were used and particles from 1 micron to larger than 100 microns in size were analyzed. Uniform dispersion of natural sediment particles coarser than 100 microns was difficult to obtain. The necessity of passing the particles through a very small aperture and of using more than one aperture for most analyses is a disadvantage.

Samples of fuller's earth, coal dust, pollens, glass beads, polystyrene beads, and sediments from natural streams were analyzed or were used as calibration materials. Particle shape, roughness, and kind of material had little effect on analysis. Determination of an accurate size or size distribution of materials for use in calibrating and checking the counter was a basic problem. Microscopic analyses were used as the standard of comparison with the counter. A counter analysis was about as accurate as microscopic analysis of size distribution and took nearly as long.

The electronic-sensing method is shown to be usable for size analysis in the laboratory. The method can be used to determine concentration by volume but the process is long and involved. However, the basic electronic approach determined accurately the volume of a single particle in an aperture. Although several problems are involved, the basic approach might be extended to the determination of concentration of particles in a larger aperture or sensing zone.

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Progress Report R

ELECTRONIC SENSING OF SEDIMENT

I. INTRODUCTION

1. Purpose and scope of the investigation--Suspended-sediment discharge of streams is being measured at hundreds of places in the United States. The discharge is obtained from manual measurements of sediment concentration and stream discharge. Such records are costly and often they are incomplete. A method of automatically determining suspended-sediment concentration in streams is needed to improve accuracy and continuity of sediment records and reduce the cost inherent in frequent manual measurements. The possibilities of using electronic sensing of sediment in suspension seem to justify an intensive study of this method.

This report is a study of the method of electronic sensing of suspended sediment. Ultimately the method should be evaluated completely. This first project report is limited to an evaluation of the only commercial instrument available for electronic sensing of sediment size and concentration.

A commercial electronic device for measuring and counting particles in suspension was purchased, and its accuracy, stability, and dependability were tested to determine the potentialities and competence of the apparatus and method. Basically the electronic-sensing device determines the number of particles larger than each of a series of selected sizes, and size distribution is computed from the data. After size distribution is determined, the concentration of sediment can be obtained. If the commercial instrument proves capable of accurate size analysis of sediment samples it will fill a need in the laboratory. However, development of an instrument for field determination of concentration is the ultimate goal.

If electronic sensing can determine accurately the size of an individual particle, the change in resistance caused by a cloud of particles in a larger aperture can perhaps be measured to give sediment concentration directly. Also measurement of concentration within limited size ranges might simplify direct determination of total sediment concentration. Electronic sensing is important because of its possible future use for determining sediment concentration in the field.

Tests of the commercial instrument were made both on known size distributions of various materials and on natural sediment samples of unknown size. The investigation was divided into two main parts: Instrument testing on known particulate systems to establish operator technique, and analysis of samples of known and unknown sizes. Some of the first tests were run to evaluate effects of temperature, counted volume, and electrical environment on the operation of the electronic

counter. Later tests were run to establish routine techniques for analysis of unknown samples.

Experience showed that the counter needed several minor improvements for best use over a wide range of sediment sizes. In most applications the instrument is used to count particles of a narrow size range or to monitor suspensions of particles of a known or limited size distribution. Such analyses can be made far more simply and more quickly than an analysis of an average sediment.

Throughout the investigation of the electronic counter, short cuts in methods of instrument operation and in data processing were studied. Because the present method of obtaining concentration involves a long and intricate process of integration to determine the concentrations of many size ranges of particles, the most important short cut would be one to permit direct determination of sediment concentration.

In this report "electronic counter" or simply "counter" will designate the complete set of commercial equipment for electronic sensing of sediment.

2. History of instrument and method--The electronic counter was developed originally for counting blood cells and particles that contaminate fluids and liquid foods in the medical-biological field. In 1956 W. H. Coulter [3]* presented a paper on a high-speed, automatic blood counter and cell-size analyzer.

Mattern, Brackett, and Olson [9] of the National Institute of Health discussed the determination of size and number of cells passing through a small orifice or gate. At the present time (1964) the counter is used widely in particle analysis of powders, slurries, and emulsified materials.

3. Basic principles of the electronic counter--The electronic counter was developed to measure and count individual particles as they pass through a small aperture. A known volume of a conductive liquid (electrolyte) containing the particles in dilute suspension is drawn through the aperture. Resistance between electrodes at each end of the aperture changes whenever a particle displaces part of the liquid in the aperture. A nearly constant electrical current flows between the electrodes so that the change in resistance produces a voltage pulse that is proportional to particle volume. The electrical pulses are amplified, screened to size, and counted.

The counter has an adjustable threshold level below which electrical pulses are not counted. The threshold can be set so high that none or only a few of the largest particles are counted. Then it can be lowered in successive steps and a count of pulses larger than each step can be taken. The relation of pulse height

* Numbers in brackets indicate references listed on page 80.

to particle volume can be established by calibration with particles of a known size. Thus, the number of particles in each size range can be determined. Threshold adjustment can provide only for a 3 to 1 range in particle diameter. However, adjustments of amplification (gain) and voltage across the electrodes permit analysis of particle diameters over a range of 20 to 1. If apertures having diameters of 50, 140, and 400 microns are used, a range of particle diameters from 1 to 160 microns can be measured. Because the measurement is of particle volume, particle diameter merely expresses the diameter of a sphere that has the same volume as the particle.

II. METHOD OF OPERATION

4. Equipment location and power supply--The equipment should be placed on a rigid table or bench that has ample room for each component. Crowding of any part increases the probability that controls or settings will be inadvertently bumped or jolted out of adjustment. The instrument should be at a height that is comfortable for operation from a chair or stool. A second table placed near the instrument provides space for preparation of samples, electrolytes, and dispersing agents. The location should be as free as possible from vibration and airborne dust. A dust cover is recommended whenever the equipment is not in use. The apparatus may have to be electrically shielded if it is near high-frequency devices, fluorescent lights, calculating machines, starters, and similar sources of electrical noise or radiation.

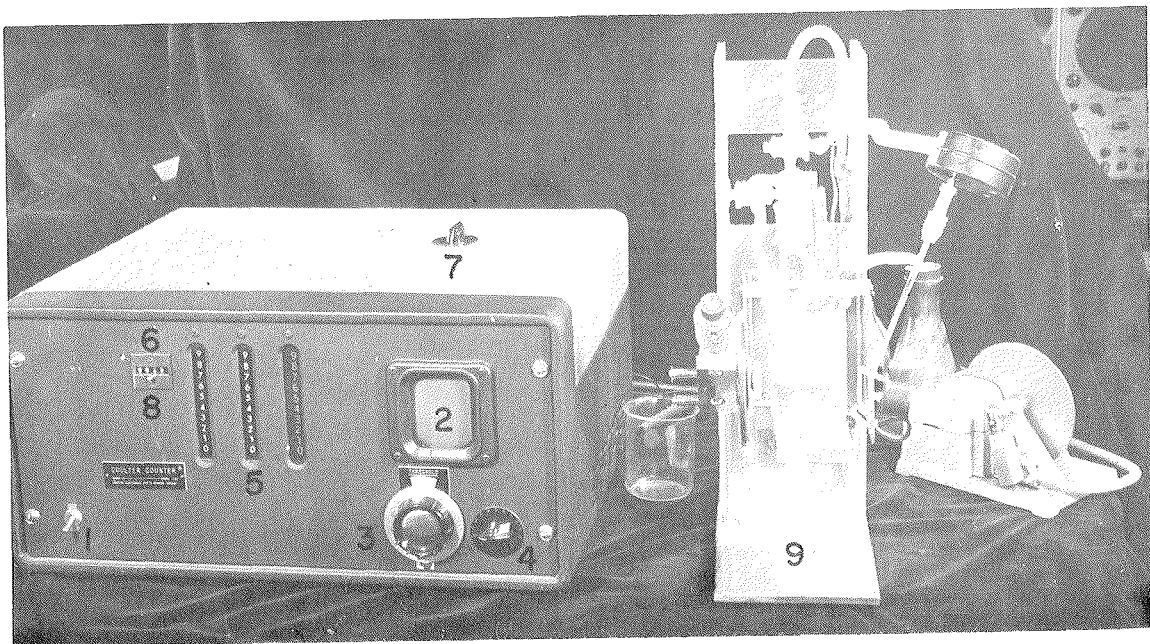
A regulated power supply relatively free from fluctuations, is necessary. The electrical equipment must be grounded carefully to protect operators from high voltages and to minimize external electrical interference.

5. Components of the counter--The electronic components form one main section of the electronic counter. These are housed in a cabinet that contains the counting system and controls for threshold, gain, aperture current, and various minor adjustments. The external features of the cabinet (Fig. 1) are: (1) an on-and-off switch; (2) an oscilloscope; (3) a threshold dial and vernier; (4) an aperture-current selector dial, or I-current dial, (with ten separate settings); a digital recorder consisting of (5) a series of three decade counters, and (6) a digital register; (7) a gain-selector switch (with six separate settings); and (8) a count reset switch.

The sample stand (9 on Fig. 1) with auxiliary equipment is the other of the two main sections of the counter. (See Figs. 1 & 2.) The principal components include the sample platform, sample beaker, motor-driven agitator, aperture tube, microscope, vacuum supply, volume-control manometer, tube-flushing beaker, and trap flask.

The platform (1 on Fig. 2) for the sample beaker may be moved up and down on a guide rod. It is held in position by a supporting spring. A 300-ml sample beaker (2) having a bottom that is rounded to simplify the suspension of the particles sets on the sample platform. The sample is agitated by a propeller on a stirring rod (3), that can be driven at a selected speed (4) by a brushless motor (5). The propeller on the end of the stirring rod revolves in the sample beaker to keep the particles in suspension.

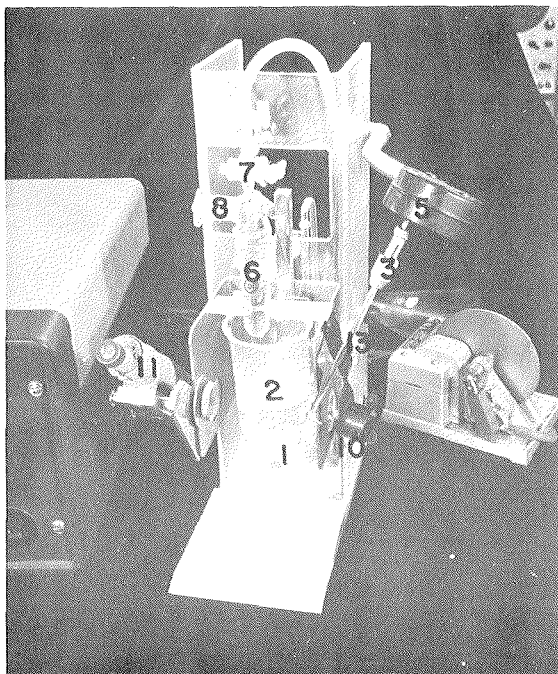
A stopcock and connections unit provides a mounting (below 7, 8) for the aperture tube (6); it allows alternate connections through stopcock (7) to the vacuum supply (through the trap flask), to the mercury manometer (between 15, 16) or to the auxiliary flushing system, which consists of stopcock (8) and a tube to the



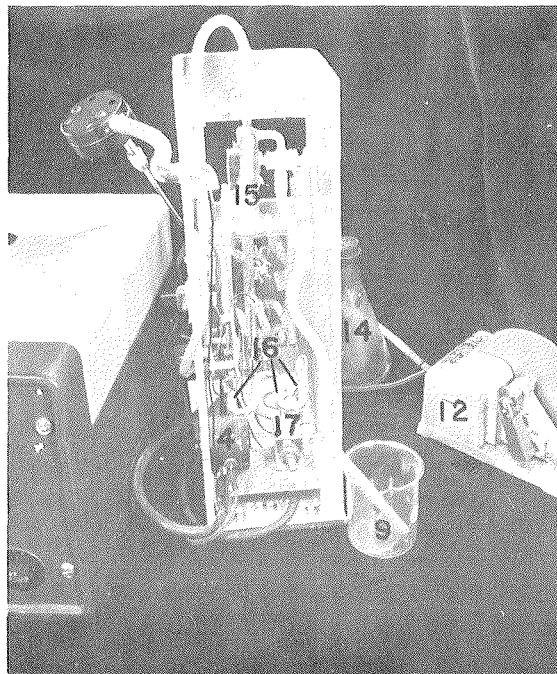
EXPLANATION

Part Number	Description
1	On-off switch
2	Oscilloscope
3	Threshold dial
4	Aperture-current selector
5	Decade counters
6	Digital register
7	Gain-selector switch
8	Count reset switch
9	Sample stand (Fig. 2)

FIG. 1--ELECTRONIC-SENSING EQUIPMENT



Front



Rear

EXPLANATION

Part Number	Description
1	Sample platform
2	Sample beaker
3	Stirring rod (agitator)
4	Agitator speed control
5	Agitator motor
6	Aperture tube
7	Vacuum stopcock
8	Tube flushing stopcock
9	Auxiliary beaker
10	Aperture light
11	Microscope
12	Vacuum pump
13	Vacuum control
14	Trap flask
15	Mercury reservoir
16	Start-and-stop electrodes
17	Volume selector switches

FIG. 2--SAMPLE STAND AND ACCESSORIES FOR ELECTRONIC SENSING

auxiliary beaker (9). The open top of the aperture tube is joined to the stopcock unit with a ground-glass joint that is sealed with vacuum grease. The aperture is a small opening in a quartz disc that is fused into the side of the aperture tube near the bottom. The area near the aperture is lighted (10) and a microscope (11) can be used to observe sediment particles in suspension near the aperture, or particles plugging the aperture.

A pump (12) develops a controlled vacuum (13) in a trap flask (14). The flask prevents accidental damage to the pump if sediment in suspension is drawn into the vacuum system. The vacuum is connected to the stopcock unit (7, 8) through a hose so that the vacuum can be used to start flow through the aperture and to unbalance the mercury in the volume-control manometer. The manometer consists of a mercury reservoir (15), glass tubing, and start-and-stop electrodes (16). As the mercury drains backward through the manometer, the start-and-stop electrodes limit the particle count to a definite volume of sample as selected by the volume-selector switches (17). An auxiliary connection to a beaker of electrolyte (9) provides for flushing or draining the system.

The electronics cabinet is connected to electrodes that span the aperture and also to those in the volume-control manometer (Fig. 3). Power for the vacuum pump and for the agitator motor comes from the power supply in the cabinet. Voltage pulses from the passage of particles through the aperture are fed into the electronic circuits in the cabinet. These circuits provide for amplifying, screening, counting, and displaying the pulses on the oscilloscope.

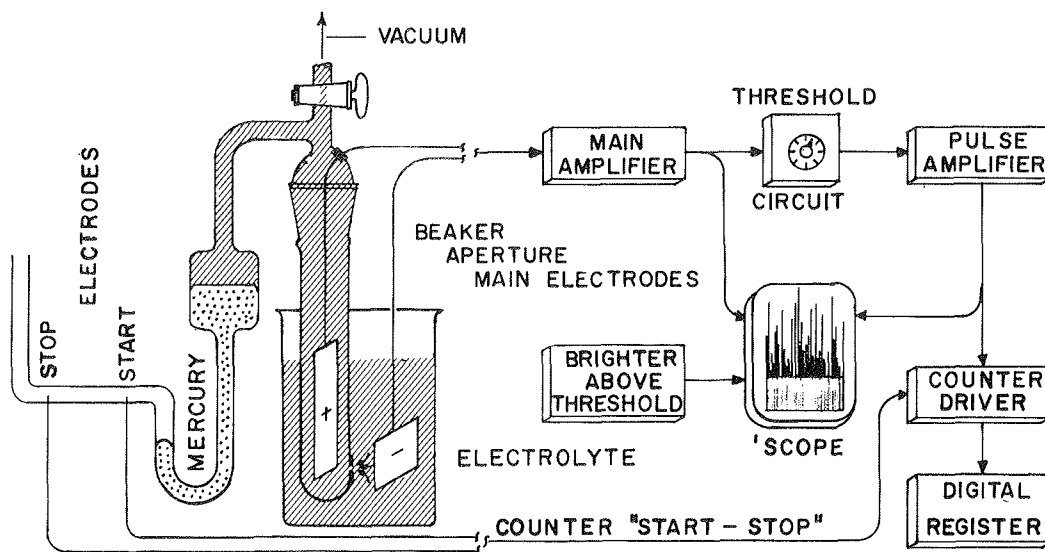


FIG. 3--SCHEMATIC DIAGRAM OF ELECTRONIC-SENSING SEDIMENT ANALYZER

6. Electrolyte--The first step in preparing for analysis of a sample is to select a suitable electrolyte. The requirements for an electrolyte for use in the counter are: a liquid that has a dielectric constant of 10 ohm-cm or more; that is miscible with all other liquids used; that will dissolve some salts to form ions but will not dissolve the sample particles; that has rather low volatility, toxicity, flammability, and cost. For sedimentation work a 1-percent solution of sodium chloride in distilled water is satisfactory and easy to prepare. It has a resistivity of 55 ohm-cm. If resistivity is much lower, accuracy is lost; and if it is much higher, electrical noise levels are a problem. Many different electrolytes have a satisfactory level of resistance. A choice of electrolytes can be based on convenience and on the material to be analyzed.

Even the best distilled water contains some fine particles. Unless the particles in the sample are large in comparison with the particles in the electrolyte, the electrolyte should be filtered to remove fine particles that might interfere with the analysis of the fine sediment particles. Most samples from natural streams contain some sediment particles so fine that the electrolyte should be filtered. As an alternative to filtering, the background count in the electrolyte may be determined as a basis for correcting the analysis, but usually filtering is more desirable. A simple system composed of a filter flask, a holding clamp, and a graduated funnel with fritted-glass filter support was used for filtering the electrolyte. A fine-membrane filter placed over the coarse fritted-glass filter further reduced the electrolyte particle interference to the 0.5 micron level.

The addition of 0.1 percent of formaldehyde prevents growth of microorganisms in the electrolyte [2].

7. Sample preparation--If the sample to be analyzed contains only a small amount of material, the entire quantity may be used in the counter, but generally, only a small sample of the available material can be used. A good sample must accurately represent the parental distribution. Representative samples of dry sediment may be obtained by splitting the original sample with microsplitters [1, 10] or by quartering and recombining opposite quarters of the sample repeatedly [8]. Representative samples from a suspension of particles may be obtained by pipette withdrawal from a thoroughly mixed sample [5]. Samples may be added to the electrolyte or diluted with electrolyte as necessary. Occasionally, a sample in dilute suspension may be analyzed directly using native water as the electrolyte. The native water may be diluted with distilled water if desirable.

Usually, organic matter in the sample should be removed. Sometimes the organic matter can be conveniently burned out of a dry sample. If the sample is in water the organic material may be removed by adding hydrogen peroxide and heating [6]. Organic matter must be removed carefully to avoid destroying any sediment particles. If treatment increases the tendency of fine particles to cling together, extra effort may be required to disperse the particles satisfactorily. Normally, the use of a dispersant is desirable regardless of prior treatment of the samples.

Particle concentration should be somewhere between 1 and 1,000 ppm by volume depending on the aperture size to be used and the particle size. Concentration must be high enough to provide statistically significant counts, but low enough to avoid an undesirably large number of coincidental passages through the orifice (Section 19). Also, if the concentration is kept low the sample is more easily dispersed. The sample may be diluted with additional electrolyte if necessary to reduce the coincidence effect or to provide additional volume for continued counting. Dilution simply reduces the number of counts. The counts can be overlapped to obtain the relationship of the counts before and after dilution.

8. Basic procedure for analysis--A size analysis with the counter is begun by placing a sediment sample in the 300-ml round-bottom beaker which is then nearly filled with filtered electrolyte to which a dispersant has been added. Next the beaker is placed on the sample platform (Fig. 2) and a spring attachment raises the beaker to the operating position in which the opening in the aperture tube is about an inch above the bottom of the beaker. The agitator motor is placed so that the stirring propeller is near the bottom of the beaker. Then the motor is started, and its speed is adjusted to obtain a uniform sediment suspension at a minimum speed.

An aperture size is chosen to fit the size range expected for the particles in the sample. Normally the smallest aperture that will not plug with sediment is chosen. (If the range of sediment sizes is large, additional apertures of smaller sizes may be required, but the discussion of the basic procedure will be confined to analysis with a single aperture.) The microscope can be used to note the size of particles in suspension as a basis for estimating the size of aperture required. Also when the sample is being drawn through the aperture, the microscope can be used to observe any plugging. When the aperture is plugged, the counting rate is reduced abnormally.

Particles are drawn through the aperture by opening a stopcock at the top of the aperture tube (Fig. 3). The controlled vacuum created by the vacuum pump draws fluid simultaneously from the sample beaker and pulls the mercury into the upper reservoir to unbalance the mercury manometer. After the mercury is drawn beyond the starting switch for the counter, the stopcock is closed to shut off the vacuum. The siphoning action of the mercury manometer continues the flow of the sediment suspension through the aperture. The mercury column drains from the upper reservoir through the volume-control manometer toward the starting electrode. When the mercury reaches the starting electrode, the counter drive in the electronic-components cabinet is activated. As a particle passes through the aperture, it displaces its own volume of electrolyte and changes the resistance in the aperture. The change in resistance produces an electrical pulse that is picked up and amplified. The amplified voltage pulse is displayed on an oscilloscope as a vertical spike whose height indicates the relative volume of the particle. The amplified pulses are fed simultaneously to a threshold circuit having a dial adjustment for screening the pulses according to size. The pulses smaller than the threshold level are not counted, but those that exceed the threshold level have bright segments above that level (as shown on the oscilloscope), and they are

counted individually. After the mercury has metered a certain volume of sediment suspension through the aperture, a stop electrode deactivates the counting circuit and the number of particles larger than the threshold size can be read from the digital register. The electrodes are spaced accurately to meter a definite volume of sample. Some instruments have provision for connecting the circuit to alternate electrodes to provide a choice of two or more sample volumes. The counter used in these tests had three metered volumes from which to choose.

Generally the threshold dial is first set high enough so that all, or all but a very few, of the largest particles are screened out. Such a setting gives a zero, or near zero, count to define the coarse end of the size distribution. A series of counts is taken at the first threshold setting, and additional series of counts are taken at 10 or more successively lower threshold settings. When the lower limit of the threshold dial is reached, the readings may be extended to smaller sizes by increasing the aperture current and by increasing the gain setting.

Particles counted at the highest threshold settings are large and each represents a significant part of the total size distribution. The numbers counted are small, and random variations cause large percentage variations in the numbers in each count. Therefore, the count should be repeated six or eight times to obtain a good average. At lower threshold settings more particles are contained in each count, the readings are less variable, and two or three duplicate counts are generally sufficient.

9. Combination of apertures for a wide range of sizes--A given size of aperture can be used only for analysis of particles having nominal diameters from about 1 to 40 percent of the aperture diameter. A few particles as large as 50 percent of the aperture diameter may be present in the sample if the aperture does not clog too frequently. The largest particle size in the sample to be analyzed determines the smallest aperture that can be used. Normally, this aperture is chosen for starting the analysis. Inspection through the microscope, absence of excessive aperture plugging, and data on the coarsest particles soon show whether the proper aperture was chosen. Analysis is continued throughout the usable range of the first aperture and then is extended to smaller sizes of apertures if additional definition of the fine-particle distribution is required. Apertures are chosen so that the particle sizes analyzed in one aperture overlap those in the next.

As an analysis is continued to smaller apertures, the coarser particles in the sample must be removed by sieving, or by sedimentation methods. Also the sample may have to be diluted to provide enough volume for analysis or to reduce the number of fine particles to be counted.

Sediment particles from 1 to 160 microns in diameter can be analyzed in a series of three apertures that have sizes of 50, 140, and 400 microns. The manufacturer of the counter can supply aperture sizes ranging from 11 to 2,000 microns.

10. Care and cleaning of instrument--The sample beaker and the aperture tube must be kept clean and free from air bubbles. Bubbles in either one cause spurious counts, and the bubbles cling to dirty glass surfaces more readily than to clean glass. All auxiliary glassware, including the filtering system and sample pipette, must be washed occasionally in a detergent solution to remove any film that clings to the glass.

Normally the mercury manometer will operate for long periods of time without cleaning. However, in the bulb-shaped reservoir near the top of the manometer the sample in the aperture tube comes in contact with the mercury surface, and in time some particles will work down into the mercury. Also, some particles may be drawn into the metering section while the vacuum pump is being regulated. If sediment particles form a scum on the platinum electrodes that start and stop the counter, the metered volume may be inaccurate, and the manometer must be cleaned. The metering section and the platinum electrodes can be cleaned by washing, rinsing, and drying with nitric acid and ethyl alcohol. After the manometer has been cleaned, clean mercury is added through the free end of the manometer to fill the manometer to the widest part of the upper reservoir. This level gives the best metering accuracy. If separations form in the mercury column of the manometer, they may be drawn into a small round reservoir called the coalescing section, where they are eliminated.

Electrical connections must be kept tight, dry, and free from dust. The connections on the sample stand require the most attention because they may become encrusted from spillages.

When not in use, the instrument and sample stand are covered with a plastic sheet to protect them from dust.

III. ELECTRICAL RESPONSE

11. Change in resistance resulting from particle passage--If the electrical effect of a particle in the aperture is expressed in terms of a right cylinder that is aligned with the aperture axis, it can be shown [4] (Appendix, Section 38) that the change in aperture resistance, ΔR , caused by the particle is

$$\Delta R = R - R_0$$

$$= \frac{\rho_0 V}{A^2 \left(\frac{1}{1 - \rho_0/\rho} - \frac{a}{A} \right)} \dots \dots \dots (1)$$

When:

R = resistance with a particle in the aperture
 R_0 = resistance of electrolyte without the particle
 ρ_0 = electrolyte resistivity
 ρ = effective resistivity of the particle
 V = particle volume
 A = aperture area normal to aperture axis
 a = area normal to aperture axis of equivalent right cylinder for the particle as oriented in the aperture

Equation 1 shows that response ΔR , depends almost directly (see also discussion of ρ_0/ρ on next page) on the resistivity of the electrolyte. This resistivity can be measured separately for each analysis. However, one electrolyte is commonly used for many analyses. The resistance is then measured once and checked occasionally. Temperature is held essentially constant or the effect of temperature changes on the resistivity is determined and corrections are made for temperatures different from that for which the resistivity was measured (Section 25.)

The last term in the denominator of Equation 1 is not significant for very low a/A ratios. For spherical particles with diameters that are 30 percent of the aperture diameter the ratio a/A is 1:11 and the equation indicates a greater than linear response. The effect of the a/A ratio is correctable whenever correction is justified. However, the larger the particle in relation to the aperture, the greater the current density around the particle at the time of passage through the aperture and the greater the electrical heating of the electrolyte. The heating of the electrolyte momentarily lowers the resistance and reduces the response somewhat. Also for the larger particles, the entire volume may not be entirely within the aperture and thus may not cause maximum response at the instant of passage. Elongated particles that align with the stream flow may have the greatest reduction in response. Preliminary investigations indicate that the effect of the a/A factor is negligible if particle diameter is less than 40 percent of aperture diameter. If the a/A term is negligible, then the response ΔR , is inversely proportional to the square of the aperture area.

Normally, particle resistivity is effectively many orders of magnitude greater than that of the electrolyte. Powders of metals that are good conductors behave like nonconductors, perhaps because of oxide surface films and ionic inertia of the Helmholtz electrical double layer and associated molecules at the surfaces of such particles [2].

Particle density does not affect response unless the particle surface contains large pores. Particle shape has little effect on response; the distortion of the electrical field in the aperture probably conforms to the general surface without following each surface indentation or protrusion on the particle.

Within the limits of particles that are likely to be found in sediment samples the shape, structure, density, and resistivity of the particles have little effect on the relation of particle volume to electrical response. Therefore, for analyses in a given electrolyte at a nearly constant temperature, and for particles with diameters that do not exceed 40 percent of the aperture diameter, the response, ΔR is proportional to particle volume and

$$\Delta R K = V \quad (2)$$

Where K is a constant to be established by calibration for a given aperture, electrolyte, and temperature.

12. Height of voltage pulse--If an electrical current is passed through the aperture, each change in resistance, ΔR , will produce a change in voltage across the aperture, thus:

$$\Delta E = G I \Delta R \quad (3)$$

When

ΔE = voltage change or magnitude of voltage pulse

G = gain factor in the circuit (determined from gain setting)

I = electrical current through the aperture (determined by the setting of the aperture-current selector dial).

If Equations 2 and 3 are combined and solved for the volume, V, of the particle that causes the voltage pulse ΔE ,

$$V = K \Delta E / (G I) \quad (4)$$

Relative values of G and I (based on the dial settings of the counter) can be used in equation (4) because the calibration constant K will take care of the difference between relative and absolute values.

13. Threshold dial and circuit--The pulse height, ΔE , is the record of a single particle passage, that is displayed on the oscilloscope and that is screened and counted. The threshold circuit provides the scale by which pulse heights are screened. The threshold dial is arbitrarily divided into equal units from 0 to 100. A dial setting establishes a sill, or minimum pulse height, that must be exceeded if a pulse is to be counted. On the oscilloscope, pulses that are below the pulse height set on the dial are shadowed and those that extend above that height are brightened above the reference level. The threshold-dial setting can be used in Equation 4 in place of ΔE . Then V becomes the minimum particle volume

that will be counted for this threshold-dial setting and for specified gain-dial and aperture-current dial settings.

14. Gain setting and circuit--The pulse height, ΔE , is extremely small until it is amplified. Before it is displayed on the oscilloscope, or screened by the threshold-dial setting, the pulse passes through an amplifying circuit that has an adjustable gain. The gain setting has six positions. If the gain at the first setting is given a unit value, then successive settings, G' , have gains, G , of 1.41, 2.00, 2.83, 4.00, and 5.67. The variable gain permits scaling of a much wider range of R values than would be possible on a threshold dial having a single gain setting.

15. Aperture-current selector dial and circuit--Ten settings are provided for varying the electrical current through the aperture by changing the electrical resistance in series with the aperture. At the first setting an electrical resistance of 25.6 megohms is placed in series with the aperture resistance, which is usually less than 0.1 percent of that amount. A constant voltage of 300 volts is imposed on the total resistance to give the aperture current, I . The relatively minor change in total resistance that is caused by the passage of a particle through the aperture is so small that the I current is not changed significantly. However, the voltage pulse, ΔR , which is measured across the aperture only, is significantly changed by the passage of the particle.

At the second setting for the aperture-current selector dial (or more simply the I -current dial), the electrical resistance in series with the aperture is 12.8 megohms and the I current is doubled. As long as the resistance across the aperture plus the small fixed resistance in the aperture circuit is a very small part of the total resistance, each increase in dial setting cuts the total resistance in half and doubles the I current. At the higher settings the aperture resistance becomes a significant part of the total circuit resistance and the change is no longer proportional. (See Table A-4 in the Appendix.) The table is made up in terms of a scale factor, F , for each I -current dial setting and the scale factors can be substituted for $1/I$ in Equation 4.

At all but the first few settings of the I -current dial the aperture resistance affects the I current. Resistance is measured directly across the aperture by means of a d-c voltmeter (Appendix, Section 39).

16. Calibration constants--Equation 4 is the basis for calibration of the counter. The equation can now be stated as

$$V = k_v T F/G = k_v S_e \dots \dots \dots (5)$$

When

T = threshold-dial setting

F = scale factor corresponding to the I -current dial setting

G = gain setting ratio (1.00, 1.41, 2.00, 2.83, 4.00, and 5.67 for settings 1 to 6 respectively)

S_e = $T F/G$ or electrical size of the particle

k_v = a calibration constant for relating volume of particle to S_e

Also

$$V = \frac{\pi d^3}{6} = k_v S_e$$

$$\text{And } d = k_d \sqrt[3]{T F / G} = k_d \sqrt[3]{S_e} \dots \dots \dots (6)$$

When d = diameter of a spherical particle or nominal diameter of an irregular particle

k_d = calibration constant for relating nominal diameter of the particle to S_e

$$\text{Then } k_v = \pi k_d^3 / 6 \dots \dots \dots (7)$$

A constant for relating particle size to electrical size applies only to one aperture and one electrolyte that is held at a nearly constant temperature (or for which temperature correction has been made).

17. Calibration with uniform particles--If uniform particles (used in this report to mean particles of limited size range) with a known mean size as determined by microscope or by other accurate means are available, the counter may be calibrated by direct comparison. This method is fast, simple, and accurate. The particles are placed in suspension in a concentration so low that the primary coincidence effect will be less than 2 percent. (See Section 19.) Particle counts are made repeatedly on this suspension. Because the particles are nearly the same size, the pulses on the oscilloscope will be of nearly uniform height. The instrument is adjusted so that average pulse height is between 10 and 30 on the threshold dial; gain setting is 3 or 4; and aperture-current dial setting is about 2. The threshold dial reading that corresponds to the average pulse height should be determined visually. The threshold dial is then set at 1/2 the average pulse height and several "full" counts, n_f , are taken to obtain an average n_f . Next the threshold is set at 3/2 the average pulse height and several counts are taken to establish an average "oversize" count, n_o . The median, or half count, n_h , can be computed from

$$n_h = n_o + (n_f - n_o) / 2 = (n_f + n_o) / 2 \dots \dots \dots (8)$$

It is assumed, and the assumption can be proved by test, that a system of particles of a narrow size range has an approximately normal distribution of sizes. Therefore, the half count, n_h , is the number of larger than average size particles in the suspension. Then, if the threshold-dial setting, T , that gives the half count of Equation 8 is found by trial, the average particle diameter, d , and the instrument setting factors, T , F , and G , can be used to determine the calibration constant k_d in Equation 6. (See Appendix Section 40.)

The method of calibration based on uniform particles is ideally suited to checking the relative effects of instrument settings. The electrical size, S_e , should remain unchanged if: (1) T and G are both doubled, (2) T is doubled and F is reduced by half, or (3) G and F are both doubled. If one variable is held constant and the other two are varied, the particle count will not remain the same unless the two variables are in correct ratio. Particle counts over a wide range

of variation of T and F were used to determine whether the zero of the threshold scale was at true zero and to adjust to true zero when necessary. Similarly, combinations of T and G were used to adjust the gain in our counter to more precise relations of the gain steps than those in the instrument originally. The gain adjustments required an actual change in the individual electrical resistances for each setting of the gain circuit.

18. Calibration with particles in known concentration--The counter can also be calibrated with a suspension having a known volume concentration of particles [4]. The particles should be free of pores or excessive surface roughness and have diameters between 2 and 40 percent of the diameter of the aperture. The density of the solid particles should be known accurately or determined carefully.

The volume of particles V_p , in the metered volume of suspension will be

$$V_p = (v_s/V_s) (W_p/\rho_p) \dots \dots \dots (9)$$

When

v_s = volume of the suspension that is metered for each count
 V_s = total volume of suspension made up for measurement
 W_p = total weight of particles in the total suspension
 ρ_p = density of the particles (actual, not bulk)

A size analysis with the counter consists of measurements of the numbers of particles $n_1, n_2, n_3, n_4, n_5, \dots, n_n$ that have electrical responses greater than $S_1, S_2, S_3, S_4, S_5, \dots, S_n$, which are a series of electrical-size settings that cover the range of sizes in the sample. Let S_a represent average electrical size of particles between two adjacent size settings and Δn represent the difference between counts for the same two settings. Then the total volume of particles is proportional to the summation of the products of Δn and S_a for all the intervals between adjacent size settings. Thus,

$$V_p = k_v \Sigma(\Delta n S_a) = (\pi/6) k_d^3 \Sigma(\Delta n S_a) \dots \dots \dots (10)$$

Because V_p is known from volume concentration and metered volume and $\Sigma(\Delta n S_a)$ can be obtained by arithmetic summation of the data from the counter, the relation, k_d , between electrical size and particle diameter can be determined. This value of k_d should be the same as that obtained from calibration with uniform particles.

Once the count at a definite size level has been established, the sample for which the volume concentration is known can be used to check drift in calibration or normal variations in instrument readings. However, if the sample having a known volume concentration has a wide range of sizes, the change in count will not be so sensitive to changes in instrument factors as for a more uniform sample of small size range. Also, errors from nonuniform dispersion are larger if a sample has a wide range of sizes.

19. Coincidence effects--Previous discussion has shown the effect of a single particle in the aperture of the counter. Particle concentration in the

sample to be analyzed should be high enough to provide statistically significant numbers at the coarsest sizes for which the distribution of sizes is to be defined. As concentration of particles is increased, the probability also increases that more than one particle will be in the sensing zone of the aperture at one time.

A study of the electrical effect of particles passing through a small orifice was made by Mattern, Brackett, and Olson [9]. They determined that for an aperture having a diameter of 100 microns and a length of 75 microns a critical volume, or sensing zone, about three times the aperture volume exists in which two or more particles will not produce individual impedance pulses. Fig. 4 shows the sensing zone of the aperture; some particle combinations in the zone, and the resulting electrical pulses. Two particles of about the same size, following each other at about the length of the sensing zone, register as a single particle of the given size. If they follow more closely, they register as a single particle that is larger than the individual particle size.

The primary effect of coincident passages is a loss of count. Mattern, Brackett, and Olson showed that the coincidence loss followed the Poisson distribution. A secondary coincidence effect is a possible, occasional increase of count of large particles. However, to register as an oversize particle, two particles of nearly the same size must pass through the aperture very close together. The probability of such restrictive occurrences is, therefore, small.

Coincidence effect can be determined experimentally [2, 9]. A high concentration of particles in suspension can be analyzed, and successive dilutions of the same suspension can be run until the concentration is so low that coincidence becomes negligible. The error in the analysis for a high concentration can then be computed from the deviation from a straight-line extension of the low-concentration analyses. The manufacturer of the counter obtained the following equation for p , the coincidence factor,

$$\begin{aligned} p &= 2.5 (D/100)^3 (500/V_m) \dots \dots \dots (11) \\ &= 1.25 (D/10)^3 / V_m \end{aligned}$$

In which

D = aperture diameter in microns

V_m = metered manometer volume in microliters (usually the nominal metered volume is sufficiently accurate)

A count-loss correction, n'' , to the observed count, \bar{n}' , can be computed as

$$n'' = p (\bar{n}'/1,000)^2 \dots \dots \dots (12)$$

The correction to the count checks the Poisson function within 1 percent up to a ratio of count loss to actual count of 1:10. Equations 11 and 12 were based on counts of uniform particles, and correct only for loss of counts. No adjustment has been developed to correct for the few oversized pulses that may be caused by two particles of about equal size passing through the aperture very close together.

The loss of count correction for a range of analytical conditions was computed from Equations 11 and 12. (See Table A-3 in the Appendix.)

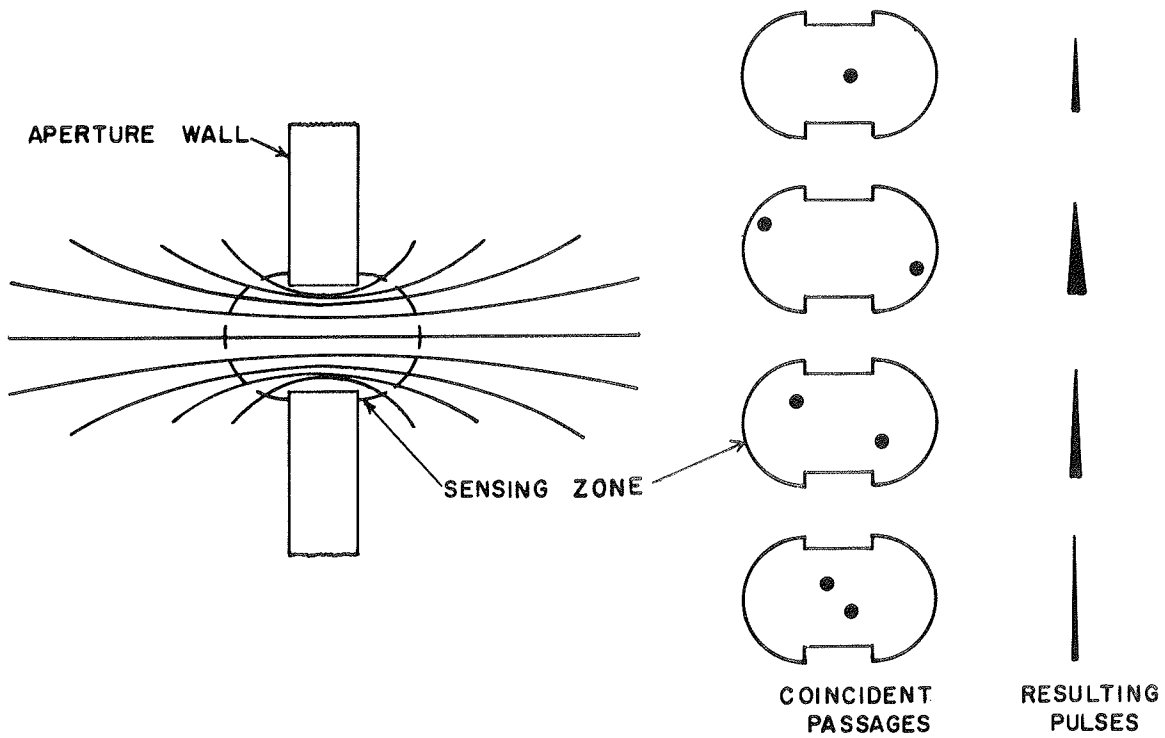


FIG. 4--SENSING ZONE AND COINCIDENT PASSAGES OF PARTICLES

Reference 9

IV. TEST PROGRAM

20. Operator training--Preliminary analyses with the counter were made to give the operator experience with the equipment and to develop skill in its use. Most operators require several days time to understand the instructions that come with the counter; to learn how to handle the equipment without too many mistakes, and to become reasonably accurate and proficient in recording and computing the data.

21. Possible sources of incidental errors in analysis--As soon as operator errors had been minimized, a study of other possible errors in the analysis was begun. Several sources of errors were considered. Many were incidental to unfavorable operation or because of inadequate maintenance of the equipment. Most of these were not subject to quantitative evaluation but their effects could be observed and procedures could be used to reduce them to negligible size.

a. Electrical interference may come from electrical switching, sudden voltage changes, or bad contacts. The interference may be in the counter or may reach the counter in the power supply or from radiation by external sources.

The counter was first set up in a part of the laboratory where interference was so bad that consistent analyses were almost impossible to obtain. The counter was then moved to a location remote from most other electrical equipment. The power supply at the second site was less variable than at the first site but a constant voltage transformer for the power supply was desirable even at the second site. If a suitable location for the counter is not available, the equipment can be enclosed in a grounded metal shield.

b. Air bubbles may form in the aperture tube. Air in the aperture tube destroys the relative incompressibility of the system, and the metered volume of sample drawn into the aperture during the counting period will not be exactly equal to the metered displacement of the mercury. Air bubbles on the inner electrode cause electrical noise pulses that may be counted. Air bubbles in the aperture tube can be eliminated by draining the tube and refilling it with electrolyte. If a dispersing or wetting agent is used in cleaning the inside of the aperture tube, bubbles or grime are less likely to cling to the tube.

c. Transient temperature differences, (or differences in electrolyte), between the sample beaker and the inside of the aperture tube may cause rapid changes in aperture resistance and cause voltage pulses that will be counted. The instrument should be operated in a relatively constant room temperature, and samples and electrolyte should be kept at that temperature for some time before analysis.

d. Aperture plugging may cause abnormal counts. Usually, aperture plugging results in particle counts or counting rates that are so far out of line with adjacent counts as to be obviously incorrect. A glance through the aperture

microscope will reveal any plugging. Generally, the aperture can be cleared by opening the stopcock leading to the auxiliary flushing system. Sometimes the aperture opening will require rubbing or brushing. Any counts made while the aperture is plugged should be disregarded.

The aperture may plug because particles are not fully dispersed, but the usual reason is that the aperture is small in relation to the largest particles in the sample. Occasional plugging from odd-shaped particles is to be expected, but frequent plugging indicates that a larger aperture should be used or some of the coarsest particles should be removed from the sample before it is analyzed.

e. Impurities such as organic matter in the sample may destroy the accuracy of analysis. Also impurities in the mercury at the volume-metering section may upset the volume accuracy. Careful cleaning of the equipment and of the auxiliary glassware and adequate maintenance of the manometer will avoid most of the effects of contamination.

f. Nonuniform dispersion in the sample beaker will obviously affect the analysis. Fine sediments and sediments of low densities are easy to disperse uniformly with the stirring mechanism that is an intrinsic part of the counter. Sediment particles usually have a specific gravity greater than 2.5, and particles over 100 microns in diameter are difficult to maintain in suspension in the sample beaker. The speed of the stirring motor is adjustable, but if it is too high air bubbles may be drawn into the sample and pass through the aperture to be counted and to accumulate in the aperture tube. If the particle count changes with a small change in speed of the agitator motor, the dispersion is probably not uniform. A relatively high stirring speed can be used while counting the coarser particles and while the sample covers the aperture to a depth of an inch or more. Then the stirring speed can be reduced slightly when the counting level is lower and the sample does not cover the aperture so deeply. However, the particle count is supposed to include the coarser particles regardless of the counting level and, even a slight departure from complete dispersion is allowable only after the total count becomes so great that the number of the larger particles is relatively very small.

g. Particles sometimes collect in the aperture tube. Most particles that pass through the aperture are carried out of the tube, but some of the larger or more dense particles may collect inside the aperture tube near the bottom. Some of these particles may be drawn up into the portion of the electrically sensitive zone just inside the aperture and be counted. If these particles accumulate excessively, the extra counts may become significant. Accumulation of particles may be reduced by flushing with the auxiliary flushing system, but sometimes only complete draining and refilling of the aperture tube will eliminate the accumulation.

h. Some minor items of procedure and possible sources of incidental errors should also be considered:

Particle counts near the lower limit of the threshold scale should be avoided or viewed with suspicion because the background electrical noise may be great enough to contribute extraneous counts.

At I-current dial settings of 7 or 8 and higher, erratic pulses may show on the oscilloscope. They may indicate overdriving of the amplifying circuit or excessive heating effects because of the large current through the aperture.

The volume of electrolyte in the sample beaker has no effect on the results of analyses unless the electrolyte level drops close to the aperture. However, a very low level combined with intense stirring may permit air bubbles to enter the aperture.

Surface roughness and porosity of the particles probably is not a significant problem for normal sediments. However, indentations, wrinkles, and gross porosity at the surface of particles, such as found in the corn pollen used in some of the calibrations, may have minor effects. Particles with internal voids register as solid particles.

22. Errors inherent in procedure--In contrast to the incidental errors, which are generally small, avoidable, and hard-to-define quantitatively, other possibilities of error are inherent in the procedure or equipment. Although inherent errors are not entirely avoidable, the different sources of these errors can usually be identified. Then the effects of the errors can be reduced or eliminated by proper procedure, by instrument adjustment, or by calibration and correction. Errors inherent in procedure are discussed in the remainder of this section.

Excessive concentration of particles in the sample is one source of inherent error in procedure. Because a concentration that is excessive for one aperture and particle size may not be excessive for another aperture and particle size, arbitrary limits on allowable concentrations are not easy to establish. The largest effect of concentration is probably the coincidence effect that was discussed in Section 19. However, if particle counts are very high, the counting rate may be too fast for the instrument to record with satisfactory accuracy. The total number of counts for a given aperture and metered volume is probably the best basis for determining the corrections that may be necessary.

The manufacturer of the electronic-sensing counter furnished Equations 11 and 12 (p. 25) for correcting the particle count for loss from the effect of primary coincidence. If this coincidence loss is kept below 10 percent and is corrected according to Appendix Table A-3, the other effects of high concentration on counting of the particles are probably negligible. Sometimes, however, complete dispersion of particles in the sample is difficult to achieve at high concentration.

Fortunately, the concentration of particles in the sample beaker can be reduced by dilution with electrolyte. Also, the minimum concentration is not closely related to accuracy. The main requirement is that sufficient particles must be counted to be statistically significant.

At present (1964), the manufacturer's data and explanation of concentration effects are accepted as adequate. However, concentration effects might well be investigated further.

Extreme ratios of particle diameter to aperture diameter are a second source of inherent error. The particle must be large enough relative to the aperture to cause a measurable voltage pulse when the particle passes through the aperture; it must be small enough relative to the aperture so that the relationship of resistance change to particle volume is essentially linear. Equation 1 indicates the basic relationships. According to general observation, the counter operates consistently when the particle diameter is from 2 to 40 percent of the aperture diameter, which is the approximate range suggested by the manufacturer. Eventually, the assumption of a linear relationship should be investigated carefully for a range of particle to aperture diameters.

The range of particle sizes that can be analyzed satisfactorily depends on the aperture or apertures available, or, in other words, the apertures required depend on the range of particle sizes to be analyzed. If necessary, coarse particles in a sample may be removed before analysis. The necessity for maintaining a uniform distribution of particles in the sample beaker sometimes limits the maximum particle size for analysis regardless of the apertures available. Hence, the upper limit of particle size may be determined partly by the ratio of the density of the particles to the density of the electrolyte.

Temperature effects may be significant if operation is not restricted to a narrow temperature range. Changes in temperature of the electrolyte cause changes in the resistance through the aperture, and resultant changes in the relation of particle size to electrical response.

Special tests were made to define the effect of temperature changes on the resistance through the aperture. During the tests, a 1-percent sodium chloride solution (electrolyte) flowed through a 50-, a 140-, or a 400-micron aperture.

In the first tests, the voltage drop across the aperture was measured with a d-c voltmeter while the electronic-sensing equipment was in the usual operating state. A nearly constant voltage was applied to the aperture circuit, which includes (1) the resistance through the aperture and (2) the resistance selected by the aperture-current dial setting plus some additional fixed resistance in the aperture circuit. The observed voltage drop across the aperture was used to compute the aperture resistance (Appendix Section 39). The resistance selected by the aperture-current dial setting and the fixed resistance are grouped together in this report under the term "aperture series resistance" and should be distinguished carefully from the resistance through the aperture or more simply the aperture resistance. In general, the aperture resistance decreased as the temperature increased. (See Fig. 5.) Some irregularities were caused by polarization, line-voltage change, errors in voltmeter readings, and slight changes in characteristics of the electrolyte. The effect of polarization was particularly large for the 50-micron aperture. However, percentage departures of individual

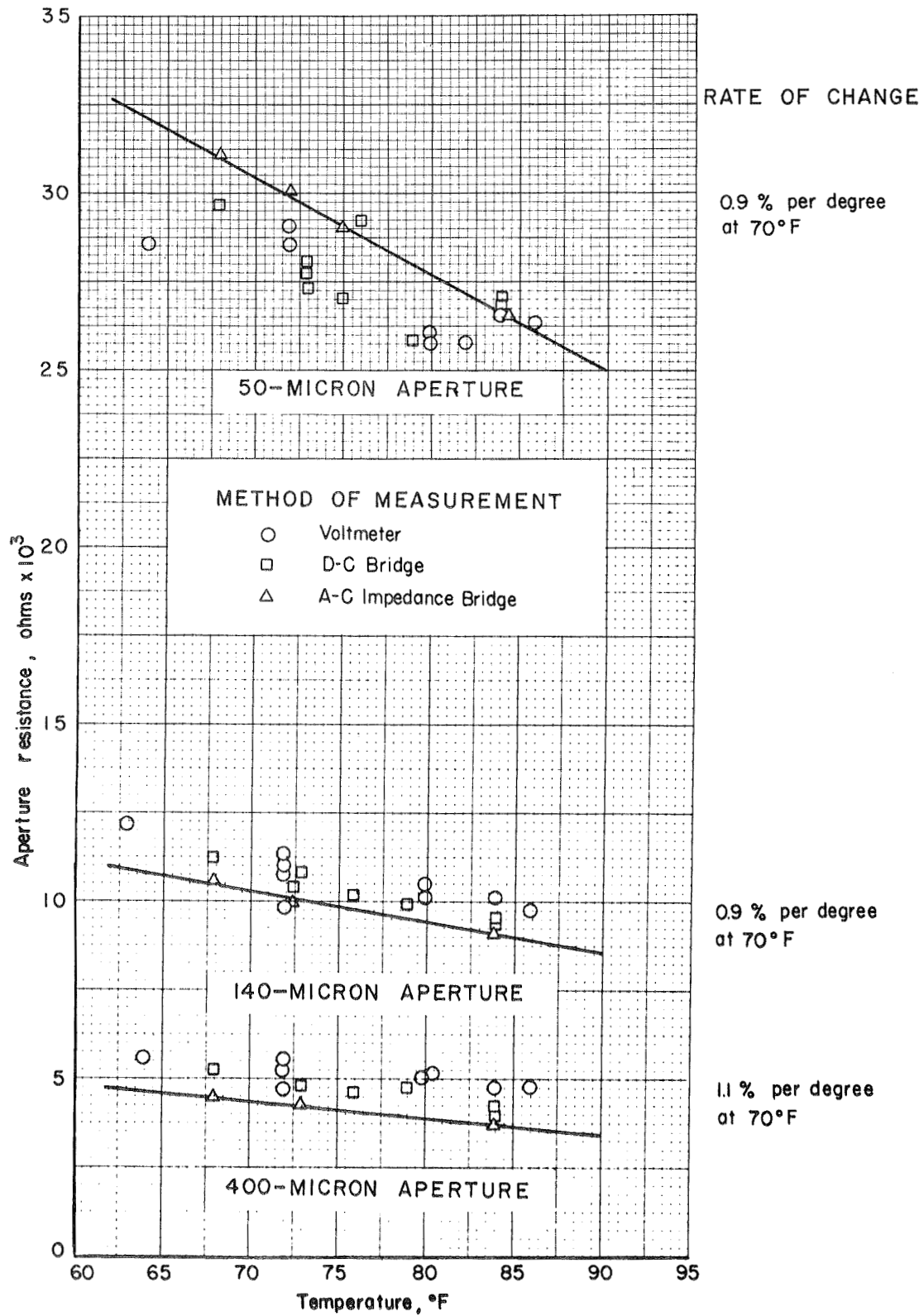


FIG. 5--EFFECT OF TEMPERATURE ON APERTURE RESISTANCE

observations of aperture resistance from an average relation were no greater for this aperture than for the 140- or 400-micron aperture (Fig. 5). The voltmeter measurements gave only a general idea of the relation between aperture resistance and temperature of the electrolyte.

Additional information on the relation of aperture resistance to temperature was obtained from measurements with d-c bridge and a-c impedance bridge circuits while electrolyte was flowing through the aperture. The a-c impedance bridge measurements of aperture resistance avoided polarity effects and seemed to be most consistent. The d-c bridge measurements of aperture resistance were somewhat inconsistent among themselves but, in general, agreed roughly with the a-c bridge measurements. Both the d-c bridge measurements and the voltmeter measurements indicated somewhat greater aperture resistances for low aperture resistances than did the a-c bridge measurements. (See Fig. 5.) Part of the difference may be caused by the slight potential that is generated by the battery action of the electrolyte.

Because the a-c impedance bridge measurements were the most consistent they were used to define relationships between temperature and electrolyte resistance. A decrease in resistance of 1 percent for a 1°F . rise in temperature was indicated for the range of normal room temperatures. This agrees with data obtained by others [2].

Equation 1 shows that the indicated volume of a particle analyzed by the counter will change almost directly as electrolyte resistance changes. Therefore, accurate results can be obtained only if analyses are confined to a narrow range of temperatures, or if results are corrected for temperature effects. One way of correcting would be by computations based on a change of resistance of 1 percent per degree change in temperature; another way would be to use the results of instrument calibration (Section 25).

23. Errors inherent in equipment--For a given aperture, electrolyte, and temperature the electrical size of a particle is determined from the three dial factors T, F, and G (Eq. 5). If particles of known size are used each factor can be calibrated. However, the amount of calibration can be reduced by adjusting the dial factors to conform to simple well-defined relationships.

The threshold dial with its calibration in units from 0 to 100 was taken as the basic size measure. The threshold dial controls rheostat resistance and should be linear. Linearity was also checked by comparison to changes in gain, G, and aperture current factor, F. The zero trim of the threshold dial may require occasional adjustment and should be checked every few months.

Zero trim of the threshold dial was adjusted by a trial-and-error procedure. First, a suspension of particles having a narrow range of sizes was analyzed repeatedly for a constant gain setting and a wide range of F values. The threshold dial was set to give about a median count for the first F values. Then, the

threshold was adjusted by trial and error to give the same particle count for other F values. The product of F and threshold-dial reading T, was not a constant. A small amount would have to be subtracted from each threshold setting to keep the product constant. Finally, the threshold zero trim was adjusted slightly until counts were about constant at threshold-dial readings that varied inversely with F.

The gain ratios were supposed to increase the pulse height by a ratio of 1.41 to 1.00 for each step increase in gain setting. Instead, slightly different gain ratios were observed when series of threshold-dial readings were made at several different gain settings and constant temperature and constant aperture-current readings. Also, theoretically equivalent combinations of gain setting and aperture-current dial setting sometimes gave appreciably different threshold-dial readings for a given particle count. Hence, variable resistances were added to the fixed resistances that originally controlled the relative gains in the amplifying circuit. Series of threshold-dial readings at each gain setting and at several current-selector dial readings were repeated, and the variable resistances were adjusted by trial-and-error until the gain ratio from one gain setting to another was essentially constant at 1.41. Improving the accuracy of the gain ratios was not a simple adjustment of the commercial instrument; it involved modification of the amplifying circuit.

The gain ratios in the commercial instrument were probably accurate enough for many uses. However, in the analysis of sediments, the limits of use of an aperture are often stretched as far as possible. Hence, the gain setting is changed much more frequently for sediment analyses than for simply monitoring the particle size or size limit in some commercial process. A simple, uniform gain ratio facilitates calibration and the computation of sediment-size analyses.

The F values furnished by the manufacturer for different apertures and for different I-current dial readings (Table A-4 of the Appendix) were checked against the T and G values. After the gain circuit had been modified and the zero trim of the threshold dial had been adjusted, all three were found to be in agreement. Agreement means that calibration of electrical response is necessary only at one value of T, one of F, and one of G, because the relation to all other values of the three is known. Calibration (Section 25) can be used as a check on the instrument adjustments, but calibration is much simpler if the instrument is in proper adjustment.

The actual volumes metered by the manometer of the counter were checked for relative size by repeatedly counting the same particle suspension for each volume setting. According to average counts of one of five sets of tests (Table 1), the 0.500- and the 0.0500-ml nominal volumes were about 1 and 7 percent, respectively, too large as compared to the 2.000-ml nominal volume. (These percentages were averaged according to the number of individual determinations.) The percentages were confirmed by additional particle counts that are not listed in Table 1. These additional counts indicated that the 0.500-ml nominal volume was about 0.9

TABLE 1

PARTICLE-COUNT CHECKS OF RELATIVE MANOMETER VOLUMES
[Blood cells through a 140-micron aperture]

		Individual particle counts for nominal volumes														
Threshold dial		20			15			15			18			20		
I-current dial		6			6			1			2			7		
Nominal volume (ml)		2.0000	0.5000	0.0500	2.0000	0.5000	0.0500	2.0000	0.5000	0.0500	2.0000	0.5000	0.0500	2.0000	0.5000	0.0500
Trial number	1	13,506	3,456	379	43,088	11,078	1,137	37,531	9,601	1,005	119,969	30,816	3,191	100,344	25,648	2,639
	2	13,693	3,491	372	43,950	10,972	1,169	35,595	8,675	957	121,004	30,566	3,224	101,695	25,347	2,640
	3	13,767	3,436	392	43,833	11,012	1,162	35,021	9,833	997	122,227	30,846	3,299	101,502	25,718	2,675
	4	13,795	3,371	388	43,032	10,919	1,136		9,592	916		30,620	3,176	101,389	25,510	2,650
	5	13,368	3,362	396	43,564	11,071	1,141		8,751	1,033		30,763	3,184		25,479	2,639
	6	13,557	3,374	391	43,236		1,151		8,346	971			3,264			
	7	13,379	3,386	363	43,237		1,157		9,163	880						
	8	13,360	3,417	350	43,150		1,188		9,310	858						
	9		3,409	360	44,239		1,213		9,297	873						
	10		3,387	401			1,150		8,128	915						
	11			352					8,971	901						
	12			359												
	13			344												
	14			382												
	15			350												
	16			364												
	17			324												
	18			427												
	19			347												
	20			360												
	21			349												
Average count		13,553	3,409	369	43,481	11,012	1,160	36,049	9,061	937	121,067	30,722	3,223	101,233	25,540	2,649
Nominal factor		1	4	40	1	4	40	1	4	40	1	4	40	1	4	40
Count for 2,000 ml		13,553	13,636	14,760	43,481	44,048	46,400	36,049	36,244	37,480	121,067	122,888	128,920	101,233	102,160	105,960
Relative count		1.000	1.006	1.089	1.000	1.013	1.067	1.000	1.005	1.040	1.000	1.015	1.065	1.000	1.009	1.047

percent (average of five sets of tests) and the 0.0500-ml nominal volume was about 8 percent (average of two sets of tests) too large if the 2.000-ml volume is correct.

The accuracy of the nominal metered volumes was also checked by weighing quantities of distilled water that were removed from the sample beaker. A rubber covering was placed over the aperture so that no water could be drawn from the beaker through the aperture. Then, a known weight of distilled water at 79°F. (density, 0.9968 g/ml) was placed in a beaker of known weight and the mercury in the manometer was drawn to the start probe. The rubber covering over the aperture was moved aside, and the mercury was allowed to siphon the sample from the beaker. When the mercury surface reached the stop probe, the aperture was covered quickly to stop the flow through the aperture. Thus, one volume of sample was removed from the beaker. Sometimes the process was repeated until several volumes had been removed. The accumulated quantity of water that was removed was determined by weighing the sample beaker and its contents after the withdrawals had been made.

Average observed volumes were 2.0074, 0.5117, and 0.06773 ml (Table 2) for the nominal volumes of 2.0000, 0.5000, and 0.0500 ml, respectively. Thus, the direct measurement of quantities of sample withdrawn from the beaker checked the nominal volume of 2.0000 ml within less than 1 percent, which is also the assumed accuracy of determination for this volume. The probable accuracy is, of course, much less for the smaller nominal volumes. Hence, the excess of observed over nominal volumes of about 2 and 35 percent for the nominal volumes of 0.5000 and 0.0500 ml probably are not as good measures of actual volumes as are the ratios that were based on the particle counts. That is, the observed volumes confirm the accuracy of the nominal volume of 2.0000 ml, and the ratios of the particle counts are reasonably good indications that the other actual volumes are about 0.505 ml (1 percent larger than nominal) and about 0.0535 ml (7 percent larger than nominal).

An additional approximate check of the accuracy of the 2.000-ml manometer volume can be based on comparison of calibration constants k_d , which are determined from volume calibration constants k_v (see Section 18), against constants k_d that are determined directly from particles of approximately uniform size (see Section 17). For known concentrations, k_d is computed from k_v , and both k_v and k_d are too small if the manometer volume is larger than its nominal size; that is, if the integrated sum of the particle volume ($\Delta n S_a$) is too large. However, k_d computed from counts with particles of about uniform size is independent of manometer volume. Hence, the approximate general agreement (Table 3, p. 40) between some k_d values determined from known concentrations and some average k_d values from Fig. 6 (p. 39) indicates that the actual volume is about equal to the nominal volume of 2.000-ml.

Correction of nominal metered volumes is seldom necessary. For size distribution analysis no error is introduced if the analysis is in one nominal metered volume throughout. Even if more than one size of metered volume is used, no error

results if an overlap in counts is used to establish a count ratio between the metered volumes. However, the accuracy of a concentration analysis depends directly on the accuracy of the metered volume. If more than one size of metered volume is used and a count ratio between volumes is established by overlapping counts, only the first, or base, volume for the analysis affects the accuracy of the concentration determination.

TABLE 2

CHECKS OF MANOMETER VOLUMES BY QUANTITIES OF DISTILLED WATER AT 79°F
[Density, 0.99681 g/ml]

	Number of runs or volumes	Weight of water (grams)		Volume of water (milliliters)		Volume ratio, average to nominal
		Total	Average	Average	Nominal	
Trial 1	1	2.0347	2.0347	--	--	
Trial 2	1	2.0176	2.0176	--	--	
Trial 3	7	13.9687	1.9955	--	--	
Trial 4	8	15.9967	1.9996	--	--	
Total or average	17	34.0177	2.0010	2.0074	2.0000	1.004
Trial 1	7	3.5822	.5117	--	--	
Trial 2	6	3.0816	.5136	--	--	
Trial 3	5	2.5178	.5036	--	--	
Total or average	18	9.1816	.5101	.5117	.5000	1.023
Trial 1	7	.4792	.0685	--	--	
Trial 2	3	.2119	.0706	--	--	
Trial 3	3	.2159	.0720	--	--	
Trial 4	6	.3756	.0626	--	--	
Total or average	19	1.2826	.06751	.06773	.0500	1.355

24. Calibration materials--The instrument adjustments that have been discussed could be made with almost any type of relatively stable particles that would pass through a given aperture, because only a relative level of particle size was required. If the specific gravity of such particles is known, they can be used also for calibration by the known concentration method. However, special materials of narrow particle-size range and known mean size facilitate calibration. Calibration with materials of known size is also much more direct, and it is not dependent on such secondary factors as metered volume, specific gravity, and uncertainties in data analysis and interpretation.

CALIBRATION MATERIALS USED

Material	Median size, in microns			
	From Appendix Table A-2	Microscopic size		Average size
		Ours	Others [7]	
Glass beads		135.		135.
Corn pollen	87.5	80.0		83.8
Pecan pollen	47.5	45.0		46.3
Glass beads		37.4		37.4
Ragweed pollen	19.5	19.5	18.6	19.2
Polystyrene beads	3.04			3.04
Polystyrene beads	1.88			1.88
Polystyrene beads	1.30			1.30

The glass beads are spherical, smooth, and easy to analyze with the microscope. Corn and pecan pollens are rougher, somewhat wrinkled and pitted, and their microscopic analyses are possibly less accurate. Ragweed pollen is fairly uniform but rather small for analysis with our microscope. Because pollens vary slightly depending on source, and because the sizes change slightly with electrolyte, time of submergence in the electrolyte, and perhaps with other conditions, an average size was used for each one.

Samples of glass beads were obtained as follows: First, several pounds of beads having a rather wide range of sizes were washed thoroughly and sieved. One sieve fraction was then further separated by letting the beads settle through a column of water. Either the middle half or the third quarter from the bottom of the deposited beads was retained for use as a calibration material. The bead sizes so obtained were as uniform as the pollens. (See Fig. 8 p. 42.)

25. Calibration of equipment--Basically, instrument calibration consists of establishing a relationship, k_v or k_d , between particle size and electrical response for a given aperture, electrolyte, and temperature. (See Eq. 5, 6, and 7.)

This discussion is mainly in terms of k_d because k_d is the constant that enters directly into the usual computation of an analysis.

Fig. 6 shows values of k_d for a 1-percent sodium chloride solution and for 50-, 140-, and 400-micron apertures. The values are plotted to show temperature effect. There is a tendency toward consistent differences among the k_d values from different calibration materials. In the 400-micron aperture, the main materials used were corn pollen, pecan pollen, and glass beads. Corn pollen gave high k_d values and pecan pollen gave low values. In the 140-micron aperture, ragweed pollen gave high k_d values and pecan pollen gave low values again. In the 50-micron aperture, 3.04-micron polystyrene beads gave average results in comparison with 1.88- and 1.30-micron polystyrene beads. Glass beads seem to be a good calibration material. They are smooth, nearly spherical, and do not expand or contract with moisture content. The k_d values plotted on Fig. 6 were taken over a period of nearly two years. Values tended to run a little high or low on certain days, but there was no definite trend with time.

Median sizes of the polystyrene beads could not be determined with available microscopes. Sizes from microscopic analysis of the pollens did not seem to improve calibration consistency as compared with sizes given by the suppliers.

Apparently calibration materials should be investigated further. However, the average calibrations are probably accurate within 5 percent or less and consistent within 3 percent or less. At 75°F the k_d values are about 63.4, 16.0, and 4.20 for the 400-, 140-, and 50-micron apertures, respectively, and the change in k_d with temperature averages about 0.45 percent per degree Fahrenheit. The corresponding change in k_v would be about 1.35 percent as compared with a change in aperture resistance of about 1.00 percent per degree Fahrenheit (Section 22). The difference may indicate a change in the electrical response of the counter circuits or it may mean a lack of precise determination.

If all analyses in the counter are run with a 1-percent sodium chloride electrolyte and at a nearly constant temperature, a single calibration constant can be used for a given aperture. If temperature varies, the calibration constant can be corrected 0.45 percent per degree Fahrenheit or 1.35 percent per degree Fahrenheit for k_d and k_v respectively. Different k_d and k_v values would be required for an electrolyte with a resistivity different from that for a 1-percent sodium chloride.

Calibration constants can also be obtained by the known-concentration method of Section 18. Table 3 lists seven known-concentration values of k_d and the average uniform-size calibration values of k_d from Fig. 6 for the same aperture size and temperature. The known-concentration calibrations were metered in the largest manometer volume, which was taken as 2.000 ml. The generally good agreement between the k_d values from the two types of calibration indicates that the 2.000 ml volume is at least approximately correct.

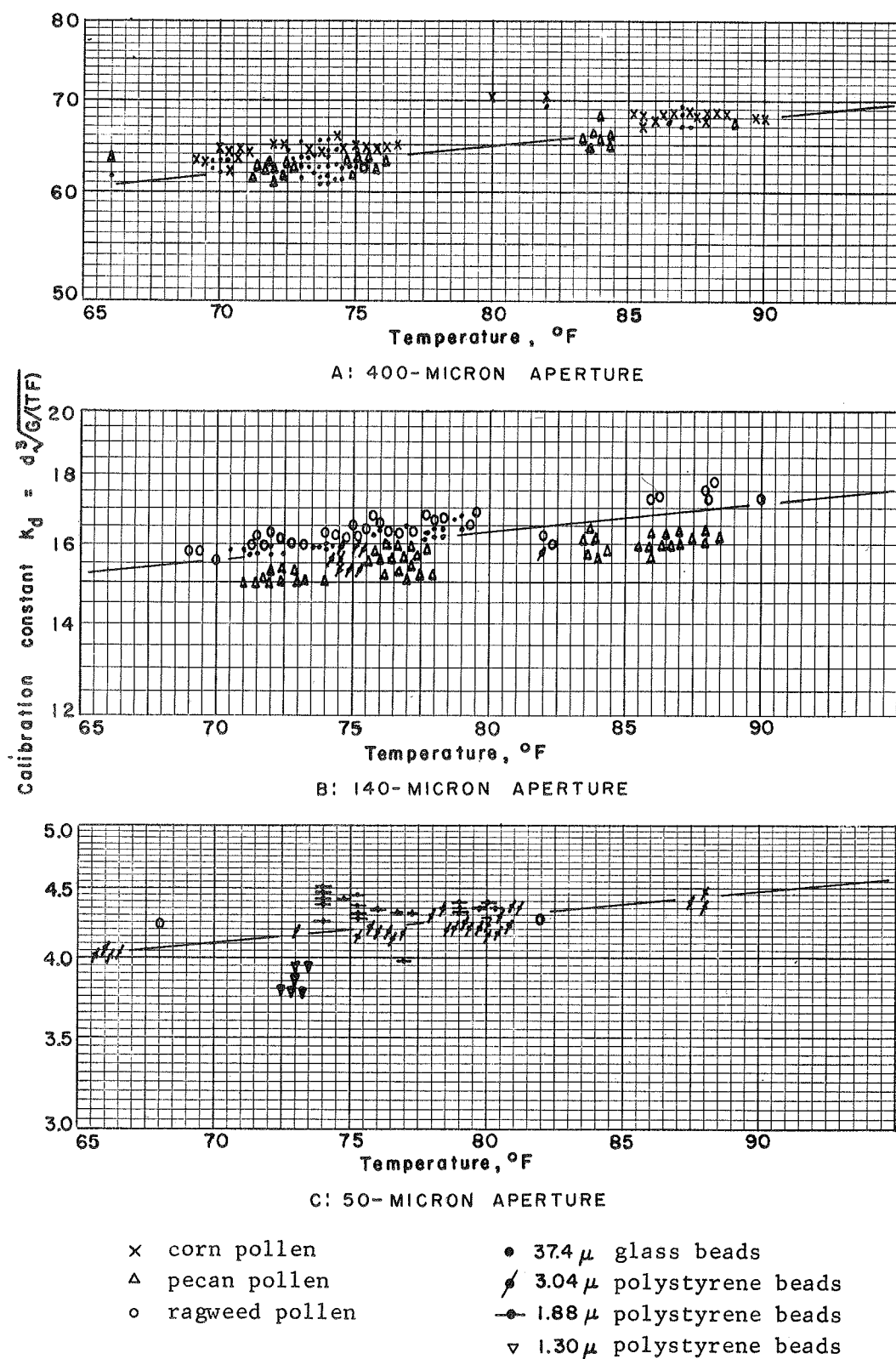


FIG. 6--CALIBRATION CONSTANT

The data from Fig. 6 for the 400-micron aperture are plotted on Fig. 7 to show how the F values can be checked by calibration. From Equation 6, $\sqrt[3]{F} = d \sqrt[3]{G/T} / k_d$. In Fig. 7, the F values from $d \sqrt[3]{G/T} / k_d$ for several I-current dial settings are plotted against the manufacturers F values for the dial setting. The 45° line of perfect agreement lies very close to all plotted points, so the manufacturers F values (Appendix Table A-4) were accepted as correct.

TABLE 3

COMPARISON OF CONSTANTS k_d FROM KNOWN CONCENTRATION AND UNIFORM SIZE

Aperture diameter (microns)	Temperature (°F)	k_d	
		Known concentration	Uniform size (from Fig. 6)
400	68	67.9	61.3
400	70	60.9	62.0
400	72	62.1	62.6
140	70	15.7	15.6
140	74	15.4	15.8
140	76	15.5	16.1
140	78	16.7	16.3

26. Analyses of calibration materials--Size distributions of several calibration materials that cover a range of sizes were determined with the counter from the average calibration values of k_d from Fig. 6. The agreement between these size distributions and size distributions determined from microscopic analyses was generally good (Fig. 8). The major difference was a general displacement of the comparative curves vertically and this difference, which is generally less than 5%, would be almost entirely eliminated if k_d were determined immediately before each analysis. Thus, Fig. 8 is a further indication that an average calibration factor is reasonably good, but not precise, for a range of materials and over an extended period of time.

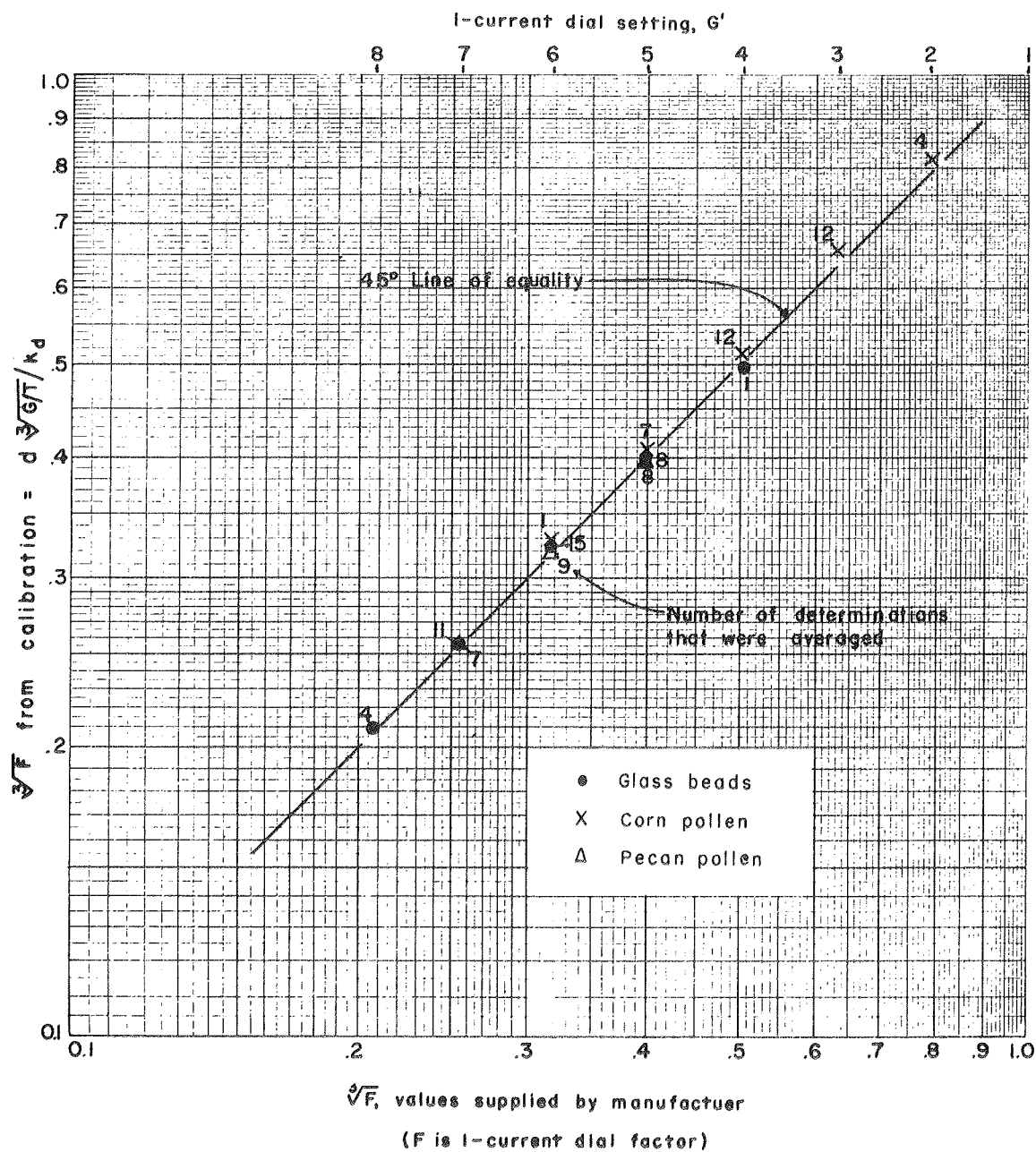


FIG. 7--CALIBRATION CHECK OF I-CURRENT DIAL FACTORS
(APERTURE--400 MICRONS, 4,400 OHMS AT 75°F)

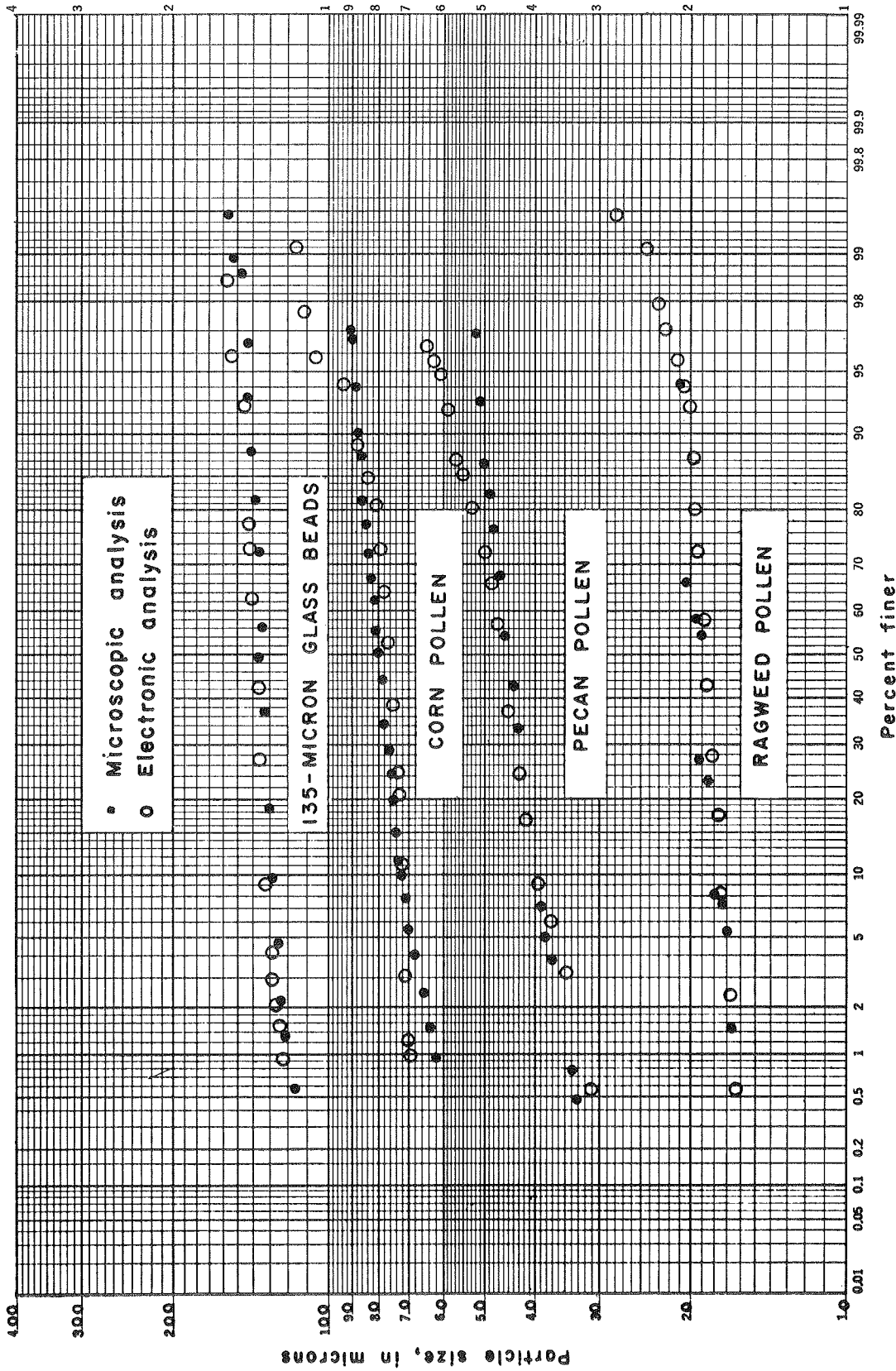


FIG. 8--ANALYSES OF CALIBRATION MATERIALS

27. Analysis of sediment samples--The size analysis of sediment samples from streams involves some steps that usually are not required in the size analysis of a calibration material. The added steps are necessary because of the wide range (1) of conductivity of the waters in which sediment samples are contained and (2) of particle size of the sediments.

If much of the stream water of a water-sediment mixture is placed in the sample beaker, the unknown conductivity of the stream water necessitates a determination of the aperture resistance for each sample. Usually, a small, representative fraction of the mixture can be taken and added to the 1-percent sodium chloride electrolyte. The relative amount of sample and electrolyte should be such that the concentration in the sample beaker will be satisfactory for the first few particle counts.

A sample may contain some particles too large to pass, without plugging, through the largest aperture that is to be used or too large to be kept in uniform suspension in the sample beaker. These large particles can be removed by sieving or by selective settling through a column of water.

The wide range of particle sizes and the usually rapid increase of particle numbers with decreasing size may cause the coincidence correction (Section 19) to exceed 10 percent of the observed particle count at some particle sizes. Whenever dilution of the sample is necessary to avoid excessive coincidence, overlapping counts should be made at three or more of the same dial settings to establish the relative particle counts before and after dilution. Of course, the ratio could be determined from relative volume of the sample after dilution to volume before dilution. However, the method of overlapping count is generally simpler and more satisfactory.

The wide range of particle sizes generally requires use of more than one aperture for a size analysis. Whenever a change is made from a large aperture to a smaller one, some of the coarser particles in the sample may have to be removed by sieving or by selective settling in a column of water to avoid plugging of the smaller aperture. If large particles are removed or the counting of the large particles is questionable for the smaller aperture, both the sample that was used for the larger aperture and the sample that is to be used for the smaller aperture should each be run at two or more of the same values of S_e ($= T F/G$). As an alternative, overlap counts can be made for S_e values that are not identical but that cover a satisfactory range so that two curves of S_e versus particle count can be plotted and an overlap relation determined from the curves. Overlap values of S_e should be for particle sizes that were too small to be removed, even in part, by the sieving or the selective settling. Overlapping counts are necessary to define a relationship between the numbers of particles within a given size range for the two samples. That is, a ratio is needed between the difference in particle count from one size to a larger size for one sample and the difference in particle count for the same size range and the other sample. The overlapping counts must be at the lower end of the size range for the larger aperture rather than at the

upper end of the size range for the smaller aperture unless all particle sizes of both samples pass readily through the smaller aperture.

The lower limit of size analysis with the 50-micron aperture is about 1 micron. However, some sediments have an appreciable volume of particles finer than 1 micron. Also, an arbitrary lower limit for analysis may be set by judgment on the assumption that the volume of smaller sediment particles is an insignificant proportion of the total volume. The quantitative estimate of the volume of uncounted fine sediment can be checked, at least crudely, by a graphical procedure that will be discussed later.

Some particles may break down or dissolve while in the electrolyte. Possible changes in size of the particles can be checked readily by comparing particle counts for a given electrolyte and S_e value at the beginning of the analysis with particle counts for the same temperature and S_e value at the end of the analysis of the same sample. Corrections to the counts must be applied for any dilution of the sample during analysis.

In general, the natural inorganic sediments that have been analyzed showed no decrease in size because of breaking down or dissolving of the particles. Some coal particles did break down enough to give a slight increase in count of particles at the smallest sizes. The small amount of breaking of particles can be decreased by agitating and stirring the sample no more than necessary to obtain discrete particles uniformly suspended in the sample beaker.

Some stream samples contain organic matter that should be removed prior to analysis. (See Section 7.)

28. Repeatability--Consecutive counts on a particular sample at the same settings of the gain dial, the I-current dial, and the threshold dial usually check within a very few percent, especially if each count includes several thousand particles. As counts decrease the percentage variation from count to count increases. It may be several percent at counts of 500 particles or less (See Table 5 on p. 50 and Table 6 on p. 56.) However, the average of several counts of a few hundred particles each should be reasonably accurate. Of course, a uniform suspension of the particles should be maintained in the sample beaker.

Repeated determinations, with two apertures, of the median size of a sample of glass beads were closely consistent among themselves (Table 4) and checked the median size of 37.4 microns that was obtained by microscopic analysis. The k_d values used in the analyses were not as completely determined as those of Fig. 6. Values from Fig. 6 give diameters for the 400-micron aperture about 1 1/2 percent smaller and for the 140-micron aperture about 1 1/2 percent larger than the diameters listed in Table 4. However, the results would be slightly more consistent between apertures if they were computed for values of k_d from Fig. 6. The excellent agreement with the microscopic size was due partly to chance because the calibration constant is not known to an accuracy of a fraction of 1 percent. The

repeated size determinations were based on one calibration constant for each aperture size. They were made on the same day by the same operator and with the same electrolyte, and at about the same temperature. The median sizes in Table 4 are for a sample with a small range of particle sizes. They indicate the repeatability of instrument readings. Computation techniques and inability to measure sizes smaller than one micron would make analyses of samples with a wider range of sizes less accurately repeatable.

TABLE 4

CONSISTENCY OF REPEATED DETERMINATIONS OF SIZES OF GLASS BEADS

Order number	Median size, in microns		Order number	Median size, in microns	
	400-micron aperture	140-micron aperture		400-micron aperture	140-micron aperture
1	38.2	--	16	39.0	--
2	37.6	--	17	38.2	--
3	38.0	--	18	37.0	--
4	37.4	--	19	36.5	--
5	37.4	--	20	37.2	--
6	38.0	--	21	37.5	--
7	38.0	--	22	--	36.5
8	37.8	--	23	--	36.2
9	37.8	--	24	--	37.5
10	--	36.8	25	--	36.6
11	--	36.8	26	--	36.5
12	--	36.6	27	--	37.3
13	--	37.0	28	--	37.2
14	39.0	--	29	--	37.0
15	38.4	--	30	--	37.2
			31	--	37.2

Average sizes in microns:

400-micron aperture	37.8
140-micron aperture	36.9
Both apertures	37.4

Apparently the electronic components of the equipment are stable. Hence, size determinations generally can be repeated consistently provided that a standard procedure is always followed in analyzing a particular sample and particle sizes are not large enough to plug the aperture or to prevent uniform dispersion in the sample beaker. Some consistency of repeated determinations obviously will be lost if the procedure is varied from time to time or if limitations of the equipment and its calibration are not recognized.

29. Accuracy of analysis--Several factors affect the accuracy of analysis. Preparation of the sample in terms of representativeness of the parental material, removal of extraneous or contaminating substances, and dispersion of the particles are important. Careful operation of the equipment and a constant watchfulness will detect most malfunctions or electrical interference. An occasional glance at the oscilloscope should show abnormal electrical interference or erratic operation. Conditions at the aperture, especially plugging, can be observed through the microscope. Questionable particle counts should be noted and repeated.

The statistical accuracy of particle counts is generally good when many particles are counted. However, one particle 100 microns in diameter is the equivalent of 1,000 particles 10 microns in diameter. A sufficient number of large particles must be counted to determine the relative volume of the coarser particles.

Particle characteristics such as shape, roughness, refractive index, density, and electrical conductivity have little or no effect on the accuracy of analysis. Orientation within the aperture probably has no measurable effect for sediment particles.

Adequacy of the calibration coefficient is perhaps the most important factor in accuracy of an analysis. A calibration factor that has been determined for a given aperture, electrolyte, and temperature is good only for that combination of basic factors. It must not be used inadvertently and without correction for another aperture, for an electrolyte of a different resistivity, or for a temperature much different than that for which it applies. Although an accurate determination of aperture resistance might be used to correct the calibration for changes in electrolyte, the d-c voltmeter determinations (Appendix Section 39) do not seem sufficiently accurate for that purpose. If a standard calibration factor is to be used, the electrolyte should be very carefully prepared and temperature corrections should be made when necessary.

Unfortunately the absolute accuracy of a complete analysis of a normal sediment sample was not established directly because no particle distribution of completely known size and size distribution was available against which to check the analyses. The calibration procedure and analysis of calibration materials established accuracy of analysis at certain specific particle sizes, spread over a wide range of sizes. Hence, a sediment analysis in a single size of aperture tube almost certainly will be reasonably accurate except outside, at, or near, the size limits for analysis in the aperture. However, the procedure for combining data

from two or more apertures may introduce significant errors in the combined analysis.

Six duplicate samples of fuller's earth were analyzed to determine consistency of analysis over a wide range of particle sizes and combination of apertures. The particle sizes in the samples ranged from less than 1 to more than 100 microns. The 400-, 140-, and 50-micron aperture tubes were all required.

A one-gallon master sample of 30,000 ppm (parts per million) of fuller's earth in electrolyte was made up. About ten drops of Triton X-100 were added for a dispersant. The 400-micron aperture was used first. The master sample was mixed thoroughly, and a pipette sample was immediately withdrawn for analysis. Pipette samples for analysis in the 140-micron aperture were withdrawn at a depth of 5 cm from the surface and 1 minute after stirring of the master sample ceased. Pipette samples for analysis in the 50-micron aperture were withdrawn at a depth of 5 centimeters and 6 minutes after stirring ceased. Most of the 50-micron particles, but few of the 20-micron and smaller particles, were removed from the sample for analysis in the 140-micron aperture. Most of the 20-micron, but few of the 10-micron or smaller particles, were removed from the sample for analysis in the 50-micron aperture. These samples did not plug the apertures and the undisturbed size distribution analyzed in each aperture provided a good overlap with counts in the next larger aperture.

The computed size distributions for the six samples were reasonably consistent. The median diameters for the six analyses ranged from 20.0 to 23.4 microns and three of the six were between 21 and 22 microns. Minor differences between analyses could often be traced to an obvious source such as insufficient overlap in readings before and after dilution, or failure to count a sufficient number of particles to provide an accurate average count at some sizes. The analyses were run by two rather inexperienced operators. No consistent differences could be seen between their results. Although better consistency of analysis could be obtained by more careful operation and experienced operators, the data of Fig. 9 probably represent the accuracy to be expected from routine analysis.

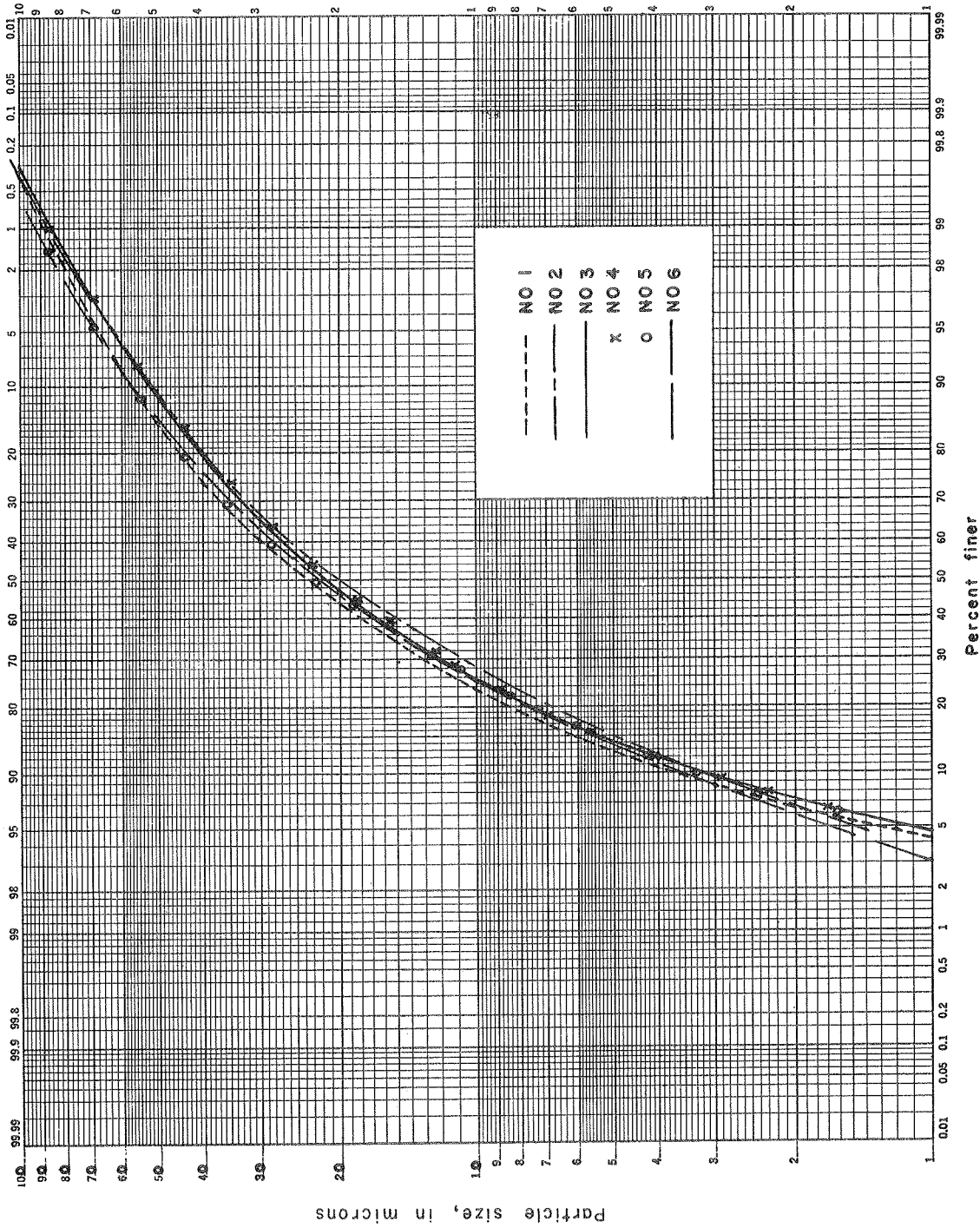


FIG. 9--ANALYSES OF SIX DUPLICATE SAMPLES OF FULLER'S EARTH

V. COMPUTATION OF SIZE AND CONCENTRATION

30. Basic data required--Before particle counts are taken for an analysis, the equipment must be set up properly and adjusted. Preliminary data on temperature, calibration constants, and aperture resistance must be obtained. Temperature of the room in which the equipment is operated should be fairly constant and the electrolyte and sample should be at room temperature. The calibration constant (Section 25) may have been determined previously or may be determined just before analysis. Calibration constants should be checked occasionally. The gain trim in the instrument may be used to bring the instrument back to a previous calibration if drift in calibration occurs. If the same electrolyte is used repeatedly and only a very small sample of water-sediment mixture is used, the resistance through an aperture should not change except with temperature, and resistance need be checked only occasionally.

After the aperture and the manometer volume are chosen for an analysis, they are recorded and a coincidence factor, p , is computed from Equation 11 on page 25.

Particle counts are taken at several size levels to obtain a count of particles coarser than each size level. (See Section 8.)

31. Recording and computing data--A method of data recording for an analysis in a single aperture is illustrated in Table 5. Data on the sample and instrument factors are recorded at the top. Values F_1 to F_{10} are taken from Table A-4 in the appendix and correspond to I-current dial settings of 1 to 10, respectively.

The first three columns from the left side of the table contain the gain setting, G' , (and the corresponding gain value, G); the threshold dial setting, T ; and the I-current dial setting, I .

Particle counts at the various combinations of G , T , and I settings are recorded in the next several columns. An average count for each combination is recorded as \bar{n}' . The coincidence count adjustment is computed from p and Equation 12 on page 25, and entered in column n'' . Column n (n_2 after dilution) is the average count of column \bar{n}' , corrected by addition of values in column n'' . Column C is for background-count corrections, for correction ratios n/n_2 of counts before and after dilution, and for n_2 counts that were corrected because of sample dilution (see the following section also). The F/G column contains the basic values of F_1 to F_{10} that correspond to the I-current dial setting, divided by the value of the gain setting, which is 1.00, 1.41, 2.00, 2.83, 4.00, or 5.66 for the 1 to 6 settings respectively. Column S_e is the product of columns T and F/G .

The cube root of S_e is multiplied by k_d , to obtain the particle diameter, in microns, shown in column d . The analysis at this point shows number of particles larger than the sizes tabulated, and for some purposes such an analysis is sufficient.

TABLE 5
BASIC FORM FOR RECORDING DATA AND COMPUTING SIZE DISTRIBUTION

Sample Special		Source										Operator TFB		Date 7-15-64	
Aperture Diameter 140 μ		Aperture resistance 11,000 Ω		Coincidence factor, p 1.715		Calibration factor, k_d 16.0		Notes:							
Manometer Volume 2,000 μ l		Temperature 75 $^{\circ}$ F		Dispersant Triton X-100		Electrolyte 1% NaCl									
F1	F2	F3	F4	F5	F6	F7	F8	F9	F10	G1	G2	G3	G4	G5	G6
1.000	.501	.251	.1262	.0639	.0327	.01707	.00930	.00544	.00360	1.00	1.414	2.00	2.83	4.00	5.66
I-current dial factors, F															
G', G	T	I	n'	n'	n'	n'	n'	n'	n'	n'	n'	C	F/G	S [*] TF/G	d [*] k _d ² /S [*]
3 = 2.00	80	1	1	0	0	0	0	0	0	0	0	0	0.500	40.0	54.7
2.00	40	1	3	4	5	4	4	4	4	4	4	4	.500	20.0	43.4
2.00	20	1	26	24	25	26	25	25	25	25	25	25	.500	10.0	34.5
2.00	20	2	58	65	61	64	73	64	64	64	64	64	.2505	5.01	27.4
2.00	20	3	131	134	153	158	128	137	137	137	137	137	.1255	2.51	21.8
2.00	20	4	305	305	290	313	303	303	303	303	303	303	.0631	1.262	17.3
2.00	20	5	681	677	677	694	684	684	684	684	684	684	.0320	0.640	13.8
2.00	20	6	1,494	1,479	1,491	1,491	1,488	1,488	1,488	1,488	1,488	1,488	.0164	.328	11.0
S = 4.00	20	6	3,304	3,304	3,388	3,343	3,343	3,343	3,343	3,343	3,343	3,343	.00820	.1640	8.75
4.00	20	7	7,191	7,100	7,100	7,074	7,122	7,122	7,122	7,122	7,122	7,122	.00427	.0854	7.04
4.00	20	8	13,083	12,964	13,012	13,020	13,020	13,020	13,020	13,020	13,020	13,020	.00232	.0464	5.75
4.00	20	9	21,674	21,654	21,668	21,665	21,665	21,665	21,665	21,665	21,665	21,665	.00136	.0272	4.81
4.00	20	10	30,120	30,320	30,526	30,332	30,332	30,332	30,332	30,332	30,332	30,332	.00090	.0180	4.19
Diluted here															
S = 4.00	20	6	1,506	1,543	1,562	1,570	1,570	1,570	1,570	1,570	1,570	1,570			
4.00	20	7	3,170	3,256	3,145	3,190	3,190	3,190	3,190	3,190	3,190	3,190			
4.00	20	8	6,195	6,092	6,020	6,102	6,102	6,102	6,102	6,102	6,102	6,102			
4.00	20	9	10,225	10,050	10,180	10,132	10,132	10,132	10,132	10,132	10,132	10,132			
4.00	20	10	13,780	14,120	14,095	13,998	13,998	13,998	13,998	13,998	13,998	13,998			
6 = 5.66	15	10	17,826	17,852	17,838	17,839	17,839	17,839	17,839	17,839	17,839	17,839			
5.66	10	10	23,182	23,406	23,650	23,413	23,413	23,413	23,413	23,413	23,413	23,413			
5.66	10	10	31,030	30,821	31,200	31,017	31,017	31,017	31,017	31,017	31,017	31,017			
5.66	7.5	10	37,160	37,140	36,990	37,097	37,097	37,097	37,097	37,097	37,097	37,097			
5.66	5.0	10	47,405	47,720	47,250	47,458	47,458	47,458	47,458	47,458	47,458	47,458			
For total of 4,577															
For estimated total of 4,900															

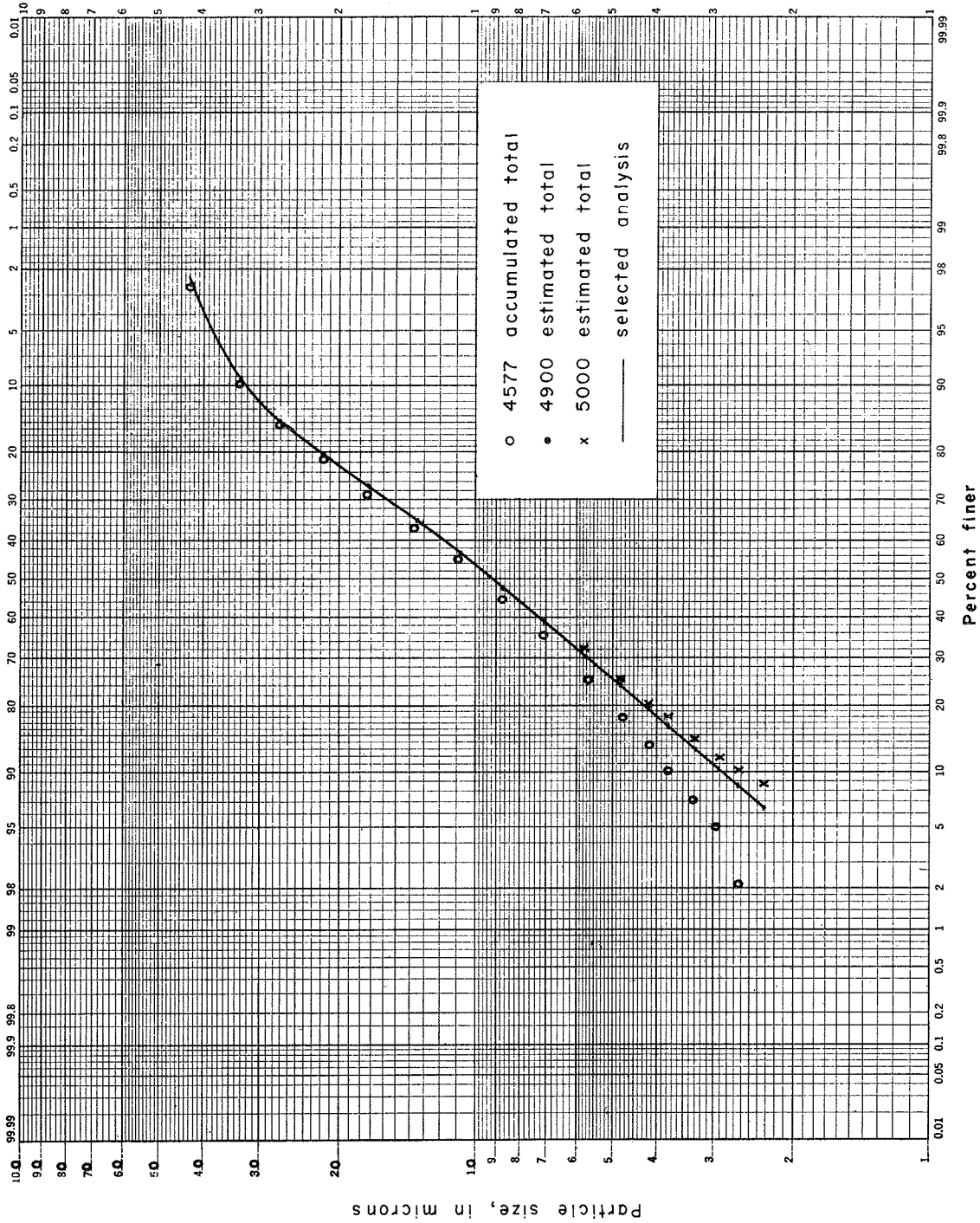
The last six columns show computation of size distribution by volume. Δn is change in counts between adjacent sizes, S_e . \bar{S}_e is the average of the adjacent sizes. The product of Δn and \bar{S}_e is the average relative volume of particles in the size interval. $\Sigma(\Delta n) \bar{S}_e$ is the cumulative total of the relative volumes. The column headed "Coarser, % vol." contains the percentage by volume coarser than the diameters of column d. It is found by dividing the cumulative values of $\Sigma(\Delta n) \bar{S}_e$ by the total for the column. The use of the final column and the analysis of diluted samples are discussed in the following section.

32. Special computations and data extension--In many analyses the particle count is not, or cannot, be carried to small enough sizes to cover the entire particle size distribution. That is, the total of the cumulative volumes of the $\Sigma(\Delta n) \bar{S}_e$ column of Table 5 is perhaps 90 or 95 percent of the true total for a complete analysis. Visual inspection of the $\Delta n \bar{S}_e$ values in Table 5 will show whether or not they are approaching zero.

If the percentages coarser than the particle-diameter steps of Table 5 are plotted on log-probability paper as in Fig. 10, the plot will be a straight line if the sediment has a log-normal size distribution. Because most sediments approach a log-normal distribution, any abrupt flattening in the relationship at the fine particle sizes usually indicates an incomplete analysis. The total cumulative volume from Table 5 may then be increased and new percentages based on the larger total may be computed and plotted. By trial and error a distribution can be obtained that will straighten the fine-particle end of the log-probability curve, and that distribution may be used as the proper analysis of the sample on the assumption that the distribution is at least approximately log normal. In the data used for Table 5 and Fig. 10, the total of the $\Sigma(\Delta n) \bar{S}_e$ column is 4,577. Percentages by volume were computed and plotted on Fig. 10 using total volumes of 4,577, 4,900, and 5,000. The 4,900 total gives the straightest line at the lower end on Fig. 10. The volume percentages based on the 4,900 total are recorded in the last column of Table 5 as the size distribution for the sample.

If concentration in the sample to be analyzed is sufficient to provide statistically significant counts, the coincidence effect may become excessive (10 percent or more) at small-particle volume settings. The sample should then be diluted. After dilution, counts of the diluted sample should be repeated at two to four of the sizes already counted. The ratio of old counts to new can be determined directly from the overlapping data and used as a multiplier to change all diluted sample counts to the basis of the original sample concentration.

Table 5 shows the analysis of a sample that was diluted. Overlapping counts were taken, and counts after dilution were related to those before dilution. The average ratio of counts before and after dilution was 2.19. Counts after dilution were multiplied by 2.19 to obtain counts on the basis of the concentration of sediment in the sample on which the analysis was begun.



When an analysis is made in more than one aperture, the particle-size steps of one aperture must be related to those of other apertures. The S_e value of one aperture can be converted to the basis of a second aperture from the cube of the ratios of the k_d values for each aperture. Perhaps a better method is to use the d^3 values for each size step in place of S_e of Table 5. The spherical volume $\pi d^3/6$ could be used in place of S_e if desired.

After the size steps are correlated from one aperture to another, a dilution or change in sample concentration, or a change in metered volume, can be handled as a simple dilution and the ratio of overlapped counts from one condition to another can be used to relate the counts for the second condition to those of the original sample.

If x is the number of counts before a dilution, y is the number after dilution, and $x_1 = K_c y_1$ at any overlapping count, then $K_c y$ is the count on the original basis. ($K_c = 2.19$ in Table 5.) If after analysis in a large aperture some coarse particles are removed by sieving or by sedimentation to avoid plugging in a smaller aperture, additional problems are involved. The removal of some coarse particles reduces all counts throughout the analysis in the smaller aperture. If a number of coarse particles, q , is removed before the count is taken $x - q = K_c y$, and the ratio of x to y is not a constant multiplier over a range of values of x and y . As x and y become very large q becomes relatively small, but q may not be small enough to neglect.

$$\text{Because } (x_1 - q) - (x_2 - q) = K_c y_1 - K_c y_2$$

$$x_1 - x_2 = K_c (y_1 - y_2)$$

and the ratio of the differences, Δn , between counts at successive size steps is a constant. The count ratio K_c can be used as a multiplier to adjust Δn_2 counts to equivalent Δn counts of the original sample and to determine n after dilution. The above usage of differences in counts between successive steps applies only for sizes smaller than any of the particles, q , that were removed. If sieving is incomplete, or separation by settling is not accurate, many particles much smaller than the sieve or sedimentation size may be removed from the sample.

A log-log plot of total count against particle volume (or d^3) can be prepared and the ratio of counts before and after a dilution, a counted volume change, or removal of coarse particles can be taken from the plot. Fig. 11 shows data from an analysis of a sample of fuller's earth. The x symbols on the right show the counts (above 100) for the first part of the analysis in the 400-micron aperture. The $+$ symbols show the second part of the analysis after dilution. In the overlap region the ratio of counts on curve x to counts on curve $+$ averages about 23.7. (The same data are shown in Table 6 with a computed ratio of 23.6). Usually greater weight is given to the part of the overlap where the counts are largest unless the higher counts exceed an n'' correction of 10%. If the n'' correction exceeds 10%, the corrected count is generally too low even after correction. The \oplus symbols show the $+$ counts multiplied by 23.6. The solid line represents the

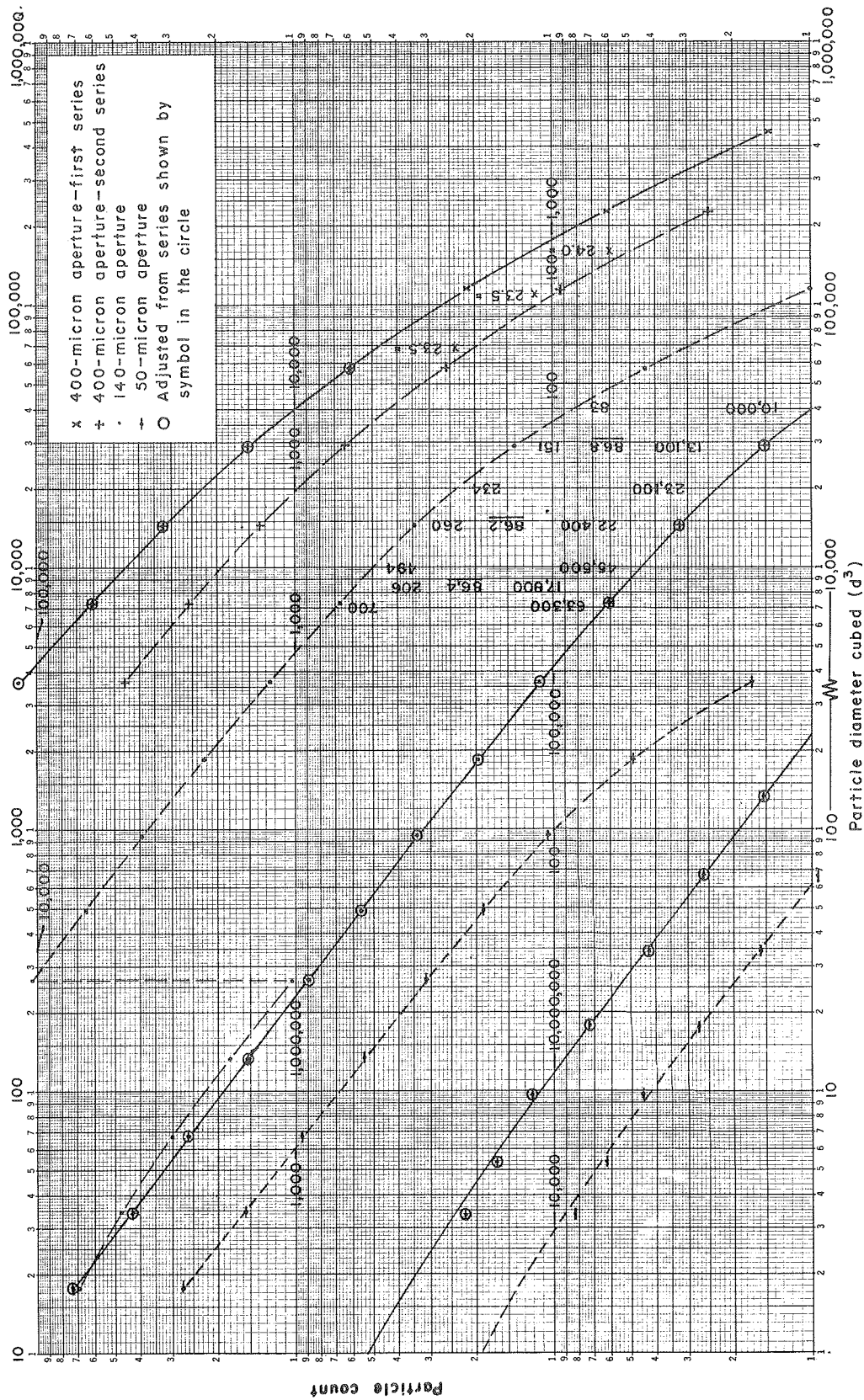


FIG. 11--COUNT-DATA PLOTTING AS A COMPUTATION AID

relation between particle count and d^3 for the concentration of sediment in the sample with which the analysis was begun.

The \bullet symbols on Fig. 11 show the counts from analysis in the 140-micron aperture. Because some of the particles coarser than about 30 microns were removed prior to analysis in the 140-micron aperture, the ratio of \bullet counts to \oplus counts is not a constant in the overlap region. If counts are taken from the \bullet and \oplus curves at several of the same d^3 values and the differences in counts between adjacent values are taken, the ratio of difference to corresponding difference is essentially constant. A difference of 17,800 in counts between d^3 values of 7,000 and 10,000 on the solid curve corresponds to a difference of 206 on the 140-micron curve and the ratio is 86.4. Other ratios of 86.2 and 86.8 are shown on Fig. 11 for ranges of d^3 from 10,000 to 20,000 and 20,000 to 40,000, respectively. (The average computed ratio in Table 6 is 86.5). If the \bullet counts at the large-count end of the overlap are multiplied by 86.5, they fall about 3,100 counts below the solid line. On the basis of the counts for the concentration with which the analysis started, 3,100 of the coarser particles were removed before analysis in the 140-micron aperture. Each \bullet count was multiplied by 86.5, and 3,100 was added to obtain the \odot counts on which the solid curve was based.

In a similar manner the \ast counts from the 50-micron aperture were adjusted and used to define the fine-particle end of the curve.

If overlapping counts are not taken at the same size (or d^3 or S_e values), direct arithmetic computation is very difficult and a curve of counts at various particle sizes should be plotted. If counts are inconsistent, plotting may be desirable. Sometimes the slope of a curve of counts after dilution can be used directly to extend the curve for counts on the basis of the original sample. In general, plotting of counts, as in Fig. 11, at various particle sizes is not necessary and it adds little to the accuracy of analysis.

33. Comprehensive form for computation of data--The range of particle sizes of natural sediments is so great that more than one aperture size is usually required. A form, Table 6, which can be used over a range of 1 to 160 microns, was made up based on analyses in 50-, 140-, and 400-micron aperture tubes. Besides the data on sample and preparatory treatment, only the particle counts, count differences, volumes of particles, and percentage by volume are recorded and computed. In Table 6 all information that is printed on the form is typed, and information that is added later for an individual analysis is hand lettered.

Aperture size, and the dial settings for gain, threshold, and I-current are printed on the form of Table 6. The basic counts, average basic count, coincidence correction, and counts between size steps are recorded or computed as in Table 5 and as explained in Section 32. A universal relative size d^3 is used instead of the electrical size S_e . The actual average particle volume in cubic microns is used as a size multiplier in each size interval. The remainder of the computations follow the general procedure for Table 5. The form of Table 6 can

TABLE 6

COMPREHENSIVE FORM FOR RECORDING DATA AND COMPUTING SIZE DISTRIBUTION

Notes:									
d 75°F	d ³	Δn	C ₂	Adj. Δn	Volume, π d ³ /6	(Δn) vol. ÷ 1,000	Σ(Δn) vol. ÷ 1,000	Coarser, % vol.	Adj. coarser, % vol.
400-Micron Aperture								Based on 1823400	Based on 1850000
243	14,400,000								
193	7,200,000				5,655,000				
153	3,600,000	0.7		0.7	2,827,000	1980	1980	0.1	0.1
122	1,800,000	1.6		1.6	1,414,000	2260	4240	0.2	0.2
96.6	900,000	19.7		19.7	707,000	13930	18170	1.0	1.0
76.7	452,000	124		124	354,000	43900	62070	3.4	3.4
61.0	226,600	466		466	177,700	82810	144900	7.9	7.8
48.5	114,000	1522	-217	1522	89,200	135800	280700	15.4	15.2
38.7	57,800	3956	-116	3956	45,000	178000	458700	25.2	24.8
30.7	29,000	9010	-93	9010	22,700	204500	663200	36.4	35.8
24.4	14,500	17350	85.0	17350	11,390	197600	860800	47.2	46.5
19.4	7,260	28510	88.0	28510	5,700	162500	1023300	56.1	55.3
15.4	3,650	47640	-81.0	-47640	2,860				
12.3	1,850		Avg. 86.5		1,440				
9.81	943				731				
140-Micron Aperture									
61.0	226,600								
48.5	114,000	7	186.5 =	-606	89,200				
38.7	57,800	34		-2944	45,000				
30.7	29,000	97		-8340	22,700				
24.4	14,500	204		-17650	11,390				
19.4	7,260	324		-28030	5,700				
15.4	3,650	588	3633	50860	2,860	145500	1168800	69.1	63.2
12.3	1,850	1002	2708	86670	1,440	124800	1293600	70.9	69.9
9.81	943	1676	2636	145000	731	106000	1399600	76.8	75.7
7.89	491	2558	2604	221300	375	83000	1482600	81.3	80.1
6.43	266	3848	2663	332900	198	65900	1548500	84.9	83.7
5.09	132	7663	2857	662800	104	68900	1617400	88.7	87.4
4.06	66.7	12057	2544	1043000	52.3	54500	1671900	91.7	90.4
3.24	34.1	17270	-2344	-1444000	26.4				
2.61	17.8	21702	-1629	-1877000	13.6				
			Avg. 2667						
50-Micron Aperture									
19.4	7,260								
15.4	3,650	14	2669 =	-37370	2,860				
12.3	1,850	32		-85410	1,440				
9.81	943	55		-146800	731				
7.89	491	85		-226900	375				
6.43	266	125		-333600	198				
5.09	132	232		-619200	104				
4.06	66.7	410		-1094000	52.3				
3.24	34.1	624		-1665000	26.4	44000	1715900	94.1	92.8
2.61	17.8	1152		-3076000	13.6	41800	1757700	96.4	95.0
2.13	9.65	1845		-4924000	7.21	35500	1793200	98.3	96.9
1.75	5.36	1678		-4471000	3.93	17600	1810800	99.3	97.9
1.50	3.38	2068		-5519000	2.29	12600	1823400	100.0	98.6
1.25	1.95				1.40				
1.00	1.00				0.772				
0.75	0.42				.372				

Est. total → 1,850,000

be used for a single aperture tube or any combination of the three aperture tubes. The work of recording and computing the data is reduced considerably by using standard settings.

In Table 6 the data for the 400-micron aperture show a dilution. Computations were made as in Table 5, but an additional column for the n count adjusted for dilution and the restrictions of the printed G , T , and I readings makes the arrangement of the data somewhat different.

Because the dilution between counts in the different apertures included also the removal of some of the coarser particles, the overlap computations were not made like those in Table 5. The ratios of counts before and after dilution were determined. (See columns headed " Δn ", " C_2 ", and "Adj. Δn ".) The average ratio between Δn counts for the 400- and 140-micron aperture was 86.5 on the basis of counts at diameters of 24.4 and 19.4 microns. Some 30.7-micron particles were probably removed before analysis in the 140-micron aperture. The count at 15.4 microns requires an excessive n correction and was probably too low. The Δn counts from analysis in the 140-micron aperture were multiplied by 86.5 to obtain Δn counts on the basis of the sediment concentration with which the analysis was started in the 400-micron aperture. The adjusted n count of 60,960 at 19.4 microns was taken as correct, and higher adjusted n counts were obtained by adding the adjusted Δn counts in succession beginning with the 50,860 between diameters of 19.4 and 15.4 microns. The 669 count at a diameter of 19.4 microns was multiplied by 86.5 to show a count of 57,870 in the 140-micron aperture compared with the 60,960 count from the 400-micron aperture. The difference is about 3,100. After n counts from the analysis in the 140-micron aperture were multiplied by 86.5, the 3,100 could be added to each product to obtain the adjusted n counts. Note that the adjusted n counts in the 140- and 50-micron apertures and the d^3 column of Table 6 are unnecessary unless the curve of counts at various d^3 values is to be plotted.

The data from the 50-micron tube were adjusted by the same methods that were used for the data from the 140-micron tube.

The $\Sigma(\Delta n)$ vol. column shows the volume (in cubic microns) of particles coarser than the corresponding size. The estimated total of 1,850,000,000 cubic microns shows a volume of 1.85 microliters of particles in the metered volume of 2,000 microliters on which the analysis was begun. The volume concentration was therefore .0925 percent, or 925 ppm, in the original sample. The volume concentration between any two particle sizes, or coarser than any particle size, can be obtained in the same way.

Besides the basic computations of particle size and concentration other types of data reduction may be desirable under some circumstances.

The size distribution from Table 6 may be plotted as in Fig. 10. If this is done (Fig. 12) a total of about 1,870,000,000 cubic microns would probably be used as the basis for computing size distribution.

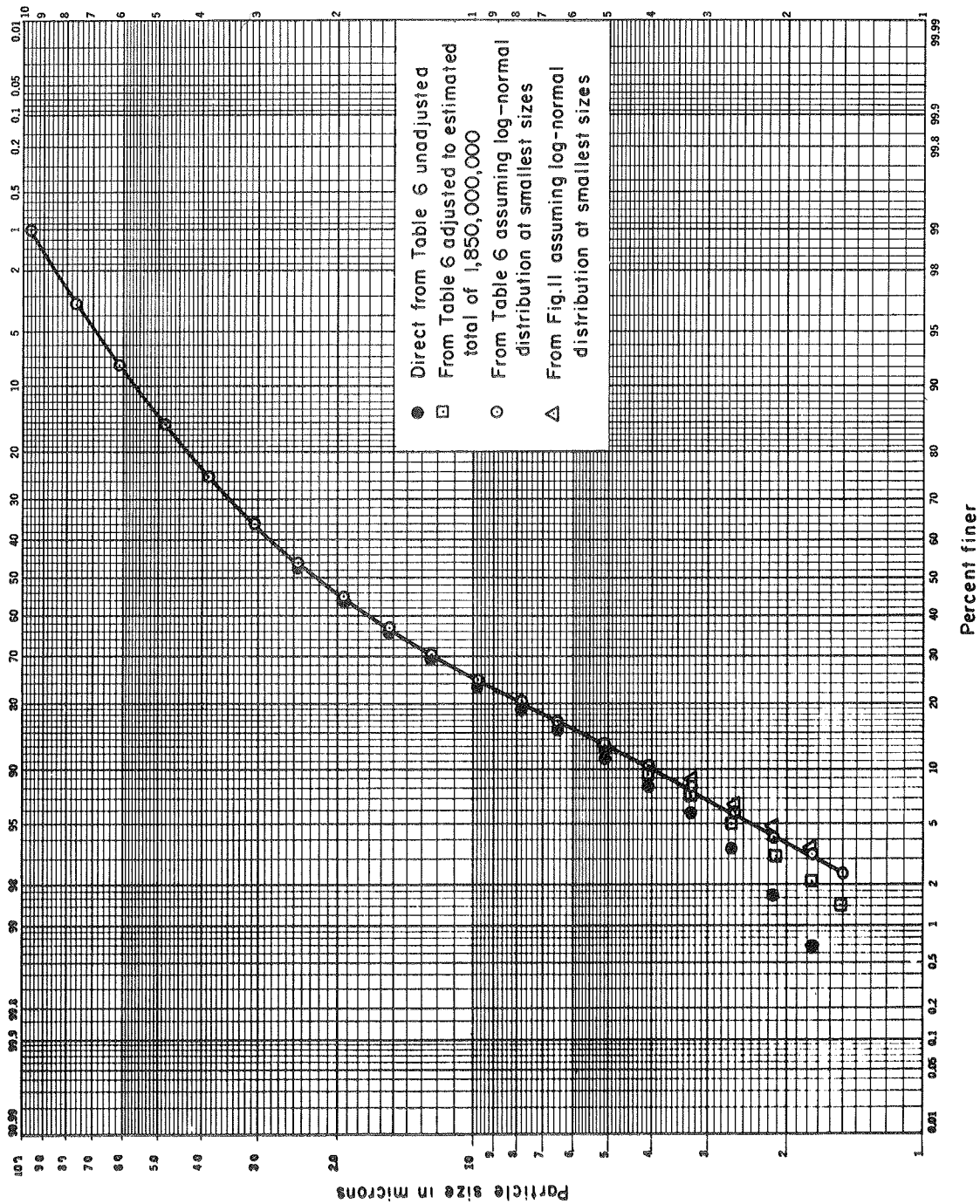


FIG. 12--COMPARISON OF DIFFERENT METHODS OF COMPUTING SIZE ANALYSIS DATA

When counts are somewhat erratic, the counts for computing Δn may be taken from an average curve. If this is done for the data on which Table 6 and Fig. 11 are based, the final analysis would be that shown by the Δ readings of Fig. 12.

Table 6 was made up on the basis of a temperature of 75°F. If temperature had been outside the range 73° - 77°F, an adjustment of the final analysis might be desirable. (See Section 25.) For example, if the average temperature during analysis had been 80°F, the particle diameters should be increased 5×0.45 , or 2.25 percent, for the analysis--and a median particle diameter of 22 microns would be changed to 22.5 microns.

VI. DISCUSSION

34. Short review of size analysis methods--Although the basic reason for studying electronic sensing of sediment was the possibility of its use to determine sediment concentration, the instrument in its present form is better adapted to size analysis.

A brief review of size analyses with other equipment is given as a basis for evaluating the potential of the electronic sensing device as a laboratory instrument for size analysis. Accuracy in terms of size analysis implies possible accuracy in determining concentration directly.

- a. Sieves--In size analysis by sieving, particles are allowed to fall as far as they will through a nest of sieves that have coarse openings at the top and successively smaller openings toward the bottom. Results of an analysis may be expressed in percentage coarser (retained on or above) each successive sieve size. Materials may be sieved wet or dry. Sieves are agitated mechanically or by hand. Changes in duration or method of sieving and wide variations in openings in sieves of the same nominal size make sieving inexact, especially for particles of irregular shapes.
- b. Visual-accumulation tube--In the visual-accumulation tube method the particles fall from a common level in a sedimentation tube filled with water. The particles form a stratified sedimentation system as they fall. The accumulation of sediment at the bottom of the tube is recorded against time of fall. The method is a fast, economical, and accurate means of determining the fall-velocity size distribution of sands.
- c. Bottom-withdrawal tube--In the bottom-withdrawal tube method of size analysis, particles fall from an initially dispersed suspension of particles in water. Several withdrawals of water and deposited sediment are made from the bottom of the sedimentation tube at timed intervals. The sediment is dried and weighed, and the size distribution is determined by the Odén curve procedure. The method is accurate for samples of silt and clay sizes. A single analysis of a sample containing sands is likely to be erratic.
- d. Pipette--In the pipette method the sediment is dispersed thoroughly in water in a cylinder and then allowed to settle. After a definite settling time a small sample is withdrawn at a fixed point below the water surface. The depth and time give fall velocity, which determines the maximum sediment size at the point of withdrawal. From several withdrawals that represent different particle sizes, the concentration of sediment finer than these selected sizes can be obtained.

- e. Hydrometer--The sedimentation principles in hydrometer analysis are the same as those in pipette analysis. However, concentration of sediment is determined from a hydrometer reading of the density of the water-sediment mixture at the reference depth and time. Because concentration determination with the hydrometer is not very sensitive, sediment is usually analyzed in too great a concentration for unhindered settling of the sediment particles. The hydrometer method is used mainly in analysis of soils.
- f. Centrifuge--Centrifuge methods of size analysis are sedimentation methods in which a centrifuge is used to hasten particle settling. They are especially valuable for analysis of fine silt and clay.
- g. Microscope--Sediment particles may be magnified with a microscope so that their size may be determined. In general, microscopic size is determined from measurement of length along two axes of the particle, and the depth of the particle is estimated or assumed in relation to the measured dimensions. For spherical particles the microscopic method is accurate within the limitations of the available microscope. An analysis requires measurement of at least a few hundred particles. Thousands of particles must be measured to obtain an accurate size distribution for a sample containing a wide size distribution of irregular particle shapes. Microscopic size analysis is tedious and expensive but provides a dependable standard by which to judge the accuracy of other size analysis methods.

35. Comparison of the electronic counter method with other methods--The electronic counter is well adapted to analysis of sediment particles from 1 to 160 microns in size, if apertures from 50 to 400 microns in diameter are used. Apertures up to 2,000 microns are available but the problem of keeping sediment particles larger than 160 microns in suspension makes other methods such as the visual-accumulation tube or sieve methods more desirable for larger particles. Apertures as small as 11 microns are available, but an analysis with so small an aperture requires additional time and the prior removal of all but the clay particles.

A comparison of the electronic counter with other size analysis methods (Table 7) indicates that it is relatively accurate. The accuracy is probably better than any other method except the generally more tedious and often more expensive microscopic analysis of several hundred particles. Because irregularly shaped particles are not accurately sized by microscope, the physical volume of such particles may be more accurately determined by the counter than by the microscope.

The counter requires only a small sample and the concentration can be low. For sand sizes the visual-accumulation tube method is satisfactory so that the upper size limit for electronic analysis is no serious disadvantage. The lower size limits should be studied further -- especially the desirability of using smaller apertures. The cost of an electronic counter analysis is relatively high.

TABLE 7
COMPARISON OF SIZE-ANALYSIS METHODS

Method	Average time per analysis (1)	Size range (microns)	Concentration minimum (ppm) (2)	Estimated accuracy (3)	Cost
Counter	8 hours	1-160	Unlimited (4)	+ 10%	\$30
Sieve	1 hour	44-up	500	+ 25%	4
Visual accumu- lation tube	1/3 hour	62-1000	200	+ 10%	2
Bottom- withdrawal tube	4 hours	2-100	300	+ 40%	15
Pipette	1 hour	1-62	1,000	+ 20%	4
Hydrometer	1/2 hour	1-62	10,000 (5)	+ 40%	2
Centrifuge	3/4 hour	1-62	200	+ 20%	4
Microscope	8 hours or more	1-up	Unlimited (4)	+ 10%	30 and up
Electron microscope	30 hours or more	.05-70	Unlimited (4)	+ 10%	100 and up

- 1) Does not include sample preparation, and assumes several samples are run routinely.
- 2) In a one-pint sample.
- 3) Of median diameter at 95% confidence level.
- 4) Unlimited as long as sample represents the original size distribution
- 5) Extremely variable but generally high.

Information in Table 7 is very general and approximate. In many instances accuracy and time of analysis depend on the size and size range of the sample and on how completely and carefully the size distribution is determined.

Comparative analyses of a glass-bead sample are shown in Fig. 13. The sieve analysis extended down to 44 microns, and the visual-accumulation tube analysis extended down to 62 microns. (Glass beads may be sieved easily and with reasonably good accuracy.) In general, the electronic-counter analysis agreed well with the other two. Electronic sensing of sediments could be combined with visual-accumulation tube or sieve analysis to cover analysis of sizes too coarse for analysis in the counter alone.

Analyses of samples of carborundum grits by the Andreasen pipette and the counter are shown in Fig. 14, which is adapted from Fig. 5 of reference 2. Although the pipette determines fall velocity and the counter measures particle volume, the two methods agree fairly well.

A bottom-withdrawal tube analysis of a sediment sample from the Kentucky River is compared with counter analysis of the same sample in Fig. 15. Eight percent of sand was removed before analysis in the electronic counter. The size distribution from the counter analysis is shown both without the sand and with 8 percent of sand added after analysis. The analyses do not agree well at the fine-particle end of the distribution.

Analyses of a coal dust sample before and after an attrition process are shown in Fig. 16. The two size distributions plot consistently and logically.

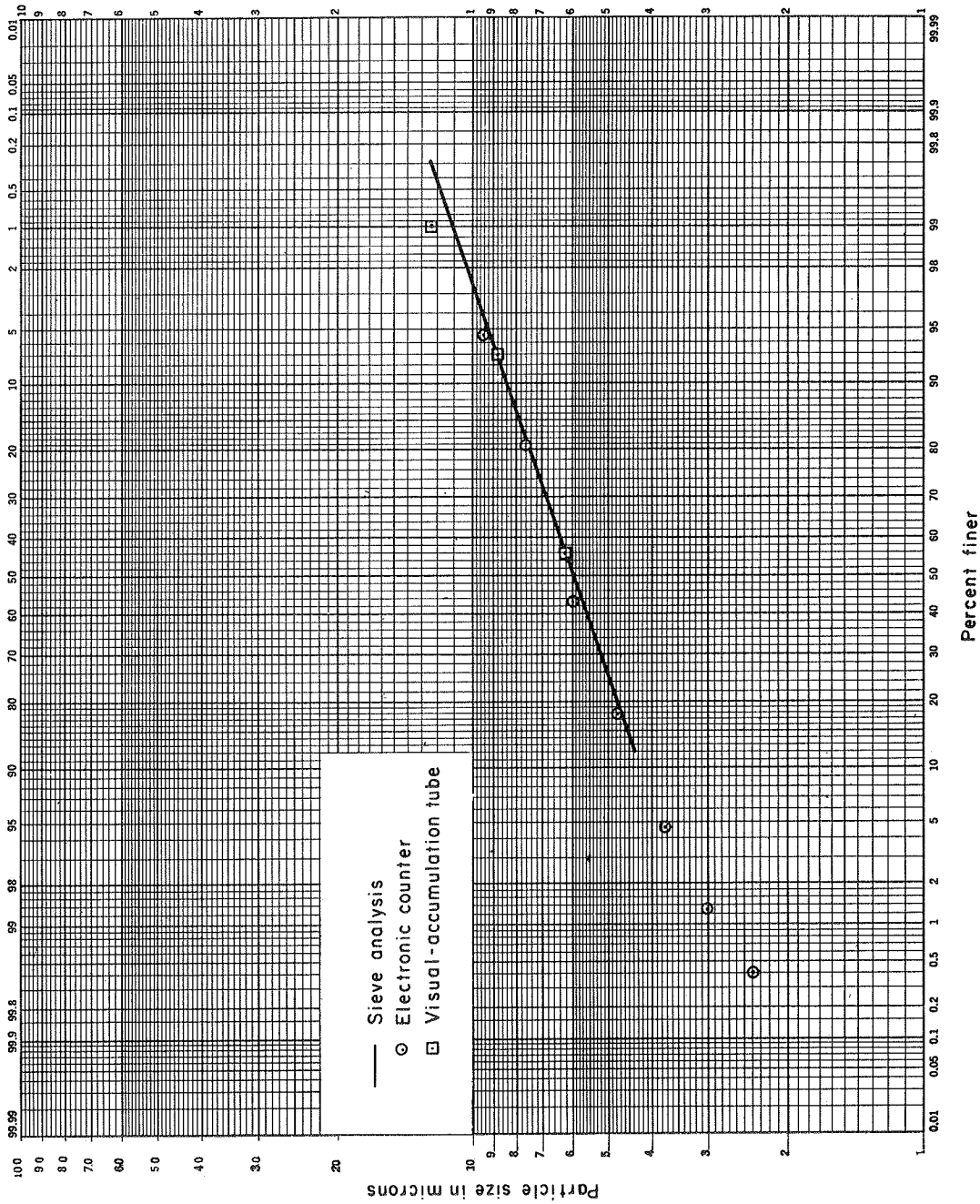


FIG. 13--SIEVE, VISUAL-ACCUMULATION TUBE, AND ELECTRONIC-COUNTER ANALYSES
 OF A GLASS BEAD SAMPLE

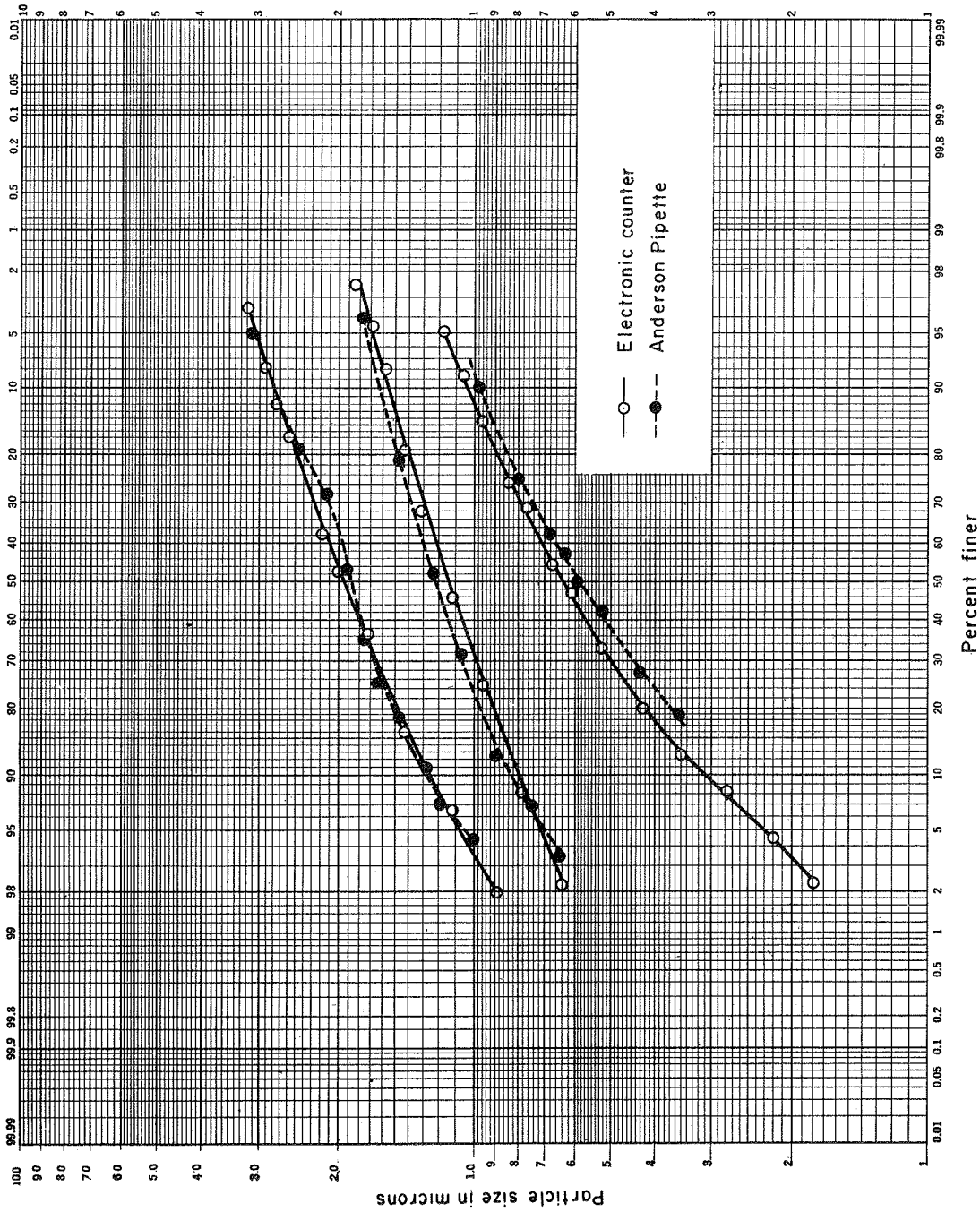


FIG. 14--ELECTRONIC-COUNTER AND PIPETTE ANALYSES OF CARBORUNDUM GRITS

Reference 2

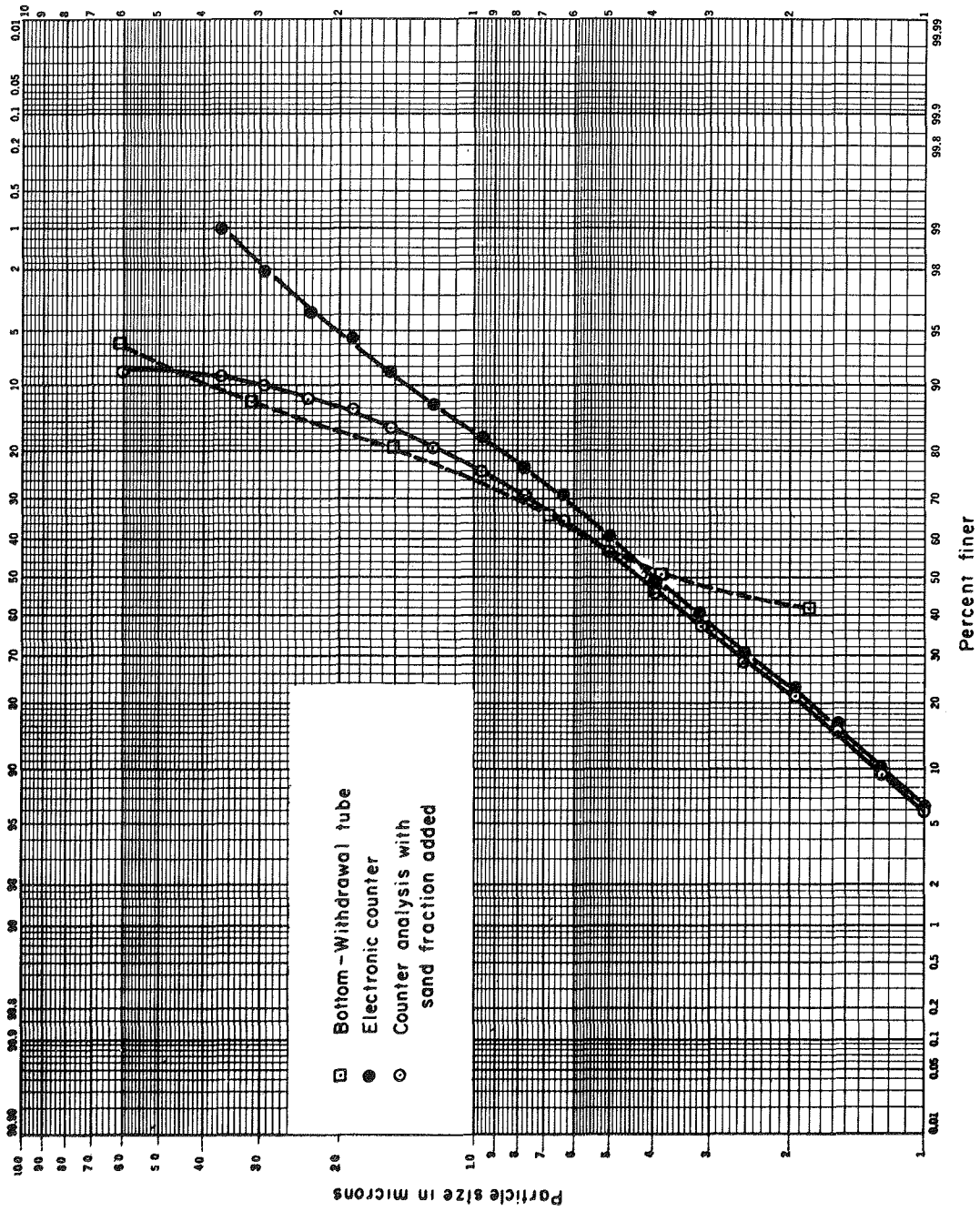


FIG. 15--BOTTOM-WITHDRAWAL TUBE AND ELECTRONIC-COUNTER ANALYSES
OF A KENTUCKY RIVER SAMPLE

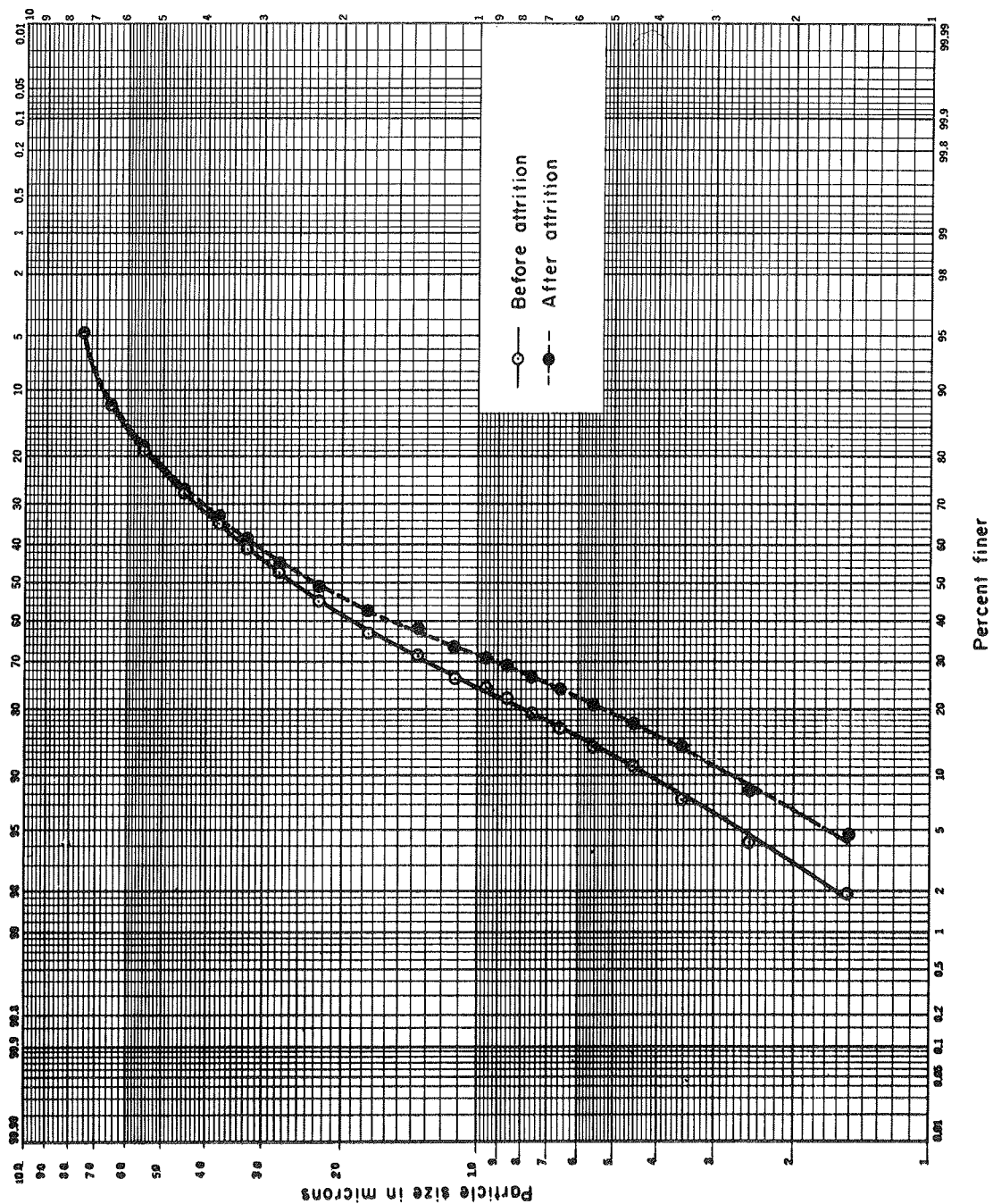


FIG. 16---ELECTRONIC-COUNTER ANALYSES OF A COAL DUST SAMPLE

VII. CONCLUSIONS

36. Conclusions--Electronic sensing is a relatively accurate method of determining the size distribution of sediments between 1 and 160 microns in diameter. In cost of original equipment and time and cost of analysis it is comparable to the microscopic method of size analysis. (See Table 7 on page 63). For irregularly shaped particles the size distribution by the electronic counter is probably more accurate than that from an average microscopic analysis.

The electronic counter as now developed is a valuable laboratory instrument for determining size distribution as a standard to compare with other less accurate but faster methods, which are generally used for routine analyses.

The small aperture through which the particles must pass and the limited range of particle sizes that can be analyzed in a single aperture are serious limitations of the electronic-sensing method. The small aperture size makes direct analysis of sediment on a flow-through basis almost impossible. Analysis in more than one aperture is generally required. Samples must be prepared for analysis in each successively smaller aperture by removing particles so coarse that they would plug the aperture. Computations of the analysis by overlapping data in two or more apertures are time consuming and introduce the possibility of additional errors.

Particles coarser than a given reference size are counted. In many ways analysis would be simpler if only particles within a limited size band were counted. Problems of keeping the coarse particles in suspension throughout the entire analysis and of correcting for coarse particles that were removed prior to analysis would be simplified.

The electronic-sensing method is not well adapted to determination of sediment concentration. Concentration can be determined by the same process as size distribution, but the method is far too time consuming and costly for a routine concentration determination. A program for determining concentrations with the counter was not followed, because such an investigation did not seem to be justified.

Work with the counter has shown that the passage of a single particle through the aperture produces a voltage pulse that is proportional to the volume of the particle. The pulses are practically independent of particle shape, roughness, and material. Also the pulses and particle volumes can be related from one aperture size to another. Progress toward electronic sensing of sediment concentration depends on using the basic facts to develop a more direct method of determining concentration. For example, the single-particle effect suggests that a cloud of particles in suspension must change the resistance of the suspension in proportion to the volume of the cloud of particles, or in proportion to the volume

concentration of particles in suspension. Presumably this relation is not limited to a small aperture.

37. Recommendations for future work--A method of analysis using several apertures in the side of a sedimentation chamber could be studied. The apertures might be connected alternately to the manometer part of the sensing device. The sediment could be dispersed thoroughly and then allowed to settle. Counts in the larger apertures could be made first; and, after the coarser particles had settled, counts in the smaller apertures could be taken.

The feasibility of counting only the particles within a certain size range instead of counting all particles coarser than a given size should be studied. If the electrical pulses could be added electrically to show directly the volume of particles within a size range, the computation of size distribution and concentration would be simplified.

Direct electronic determination of sediment concentration in a large orifice or sediment chamber is so promising that it should be studied and tested carefully. In comparison with the present electronic-sensing method, the problems seem to be: (1) effect of temperature of the fluid suspension, (2) changing concentration of dissolved solids, and (3) need for a basic reference or zero reference to cancel out, or to provide correction for, changes in the electronic circuitry and perhaps in items (1) and (2) above. The advantages would be: (1) fast and direct information on concentration, (2) limitations imposed by very small apertures could be avoided, and (3) the equipment could probably be simplified.

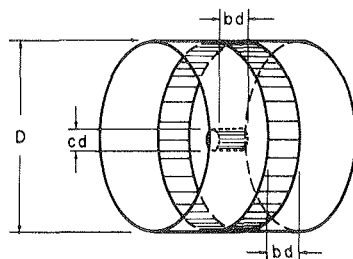
VIII. APPENDIX

38. Derivation of electrical response to particle size--A knowledge of the basic electrical response may help in understanding the operation of the electronic-sensing equipment. The following derivation is based on that of the manufacturer of the electronic-sensing equipment [4].

Assume that:

1. The aperture contents form a cylindrical resistor in which current density is uniform.
2. If the length of the cylindrical resistor is multiplied by an appropriate factor, the cylinder represents the electrically effective zone of the aperture.
3. Individual particles pass through at random and are evenly distributed within the aperture cross section.
4. The electrically effective volume of a particle in the aperture may be expressed as a cylinder having the same resistivity as the particle.

Let the effective cylinder that represents the particle be bd in length, cd in diameter, and a in cross-sectional area, where d is the diameter of a sphere having the same volume as the cylinder.



Consider the aperture as having a disc segment that contains a given particle and has a diameter, D , and a cross-sectional area, A , equal to that of the aperture and a thickness bd equal to the length of the particle cylinder. Let ρ_0 and ρ be the resistivities of the liquid and the particle, respectively.

Then the resistance of the disc segment without the particle is

$$R_0 = \frac{\rho_0 bd}{A}$$

And the resistance with the particle is that of two resistors in parallel

$$R = \frac{1}{\frac{1}{r_1} + \frac{1}{r_2}} \quad \text{or} \quad \frac{r_1 \cdot r_2}{r_1 + r_2}$$

Thus the particle makes a resistance change

$$\Delta R = R - R_0$$

$$\begin{aligned} &= \frac{\frac{\rho_0 bd}{A-a} \cdot \frac{\rho bd}{a}}{\frac{\rho_0 bd}{A-a} + \frac{\rho bd}{a}} - \frac{\rho_0 bd}{A} \\ &= \frac{\rho_0 bd}{A} \left[\frac{A \rho}{a \rho_0 + \rho(A-a)} - 1 \right] \\ &= \frac{\rho_0 bd}{A} \cdot \frac{A \rho - a \rho_0 - A \rho + a \rho}{a \rho_0 + A \rho - a \rho} \\ &= \frac{\rho_0 bd}{A} \cdot \frac{a(1 - \rho_0 / \rho)}{A - a(1 - \rho_0 / \rho)} \\ &= \frac{\rho_0 bd}{A} \cdot \frac{a}{\frac{A}{1 - \rho_0 / \rho} - a} \end{aligned}$$

For an equivalent sphere and cylinder of equal volume, abd equals the particle volume, V

$$\text{Then } \Delta R = \frac{\rho_0 V}{A^2 \left(\frac{1}{1 - \rho_0 / \rho} - \frac{a}{A} \right)} \quad \text{which is Equation 1}$$

With $V =$ particle volume.

39. Method of measuring aperture resistance--Aperture resistance is measured with a d-c voltmeter that is connected to the leads from the electrodes inside and outside of the aperture tube. The aperture resistance is

$$R = \frac{R_s V_0}{300 - V_0} \dots \dots \dots (15)$$

Where R_s = resistance in series with the aperture
 = 215,000 ohms for I setting 8; 415,000 ohms for I setting 7
 = sum of all resistances in series with the aperture, any setting
 V_o = voltage from voltmeter
 300 = voltage across resistances $R + R_s$
 $300 - V_o$ = voltage across R_s

If the average reading of the voltmeter for both polarity orientations is 9.5 at an I setting of 7, the computed aperture resistance is 13,6000 ohms.

Compute the internal resistance of the voltmeter by multiplying its sensitivity in ohms per volt by the full scale voltage of the dial range. If this internal resistance is not at least 100 times greater than the resistance from Equation 14, correct for voltmeter shunt effect as follows:

$$\text{From Equation 14} \quad R = \frac{R_m \cdot R_c}{R_m + R_c}$$

$$\text{and} \quad R_c = \frac{R_m \cdot R}{R_m - R}$$

Where R_c = the corrected aperture resistance
 R_m = the voltmeter resistance for the scale used

For a voltmeter with a sensitivity of 10,000 ohms per volt and for the 10-volt scale, the resistance would be 100,000 ohms.

$$\text{Then} \quad R_c = \frac{100,000 \times 13,600}{100,000 - 13,600} = 15,700 \text{ ohms}$$

Table A-1 shows several aperture resistances and suggests some limitations of the d-c voltmeter method.

An approximate resistance for the aperture alone was added to the aperture series resistance that is indicated by the current-selector dial reading given in column 1, and the resulting approximate total aperture-circuit resistance is listed in column 2. Polarity differences are shown by the measured voltages in columns 3 and 4; they generally are large for the 50-micron aperture.

Each calculated value of current (col. 5) was computed from the applied constant voltage of 300 volts and the approximate total resistance of the aperture circuit (col. 2). Although calculated current usually agreed well with measured current (col. 6), any small difference in current may cause a comparably small difference between the computed resistances through the aperture (cols. 7 and 8).

The resistance in column 7 is computed by dividing average voltage by the measured current; the resistance in column 8 is computed by dividing average voltage by a calculated current. However, the calculated current used in the

TABLE A-1

SOME APERTURE RESISTANCE TESTS AT HIGH SETTINGS OF THE CURRENT-SELECTOR DIAL

Current selector dial setting	Approximate total aperture-circuit resistance (1,000 ohms)	Measured voltages		D-c current (milliamperes)		Computed aperture resistance (1,000 ohms)	
		Current flowing one way	Current flowing other way	Calculated from constant 300 volts	Measured	From average voltage and measured current	From average voltage and calculated current
(1)	(2)	(3)	(4)	(5)	(6)	(7)	(8)
140-micron aperture - 75°F							
5	1,625	2.2	2.25	0.185	0.18	12.4	12.1
6	825	-	-	.364	.36	-	-
7	425	7.8	7.8	.706	.72	10.8	11.1
8	225	14.3	14.3	1.33	1.33	10.8	10.8
9	125	24.1	24.5	2.40	2.38	10.2	10.1
10	75	40.5	39.5	4.00	4.00	10.0	10.0
400-micron aperture - 70°F							
4	3,220	-	-	.093	.10	-	-
5	1,620	1.25	1.65	.185	.185	-	-
6	820	2.4	2.5	.366	.365	6.70	6.70
7	420	4.3	5.0	.714	.725	6.41	6.53
8	220	7.5	8.2	1.36	1.36	5.77	5.78
9	120	13.5	13.5	2.50	2.49	5.42	5.42
10	70	21.7	22.0	4.29	4.25	5.14	5.11
140-micron aperture - 70°F							
5	1,625	2.25	2.68	.185	.18	13.7	13.4
6	825	4.5	5.2	.364	.36	13.5	13.4
7	425	8.0	8.8	.706	.72	11.7	12.0
8	225	14.5	14.9	1.33	1.33	11.1	11.1
9	125	25.6	25.6	2.40	2.38	10.8	10.7
10	75	41.5	41.5	4.00	4.00	10.4	10.4
50-micron aperture - 70°F							
4	3,245	3.0	3.45	.092	.10	32	35
5	1,645	6.2	6.0	.182	.185	33	34
6	845	10.8	11.6	.355	.36	31.1	31.6
7	445	20.8	22.0	.674	.67	31.0	31.9
8	245	33.0	39.0	1.22	1.225	29.4	29.3
9	145	62.0	58.0	2.07	2.05	29.3	28.8
10	95	77.0	87.0	3.16	3.22	25.5	24.4

computation is not exactly the same as the calculated current listed in column 5 unless the aperture resistance is exactly equal to the aperture resistance that was assumed in computing the approximate total aperture-circuit resistance in column 2. More specifically, the calculated current used in computing aperture resistance in column 8 is based on the ratio of the difference between 300 volts and the voltage drop through the aperture to the aperture series resistance, whereas the calculated current in column 5 is based on the ratio of the 300 volts to the approximate total aperture-circuit resistance. Resistances in column 7 are practically the same as those in column 8 despite these slight differences in computing the calculated current and the slight differences between the calculated and the measured currents in columns 5 and 6.

Each of the four groups of data in Table A-1 shows a general decrease of computed aperture resistance as aperture-circuit resistance decreases. The computed resistances generally change less from one setting of the I-current dial to the next at dial settings of about 7-10 than at lower dial settings. Instructions with the electronic-sensing equipment suggest that resistance be measured at a selector dial setting of 7 or 8--a setting high enough to make the voltage across the aperture appreciable.

40. Procedure for median-count calibration--Calibration with uniform particles of known size was discussed in Section 17. The method can be illustrated as follows:

Equation: Median count (count for median size) = $n_o + (n_f - n_o) / 2 = (n_f + n_o) / 2$

Material: Corn pollen, 83.8-microns median diameter

Aperture: Diameter 400 microns

Electrolyte: 1% NaCl solution, temperature 75°F, aperture resistance 4,100 ohms

Instrument dial settings: G = 3, I = 3 (Select a combination that gives a pulse height between 10 and 30 on the threshold dial)

Threshold dial reading for approximate average pulse height: 20 (use threshold dial settings of 10 and 30 for n_f and n_o counts respectively).

Median count

Count number	Threshold dial	
	10	30
1	424	5
2	474	12
3	451	6
4	454	10
5	412	15
6	512	8
7	462	15
Total count	3,189	71
Average	456	10

$$\text{Median count} = (n_f + n_o) / 2 = (456 + 10) / 2 = 233$$

Threshold reading to give median count

By trial and error find the threshold-dial setting that will give a count of about 233 at a desired combination of settings of the G and I dials. For example, use a gain dial setting of 2 for which $\sqrt[3]{G} = 1.122$ and an I-dial setting of 4 for which $F = 0.1258$.

Trial setting of threshold dial	Counts		
	1	2	3
22	373	-	-
25	247	254	-
26	239	225	236

From Equation 6

$$k_d = d \cdot \sqrt[3]{G/(TF)} = 83.8 \cdot 1.122 / \sqrt[3]{26 \cdot 0.1258} = 63.3$$

41. Calibration materials that are commercially available--The electronic counter can be calibrated quickly and easily with essentially mono-sized particles such as those listed in Table A-2. Sizes for calibration should be between 5 and 20 percent of the aperture diameter.

42. Table of coincidence corrections--Adjustments to observed particle count to correct for coincident passages may be computed from Equations 11 and 12 of Section 19. However a table of corrections is often more useful. Table A-3 shows corrections n'' to be added to the average observed count n' to adjust for loss of count from coincident passages for the apertures and counting volume generally used in this work. An operator should remember the count limits for keeping corrections within 10 percent and dilute the sample when the limits are reached.

43. Table of aperture-current factors, F--Aperture current factors F_1 to F_{10} for I-current dial settings from 1 to 10 are shown in Table A-4 for the range of aperture resistances normally encountered with 50- to 400-micron apertures and a 1-percent sodium chloride solution as electrolyte. The values are taken from the manufacturers more extensive but less detailed table [4].

TABLE A-2

CALIBRATION MATERIALS THAT ARE COMMERCIALY AVAILABLE

Particle diameter (microns)	Material	Cost	Source
0.557 0.796 1.30 1.88 2.00 3.04	Polystyrene latex (some other sizes depending on stocks)	Prices on request	The Dow Chemical Co. Order from: Coulter Electronics Industrial Division
4.8 7.2 8.6	Puff ball spores (request debris removal instructions from supplier)	\$10.00 service charge	Particle Information Service 600 S. Springer Road Los Altos, California Paul L. Magill (has catalog of various reference materials)
12.0-13.0 19.0-20.0 45.0-50.0 85.0-90.0	Paper mulberry pollen Ragweed pollen Pecan pollen Corn pollen	\$5.00 minimum charge	Hugh Graham Laboratories Div. Hollister-Stier Laboratories P.O. Box 14197 Dallas 34, Texas (has an extensive catalog of pollens, but not all are mono-sized)
Bulletin 12P-1 prices a series of very narrowly distributed plastic beads, peaking from 15 microns through 95 microns			Ionics, Incorporated 152 Sixth Street Cambridge 42, Massachusetts Morton Rosenstein

Adapted from [4] and slightly expanded

TABLE A-3
CORRECTIONS FOR COINCIDENT PASSAGES

Aperture size in microns							
50		140		140		400	
Counted volume in microliters							
50		500		2,000		2,000	
Count	Correc- tion	Count	Correc- tion	Count	Correc- tion	Count	Correc- tion
	p = 3.125		p = 6.86		p = 1.715		p = 40.0
200	.1	100	.1	500	.4	100	.4
500	1	500	2	1,000	1.7	200	1.6
1,000	3	1,000	7	2,000	7	300	3.6
2,000	12	1,500	15	3,000	15	400	6.4
3,000	28	2,000	27	4,000	27	500	10
4,000	50	2,500	43	5,000	43	600	14
5,000	78	3,000	62	6,000	62	700	20
6,000	112	3,500	84	7,000	84	800	26
7,000	153	4,000	110	8,000	110	900	32
8,000	200	4,500	139	9,000	139	1,000	40
9,000	253	5,000	172	10,000	171	1,100	48
10,000	312	5,500	208	11,000	207	1,200	58
12,000	450	6,000	247	12,000	247	1,300	68
14,000	613	6,500	290	13,000	289	1,400	78
16,000	800	7,000	336	14,000	335	1,500	90
18,000	1,012	7,500	386	15,000	385	1,600	102
20,000	1,250	8,000	439	16,000	438	1,700	116
22,000	1,512	8,500	496	17,000	494	1,800	130
24,000	1,800	9,000	556	18,000	554	1,900	144
26,000	2,112	9,500	619	19,000	618	2,000	160
28,000	2,450	10,000	686	20,000	685	2,100	176
30,000	2,812	11,000	830	25,000	1,070	2,200	193
32,000	3,200	12,000	988	30,000	1,540	2,300	212
34,000	3,612	13,000	1,159	35,000	2,100	2,400	230
36,000	4,050	14,000	1,345	40,000	2,740	2,500	250
38,000	4,512	15,000	1,544	45,000	3,470	2,600	270
40,000	5,000	16,000	1,756	50,000	4,290	2,700	292
42,000	5,512	17,000	1,983	60,000	6,170	2,800	314
44,000	6,050	18,000	2,223	70,000	8,400	2,900	336
46,000	6,612	19,000	2,476	80,000	10,980	3,000	360
48,000	7,200	20,000	2,744	90,000	13,890	3,100	384
50,000	7,812	21,000	3,025	100,000	17,150	3,200	410
52,000	8,450	22,000	3,320	120,000	24,700	3,300	436
54,000	9,112	23,000	3,629	140,000	33,610	3,400	462
56,000	9,800	24,000	3,950	160,000	43,900	3,500	490

TABLE A-4

APERTURE-CURRENT FACTORS, F

Aperture Resistance in ohms	Aperture-current dial readings									
	F ₁	F ₂	F ₃	F ₄	F ₅	F ₆	F ₇	F ₈	F ₉	F ₁₀
0	1.00	0.500	.250	.1255	.0630	.0317	.01620	.00839	.00449	.00254
4,000	1.00	.500	.251	.1258	.0633	.0321	.01650	.00872	.00484	.00290
5,000	1.00	.500	.251	.1259	.0634	.0322	.01659	.00880	.00492	.00300
6,000	1.00	.500	.251	.1260	.0635	.0323	.01668	.00888	.00500	.00310
7,000	1.00	.500	.251	.1260	.0636	.0324	.01676	.00896	.00509	.00320
8,000	1.00	.501	.251	.1261	.0636	.0324	.01684	.00904	.00518	.00330
9,000	1.00	.501	.251	.1261	.0637	.0325	.01692	.00912	.00526	.00340
10,000	1.00	.501	.251	.1262	.0638	.0326	.01699	.00921	.00535	.00350
11,000	1.00	.501	.251	.1263	.0639	.0328	.01706	.00930	.00544	.00360
12,000	1.00	.501	.251	.1264	.0640	.0329	.01714	.00938	.00553	.00371
14,000	1.00	.501	.251	.1265	.0642	.0330	.01729	.00954	.00572	.00392
16,000	1.00	.501	.251	.1266	.0643	.0331	.01744	.00970	.00590	.00414
18,000	1.00	.501	.252	.1267	.0644	.0332	.01760	.00986	.00608	.00436
20,000	1.00	.501	.252	.1268	.0645	.0333	.01776	.01003	.00626	.00457
22,000	1.00	.501	.252	.1270	.0646	.0334	.01790	.01020	.00644	.00480
24,000	1.00	.501	.252	.1271	.0648	.0336	.01805	.01036	.00663	.00503
26,000	1.00	.501	.252	.1272	.0649	.0337	.01820	.01052	.00682	.00526
28,000	1.00	.501	.252	.1274	.0650	.0338	.01835	.01068	.00700	.00549
30,000	1.00	.501	.252	.1275	.0652	.0340	.01850	.01083	.00718	.00572
32,000	1.00	.501	.252	.1276	.0653	.0341	.01864	.01098	.00736	.00596
34,000	1.00	.502	.252	.1277	.0654	.0343	.01878	.01114	.00755	.00620
36,000	1.00	.502	.252	.1278	.0656	.0344	.01892	.01130	.00773	.00644
38,000	1.00	.502	.253	.1279	.0657	.0346	.01906	.01146	.00791	.00668
40,000	1.00	.502	.253	.1280	.0658	.0347	.01920	.01161	.00809	.00691

Adapted from [4]

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