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Report 00

**Pipe and X-RAY Grain-Size Analyzers:
Comparison of Methods and Basic Data**

May 2000

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REPORT OO

PIPET AND X-RAY GRAIN-SIZE ANALYZERS: COMPARISON OF METHODS AND BASIC DATA

May 2000

By

John Skinner

Published by

FEDERAL INTERAGENCY SEDIMENTATION PROJECT

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CONVERSION FACTORS AND ABBREVIATIONS

For readers who prefer English units instead of the metric units used in this report, values may be converted by using the following factors:

Multiply	by	to obtain
micrometer (mm)	3.937×10^{-5}	inch
millimeter (mm)	3.937×10^{-2}	inch
centimeter (cm)	3.937×10^{-1}	inch
cubic centimeter (cm ³)	6.102×10^{-2}	cubic inch
gram (g)	3.527×10^{-2}	ounce, avoirdupois

Temperature in degrees Celsius (°C) can be converted to degrees Fahrenheit (°F) by using the following equation:

$$^{\circ}\text{F}=1.8(^{\circ}\text{C})+32$$

Suspended-sediment concentration is expressed in this report as milligrams per liter (mg/L). It is computed as one million times the ratio of the dry weight of sediment in grams to the volume of the mixture in cubic centimeters.

Abstract

Size distributions of sediment grains in rivers, estuaries, and reservoirs are important factors in sedimentation processes. The traditional measurement technique for determining grain-size distributions is the pipet procedure; however, it has come under increasing criticism because of its high cost and heavy demands on labor. After reviewing several methods, an X-ray instrument was selected as a candidate for replacing the pipet. This report reviews the rationale leading to selection of the X-ray technique and presents data comparing the two methods. The data indicate that compared with a pipet analysis, an X-ray analysis gives grain-size distributions biased toward finer particle sizes.

Introduction

A major function of sediment laboratories is measuring grain-size distributions of sediments in rivers, lakes, and estuaries. One size class of particles, those smaller than 62 μm (micrometers), has traditionally been analyzed by the pipet procedure, which is based on sedimentation rates (Guy 1969). Particles are mixed with water in a cylinder and then allowed to settle under the influence of gravity. As sedimentation proceeds, subsamples are withdrawn at prescribed times and depths in the cylinder. Sediment concentrations in these subsamples are then used along with Stokes' Law to determine particle-size distributions.

Although the pipet method has been a standard for many years, it has become increasingly objectionable because of its high analysis costs and heavy demands on labor. Efforts to automate the method have been only marginally successful. Consequently, the Technical Committee of the Interagency Sedimentation Committee and the Sediment Action Committee of the U.S. Geological Survey have attempted to locate an instrument that is not only more efficient and economical but also one that ensures consistency in particle-size databases.

Since about 1970, the Federal Interagency Sedimentation Project has encouraged the testing of new instruments for replacing the pipet method for grain-size analysis of soils and fluvial sediments. Attention has focused on a commercially-made instrument that measures grain-size distributions automatically. Samples are dispersed chemically and mechanically to form water-sediment mixtures, which then undergo gravity-induced settling in a special glass-walled cell. As sediment particles settle, their concentrations are sensed with a collimated X-ray beam that, after passing through the cell, enters a detector that registers beam intensity. Sediment grain sizes are automatically computed from Stokes' Law that gives fall rates for spheres settling in fluids. Concentrations and particle diameters are recorded continuously on a x-y plotter, an integral part of the analyzer.

Purpose

The purpose of this report is to (a) review the rationale leading to adoption of the pipet procedure and subsequently to adoption of the X-ray instrument as a possible replacement for the pipet, (b) review theories supporting the pipet and X-ray technique and to examine their similarities and differences, and (c) present the data collected for comparing the two methods. This report supplies data referenced in the report entitled, "Evaluation of pipet and X-ray procedures for determining particle-size distributions of sediment," by Rollin Hotchkiss, 1994.

Scope

This report focuses on instruments for analyzing fluvial sediment in rivers, estuaries, and reservoirs. Furthermore, the instruments are those suitable for analyzing particles that are (a) *smaller* than about 62 μm , (b) *shaped* irregularly as is characteristic of mineral fragments that are eroded, transported, and deposited by flowing water, and (c) *chemically* identical or similar to material suspended and deposited in natural water bodies.

Sizes of Sediment Particles

At first examination, designating the size of a sediment particle seems a simple, almost trivial problem. In the case of spheres, only their diameters must be specified. Most spherical particles are, however, synthetic products manufactured for calibrating particle-sizing instruments. In sedimentation work, naturally occurring spherical particles are rare. In nature, particles consist of crystals, plates, rods, or random shapes produced by grinding and crushing processes. Sizing nonspherical, irregularly shaped particles involves several techniques each yielding different results.

One popular size measurement technique is to examine particle images projected as two-dimensional (2-D) shapes. Size measurements are then made manually with the aid of a microscope or made automatically with the aid of sophisticated image-processing equipment. With the latter, outlines of particles are projected onto a television (TV) screen. Particle grain-size signals are coded in the fluctuating intensity of the electronic beam, which strikes the backside of the screen. These signals are then processed by a computer to obtain particle diameters. Even after particles have been reduced to 2-D images, several choices still exist as illustrated by the following definitions for sizes of 2-D images.

Projected area diameter—A particle's image is projected onto a 2-D screen. The enclosed area of the image is then measured and equated to the diameter of a circle with the same area. Areas are usually calculated automatically by image-processing analyzers.

Feret's diameter—Two parallel lines are drawn through points on the extremities of the particles perimeter as shown in Figure 1. Feret's diameter is the distance between the parallel lines. The diameter measurement is usually made automatically by an image-processing analyzer.

Martin's diameter—A chord is drawn across the particle outline so that the enclosed area above the chord equals the area below the chord. The chord's length is designated Martin's diameter.

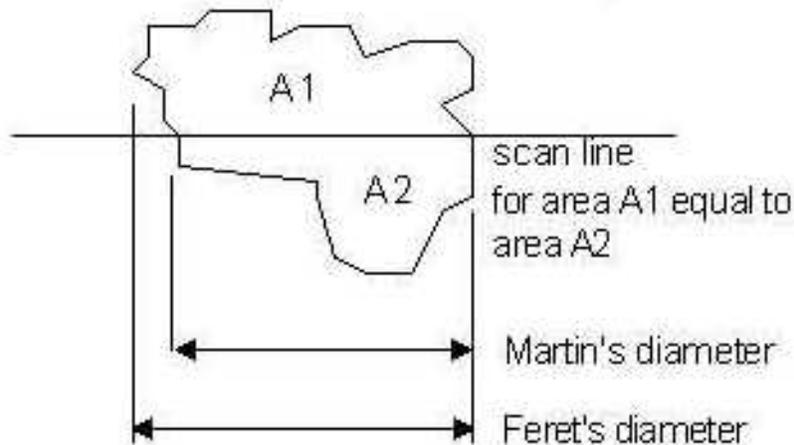


Figure 1. Feret's and Martin's diameters.

Feret's diameter is measured between parallel lines drawn through the particle's extremities.

Martin's diameter is measured between intercepts along the scan line dividing the image's area in half.

Crofton's diameter—Chords are drawn through the particle's outline in random directions. Crofton's diameter is the mean of the chord lengths.

Equivalent perimeter diameter (EPD)—The perimeter of the particle's image is measured. The particle's EPD is the diameter of a circle having the same perimeter.

Minimum linear diameter (MLD)—A particle's MLD is the shortest chord that can be drawn across the particle's image.

With modern data processors, all particle diameters can be obtained without displaying particle outlines. Unfortunately, the problem with diameters obtained from 2-D images is that most depend upon the image's orientation relative to the grid lines or scan lines on the display. Another problem is that a particle's dimensions that are *normal* to the projection screen are hidden: a round, plate like particle is indistinguishable from a sphere. Parent (1967) comments, "It is known that direct microscopic measurement of irregular particles yields results which are at least 25% larger than results obtained by other methods." The problem is that the longest dimensions of particles tend to align parallel to the slide's surface; consequently, only these longest dimensions are seen through the microscope. To correct these inaccuracies, the following diameters based on *three* dimensions are used:

Equivalent volume diameter—This diameter is sometimes referred to as *the nominal diameter*. The particle's volume is determined and equated to the diameter of a sphere having an equal volume.

Aerodynamic diameter—This diameter is based on drag forces exerted on the particle as it moves through a fluid, usually air. The diameter is commonly used in gauging characteristics of aerosols and is usually measured with the aid of cascade impactors.

Equivalent diffusion diameter—This diameter is derived from the diffusion (scattering) rate that a particle undergoes as it moves randomly about in a fluid under impacts of molecular collisions. This movement is sometimes referred to as *Brownian motion*.

Sieve diameter—This is the length of the side of a square sieve opening through which a given particle will just pass.

Sedimentation diameter—A particle's settling rate is measured in a specified fluid at a specified temperature. The particle's diameter is equated to the diameter of a sphere, which settles at the same rate.

Standard sedimentation diameter—A particle's settling rate is measured in quiescent distilled water at 24 °C. Its diameter is equated to the diameter of a sphere that has the same density as the particle and that settles through the fluid at the same rate.

Fall diameter—A particle's settling rate is measured in quiescent distilled water at a temperature of 24 °C. The particle's fall diameter is equated to the diameter of a sphere having a density of 2.65 g/cm (the density of quartz) and settling at the same rate as the particle. Fall rates must be free of interference from container walls and other particles.

Particle-Sizing Needs In Sedimentation

With several particle-diameter definitions to choose from, Oden (1924), a pioneer in developing particle-size analysis methods, explained that the concept of particle size is mainly a matter of convenience and an aid in visualization. He remarked, “Though the equivalent radius is of great value for the image it gives us of the size of the soil particles, it has in this paper been introduced only as a secondary variable, whereas as the size variable chosen has been the velocity of the fall, this quantity being free from theoretical suppositions.”

Despite Oden’s preference for classifying particles by fall rates rather than diameters, many researchers still regard particle diameter as a critical factor. Scientific literature contains numerous statements stressing that particle size influences curing times for concrete, reaction times for medicines, hiding power of paints, along with flavor and nutrient values of foods. As other examples, Berg and Youngdahl (1961) comment on choosing particle-size dimensions: “Area is useful for reflective glass beads for highway signs and for many catalysts. For correlating abrasive performance, minimum and maximum linear dimensions are of great significance in assessing for large scratchy particles or excessive fines.”

Most authorities agree that the appropriate step in selecting a diameter is to choose one that reflects applications for the data. Expressing needs and applications in the field of sedimentation in the early 1940s, the Interagency Committee on Water Resources (Report 4, 1941) commented, “...the determination of the actual *settling rate* of a particle is generally of more importance in sediment studies than an exact measure of volume or diameter. The common method is to *express the settling rate in terms of a diameter of a sphere of the same specific gravity, which will fall at that rate*. Some workers have even suggested expressing sizes of particles directly as fall velocities rather than converting into hypothetical spheres. There would be a distinct advantage in having an entire sample analyzed hydraulically rather than by a comparison of hydraulic and sieving methods, as the former eliminates the rather abrupt break in the size curves that not infrequently results when the two methods are combined.” Notice the italicized phrase mentions the need to select a diameter-based settling rate. The diameter definition was gradually refined; the assumed specific gravity value for particles was fixed at 2.65; and the term *fall diameter* was introduced and became widely accepted.

A few years later, the Interagency Committee on Water Resources (Report 7, 1943) remarked, “A recent study has shown that the movement of material in rolling or sliding along the bed also is related more closely to its settling rate than to its size. Therefore, from the viewpoint of the hydraulic engineer, settling rate appears to be the most important property of sediments carried in suspension by flowing water.”

In the late 1950s, the Interagency Committee on Water Resources in Report 12 (1957) reiterated calls for fall rates by stating, “The fundamental property governing the motion of a sediment particle in a fluid is its *fall velocity*, a function of its volume, shape, density, and the viscosity and density of the fluid. As research in sediment transportation becomes more refined, it is necessary that sediments be classified on this basis.”

Researchers at the 1991 proceedings of the Fifth Federal Interagency Sedimentation Conference reinforce the notion that particle-fall diameter is still an important way of characterizing sediment grain sizes. The majority of papers at the conference dealt with the physical aspects of sediment transport and were closely linked to the physical interaction between liquid flows and sediment grains. Simons (1991) in discussing deposition patterns at dams and in reservoirs stresses the importance of gauging *fall rates of particles* as a necessary step in predicting deposition pattern. Han and He (1991) stress the importance of *particle-settling velocities* in transport equations for various particle-size groups. Dou (1991) explains that similarity scales for suspended-sediment transport are based on *particle-settling velocities*, sediment concentration, and sediment-carrying capacity. Pugh and Dodge (1991) explain that “*settling velocity* is very important in determining when a particle will remain at rest or how far it will travel once lifted into the flow.” Klumpp (1991) discusses the adjustment of *particle-settling velocities* to obtain sediment discharges in a model. Wong et al. (1991) explain that equations governing three-dimensional (3-D) sediment transport in models depend on *particle-fall velocity*. Bernard et al. (1991) discuss sediment and sediment-attached substances. They remark, “The design of tactics to trap sediment must consider the effective *particle-settling velocities* if the practice is to be effective.”

Pipet Procedure: Its Advantages and Disadvantages

The theoretical basis for relating particle-settling rates to particle sizes was established in 1851 when Stokes derived an equation for fall rates of spheres settling through fluids. Stokes assumed viscous forces govern fall rates. Experts do not fully agree on the range of particle sizes to which the theory applies; but in fluvial sedimentation, an upper limit of about 62 mm is normally used if the particles have a specific gravity of 2.65 and settle in water at 20 °C.

Applying the Stokes theory to particle sizing is straightforward. A sample consisting of a mixture of water and sediment is processed to remove organic material. Then the remaining particles are transferred to distilled water in a 1-L cylinder about 55 mm in diameter. After adjusting water temperature and adding a dispersing chemical, the operator mixes the suspension with a churning motion. When the particles are mixed and scattered throughout the column, stirring is stopped and at the same time a timer is started. At prescribed times and depths computed from Stokes' equation, small samples are withdrawn into a pipet for drying and weighing. Often, six withdrawals are made: the first being immediately after stirring, the last, 2.5 to 4 hours later depending on temperature and the chosen withdrawal depths. The six samples are dried, and the sediment weights are plotted to form a cumulative distribution curve of percent-less-than (PLT) values versus particle-fall diameter. The entire process is known as the pipet procedure.

In the pipet procedure, making the subsample withdrawals takes only a few minutes, but performing the entire analysis beginning with sample preparation and ending with a size distribution takes several hours. Much of this time is spent waiting for the small particles, those in the 4-mm and 2-mm sizes, to settle. Horiba (undated) estimates one operator can perform about four pipet analyses simultaneously each day.

Although the pipet procedure is widely used, it has drawn increasing criticism for its demands on labor and budgets. Appel (1952) commented, "Sampling equipment and techniques have been perfected to such a point that thousands of sediment samples are obtained each year, but only a relatively few of these are analyzed for particle size frequency distributions simply because present methods are too time consuming and costly." Welch's et al. comments (1979), which were offered nearly three decades later, reflected the same problem. They stated, "Besides requiring operator care and precision, the pipet procedure is laborious and time consuming, which limits the number of samples which can be analyzed. Therefore, a method is needed which eliminates some of the disadvantages of the pipet procedure for routine particle size analysis and for analysis of suspended sediment samples."

Acquisition of The X-RAY Database

In about 1976, the Interagency Committee on Water Resources attempted to locate a technique or instrument for replacing the pipet. The search was weighted in favor of instruments based on sedimentation-sizing methods to maintain consistency in the fundamental approach and thereby avoid introducing misleading changes in historical records. Much of the attention focused on an instrument combining sedimentation rates for particle sizing with X-ray attenuation for determining cumulative-distribution functions. Since the Sedimentation Project did not have access to an X-ray analyzer, Federal agencies ran many of the tests and shared the data with the Project. Some of the results were published in technical journals by the agencies. In a few instances, sedimentologists in foreign countries participated in the testing effort. Through these sources, the Sedimentation Project gradually acquired a body of data.

In 1986, the Technical Committee of the Sedimentation Project asked Dr. Rollin Hotchkiss to recommend statistical tests for analyzing the data. His response (Hotchkiss 1994) was published through the Federal Interagency Sedimentation Project. Later, in 1993, the Technical Committee asked the Project to recommend a format for tabulating pipet and X-ray data in the Project's possession. This report responds to that request.

The data in this report include the earliest tests that were conducted from 1976 to 1979 by the Agricultural Research Service at Chickasha, Oklahoma, and at Oxford, Mississippi. From 1984 to 1986, several tests were performed at the U.S. Geological Survey's Cascades Volcano Observatory at Vancouver, Washington. Some of these were made by scientists from the Peoples Republic of China (PRC). The author wishes to thank personnel from these centers for sharing results with the Federal Interagency Sedimentation Project. Also, Doctors Dennis Helsel of the U.S. Geological Survey and Paul Britton of the Environmental Protection Agency made valuable contributions by suggesting methods for evaluating differences between data collected with the pipet and the X-ray Sedigraph.

Pipet Theory and Dispersing Techniques

The theory of the pipet is based on Stokes' Law, which relates the diameter of a sphere to its fall velocity in a fluid. The fundamental equation is

$$w = [(gd^2/18\eta)][(g_s-g)/g]$$

where

w = particle fall velocity in cm/s

g = acceleration because of to gravity in cm/s²

d = particle diameter in cm

η = fluid kinematic viscosity in cm²/s

g = specific weight of fluid surrounding the particle in g/cm³

g_s = specific weight of the particle in g/cm

In deriving the equation, Stokes made four assumptions: (a) the particles are large enough to overcome Brownian motion, which is the random movement caused by impacts with molecules of the surrounding fluid, (b) the particles are smooth, rigid, and spherical or nearly spherical in shape, (c) the particles are falling at a steady rate in a fluid of infinite extent, and (d) the fall rates are governed by fluid viscosity. Beyond stating these assumptions, Stokes offered no estimates of the range of particle sizes covered by the equation.

Stokes' Law applies if momentum forces are negligible and fluid viscosity is the controlling factor; however, Lehman (1988) notes that opinions differ on the upper-size limit of particles governed by Stokes' Law. For particles settling in water, some researchers believe the law applies to particles as large as 150 μ m. Rouse (1937) shows significant deviations occur only if the spheres are larger than about 450 μ m. Horiba (undated) comments, "It seems to be fairly generally agreed that wall effects are unlikely to be important when vessels of the size normally recommended are used and *when the particles are smaller than 100 μ m.*" Guy (1969) expresses another opinion regarding the upper limit. He states, "Stokes' Law is not applicable for quartz particles larger than about 60 μ m falling in water..." In applying the equation to the pipet procedure, most sedimentologists have adopted Guy's estimate.

Before leaving the subject of sphere sizes and fall rates, it should be noted that Stokes' Law is only one of several expressions involving particle-settling rates. Rubey (1933) derived the following equation for spheres large enough to generate significant inertia forces and consequently are too large to be covered by the Stokes equation:

$$w = \sqrt{a}$$

where

$$a = [(20/3)g(g_s-g)d^3+360,000g^2-600g]/gd$$

Gibbs et al. (1971) published tables of fall rates of spheres ranging in diameter from 0.1

mm to 5,000 mm. According to his assessment, the combined precision of the measurements was about 2% at the 95% confidence level. Gibbs also derived an empirical equation that, when used with correction factors, applies to spheres as large as 50,000 mm in diameter.

In 1924, Oden presented a method for experimentally obtaining size distributions of samples containing broad ranges of particle sizes settling at Stokes' fall rates in fluid columns. In his paper, Oden (1924) describes the concept as follows: "If again the particles are of various sizes and therefore fall with different velocities, certain groups in time disappear completely from a certain layer and the concentration there decreases continuously while the sedimentation proceeds." His theoretical work was based not only on Stokes' Law but on four assumptions: (a) complete dispersion of the soil particles, and prevention of any tendency to coagulation, (b) uniform distribution of the particles at the beginning of the measurement, (c) constant temperature so that no convection currents and disturbances occur during sedimentation, and (d) a concentration so dilute that the particles do not interfere with one another during their fall through the liquid, and the density of the suspension will never vary greatly from that of the pure liquid. In the following sections, some of the practical implications of each of these assumptions are examined.

The practice of dispersing samples prior to performing size analyses has been the subject of numerous debates. In natural water bodies, some degree of flocculation is always present. It is therefore reasonable to analyze samples in their more natural, flocculated states in which a particle may actually consist of several smaller particles loosely bound together. On the other hand, suspensions of this kind are usually fragile and unstable since the flocs can combine or separate easily. The end result is that a particle-size distribution can seldom be measured with consistent results. Nelson (1948) summarized the philosophy of dispersing samples prior to making a grain-size analysis. He explained, "Undoubtedly, flocculation plays an important role in the natural sedimentation process occurring in rivers and reservoirs. Unfortunately, we do not know just what forces are involved in the natural sedimentation process... There appears to be a new field of research that should be covered in order to clarify this phase of sedimentation. In the meantime, it is my opinion that the effects of dissolved solids should be eliminated as far as possible when making laboratory size analysis with the bottom withdrawal tube... Therefore, until the effects of flocculation in the laboratory apparatus and that which occurs in nature can be correlated, the bottom tube size analysis should be made in distilled water with the assistance of a dispersing agent when necessary." Although Mr. Nelson was speaking of the bottom withdrawal-tube apparatus, his philosophy of dispersing samples has been applied to pipet analyses as well (Guy 1969).

Guy (1969) explains dispersion techniques and defines the purpose of pipet measurements. He comments, "In sediment investigations the ultimate particle size should be used unless otherwise specified. Ultimate particle size in fluvial sediment can be defined for practical purposes as the particle size obtained by standard methods of preparation and dispersion of sediment samples."

Table 1 illustrates the influence of dispersants on pipet results. Norman Welch collected all data on soil samples from a 0-inch to 6-inch layer of soil from a research watershed at Chickasha, Oklahoma. (Personal communication, Norman Welch to J. Skinner, 1977).

Table 1. Particle-size distributions obtained with and without chemical dispersants. All analyses were run by pipet. Each sample was analyzed twice, once with the dispersant and once without.

Breakpoint in mm	50	31	20	10	7.8	4	2
Percent-less-than Values							
Without Dispersant							
Sample Number							
54	99.5	79.5	63.4	48.3	45.2	37.5	25.9
56	90.6	80.0	57.4	42.8	38.1	31.8	28.2
59	104.8	89.3	74.8	22.0	13.5	15.5	9.9
60	93.5	67.2	50.9	23.1	14.3	8.8	9.7
62	101.8	91.8	82.8	15.5	13.4	11.5	10.4
66	97.1	73.1	58.6	19.3	15.6	6.5	5.8
69	101.3	81.4	58.3	14.4	10.0	8.5	8.1
70	101.5	85.5	78.1	13.2	11.1	9.5	6.2
With Dispersant							
54	97.9	78.5	60.6	48.3	42.6	35.4	35.6
56	95.8	79.8	60.1	45.6	39.1	34.7	32.6
59	94.0	78.0	66.0	50.2	48.9	40.0	36.5
60	83.8	56.4	40.3	33.8	31.9	27.0	26.6
62	96.1	83.4	76.2	66.0	63.2	52.9	48.1
66	89.7	63.2	45.7	34.5	32.7	28.7	26.3
69	88.1	66.8	56.4	46.8	48.7	41.9	38.7
70	90.3	72.5	63.6	54.3	49.3	44.0	39.0

As the data show, a dispersing agent can strongly influence grain-size distributions at the fine end of the particle-size scale. For example, on sample 62, adding a dispersant changed the PLT value at the 2-mm breakpoint from 10.4% to 48.1%. At the coarser end of the scale, the influence was smaller: at the 20-mm breakpoint, the PLT value changed from 76.2% to 82.8%. Shifts in the distributions were inconsistent among the samples. This probably indicates that influences of the dispersant depended on mineral composition of the particles. Values in the table greater than 100% were a result of measurement errors.

The efficiency of floc separation generally increases as more dispersing chemicals are added. Again, the influence is greatest for the finer particle sizes. Paul Allen (Personal communication, April 1976) reported the data in Table 2 for pipet runs on a soil sample collected near Chickasha, Oklahoma. Notice the decline in PLT values with a decrease in dispersant concentration. X-ray (Sedigraph) data for these runs are missing, but Allen reports, "The standard 5% solution gave percentages closest to the Sedigraph curve."

Table 2. Influence of dispersant concentrations on measured particle-size distributions.
Table shows percent-less-than values obtained from pipet analyses.

Dispersant (Calgon) concentration in percent	Breakpoints in mm						
	50.0	31.2	20.0	10.0	7.8	3.9	2.0
5	92.9	79.3	60.9	47.2	44.1	36.1	33.4
1.25	94.5	78.1	65.3	49.2	46.2	36.3	27.0
0.31	96.9	79.1	63.5	47.9	44.6	32.5	22.1
0.078	94.4	79.3	64.8	46.2	41.9	31.7	20.3

The *chemical composition* of dispersants influences results obtained in particle-size tests. Paul Allen of the Agricultural Research Services (ARS) at Chiskasha, Oklahoma (Personal communication, J. Skinner, June 17, 1975) reported the data in Table 3 that compares four dispersing chemicals: Calgon, sodium hexametaphosphate, Sedisperse E and Sedisperse B. Calgon is the standard chemical used by ARS at Chickasha; the U.S. Geological Survey uses sodium hexametaphosphate; Sedisperse E and B are commercial dispersants. Mr. Allen observed that Sedisperse E and B failed to mix with distilled water and produced an *oily appearance*. Notice the differences for particle breakpoints smaller than about 8 mm. All runs were on ACF, a commercially mined sediment commonly used for standard particle-size testing.

Table 3. Influence of dispersant types and concentrations on measured particle-size distributions.
Table shows percent-less-than values obtained from pipet analyses.

Dispersant Type						
Breakpoints in mm	Calgon ¹		Sodium hex ²		Sedisperse E ³	Sedisperse B ⁴
	Run 1	Run 2	Run 1	Run 2		
62.5	97.1	97.5	97.0	97.1	97.1	97.6
50.0	93.1	93.0	90.3	88.2	90.7	86.6
31.2		78.6	80.4	78.1	87.8	79.5
20	68.3	71.4	69.9	67.2	68.6	67.0
10	54.7	55.3	52.0	51.7	47.2	44.4
7.8	49.1	50.0	47.1	45.9	42.4	38.6
3.9		35.0	32.7	30.5	23.0	24.0
2	25.1	23.5	23.6	19.9	13.7	14.5

¹Concentration of 50 g/L

²Concentration of 3.7 g/L of sodium hexametaphosphate and 7.97 g/L of sodium carbonate

³Concentration of 10 g/L

⁴Concentration of 10 g/L

In summary, the presence of a dispersant, its concentration level, and its chemical composition are factors in determining the amount of sediment measured in various size ranges. In most

cases, the influence is strongest at the fine end of the size scale and can be very strong for particles smaller than about 8 mm. Dispersion may account for some of the differences between pipet and X-ray analyses to be discussed later. In future testing, the importance of adequate dispersion must not be overlooked.

Returning to Oden's theory, his second assumption was that at the beginning of the sedimentation process, particles are uniformly scattered throughout the sedimentation column. In modern practice, mixing is usually achieved by churning the mixture with a perforated paddle. At the instant the paddle is withdrawn, it is assumed that settling begins at Stokes' Law rates; however, turbulent eddies persist for several seconds; consequently, the exact instant to begin timing cannot be precisely determined. Some laboratories begin timing immediately after churning is stopped but others have a short waiting period. In testing pipet procedures (Corps of Engineers 1950), researchers made 28 pipet runs on sediment from the Missouri River. Tests were run in 500 ml cylinders with internal diameters of 4.5 cm. Initial starting depths were about 0.28 m. Other tests were run in 1000 ml cylinders with internal diameters of about 6 cm. After evaluating the results, researchers suggested procedural changes. Prior to the tests, initial withdrawals for concentration were taken as soon as possible after dispersion, but researchers commented, "It was found that in several instances the indicated concentration of the withdrawal at about 45 seconds (settling time for 62.5 m) was greater than the concentration of the sample as determined by drying and weighting all material." The initial withdrawal procedure was later revised as explained in the report, "... the time required to withdraw the concentration aliquot sample has been increased from 4 seconds to 25 seconds. This action is taken as there is the possibility that filling the pipet rapidly may not give a sample representative of all of the sediment in the suspension." Horiba (undated) states, "...it is impossible to fix precisely the moment at which turbulence ceases and laminar flow sedimentation begins. All that can be done is to standardize the technique." Differences of a few seconds in withdrawal times has little influence at small-particle breakpoints for which a few *hours* elapse before making withdrawals; however, even short delays can strongly influence results at large-particle breakpoints for which only a few seconds elapse before making withdrawals.

Oden's third assumption requires a steady temperature in sedimentation columns. In practice, temperature shifts can be minimized but not completely eliminated. Guy (1969) remarks, "In laboratories where the temperature varies considerably, it is desirable to use a constant temperature water bath for the sedimentation cylinders." Actually, two classes of temperature shifts are involved. The first class of shifts are those that change slowly and induce only weak convection currents. These influence fall rates primarily by changing the density and viscosity of the liquid. The second class of shifts are those that are localized in the column and occur rapidly. These shifts may cause convection currents strong enough to either *lift* particles or slow their rate of descent. Even under controlled conditions, temperature shifts undoubtedly contribute to degradation in repeatability of pipet analysis. Horiba (undated) states, "Even with good thermostatic control, it is difficult to keep the temperature constant over long periods, and for all practical purposes this imposes a lower size limit on the analysis with gravitational settling of *about* 2 mm when water is used as the suspending fluid."

Oden's fourth assumption requires that particle concentrations be small enough to avoid particle-to-particle interactions: that is, each particle must be free of vortices and eddies created by the settling of neighboring particles. Horiba (undated) states, "More than one worker has stated that the volume concentration should be less than 0.05% if interference between particles is to be assumed negligible but it is clear that it is impractical to use such low concentration if the amount of solid in the sample is to be determined by weighing." Appel (1952) remarks on concentration effects in sedimentation columns: "It has been known for a long time that the presence of numerous falling particles in a column of liquid would probably have an effect on the fall velocities of the individual particles. Now both theoretical and experimental evaluations of the concentration effect on fall velocity have been made and brought to the attention of the profession. For statistically homogeneous concentrations as low as 1000 parts per million, surprisingly larger reductions in velocity—10 %—were recorded. For higher concentrations, the reduction in velocity was found to be even greater." Swift et al. (1972) suggest, "For the pipet and hydrometer methods, a concentration of 15 g/L is optimum, and 5 g/L is a lower limit below which error is excessive." Guy (1969) mentions a more practical limit of 2-5 g/L for drying and weighing. For X-ray analysis, much higher concentrations are used.

Another assumption, not specifically stated by Oden but nevertheless imbedded in pipet theory, is that container walls do not influence particle-fall rates. McNown et al. (1948) derived the following correction factor for spheres for which $d/D < 0.25$ and for which inertia effects are negligible:

$$w_o/w_d = 1 + (9d/4D) + (9d/4D)^2$$

where

w_o = the fall velocity of single particle in a fluid of infinite extent in cm/s

w_d = the fall velocity of a particle in a sedimentation column in cm/s

d = the particle diameter in cm

D = the sedimentation column diameter in cm

Gibbs (1972) discusses significant wall effect and particle-interference errors in sedimentation columns in which *stratified settling* occurs. He suggests, "A settling tube 13 to 16 cm in diameter having a settling length of 140 cm and utilizing coarse sediment (0.3 to 2 mm) samples weighing 1-2 grams or fine sediment (0.02 to 0.5 mm) samples weighing 0.6 gm are recommended for particle size analysis utilizing settling tubes." He found that the greater the diameter of the settling tube, the closer the agreement with fall rates in fluids of infinite extent. A cylinder of 7.6 cm in diameter gave errors of 20% to 34% for spheres 20 mm to 30 mm in diameter. Although the pipet method requires *dispersed settling* for initial conditions, Gibbs' tests serve as a warning that wall-to-particle and particle-to-particle effects can be strong even in large columns. Most sedimentation columns for pipet analyses are about 5.5 cm in diameter.

Although disturbances to the mixture while inserting and withdrawing the pipet are often cited as a source of error, *values* for the errors have not been thoroughly investigated. Errors are known to occur during extraction of the subsamples from the sedimentation column for drying and weighing. The assumption is that the subsamples are taken from a region symmetrically

centered around the pipet tip, and that no preferential capture of particles occurs according to size. The sample extraction time is presumed short compared with the settling time of the particles. Once the pipet has been inserted in the column, the rate of subsample withdrawal is critical. Horiba (undated) discusses a test of varying sample withdrawal times between 12 seconds and 140 seconds. The company's writer concludes, "The slower the withdrawal of the sample the coarser the suspension appeared to be..." Guy (1969) recommends making a 25-ml withdrawal in 8 seconds to 12 seconds.

As pipet subsamples are withdrawn, the water surface falls in the sedimentation column. This raises questions regarding setting and measuring the pipet's depth. Should the depth of the pipet tip be set at the beginning of withdrawal, at the *end* of withdrawal, or at some intermediate depth? Horiba (undated) states, "The value to use is the depth when sampling *begins* but this need be admitted to be an approximation that may not be exactly realistic in practice."

In summary, the theory supporting the pipet procedure is straightforward, but transforming the theory to practice is more difficult. Measurement variability stems from many sources including dispersion techniques, residual eddies, temperature shifts, particle-to-particle interference, particle-to-wall interference, and withdrawal rate sensitivity. These factors act in concert to set limits on repeatability for the pipet method.

For a general estimate of pipet precision, Horiba (undated) states, "No excessive claims are now made for the precision of the pipet method; as an example B.S. 3406 is satisfied if duplicate estimates of cumulative proportions by weight do not differ by more than 4%." Millipore (undated brochure) estimates variability of the pipet procedure. They state, "Even with good control over the many test variables, the recognized 95% confidence level for variability using 600 grit (13 mm to 32 mm particles) is 15% to 18%, depending on the height percent point." In later sections, we will examine variability obtained from the database in this report.

Despite disadvantages of the pipet, its attractive features led to its adoption by the European Economic Community (Wilson 1980) for certification of reference samples used in comparing modern particle-size analyzers.

Alternate Methods

Procedures for finding alternate methods to the pipet as a way of improving efficiency seem at first to be straightforward. Several automated instruments could be evaluated, and the ones yielding results identical or very close to those obtained with the pipet would be contenders for laboratory use. Unfortunately, finding instruments that yield satisfactory data has been an elusive goal. Wilson (1980) comments, "Literature on the measurement of particle size abounds with examples where apparently different results are obtained on ostensibly identical materials. There are several possible explanations for the differences." His list includes (a) attempting to compare instruments based on different techniques for measuring size, for example, *comparing equivalent fall rate diameters* used in the pipet method with *equivalent volume diameters* used in electrical-sensing-zone methods, (b) attempting to convert size parameters that are equivalent only for spherical, fully dense particles, (c) extracting nonrepresentative samples from large samples, (d) testing inefficiently dispersed samples, and (e) failing to account for differences in instrument calibration.

Unfortunately, the particle-size analysis field has reached a consensus regarding measurement accuracy but only for spherical-shaped particles. The literature contains numerous statements attesting to this limited level of agreement. For example, American Society for Testing and Materials (ASTM) Standard C-958 dealing with X-ray monitoring of gravity-driven sedimentation states, "No absolute method of particle size determination is recognized. Therefore, it is not possible to discuss the accuracy of results by this method."

In seeking alternates to the pipet, certain instrument features are judged essential. Alternates will be considered only if they register *total quantities of material in the clay and colloid size range*, which includes all particles smaller than about 2 mm. Many instruments currently available are cited as having certain size-range capabilities. For example, 1.0 mm to 50 mm is common to many instruments but unfortunately many of these cannot detect particles smaller than their rated lower limits. Measured distributions show a *complete absence* of material smaller than about 1 mm. Within the pipet size range, many river-water samples contain a significant quantity of particles smaller than 2 mm. Particle sizes must be based on fall diameters derived from settling rates in water. Frequency distributions and cumulative distributions must be based on *masses* of sediment, not upon *numbers* of particles in chosen size ranges. Some devices such as electrical impedance instruments and laser detectors count discrete particles and display histograms based on numbers of particles, not masses. Converting from numbers to masses adds errors. Impedance instruments measure particles by vacuuming them through a small orifice that carries electric currents. As the particles pass through the orifice, they momentarily interrupt the current; the resulting electric pulse is scaled and used as an index of particle size.

Particle-sizing instruments can be divided into six categories: (a) sieves, (b) microscopes, (c) sensing-zone instruments, (d) elutriators, (e) gravity-driven sedimentometers and (f) centrifuges. Sieves are routinely used in stacks to mechanically divide samples into size-range fractions. Microscopes are used to either manually or automatically gauge 2-D particle images. Electron microscopes produce photographs that are usually analyzed manually. Sensing-zone instruments gauge particles by their interaction with acoustic beams, light beams, or electric fields.

Elutriators include (a) cyclone separators that size particles as they spin in a fluid vortex and (b) laminar flow deposition instruments in which low-speed currents carry particles horizontally as they settle. Points at which the particles cross a reference plane are calibrated according to particle size. Gravity-driven sedimentometers, which include the pipet, detect particles through their interaction with light rays or X-rays. Sedimentometers catch particles on submerged weigh pans or sense particles in suspension by measuring pressures within sedimentation columns. Modern centrifuges are equipped to sense the distribution of particles through their interaction with light rays or X-rays.

Specific Instrument Problems

Some instruments were judged inappropriate because they (a) were not in commercial production, (b) had inappropriate ranges, (c) were too slow for routine analysis, or (d) failed to measure fall diameters. The Coulter Counter was tested by the Federal Interagency Sedimentation Project. This device measures particles as they are forced through an orifice in the wall of an insulated tube that is submerged in a water-sediment mixture. The passage of a particle momentarily disrupts the flow of an electric current flowing through the orifice and thereby generates a voltage pulse, which is proportional to particle volume. The instrument, which is sensitive to low-sediment concentrations, is a useful research tool; however, it is unsuitable for rapid processing of river water because of the elaborate sample processing required to remove particles that may plug the orifices, some of which are only 10 mm in diameter. Swift et al. (1972) comment, "The Coulter Counter method is in some respects the most versatile method but it yields a volume distribution which cannot be related to settling velocity."

Lehman (1988) developed a method for recording particle-size distributions based on hydrostatic pressure records taken at taps in the walls of a sedimentation column. The instrument registers fall diameters; but because it is designed as a replacement for the visual accumulation tube, it analyzes only particles *larger* than about 62 mm.

Optical scanning devices show great speed and convenience in sample processing; however, these instruments have met with mixed degrees of success. Bohren and Huffman (1983) comment on difficulties in sizing particles with light. They report, "There are two general classes of problems in the theory of the interaction of an electromagnetic wave with a small particle. The direct problem. Given a particle of specified shape, size and composition which is illuminated by a beam of specified irradiance, polarization and frequency, determine the field everywhere. This is the easy problem; it consists of describing the tracks of a given dragon. The inverse problem. By a suitable analysis of the scattered field, describe the particle or particles that are responsible for the scattered field. This is the hard problem; it consists of describing a dragon from an examination of its tracks." The authors speak of light beams as members of the electromagnetic spectrum. As with radio waves, light is composed of electric and magnetic fields.

Many studies have been made of light passing through water-sediment mixtures and the relation between light attenuation and sediment concentration. Early efforts focused on in situ turbidity measurements in rivers. In some respects, the measurements are similar to making particle-size distribution measurements in sedimentation columns. If light beam attenuation and sediment concentration were correlated, light attenuation could replace weight measurements in the pipet procedure; unfortunately, efforts to discover the correlation have met with only limited success. Benedict (1945), after making an extensive review of turbidimeter data on Iowa streams, reported, "It appears that no method has been developed which will permit quantitative correlation of the optical properties of fluids with the weight of solids in suspension." After comparing the concentration of suspended sediment by weight with light attenuation, he concluded, "The percent difference between the two methods of determining the concentration of

suspended solids in water is thus shown to range from 0 to 500.” On the other hand, Brown and Ritter (1971) studied turbidity of the Eel River basin and concluded, “The turbidity concentration relation is very consistent for each station and throughout the basin.” Diplas and Parker (1985) encountered problems in attempting to relate light scattering to sediment particles. They remark, “The turbidity measurements with this device [a light transmission meter] were very sensitive to the color and size of the suspended material.” Pickering (1976) cites, “Perhaps the most widespread misuse of turbidity measurements data is as an indicator of the concentration of suspended sediment.” He also reports, “However, it is almost impossible to transfer the relationships between sediment concentrations and optical characteristics from one environment or type of sediment to another.”

In some aspects, estimating sediment concentration in rivers containing unknown particle-size distributions differs from estimating concentrations in sedimentation columns. Even though the *initial* mixtures in the columns have *unknown* distributions, the sedimentation process proceeds in an orderly fashion with the removal of particles of *successively finer sizes*. Swift et al. (1972) remark that the photoextinction method has been developed on a mathematical foundation based on the equation,

$$\ln(I_0/I) = -kClSKnd^2$$

where

I_0 = the light intensity emerging from the sedimentation column

I = the light intensity entering the column

k = the bulk absorption coefficient

C = the sediment concentration in g/cm^3

l = the light-beam path length in the sedimentation cell

K = the effective projected-area coefficient of the sediment particles

n = the number of particles in the light beam

d = particle diameter

D = the largest particle in the beam

In this equation the summation runs from $d = 0$ to $d = D$.

Lu Yong-sheng et al. (1983) had some success in using light attenuation for particle sizing by incorporating Rose’s extinction factors and Mie’s theory of light scattering. His instrument has been calibrated primarily with sediment from the Yangtze and Yellow rivers in China. Only a few tests have been run with the instrument on sediments from United States rivers.

During the last two decades, automatic optical equipment for examining individual particles has received considerable attention. One such instrument was tested at the University of Minnesota, Department of Soil Science. Particle images were magnified, projected onto a TV screen, and then sized automatically; however, according to researchers (Rust and Gross, Personal communication, 1975), “The principal weakness in the system is the inability to resolve the distribution below about 0.5 mm particle size.”

Laser scanning of sediment mixtures is a rapidly developing technology; however, it has certain

disadvantages for sizing mixtures containing a broad range of particle sizes. Berg (1991) observed that distributions obtained with laser instruments were always broader than the actual distributions. He comments, “The intrinsic nature of LD [laser diffraction] and SED [sedimentation] also broadens the signal range...” Figure 2 illustrates the problem with a hypothetical sample containing only thin, flat, disc-shaped particles all with the same dimensions.

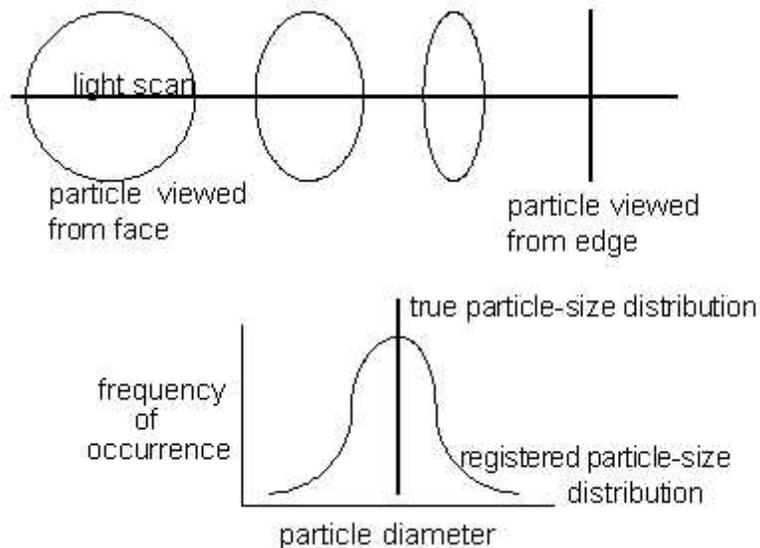


Figure 2. Size-distribution broadening caused by shifts in particle-viewing angles.

Because all particles are of the same size, the *true* frequency distribution is a vertical line as indicated on the frequency plot in the figure. A laser beam scanning the mixture crosses some particles with their broad, flat surfaces fully exposed. Other particles, because of their tilted position, appear as ellipses or thin lines. The suspension *appears* to contain a broad range of particle sizes as indicated by the bell-shaped distribution shown in the figure.

In 1989, an advertisement was placed in the *Commerce Business Daily* newspaper (refer to Appendix 1 of this report) inviting information on new particle-size measuring instruments. Five companies responded with brochures that are summarized below.

The first response described a laser light-scattering system with two sensors. One is a flow-through sensor for installation in pipelines; the other is a laboratory instrument for analyzing discrete samples. Communications with technical representatives indicated particle-size ranges of the instruments are 5 mm to 300 mm for the flow-through model and 2 mm to 125 mm for the discrete sample model. The lower limit for both sensors is inadequate to analyze most river-water samples, which, within the pipet range, contain substantial quantities of particles smaller than 2 mm. For best accuracy, multiple scattering among particles should not occur. To meet this requirement, many river-water samples must be diluted. As for the response to nonspherical particles, the sensors are sensitive to particle orientation; for example, the registered size of a disc-shaped particle depends upon its orientation as it passes through the laser beam. The

instrument's response correlates best with particle volumes but is not linked directly to particle-fall rates or fall diameters.

The second response described an instrument for measuring airborne particles and therefore had no application to analyzing water-sediment mixtures.

The third response described an instrument for sizing liquid-borne particles in water flowing through a transparent cell in a laser beam. Particles scatter the light and produce diffraction rings that depend upon particle size—the smaller the particle, the greater the angles separating adjacent rings. The rings are analyzed with the aid of optical detectors mounted in a spokelike pattern centered on the beam's axis. Signals from the detectors are processed to obtain particle size distribution functions. The instrument gauges particles in terms of optical equivalent diameters, not particle-fall diameters. The technical brochures state, "If the particles being measured have high aspect ratios, then results from laser diffraction techniques may not agree with other, more manual sizing methods...software includes a shape correction facility to allow reconciliation to the user's historic results." The instrument contains many attractive features such as built-in, particle-dispersing equipment.

The fourth response described an instrument based on laser diffraction technology. It registers particle sizes in terms of optical volumes or equivalent surface areas but not by fall diameters. The instrument's particle-size range was from 0.1 mm to 800 mm.

The fifth response described an instrument based on the electrical impedance principle. Particles are pulled by means of a vacuum through a small aperture in a glass tube. The aperture carries a steady electrical current that is momentarily interrupted when a particle passes through the opening. The resulting voltage pulse is proportional to particle volume but only if the particle's diameter is between 2% and 60% of the orifice diameter. The lower detection limit is about 0.4 mm. Particles smaller than this size produce voltage pulses that cannot be separated from the random voltage fluctuations produced by the orifice and electronic-signal-processing equipment. Many river-water samples contain substantial quantities of particles smaller than 0.4 mm. Detailed information on size distributions below this level are not normally required; however, it is desirable to register the total mass below this critical threshold.

In summary, the *Commerce Business Daily* advertisement failed to uncover instruments qualifying as replacements for the pipet. However, as part of the study, several particle-size analyzers and techniques were surveyed, and their ranges are plotted on Figure 3. The range of the pipet procedure is the top line in the figure. Certain standard reference materials used for testing and calibration are shown below the *electron microscope* line. As Figure 3 shows, many of the instruments have ranges exceeding that of the pipet. Their major shortcoming is failure to measure particles in terms of fall diameters.

Table 4. Instrument description footnotes for Figure 3.

Boldface numbers refer to range lines on the figure.

- 1.** Holve (1992) remarks that by detecting forward-scattered laser light, complete size distributions can be obtained in less than one millisecond with concentrations less than 0.1% by volume. He cites particle-size ranges from 1 mm to 500 mm and states: “Smaller sizes down to 0.2 microns can be measured when concentrations are more dilute.”
- 2.** Whitney (1960) extended the pressure-sensing principle to measuring particles as large as 5000 mm in diameter. Whitney’s apparatus consisted of a plastic column 3 in. in diameter, 2 m long, and fitted with taps through which pressures in the column could be measured as particles settle. He offered no data showing the precision or accuracy of the instrument. His motive for developing the equipment was to avoid removing material for drying and weighing.
- 3.** Shimadzu uses laser-light scattering to cover the particle-size range from 0.1 mm to 500 mm. Scattered light is analyzed by three theories: the Fraunhofer theory covering the 10 mm to 500 mm range, along with Mie and side-scattering theories covering the 0.1-mm to 10-mm range. The instrument reports the quantity of particles smaller than 0.1 mm. Particle sizes are reported in terms of equivalent latex spheres that are used for calibration. Alarms warn of excessive secondary scattering caused by sediment concentrations beyond rated limits. The Mie theory, which is used for data reduction, requires values for particle refractive indices that must be supplied by the operator. The cell size is 33 by 13.5 by 9 mm. A built-in sonifier disperses samples automatically.
- 4.** Malvern uses laser-forward-light scattering to cover the range of 0.5 mm to 1800 mm that is divided into 32 intervals. An analysis is completed in about 10 sec after the instrument has been calibrated with latex spheres.
- 5.** Lasentec focuses a laser beam and measures the backscattered light as particles are carried through the beam by flowing water moving at a controlled speed. Particles are sized not by the intensity of the scattered light but by the length of time it takes the particles to traverse the beam. The instrument is calibrated with a Community Bureau Reference (CBR) standard sample of quartz. Tests show that distributions of CBR70, CBR67, and CBR69 agree with pipet analyses to within about 5% through the sample midranges. The particle-size range of the instrument is 0.7 mm to 250 mm that is divided into 28 particle-size intervals.
- 6.** Brinkman, Inc. produces a laser instrument termed a “flying spot scanner.” A focused beam rotates within a sample cell at high speeds. Backscattered light pulses are sorted to reject those produced by particles outside the focal point of the beam or by particles that graze the edge of the beam. Pulses that meet the criteria are scaled according to the time it takes the beam to travel across the particle. The addition of a view monitor permits display of Feret’s diameter, Martin’s diameter, projected area, shape factor, and particle-aspect ratio. Calibration is performed with polystyrene microspheres. One model of the instrument covers the range of 0.7 mm to 150 mm, another the range of 2 mm to 300 mm.

7. Photon correlation light scattering senses fluctuations in scattered-light intensity. A laser beam is focused inside a cell. The phase of the scattered beam depends on particle position relative to the light sensor. As the particles move in response to Brownian motion, the phase of the light shifts and causes the scattered-light intensity to fluctuate—a phenomenon similar to Doppler frequency shifting caused by a moving target. Particle distributions are obtained from a theoretical relation linking diffusion speed to particle size. Sheppard (1988) cites photon correlation spectroscopy as capable of sizing particles in the range of 0.005 mm to 3 mm.

8. A forward-light-scattering laser unit by Leeds and Northrup reportedly has a dynamic range of 0.7 mm to 700 mm.

9. Sheppard (1988) cites an instrument by Insitec for simultaneously measuring concentration, size, and particle velocity. The particle-size range is 0.2 mm to 200 mm, and the velocity range is 0.1 m/s to 200 m/s. Typical accuracy is cited as 10% for particle size.

10. Sheppard (1988) describes an instrument by Shimadzu with a range of 0.02 mm to 150 mm. Particles settle by gravity force, by centrifugal force, or by a combination of the two. Shifts in concentrations are sensed by changes in optical absorbency.

11. Sheppard (1988) cites the Coulter Counter as having a range of 0.3 mm to 1200 mm. Coulter Counter orifices are limited to particles in the size range of 2% to 40% of orifice diameter. Signals from particles smaller than 2% become indistinguishable from electronic noise produced by the amplifiers. Particles larger than 40% sometime block the orifices. As with any sensing zone technique, coincidence effects produce errors. Coincidence occurs when two or more particles simultaneously pass through the orifice forming the sensing zone. McCave and Jarvis (1973) present equations for correcting coincidence errors.

12. High Accuracy Products (HIAC) (undated) cites a range for its instrument as 2 mm to 9,000 mm. Light from a tungsten lamp is focused on a detector. One by one, particles flow through the beam and interrupt a portion of the light. Detector voltage pulses are then correlated with particle size.

13. Horiba (undated) cites ranges of general instrument classes: elutriators, 3 mm to 75 mm; cyclone separators, 8 mm to 50 mm; centrifuges combined with mass-accumulation sensing, 0.05 mm to 25 mm; centrifuges combined with light-absorption sensing, 0.5 mm to 100 mm; centrifuges combined with X-ray sensing, 0.01 mm to 5 mm; gravity sedimentation combined with light absorption, 0.05 mm to 100 mm; gravity sedimentation combined with X-ray sensing, 0.1 mm to 130 mm.

14. Some laser-light-scattering instruments have achieved a broad range by analyzing light scattering using Fraunhofer and Mie theories. The Fritsch (undated brochure) instrument reportedly covers 0.16 mm to 1,250 mm in a single setting.

15. Atmospheric aerosols span the size range from 0.002 mm to 100 mm (Pui and Liu 1989).

16. The U.S. Department of Commerce (1994) provides a reference material for particle sizing. Known as *standard reference material 659, particle size distribution standard for Sedigraph calibration*, the material is a silicon nitride powder containing particles in the 0.2- μ m to 10- μ m range. It is issued as a set of five 2.5-g vials. *Standard reference material 1978 is a particle size distribution standard for gravity sedimentation*. It is a zirconium oxide powder for calibration in the 0.2- μ m to 10- μ m range and is issued as a single bottle containing 5 g of irregularly shaped primary particles with mean diameters of about 1.0 μ m.

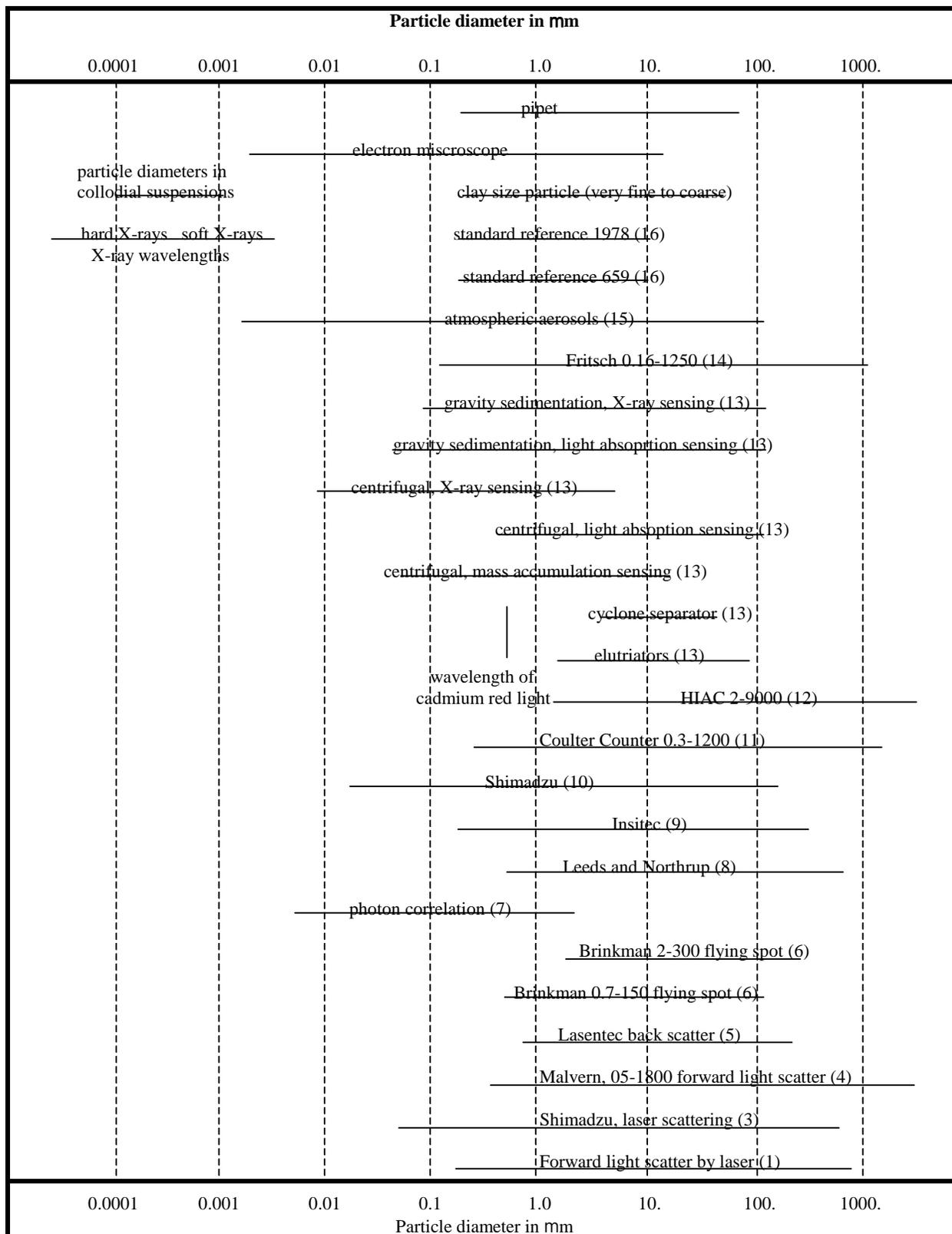


Figure 3. Ranges of particle-size analyzers and techniques.
 Details of operating principles for the instruments are given in Table 4.

Allen and Davies tested several instruments by using BCR 66, a special sediment standard that was validated in Europe by the *pipet* method. The average of deviations taken at several particle size breakpoints were Brookhaven, 2%; Ludal pipet (a centrifugal version of the pipet), 2.5%; Sedigraph 5000 ET (X-ray analyzer), 3%; Coulter Counter (corrected), 3%; Horiba 700X (disc centrifuge), 4%; Horiba LS (a light-scattering instrument), 5%; Horiba 500 (a cuvette centrifuge), 7%; Horiba 2=1, 8%; Horiba LA 500 (low-angle light scattering), 9%; Microscan (X-ray absorption), 9%; Horiba K=X, 10%; Joyce Lobel, 11%; Coulter UC (electronic orifice sensing), 11%; Microtrac SPA (laser scattering), 21%; Elzone (electronic orifice sensing), 27%; and Malvern (laser-light scattering), 48%. *As shown by these data, several instruments including the X-ray method, which forms the basis of the Sedigraph 5000ET, produce data in close agreement with results from the pipet.*

Theory of X-RAY Analysis

Surveys of particle-sizing instruments confirm that X-ray analysis is a viable contender for replacing the pipet. Several investigators indicated the range, speed, and weight-measuring principle of the X-ray method has several advantages. According to Sheppard (1988), the Sedigraph's range is 0.1 mm to 300 mm for the model 5100. In comparing light attenuation with X-ray absorption for estimating weights of particles at specific depths, Horiba (undated) states, "These methods (X-ray absorption) have the advantage over those involving the use of a light beam in that the absorption is more directly proportional to mass." Analysis time with the pipet requires several hours of attention on an intermittent basis. Particle sizing by the X-ray technique proceeds much faster. Orr and Dallavalle (1959) state, "...a material with a density of 2.6 g/cm³ requires 10 min from start to 2 mm, 20 min to 1 mm and 100 min to the lower limit of 0.2 mm."

The history of X-ray analysis contains an interesting application of nuclear radiation. As early as 1958, sedimentation combined with X-rays was used to measure particle-size distributions. Bate and Leddicotte (1958) activated a powdered sample by placing it in a nuclear reactor. The powder was then dispersed in a water column and allowed to settle by gravity. A scintillation counter clamped to the column registered radiation as the particles settled. The researchers assumed that any decrease in radioactivity was equal to the fraction of the total activity contributed by particles of a specified size that settled away from the measurement point. In a sense, the measuring scheme reflected that used in modern X-ray analyzers. There was one difference: instead of illuminating the column with a beam of X-rays from an external source and then measuring transmission *through* the column, the researchers made the particles radioactive and measured the flux *emanating* from the column. Temperature variations were found to cause only small errors. A shift from 72 °F to 78 °F contributed only a 2.5% error on the PLT scale.

Several investigators have compared Sedigraph data with pipet data and found agreement within a few percent. Ingram (undated) found that most PLT values differed by less than 4% from the mean. He took precautionary steps by running daily baseline correction tests to compensate for scratches and misalignments of cell walls in the X-ray path. In recent years, agreement between pipet and X-ray was verified in tests run by Allen and Davies (undated). In comparing 12 modern, commercial instruments, they found the X-ray data differed from pipet data by only 3%. Overall difference was defined as the average percent deviation taken without regard for sign. Deviations for the entire suite of instruments ranged from a low of 2% to a high of 11% based on measurements of BCR 66 standard test material that contains particles from 0.35 mm to 2.5 mm in diameter. On the PLT scale for the X-ray instrument, the following deviations were observed at the particle-size breakpoints in micrometers: 0.6, -4%; 0.75, 0%; 0.9, +3%; 1.0, +3.5%; 1.2, +4%; 1.3, +2%; 1.5, +2%; 1.7, +4%; 1.8, +7%.

Regarding repeatability of the X-ray instrument, ASTM committee C-21 found that sediment specimens tested in a single laboratory and by one operator yielded repeatability values of ± 0.08 mm in the range of 0.5 mm to 50 mm. At a chosen fall diameter, percent-finer values agreed to within about $\pm 1\%$.

The modern X-ray instrument merges X-ray attenuation as a weight-sensing technique with gravity sedimentation rates as a particle-sizing technique. A test suspension is mixed by a pump that circulates the water-sediment slurry through a sensing cell located in the path of a low energy X-ray beam. The flow is then stopped abruptly to allow the particles to settle toward the bottom of the cell. The weight of particles in suspension is obtained from the intensity of the X-ray beam emerging from the cell. As settling proceeds, the suspension gradually clears and the beam becomes stronger. Particle-settling rates are determined from elapsed time records; settling rates are then converted to fall diameters by using Stokes' Law. To obtain a complete particle-size distribution, the cell is slowly lowered through the beam in order to scan the full range of particle sizes.

The sedimentation column in the Sedigraph is small compared with the pipet. Orr and Dallavalle (1959) gives inside dimensions as 0.5 in. wide, 1.375 in. high, and 0.125 in. thick along the beam's path. Despite the short column, the beam focuses on a narrow range of particle sizes occupying a zone only 0.002 in. high.

Oliver et al. (undated) give the following X-ray attenuation relation that includes the cell walls:

$$I/I_0 = e^{-b}$$

where

I = the strength of the emerging beam

I_0 = the strength of the beam entering the cell

$$b = (a_l F_l + a_s F_s) L_1 + a_c L_2$$

where

a_l = the absorption coefficient of the liquid

F_l = the weight fraction of the liquid

a_s = the weight fraction of the sediment

F_s = the absorption coefficient of the sediment

L_1 = the cell thickness in the direction of radiation

a_c = the absorption coefficient of the cell walls

L_2 = the thickness of the cell walls in the direction of the X-rays

Some authors reverse I and I_0 in the equation but maintain correctness by reversing their definitions.

Potential Sources of Differences Between Methods

Some of the comparative tests show differences of several percent between the pipet and X-ray methods. Even though both are based on similar principles, some comparisons show a bias between the two methods.

Weaver and Grobler (1981) tested naturally occurring sediments containing broad ranges of particle sizes and also sediments containing only narrow ranges of sizes. The latter sediments were supplied by the U.S. Department of Commerce. After testing samples at different concentrations in X-ray analyzers, pipets, hydrometers, and sedimentation balances, they commented, "The Sedigraph [X-ray] consistently gave finer particle size distributions compared with other methods." They concluded, "Discrepancies in results obtained with the Sedigraph, pipet, hydrometer, and sedimentation balance methods are disturbing. A microscopic analysis of the samples tends to support the results obtained with the Sedigraph method."

Stein (1985) noted that analysis errors might be quite large for samples containing montmorillonite. The thixotropic nature of the clay produces a gel-like suspension that greatly slows or stops the sedimentation process. He observed, "Hindered settling effects due to too high concentrations required for Sedigraph analysis may cause too high amounts of fine fraction in Sedigraph measurements." Regarding temperature stability, Stein (1985) remarks, "Temperature changes of about 1 °C to 2 °C during the Sedigraph analysis show no conspicuous effect in the results."

Lara and Matthes (1986), after testing samples with a Sedigraph and pipet, concluded, "Agreement between the Sedigraph and pipet results is good for samples having large percentages of fine material (50% to 60% finer than one micron)." Regarding bias, their results agree with those of other researchers. They conclude, "For samples having a large percentage of coarse material (65% to 75% coarser than 10 mm), the Sedigraph method results indicate finer particle size distributions than do the pipet method results."

Singer et al. (1988) tested an X-ray instrument, an electrical impedance instrument, and a light-scattering instrument. No tests were made with a pipet. They attributed differences in data among the instruments to dissimilar particle properties. Regarding particle interference, they commented, "One factor contributing to the increased disparity in results for all instruments involving samples with high clay content is increased particle-particle interference. Eight percent clay appears to be the critical level." Below this concentration they did not observe adverse effects. In their recommendations they suggest, "Sedigraph analysis should be performed at sample concentrations less than 2 vol. %. Higher concentrations are associated with a positive (finer) shift in the data. At lower concentrations, Sedigraph analysis are both accurate and highly reproducible."

Yuquian et al. (1989) tested samples ranging in concentration from 8,000 mg/l to 16,000 mg/L and containing particles ranging from 20 mm to 500 mm in diameter. In testing the finer size range, they used a pipet, a Sedigraph (X-ray), a GDY and a NSY. The last two are optical photosedimentation instruments developed in the Peoples' Republic of China (PRC). The

researchers concluded, “All analytical methods gave about the same particle-size distributions except for a commercially available X-ray technique. Unadjusted data from the X-ray technique consistently indicated smaller particles than the other methods, but the differences in indicated particle sizes were small.”

Schroder (July 1992 technical memo) concluded from 38 comparative size-analysis runs that, “The principal feature of the data is the consistent higher [PLT] value obtained from the Sedigraph analysis.”

The reason for the finer distributions obtained with the X-ray instrument is not fully understood. Particle-to-particle interference caused by excessively high concentrations may be at fault. Welch et al. (1979) concluded that low concentrations produced best agreement between pipet and Sedigraph. Data showed a systematic trend: as sediment concentration in the Sedigraph decreased, the particle-size data approached that obtained with the pipet. The authors suggested cell concentrations of about 22,000 mg/L were appropriate. Orr and Dallavalle (1959) recommends that sediment concentration be in the range of 0.5% to 3% by volume.

In any analyzer, container walls slow the fall of nearby particles and, as a result, distort size distributions by making them appear finer than they actually are; however, Oliver et al. (undated) used an equation derived by Lorentz to show that wall-effect errors in Sedigraphs are less than 0.1%. Oliver et al. also showed that errors caused by heating due to X-ray absorption amounts to only $1.2 \times 10^{-4}\%$. Furthermore, they conclude the combination of all X-ray errors is less than $\pm 0.5\%$.

Lara and Matthes (1986) observed that air bubbles in test suspensions caused errors in X-ray analyses. Because air has a low X-ray absorption coefficient, transmission increases abruptly when the beam crosses bubbles trapped in the liquid or clinging to the cell walls.

Although unconfirmed, chemical variation among samples may have contributed to some pipet and X-ray cumulative curves crossing another at about 5 mm. Lara and Matthes (1986) attributes the crossings to the wide range of particle sizes. They noted the trends were consistently repeated among numerous replicate runs. Micromeritics comments on the influence of particle composition. “The Sedigraph 5500L (light sensing) is ideal for determining size of low atomic number material such as boron or beryllium compounds and high density organic materials... which cannot be sized using the Sedigraph 5000ET.” The 5000ET is an X-ray instrument resembling the model 5100.

Information about chemical sensitivity of sediment-analyzing instruments stems from work started in 1963 with an X-ray sediment concentration gauge developed under sponsorship of the Atomic Energy Commission. Sediment was sensed by measuring the attenuation of X-rays from a cadmium-109 radioactive source housed in an underwater probe. After passing through river water, the beam's intensity was measured and compared with beam absorption through a clear-water reference sample also housed in the underwater unit.

For this arrangement, the attenuation relation for the gamma rays is

$$I = I_0 e^{-mrx}$$

where

I = the strength of the X-ray beam emerging from the river water

I_0 = the strength of the beam entering the river water

m = the mass absorption coefficient of the river water in cm^2/g

r = the density of the absorber in g/cm^3

x = the absorber thickness (path length) in cm

The instrument registered the ratio of X-rays transmitted through river water containing sediment to X-rays transmitted through the clear-water reference. The ratio was a function of liquid densities which, in turn, was related to the concentration of sediment and dissolved solids. Caldwell (1960) gives values of absorption coefficients for various chemical elements.

Similarities between the underwater sediment gauge and the X-ray particle-size analyzer can be seen by comparing the form of the equations describing the two systems. Oliver et al. (undated) give the following X-ray attenuation relation for the X-ray particle-size analyzer

$$I/I_0 = e^{-b}$$

where

I = the strength of the emerging beam

I_0 = the strength of the beam entering the cell

$b = (a_l F_l + a_s F_s) L_1 + a_c L_2$

where

a_l = the absorption coefficient of the liquid

F_l = the weight fraction of the liquid

a_s = the weight fraction of the sediment

F_s = the absorption coefficient of the sediment

L_1 = the cell thickness in the direction of radiation

a_c = the absorption coefficient of the cell walls

L_2 = the thickness of the cell walls in the direction of the X-rays

Warnings of chemical sensitivity of X-ray measurements range from mild comments to strong admonitions. Ziegler et al. (1967), a developer of the sediment-concentration gauge, may have been apprehensive about the effects of shifts in chemical composition when he remarked, "...recalibration is necessary at suitable intervals if the gage is left in one location or if it is moved to a new location." Berg (1991) commented on chemical influences in the X-ray method, "For SED (sedimentation) employing X-rays, variant X-ray opacity seriously affects response." Referring to organic material, Maehl (1962) observed, "Hence the X-ray absorption coefficients are low and the organic solids contribute little to the density as measured by the attenuation of radiation."

Caldwell (1960) cites the presence of iron, calcium, hydrogen, manganese, and barium as having X-ray absorption coefficients that are significantly different from other elements and from potential constituents of sediment. Speaking of the calibration of a sediment-density probe, he remarks, "The presence of certain elements (notably iron, calcium or chlorine) in larger than ordinary proportions will distort the calibration of the probe and make calibration for the particular sediment necessary."

The X-ray particle-size instrument operates at 0.01 Mev, an energy level that is highly sensitive to sediment composition. Table 5 shows that absorption coefficients at levels below 0.05 Mev are highly dependent on chemical composition, but above 0.1 Mev composition plays only a minor role. For sediment samples that are chemically homogeneous, this chemical sensitivity is not important; all particles, small or large, register their proper proportions. On the other hand, a lack of homogeneity introduces errors. For example, consider a sample in which only the small particles contain a high percentage of calcium. The large particles, being devoid of calcium, attenuate the beam to a slight extent and consequently go almost undetected. Since calcium has an absorption coefficient about 250 times stronger than hydrogen, the instrument reacts strongly. It will likely overregister the fines and report a mean diameter that is too small.

Table 5. Absorption coefficients for various energy levels of gamma radiation.

Gamma Energy in Mev	Chemical Element													
	H	C	N	O	Na	Mg	Al	Si	S	K	Ca	Fe	Mn	Cl
	Absorption coefficients in cm/g for gamma radiation													
0.01	0.38	2.1	3.6	5.7	16	21	26	34	51	81	96			
0.05	0.34	0.18	0.19	0.20	0.25	0.29	0.53	0.39	0.52	0.78	0.93	1.20 ^e	1.07 ^e	0.43 ^e
0.10	0.30	0.15	0.15	0.15	0.15	0.16	0.16	0.17	0.19	0.22	0.34			
1.00	0.13	0.06	0.06	0.06	0.06	0.06	0.06	0.06	0.06	0.06	0.06			

^e estimated

Adapted from Grodstein (1957)

Rakoczi (1973) gives the following equation for the *material parameter* that controls X-ray absorption

$$P = \sum p_i (Z_i^5 / A_i)$$

where

p_i = the quantity of the i^{th} element in percent by weight in the transmission path

Z_i = the atomic number of the i^{th} element

A_i = the atomic mass number of the i^{th} element

The summation runs from $i = 1$ to $i = n$ where n is the number of elements in the transmission path.

Chu et al. (1972) cite Z_i , A_i and P_i values (Table 6) for elements at the Chi-Chi gauging station in China. In analyzing the various components, Chu found that bed-load sediment and suspended sediment had similar compositions except for $AlSi_2O_5(OH)_4$ that was present in bed load, but not in the suspended sediment.

Table 6. Computation of material parameter for sediment at the Chi Chi gauging station in China.

Elements	Zi	Ai	Pi (%)	Zi ⁵	(PiZi ⁵ /Ai)x10 ³
Fe	26	55.85	5.47	11881376	11636.73
Pb	82	207.19	0.21	370739843 2	37576.80
Cr	24	53.00	0.005	7962624	7.66
Cu	29	63.54	0.007	20511149	22.60
Mn	25	54.94	0.15	9765625	266.63
Al	13	26.98	28.57	371293	3931.74
K	19	39.10	4.62	2476099	2925.72
Ca	20	40.08	5.48	3200000	4375.25
Na	11	22.99	6.24	161051	437.13
Mg	12	24.31	1.27	248832	129.99
Ni	28	58.71	0.007	17210368	20.52
Si	14	28.09	31.61	537824	6052.20
Others			16.361		
Total					67382.97

After testing sediments from eight rivers in Thailand, Rakoczi noted, “The results of the mineral analysis revealed that all samples had relatively high iron content, ranging from 3.52% to 5.38% increasing remarkably the material parameter of the sediment, $P=20,000$.” Rakoczi also noted the presence of 10% $CaCO_3$, which increased the material parameter to 25,000.

Maehl (1962) observed, “The compositions of a given sedimentary mineral assemblage varies with grain size. In particular, the sand and silt size grains will be composed heavily of quartz, feldspar, and micas. In the clay fraction these minerals are less abundant and the clay minerals predominate. These will include kaolinite, illite, montmorillites, calcites, and the various mixed-layer minerals.” Table 7 shows the composition and densities of these minerals.

Table 7. Chemical composition and densities of minerals commonly found in sediments.

Mineral	Chemical Composition	Density in g/cm
Quartz	SiO ₂	2.65
Feldspar	KAlSi ₃ O ₈	2.54-2.76
	NaAlSi ₃ O ₈	
	CaAl ₂ Si ₂ O ₈	
Kaolinite	Al ₂ Si ₂ O ₅ (OH) ₄	2.60-2.63
Muscovite	KAl ₃ Si ₃ O ₁₀ (OH) ₂	2.76-3.00
Calcite	CaCO ₃	2.71

Rittenhouse and Thorpe (1943) commented, “In natural fluvial deposits the size distribution of heavy minerals are known to vary systematically with the size distribution of the lighter materials with which they are associated.” Rittenhouse (1944) analyzed sands from the Rio Grande and found a trend between particle size and heavy mineral content. For example, in a sample collected near Bernadillo, New Mexico, size fractions expressed in micrometers and heavy mineral content expressed in percent were 246 mm to 175 mm, 0.7%; 175 mm to 124 mm, 1.7%; 124 mm to 88 mm, 4.8%. Particle sizes smaller than 88 mm were not analyzed; however, the trend indicated that as much as 5 percent of the material in the pipet range may have been heavy minerals such as magnetite with a specific gravity of about 5.2, hornblende at 3.2, apatite at 3.2, or tourmaline at 3.1. A sample from Santa Fe Creek contained 15% heavy minerals in the 124-mm to 88-mm range.

Commenting on the sediment gauge developed under Atomic Energy Commission sponsorship and the possibility of increasing the energy of the X-ray beam to minimize attenuation shifts caused by variability in chemical composition, Maehl (1962) said, “If such high energy radiation is used however it turns out that the required source detector distance becomes undesirably large, several meters, and very high source strength and elaborate measuring equipment are required.” There is no clear path toward the elimination of chemical sensitivity. Table 5 shows that high-energy X-rays have nearly uniform attenuation coefficients across the spectrum of elements. Unfortunately, shifting to high-energy beams carries a price.

Although a new model Sedigraph has been developed, chemical sensitivity will still be a potential problem. The old model is the 5000ET; the new version is the 5100. After testing the two models in a side-by-side comparison, the manufacturer concluded (Micromeritics, undated bulletin 38), “Data from the Sedigraph 5000ET agree with data from the new Sedigraph 5100 within a 2.0 mass percent margin of difference.” The tests spanned a range of materials and densities as follows: alumina 1 (2.4), alumina 2 (3.9), barium sulfate (4.37), garnet (3.85), kaolin (2.62), yellow pigment (6.8), zirconium dioxide (5.6), and cement (3.04).

The new model Sedigraph has a few disadvantages that only an experienced operator will notice. In speaking of the new Sedigraph 5100 combined with the Mastertech, Gooding (Personal

communication to Skinner, March 1992) comments, “When the sediment is minimal, one can lower the volume to increase the concentration of the sample. In most cases this will work with the 5000 model, but with the 5100 model there is an additional pump and quite a network of tubing used. There must be enough sample to fill the tubing and sample cell so it can be circulated through the system. The Mastertech will modify this problem by adding more tubing...” Gooding also comments that “...the Mastertech is not able to retrieve 100% of the volume out of the beakers.” He also observes that with the new system, a sample is dumped into a waste container and lost unless the operator is present to retrieve the sample.

Interpolating Data and Detecting Bias

Interpolating between size breaks on cumulative-size-distribution curves is necessary in many applications. For example, the median particle diameter, D_{50} , usually falls somewhere *between* predetermined particle-size breakpoints. Interpolation can be performed by plotting the size-distribution graph, then reading the value of D_{50} opposite the 50th percentile. Another method that is more compatible with computer applications is to fit an equation to the data points and evaluate the equation at the chosen percentile. Lehman (1988) suggests using a *cubic spline* as the interpolating function.

The cubic spline is a *group* of equations each joining two adjacent breakpoints on a cumulative-distribution function. The final plot is a piecewise approximation curve that passes through all breakpoints and that overcomes a shortcoming of high-order polynomials—their tendency to produce curves with strong oscillations. Furthermore, the spline equations for two adjacent sections join one another smoothly so that first- and second-order derivatives exist throughout the entire range. At the largest and smallest particle diameters on a distribution curve, additional information must be supplied in order to evaluate all of the spline coefficients. Three choices exist: the function can merge with a first-order function (straight line), a second-order function (parabola), or a third-order function (cubic). The choices give rise to the terms *lspline*, *pspline*, and *cspline*.

Evaluating coefficients for spline functions is a tedious process involving the inversion of matrices. Fortunately the process has been coded in the MathCAD computer program (Wieder 1992) which for input data requires a table of x and y values and a choice of *lspline*, *pspline*, or *cspline*. After forming the spline equations, the program plots interpolated $y(x)$ values (PLT values) for a range of x (particle-fall diameter) values. Figure 4 shows a sample of the input and output.

$$V_s = \text{lspline}(X, Y)$$

$$x = 1, 1.1, \dots, 70$$

$$y(x) = \text{interp}(V_s, X, Y, x)$$

	x		y(x)
X=	2		13.5
	4		20.5
	8		24
	10		30
Y=	20		41
	32		58.5
	50		86
	62		99
	70		99
	27		49.714
	27.1		49.881
	27.2		50.049
	27.3		50.218
	27.4		50.387
	27.5		50.557
	27.6		50.728
	27.7		50.899
	27.8		51.071
	27.9		51.243
	28		51.416

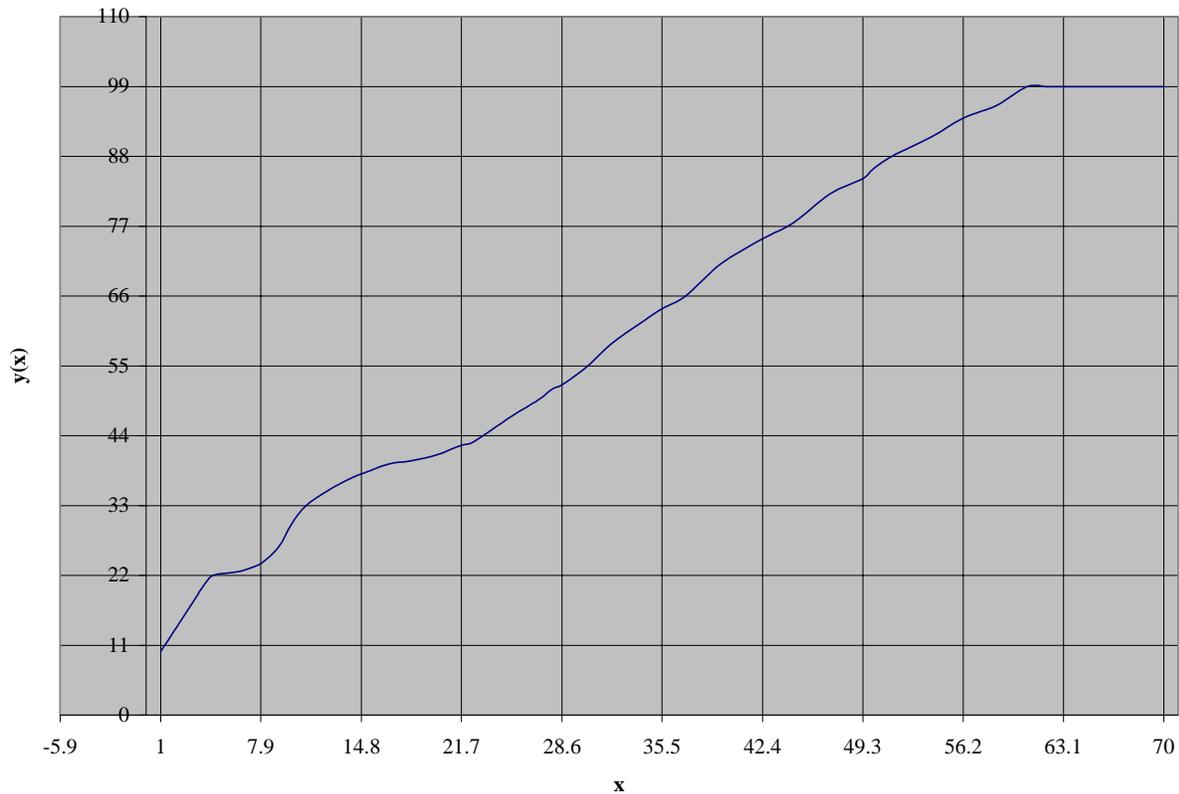


Figure 4. Sample of a lspline curve fitted to data for index 24 in the basic data table.

Hotchkiss (1994) suggests summarizing size-distribution data as standard functions such as normal distributions or lognormal distributions that can be characterized by mean values and standard deviations. Lognormal particle-size distributions are frequently produced by grinding and crushing operations; however, each distribution must be individually tested for goodness of fit before it can be used to check for bias between X-ray and pipet data.

Helsel (1991) suggests a simpler test based on Bernoulli trials. He comments that a Bernoulli *sign test* applied over a full-particle size range is preferable to a T-test applied at each particle-size breakpoint. The Bernoulli test is based on experiments having only two outcomes such as true/false, fail/succeed, plus/minus, or greater than/less than. The last two examples form the basis of bias detection in particle-size comparisons. Assume a size distribution has been obtained with a pipet and another size distribution has been obtained with a Sedigraph. If no bias exists, each breakpoint reading taken with the X-ray method has a 50% chance of exceeding the pipet reading. For 10 breakpoints, about five of the X-ray readings should exceed those obtained with the pipet. If all 10 X-ray readings exceed those obtained with the pipet, the event strongly indicates bias between the two methods.

Bernoulli trial theory requires two possible outcomes for each trial and that outcomes for each trial be independent of the others. Furthermore, the probability of the outcomes is the same for

each trial in a sequence. If these conditions hold, the probability of obtaining “k” greater than readings (outcomes) out of “n” breakpoints (trials) is

$$P(k,n) = [n!/k!(n-k)!]p^k q^{n-k}$$

where

! designates factorial

p = the probability of obtaining a *greater than* reading

q = the probability of obtaining a *less than* reading

With bias free trial, p = q = 1/2.

Table 8. Probability of events in Bernoulli trials.

The number of positive values obtained in “n” trials is labeled “k.” The probability of obtaining a positive outcome equals the probability of obtaining a negative outcome.

Number of Positive Outcomes, K	Number of trials, n							
	10	9	8	7	6	5	4	3
	Probability of Event							
0	0.001	0.002	0.004	0.008	0.016	0.031	0.063	0.125
1	0.010	0.018	0.031	0.055	0.094	0.156	0.250	.0375
2	0.044	0.070	0.109	0.164	0.234	0.313	0.375	0.375
3	0.117	0.164	0.219	0.273	0.313	0.313	0.250	0.125
4	0.205	0.246	0.273	0.273	0.234	0.156	0.063	
5	0.246	0.246	0.219	0.164	0.094	0.031		
6	0.205	0.164	0.109	0.055	0.016			
7	0.117	0.070	0.031	0.008				
8	0.044	0.018	0.004					
9	0.010	0.002						
10	0.001							

Table 8 helps estimate the minimum number of breakpoints needed to reliably detect bias. Consider a situation in which all Sedigraph readings exceed pipet readings. If the total number of breakpoints (n) is only three, the event occurs in 12.5% of all tests even if no bias exists. *Four* breakpoints are about the minimum required to draw a valid conclusion. In the case, all four Sedigraph readings will exceed pipet readings in 6% of all tests provided no bias exists.

If bias does exist, determining the amount of bias is the next step. For comparing differences between particle-size methods, Wilson (1980) takes the absolute value of differences in percent less than readings in each particle-size break and then averages the absolute values. Allen and Davies (undated) follow the same process; however, they discard differences at the extremes of the distributions before computing averages. They used only values in the 10 to 90 percentile range on the percent less than scales. The tails of distributions were disregarded because they are subject to large percentage errors.

Analysis of Data

Dispersants

The influence of dispersants on size distributions may range from minor shifts of only a few percent to major changes amounting to 20% or more. Figure 5 shows a case where adding a chemical dispersant increases the quantity of sediment particles smaller than about 18 mm. The change in the shape of the distribution curves follows a common pattern: material in the fine range *gains* mass while material in the coarse range *loses*. As shown by Figure 5, the redistribution caused by adding the dispersant shifts the left end upward and levels the midrange. Material gained in the 2-mm to 15-mm range accompanies a loss of material in the 20-mm to 50-mm range. Because the distribution curves cross one another, the Bernoulli test indicates no bias even though a major realignment has occurred.

Influence of Chemical Dispersants

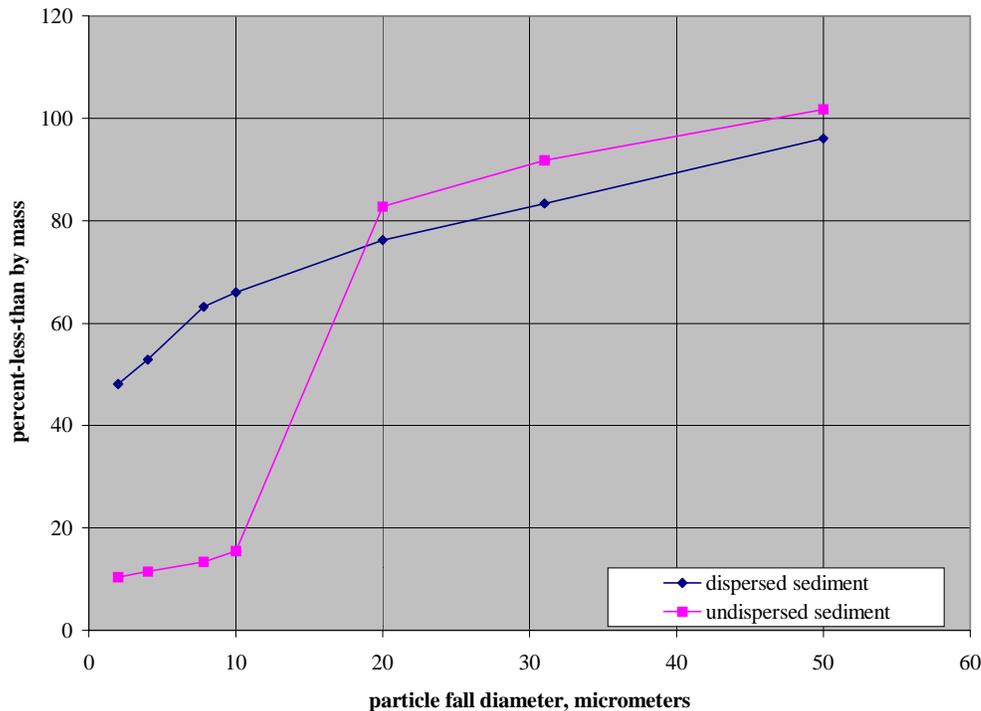


Figure 5. Comparison of measured particle-size distributions of chemically dispersed and chemically undispersed samples analyzed with the pipet procedure.

Data, which are for sample index number 62, were supplied by Mr. Welch of the ARS.

Not all shifts are as dramatic as those in Figure 5. Figure 6 shows a case where increasing dispersant concentrations produces an upward shift in the fine-particle range; however, the leveling effect in the coarse-particle range is small but spread over a broader region.

Size Distributions at Four Dispersant Levels

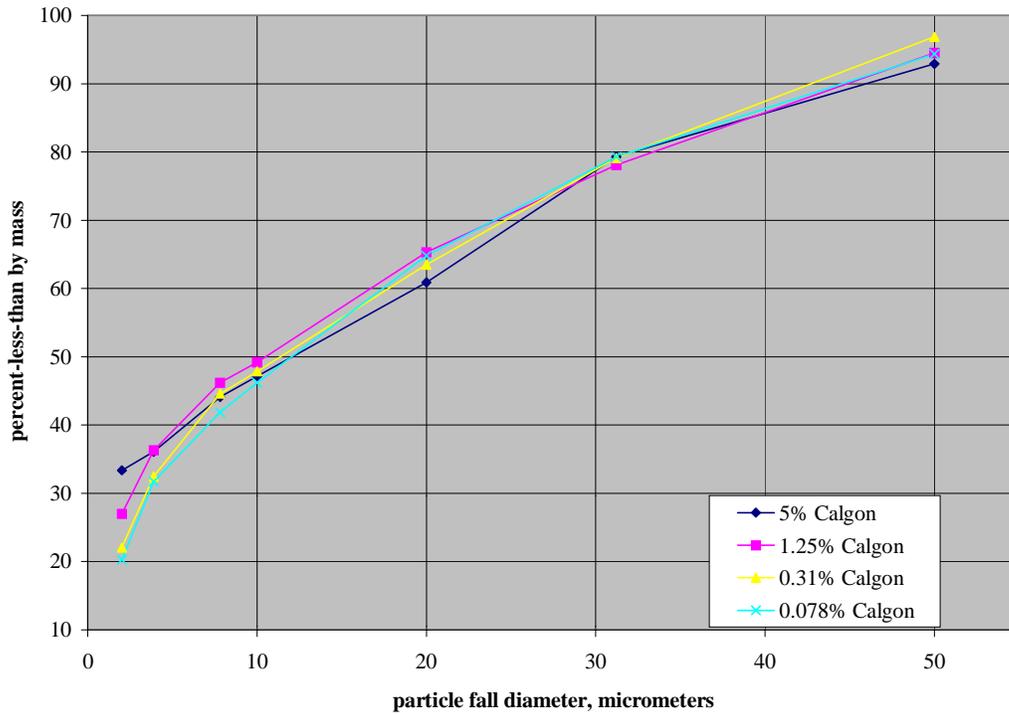


Figure 6. Influence of dispersant concentrations on pipet measurements of particle-size distributions.

Data supplied by Mr. Paul Allen, ARS.

Figure 7 charts the influence not only of adding a dispersing agent but also of introducing the X-ray analysis. The pipet sample was dispersed according to ARS routine procedures that call for about 1.0 ml of dispersant for each gram of sediment. The dispersant level of 0.5 ml/g on the X-ray run was apparently inadequate as indicated by the deficiency of material in the fine range. Increasing the dispersant level to 1.0 ml/g apparently completed the dispersing process since adding more of the chemical had little effect on the particle-size distribution. With dispersion in the X-ray instrument complete, a bias between the pipet and X-ray developed. At all particle-size breakpoints, the X-ray data were greater than the pipet data. The table of Bernoulli trials indicates that the probability of such an event occurring by chance alone is about 0.008 (0.8%). In this instance, we are led to conclude that a bias exists between the methods.

X-Ray Runs at Four Chemical Dispersant Concentrations

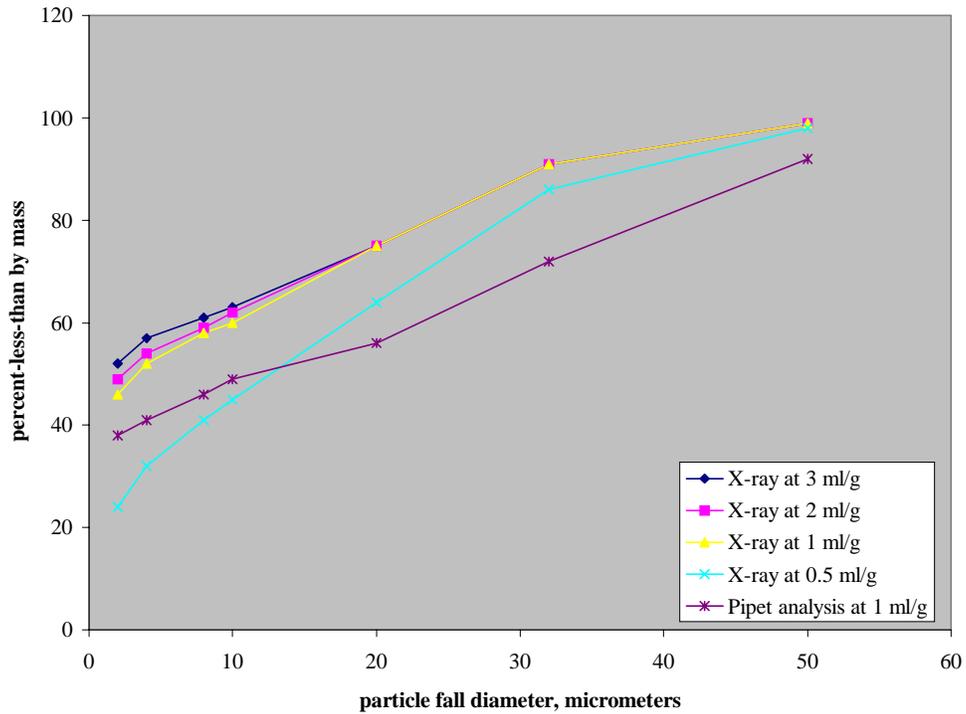


Figure 7. Influence of chemical-dispersant concentrations on X-ray measurements of particle-size distributions.

Data courtesy of ARS, Chickasha, Oklahoma. Refer to sample indices 44, 45, 46, and 47 of basic data tables.

Bias

The database was analyzed for bias between pipet and Sedigraph data. Single-pipet/X-ray runs (Hotchkiss 1994) composed of index numbers 13-43, 53-59, 76-93, 94-110, 159-160, 175-176, and 83-197 in the database were processed as indicated on the partial worksheet of Table 9.

Table 9. Partial worksheet for analysis of bias.
Column headings are explained in the text.

Index	2p	2s	2s-2p	4p	4s	4s-4p	8p	8s	8s-8p	10p	10s	10s-10p	20p	20s
13	40.5	47.5	7	42.5	53.5	11	49	59	10	51	61	10	57.5	71.5
14	50.5	53	2.5	58.5	61.5	3	66	73	7	68.5	76	7.5	76.5	87
15	51	54	3	56.5	59	2.5	62.5	66	3.5	64	67.5	3.5	74	79
16	50	54	4	51	59	8	58	66	8	64.5	68	3.5	74	80
17	48	46	-2	51	54	3	57	62	5	59	64.5	5.5	71	77
18	50	57	7	54	64	10	60.5	71.5	11	64	74	10	72	84.5
19	36	43	7	37	47	10	44	54	10	46	56	10	55.5	70
20	59	59	0	68.5	68.5	0	75	78	3	77	81	4	85.5	88.5
21	59	62	3	66	70.5	4.5	71.5	79	7.5	78	81	3	85	88
22	41.5	44	2.5	49	50	1	51	58	7	53.5	62	8.5	69.5	78
23	45	56	11	56	63	7	62.5	72	9.5	67.5	75	7.5	76.5	85
24	13.5	12	-1.5	20.5	17	-3.5	24	26	2	30	30	0	41	46
25	14	16	2	20	22	2	24	33	9	30	37	7	41	56
26	30	52	22	37	57.5	20.5	43	64	21	44	65	21	51	76
27	32.5	45	12.5	38	54	16	44	62	18	52	65	13	62	78.5
28	35.5	40	4.5	35.5	47	11.5	42.5	57	14.5	48.5	60	11.5	66	75
29	43.5	49	5.5	48.5	56	7.5	53.5	63	9.5	58.5	65.5	7	68.5	76
30	32.5	44	11.5	34.5	48	13.5	39	55	16	45.5	57.5	12	60	74
31	50	63.5	13.5	59	69.5	10.5	67.5	75.5	8	72.5	78	5.5	79.5	86
32	36.5	43	6.5	40	50	10	48.5	58	9.5	50.5	61	10.5	66	77
33	26	33	7	27	36	9	32	40.5	8.5	34	43	9	40.5	53.5

Index	20s-20p	32p	32s	32s-32p	50p	50s	50s-50p	No. of Values	No. s>p	P of Chance	Bias	Sum of Abs.	Av. Err. Pct.
13	14	67.6	88	20.4	87	99.9	12.9	7	7	0.01	yes	85.3	12.2
14	10.5	86	97	11	96	99.9	3.9	6	6	0.02	yes	41.5	6.9
15	5	87	91	4	99	98	-1	6	6	0.02	yes	21.5	3.6
16	6	80	91.5	11.5	92.5	99	6.5	7	7	0.01	yes	47.5	6.8
17	6	83	90	7	98	98	0	6	5	0.11	probable	28.5	4.8
18	12.5	84	93.5	9.5	95.5	99	3.5	6	6	0.02	yes	60	10
19	14.5	67.5	87	19.5	86.5	98	11.5	7	7	0.01	yes	82.5	11.8
20	3	93	97	20.4	99.9	99.5	-0.4	6	6	0.02	yes	30.4	5.1
21	3	91	93	2	96	98	2	6	6	0.02	yes	23	3.8
22	8.5	85	94	9	98	99.5	1.5	6	6	0.02	yes	36.5	6.1
23	8.5	86	96	10	98	99.9	1.9	6	6	0.02	yes	53.5	8.9
24	5	58.5	73	14.5	86	99	13	7	5	0.23	probable	39.5	5.6
25	15	58	83	25	86	99.9	13.9	7	7	0.01	yes	73.9	10.6
26	25	64	89	25	73	98.8	25.8	7	7	0.01	yes	160.3	22.9
27	16.5	75	90	15	95.2	98	2.8	6	6	0.02	yes	91	15.2
28	9	78.5	93.5	15	98	99	1	6	6	0.02	yes	66	11
29	7.5	84	90	6	93.5	99	5.5	7	7	0.01	yes	48.5	6.9
30	14	80	89	9	96	99.9	3.9	6	6	0.02	yes	76	12.7
31	6.5	89.5	94	4.5	94.5	99.5	5	7	7	0.01	yes	53.5	7.6
32	11	78	93	15	94	98.5	4.5	7	7	0.01	yes	67	9.6
33	13	56.5	77	20.5	84	97	13	7	7	0.01	yes	80	11.4

As a first step in analyzing the data, breakpoint PLT values were arranged in columns. For example, columns 2p and 2s in the table are PLT values at the 2 breakpoint for the pipet and Sedigraph, respectively. The column labeled “2s-2p” is the difference between the two readings. Differences are also shown for breakpoints at 2, 4, 8, 10, 20, 32, and 50.

Values at the tails of PLT distributions were then discarded as being unrepresentative of the pipet and Sedigraph methods. Distribution *tails* were arbitrarily defined as breakpoints at which *both* pipet and Sedigraph values were *greater* than 95% or *less* than 5% on the PLT scale. Agreement between the methods is almost always good for PLT values exceeding 95% because the Sedigraph is manually set to register 100% as part of the operating procedure. By the same

token, the pipet registers 100% because the sediment has been wet sieved to remove particles larger than 62 μ m. Agreement is usually good for PLT values less than 5% because most or all of the sediment has cleared the X-ray path or the sampling point. After discarding readings in the size distribution tails, the remaining readings, which are termed *midrange* values, reflect the inherent characteristics of the two methods. In Table 9, the column labeled “no. of values” is the number of midrange values in each set.

Data for bias analysis are in the columns labeled “*no. S>p*” (number of midrange range values in which Sedigraph readings exceed pipet readings), *P of chance* (probability of event occurring by chance) and *bias*. The probability of a chance occurrence is based on data in the columns labeled “*no S>p.*” and “*no of values,*” along with the Bernoulli trials tables discussed earlier. For example, consider values for index 13. Of seven values in the midrange, all show Sedigraph readings exceeding pipet readings. The probability of such an event occurring randomly and *without* bias is only 0.008 (0.8%). Such a rare occurrence strongly suggests bias does exist.

The bias descriptors *yes*, *probable*, *possible*, and *no* were arbitrarily assigned to probability event levels, P, as follows: *yes* if $P < 0.100$; *probable* if $0.101 < P < 0.200$; *possible* if $0.201 < P < 0.350$, and *no* if $0.351 < P$. These bias descriptors are shown in the “bias” column of Table 9.

The complete listing shows a strong tendency toward bias. Of the 88 pipet/x-ray runs, 49 fall in the *yes* category, 23 in *probable*, 11 in *possible*, and only 5 in the *no* category. Among the last group, the indicator was weak because of a scarcity of qualifying readings. In a few isolated instances, PLT values agreed perfectly. These readings were discarded because the Bernoulli trial model is based on the assumption of only two outcomes.

Degrees of disparity between pipet and Sedigraph runs were gauged by averaging *absolute* values, differences taken without regard for sign. Using absolute values overcomes problems in comparing distributions that cross one another. In these cases, differences taken *with* regard for sign give the illusion of good agreement even though the distributions may cross one another at steep angles and differ by wide margins.

The average of the absolute differences is tabulated in the column labeled “avg err. pct.” In the complete set of 88 runs analyzed, the largest average error is 22.9%; the smallest is 0.8%. Between these extremes, 90% are smaller than 11.4%; 70% smaller than 8.4%; 50% smaller than 6.8%; 30% smaller than 5.3%; and 10% smaller than 3.6%. In summary, the Sedigraph almost always gives PLT values greater than pipet values. The median error is about 6.8%.

Variability within X-ray and pipet runs

The best data for comparing variability within X-ray and pipet runs are those in index numbers 150-156. Values were obtained at the U.S. Geological Survey sediment laboratory in Iowa City where one operator repeatedly tested seven sediments by both techniques. The number of runs on each sediment ranged from 7 to 23; the average was about 15. As Figure 8 shows, standard deviations for the pipet were *greater* at the fine end of the scale than at the midrange or coarse end of the scale. Through the full range of particle sizes, the average standard deviation was

about 2.5%. The same sediments were tested with an X-ray analyzer. Results, which are plotted in Figure 9, show standard deviations were *smaller* at the fine end than at the midrange or coarse end. Throughout the full range of particle sizes, the average standard deviation was about 1.5%. As the charts show, *the X-ray method has better repeatability than the pipet.*

Variability within Single-Operator Pipet Runs

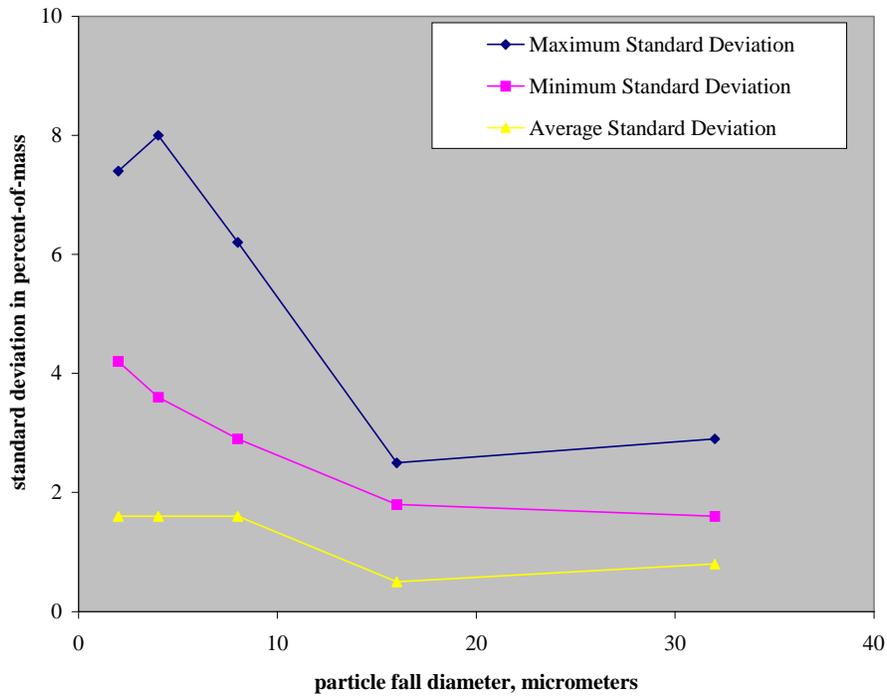


Figure 8. Variability of single-operator tests made with pipet.

Variability within Single-Operator X-ray Runs

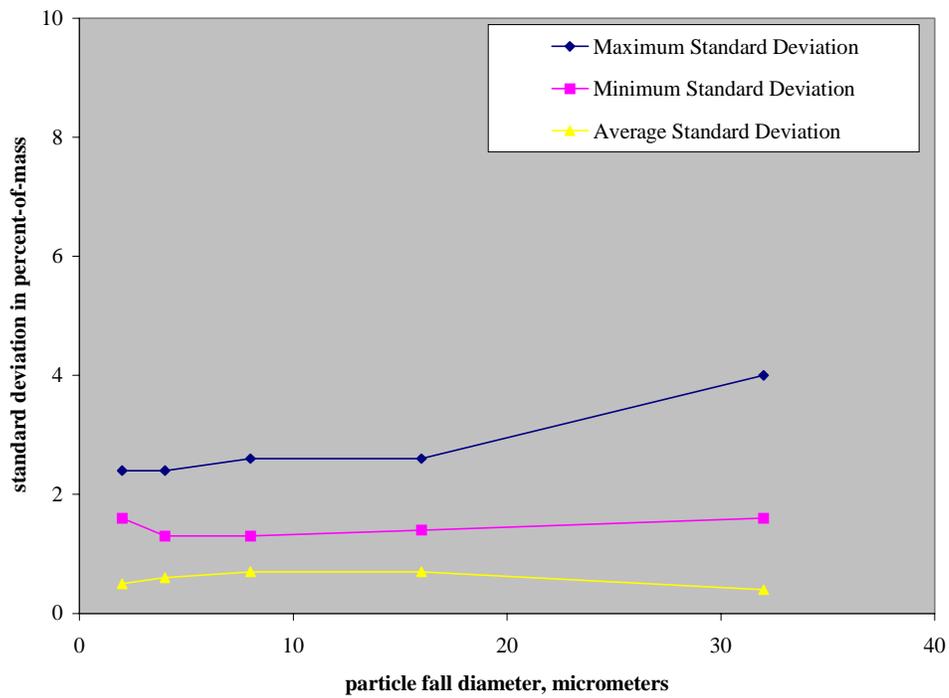


Figure 9. Variability of single-operator tests made with a Sedigraph X-ray particle-size analyzer.

Results from *multioperator* pipet runs are shown in Figure 10, which indicates standard deviation values across the full range of particle sizes is 2.5%, about the same as for single-operator runs. Data for the multioperator runs were obtained by testing two sediments at 18 laboratories. Unfortunately, comparable multioperator data for the X-ray method are not available owing to a scarcity of instrumentation.

Variability within Multioperator Pipet Runs

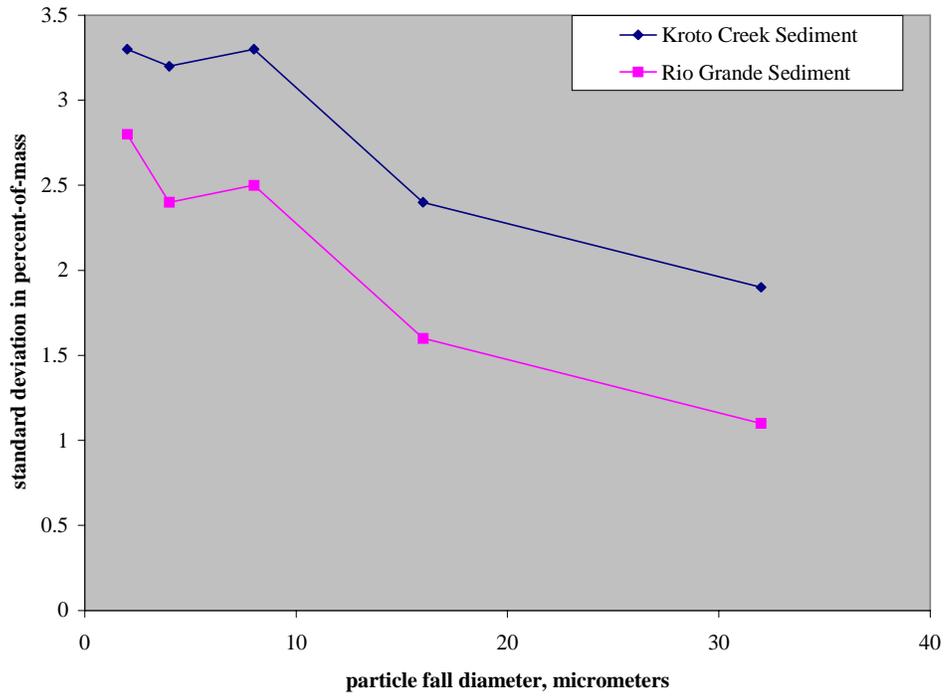


Figure 10. Variability of multioperator tests made with pipet.

Conclusions

- a. Grain-size data are an important component of many sediment-transport studies.
- b. Several grain-size descriptors have been defined, but the one favored by hydrologists is fall diameters.
- c. The pipet procedure yields the required grain-size data; but the technique is slow, expensive, and in need of modernization.
- d. Of size-measuring instruments available, the X-ray unit is a viable contender for replacing the pipet. Fall-diameter measurements are performed automatically, and the unit has better repeatability than the pipet.
- e. A bias exists between X-ray and pipet data. Of the two instruments, the X-ray reports finer particle-size distributions. On a percent-finer scale, the degree of bias ranges from about 1% to 23% with a median value of about 7%.

References

- Allen, T. and Davies R., Evaluation of Instruments for particle size analysis, Engineering Test Center, Dupont Engineering.
- Appel, David W., 1952, An instrument for rapid size frequency analysis of sediment, in Proceedings of the Fifth Hydraulics Conference, June 9-11, Iowa Institute of Hydraulic Research, p. 287-302.
- ASTM Committee C-21, 1981, ASTM Standard method for particle size distribution of alumina or quartz by X-ray monitoring of gravity sedimentation, ASTM, Philadelphia PA.
- Bate, L. C. and Leddicotte, G. W., 1958, An evaluation of the particle size distributions in multicomponent systems, in Analytical Chemistry in Nuclear Reactor Technology, Particle Size Analysis, Second Conference, Gatlinburg, TN, U.S. Atomic Energy Commission.
- Benedict, Paul C., 1945, A study of errors resulting from the use of turbidity in computing the suspended sediment discharge of Iowa streams; Iowa Academy of Science, v. 52, University of Iowa, Iowa City, IA, p. 205-209.
- Berg, Robert H., (1991), Precision in particle size analysis, International Labmate, v. 16, issue 1, p. 4.
- Berg, Robert H. and Youngdahl, Arthur C., 1961, New advances in fine particle measurement, in proceedings of the American Ceramic Society, 63rd Annual Meeting, Toronto, Canada, 11 p.
- Bernard, Jerry M., Langemeier, David L., Dumper, Thomas A., and Clark, C. D., 1991, Actions to treat sediment water quality problems, in Proceedings of the Fifth Federal Interagency Sedimentation Conference, Interagency Advisory Committee on Water Data, v. 2, sec. 11, p. 1-8.
- Bohren, Craig F. and Huffman, Donald R., 1983, Absorption and scattering of light by small particles, John Wiley and Sons, 519 p.
- Brinkman, (undated); Brinkman particle size analyzer, Brinkman Instruments, Inc. Westbury, NY, 11590, 10 p.
- Brown, William M., III and Ritter, John R., 1971, Sediment transport and turbidity in the Eel River Basin, California; U.S. Geological Survey Water Supply Paper 1986, U.S. Government Printing Office, Washington DC.
- Caldwell, Joseph M., 1960, Development and tests of a radioactive sediment density probe, Corps of Engineers, Beach Erosion Board, Technical Memo no.121, 29 p.

References *continued*

Chu, David S. L., Pan, Chi-Fen, and Cheny, I-Lung, 1972, Final report, measurement of flood flow and suspended sedimentation by radioisotope method, Taiwan Power Company, Republic of China, 21 p.

Committee D19.07, 1993, ASTM Standard D4410, Terminology for Fluid Sedimentation, ASTM, Philadelphia, PA, v. 11.01, p. 75.

Corps of Engineers, Beach Erosion Board, 1951, The use and accuracy of the Emery settling tube for sand analysis, Scripps Institute of Oceanography, Technical Memo 23, 20 p.

Corps of Engineer, MRD 1950, Correlation of bottom withdrawal test with pipet test, laboratory technical memorandum, Corps of Engineers, Missouri River Division laboratory, Omaha, Nebraska.

Diplas, Panayiotis and Parker, Gary; 1985, Pollution of gravel spawning grounds due to fine sediment: Saint Anthony Falls Hydraulic Laboratory, Minneapolis, MN; Project Report no. 240, p. 25.

Dou, Guaren, 1991, Physical modeling of sediment transport in China, in Proceedings of the Fifth Federal Interagency Sedimentation Conference, Interagency Advisory Committee on Water Data, v. 2, Sec. 8, p. 54-60.

Fritsch (undated brochure); Laser particle size; Gilson, Worthington, Ohio, 12 p.

Gibbs, Ronald J., 1972, The accuracy of a particle size analysis utilizing settling tubes; Journal of Sediment Petrology, v. 423, no. 1, p. 141-145.

Gibbs, Ronald J., Matthews, Martin D., and Link, David A., 1971, The relationship between sphere size and settling velocity, Journal of Sediment Petrology, v. 41, no.1, p. 7-18.

Grodstein, G. W., 1957, X-ray attenuation coefficients from 10 Kev to 100 Mev; National Bureau of Standards Circular No 583.

Guy, Harold P., 1969, Techniques of water resources investigations of the U.S. Geological Survey, laboratory theory and methods for sediment analysis, book 5, chapter C1, U.S. Geological Survey, Reston, VA, 58 p.

Han, Qiwei and He, Mingmim, 1991, Mathematical model of sediment transport, in Proceedings of the Fifth Federal Interagency Sedimentation Conference, Interagency Advisory Committee on Water Data, V.1, Sec.7, p. 23-30.

References *continued*

Helsel, Dennis, July 9, 1991, Personal communication to J.V.Skinner.

High Accuracy Products, undated, HIAC SS, Automatic particle counter brochure, HIAC Products, Claremont, CA, 4 p.

Holve, Donald J., 1992, Sizing particles with a laser, R and D Magazine, February 18, 1992, p. 9.

Horiba, undated, Horiba particle sizing seminar, seminar notes and workbook: Horiba.

Hotchkiss, Rollin, 1994, Evaluation of pipet and X-ray procedures for determining particle size distribution of sediment, Water Resources Investigation Report, Report KK, Sedimentation Committee, Interagency Advisory Committee on Water Data Acquisition, U.S. Army Engineer Waterways Experiment Station, Vicksburg, MS, 28 p.

Ingram, C., undated draft, Determination of silt and clay particle concentrations in marine sediments with the Micromeritics 5000 particle size analyzer, U.S. Navy.

Interagency Committee on Water Resources, Subcommittee on Sedimentation, 1941, Methods of analyzing sediment samples, Report 4 in A study of methods used in measurement and analysis of sediment loads in streams; Federal Interagency Sedimentation Project, Corps of Engineers, Vicksburg, MS, 203 p.

Interagency Committee on Water Resources, Subcommittee on Sedimentation, 1943, A study of new methods for size analysis of suspended sediment samples, Report 7 in A study of methods used in measurement and analysis of sediment loads in streams: Federal Interagency Sedimentation Project, COE, Vicksburg, MS, 102 p.

Interagency Committee on Water Resources, Subcommittee on Sedimentation, 1957, Some fundamentals of particle size analysis, Report 12 in A study of methods used in measurement and analysis of sediment loads in streams; Federal Interagency Sedimentation Project, Corps of Engineers, Vicksburg, MS, 55 p.

Klumpp, Cassie C., 1991, Sediment studies at Union Avenue Dam Denver Colorado, in Proceedings of the Fifth Federal Interagency Sedimentation Conference, Interagency Advisory Committee on Water Data v. 2, sec. 8, p. 69-76.

Lara, O. G., and Matthes, W. J., 1986, The Sedigraph as an alternate method to the pipet, in Proceeding of the Fourth Federal Interagency Sedimentation Conference: Subcommittees on Sedimentation of the Interagency Advisory Committee on Water Data, Las Vegas, Nevada, 1986, v.1, p. 1-12.

Lasentec, undated, Particle size analyzer for high and low concentrations; Laser Sensor Technology, Bellevue, WA, 98009.

References *continued*

Lehman, Roger William, 1988, Calibration and adaptation of Iowa rapid sediment analyzer to an IBM compatible microcomputer; MS thesis; University of Iowa, 116 p.

Lu Yong-sheng, Wang Xi-di, and Wang Gao-yuan, 1983, Automatic particle size analysis of fluvial sediment by photoelectric technique, in Proceedings of Second International Symposium on River Sedimentation, Nanjing, China.

Maehl, Richard H., 1962, Personal communication to B. C. Colby.

Malvern, undated, Series 2600 C droplet and particle size analyzer, Malvern Instruments, 8 p.

Micromeritics, undated bulletin, Product Bulletin 38, Agreement of data between old and new Sedigraphs; Micromeritics, Norcross, GA., 5 p.

Micromeritics, undated brochure, Sedigraph 5000 ET Sedicomp; Micromeritics Instrument Corp, Norcross, GA, 2 p.

Millipore, undated brochure, Analyzing abrasive powders with the MC system, application report AR 143, Millipore Corp, Analytical Systems Division, Bausch and Lomb.

McCave, I. N., and Jarvis, J., 1973, Use of the model T Coulter counter in size analysis of fine to coarse sand; *Sedimentology*, v. 20, p. 305-315.

McNown, J. S., Lee, H. M., McPherson, M. B. and Engez, S. M., (1948), Influence of boundary proximity on the drag of spheres, Proceedings 7th International Congress on Applied Mechanics, London, England, p. 17-29.

Nelson, Martin E., 1948, Comments in paper by C. S. Howard laboratory experience with the bottom withdrawal tube method of size analysis; in Proceedings of 1948 Sedimentation Conference.

Oden, Sven, 1924, The size distribution of particles in soils and the experimental methods of obtaining them: *Soil Science*, v. 19, no. 1, p. 1-35.

Oliver, J. P., Hicken, G. K., and Orr, Clyde, Jr., undated, Rapid automatic particle size analysis in the subsieve range, Micromeritics Instrument Corp., Norcross, GA.

Orr, Clyde, Jr., and Dallavalle, J.M., 1959, *Fine particle measurement*: New York, MacMillan, 353 p.

Parent, E. D., 1967, Ronad Robin report on Particle size measurement of Aluminum Oxide Samples, ASTM, Committee F-1, 19 p.

References *continued*

- Pickering, R. J., 1976, Measurement of turbidity and related characteristic of natural waters, U. S. Geological Survey, open file report 76-153, Reston, VA.
- Pugh, Cliff A. and Russell, Dodge A., 1991, Design of sediment models, in Proceedings of the Fifth Federal Interagency Sedimentation Conference, Interagency Advisory Committee on Water Data, V. 2, Sec. 8, p. 61-68.
- Pui, David Y. H., and Liu, Benjamin, Y. H., 1989, Advances in instrumentation for atmospheric aerosol measurement, TSI Journal of Particle Instrumentation, v. 4, no. 2, p. 3-19.
- Rakoczi, Li, 1973, Tracer techniques in sediment transport, International Atomic Energy Agency, Vienna, Technical Reports Series 145, p. 216-225.
- Ricley, W. W., 1933, Settling velocities of gravel, sand, and silt particles, American Journal of Science, v. 225, p. 325-338.
- Rittenhouse, Gordon, 1944, Sources of modern sands in the Middle Rio Grande Valley, New Mexico, The Journal of Geology, v. 12, no.3, p 145-181.
- Rittenhouse, Gordon, and Thorpe, E. M., 1943, Heavy minerals in sediment transport studies, Transaction of the American Geophysical Union, p. 524-530.
- Rouse, H., 1937, Modern conceptions of the mechanics of fluid turbulence, Transactions, ASCE, v. 102, paper no. 1965, p. 463-543.
- Rubey, W., 1933, Settling velocities of gravel, sand and silt particles: American Journal of Science, v. 23, p. 325-338.
- Sheppard, Laurel M., 1988, Automation of particle analysis; Ceramic Bulletin, v. 62, no. 5.
- Shimadzu, undated, SALD-1100 laser diffraction particle size analyzer, Shimadzu Corp., Tokyo, Japan, 7 p.
- Simons, Daryl B., 1991, Practical sediment management techniques utilized in the United States, in Proceedings of the Fifth Federal Interagency Sedimentation Conference, Interagency Advisory Committee on Water Data, v.1, p. K1-K12.
- Singer, J. K., Anderson, J. B. Ledbetter, M. T., McCave, I. N., Jones, K. P. N., and Wright, R., 1988, An assessment of analytical techniques for the size analysis of fine grained sediments; Journal of Sediment Petrology, v. 58, no. 3, p. 534-543.

References *continued*

- Stein, Rudiger, 1985, Rapid grain size analysis of clay and silt fractions by Sedigraph 5000 D: comparison with Coulter counter and Atterberg methods; *Journal of Sedimentary Petrology* v. 55, no. 4, p. 590-593.
- Swift, Donald J. P., Schubel, J. R., and Sheldon, Raymond W., 1972, Analysis of fine grained suspended sediments, a review, *Journal of Sediment Petrology*, v. 42, no.1, p. 122-134.
- U.S. Department of Commerce, 1994, NIST Special Publication 260, Technology Administration, National Institute of Standards and Technology, Gaithersburg, MD, 16 p.
- Weaver, A., Van B., and Grobler, D. C., 1981, An evaluation of the Sedigraph as a standard method of sediment particle size analysis, *Water SA*, v. 7 no.2, p. 79-87.
- Welch, Norman H., Allen, Paul B., and Galindo, Donnie Ji, 1979, Particle size analysis by pipet and Sedigraph, *Journal of Environmental Quality*, v. 8, no. 4, p. 543-546.
- Whitney, G. G. Jr., 1960, The Woods Hole rapid analyzer for sands, unpublished manuscript, reference no. 60-36, Woods Hole Oceanographic Institution, 25 p.
- Wieder, Sol, 1992, *Introduction to MathCAD for Scientists and Engineers*, McGraw-Hill, 292 p.
- Wilson, R., 1980, Reference materials of defined particle size certified recently by the community Bureau of Reference of the European economic community; in *Powder Technology*, Elsevier Sequoia, v. 27, p. 37-43.
- Wong, Sam S. Y., Combs, Phil; and Letter, Joseph, 1991, Simulation of sediment transport near structures, practical sediment management techniques utilized in the United States, in *Proceedings of the Fifth Federal Interagency Sedimentation Conference*, Interagency Advisory Committee on Water Data, v. 2, Sec. 8, p. 77-84.
- Yuqian, Long, Emmett, William W., and Janda, Richard J., 1989, Comparison of some methods for particle size analysis of suspended sediment samples, in *Proceedings of Fourth International Symposium on River Sedimentation*, Beijing, PRC, 10 p.
- Ziegler, C. A., Papadopoulos, J., and Sellers, Bach, 1967, Radioisotope gage for monitoring suspended sediment in rivers and streams: *International Journal of Applied Radiation and Isotopes*, v. 18, Pergmon Press Ltd, p. 585-59.

APPENDIX 1

CONDENSED VERSION OF
ADVERTISEMENT IN THE *COMMERCE BUSINESS DAILY* NEWSPAPER
January 23, 1989

SPECIFICATIONS FOR FLUVIAL SEDIMENT
PARTICLE-SIZE ANALYZER

1. Scope

This document presents requirements for laboratory instruments designed to measure particle-size distributions of fluvial sediment samples.

2. Supporting documents

2.1 Guy, Harold P., Techniques of water resources investigations of the United States Geological Survey, Chapter C1, laboratory theory and methods for sediment analysis.

2.2 A study of methods used in the measurement and analysis of sediment loads in streams, 1981, Report no. 4, methods of analyzing sediment samples.

2.3 National handbook of recommended methods for water-data acquisition: sediment, chapter 3, appendix 3.L.2.

2.4 Vanoni, Vito A. (editor), 1975, ASCE manual No. 54, sedimentation engineering, particle size data and methods, p. 407-426.

2.5 Swift, Donald J. P., Schubel, J. R., and Sheldon, Raymond W., 1972, Size analysis of fine grained suspended sediments.

2.6 American society for testing and materials, standard E-11, specifications for wire cloth sieves for testing purposes.

3. Definitions

3.1 Standard fall velocity—The average rate of fall that a particle attains in settling through quiescent distilled water of infinite extent and at a temperature of 24 °C.

3.2 Fall diameter of a sediment particle—The diameter of a smooth sphere having a density of 2650 Kg/m³ and having the same standard fall velocity as the sediment particle under study.

3.3 Particle size distribution—The relative mass of particles in each of several specified fall diameter ranges. An alternate definition calls for the relative mass of particles finer than (or coarser than) each of several specified fall diameters. Relative mass is unusually expressed as a percentage and is obtained by dividing the mass in the fall diameter range by the mass of the

complete sample then multiplying the quotient by 100.

4. Use of the analyzers

4.1 Workers involved in fluvial sediment investigations routinely collect and analyze samples of water sediment as an aid in studying sediment movement and deposition in rivers, estuaries, and reservoirs. One phase of the analysis consists of measuring particle-size distributions of sediment grains in the samples. By using a gravimetric settling process, the standard fall velocities of the grains are measured; then the velocities are converted to fall diameters. Last of all, the proportion of grains smaller than certain diameters are tabulated; then the proportions are converted to fall diameter distributions.

4.2 This traditional gravimetric process is not only difficult to perform but it is also subject to operator error. A more efficient means of obtaining particle size data is badly needed; however, it is highly desirable that new particle-size analyzers yield data compatible with data obtained by the traditional method. Particle-size measurements should continue to be based on fall diameter concepts outlined in the definitions of section 3.

5. Technical requirements

5.1 Operating environment—The particle-size analyzer discussed in these specifications should be designed for use in clean laboratory environments.

5.2 Sample processing mode and sample volume requirements—The analyzer shall accept discrete samples ranging from 300 ml to 1000 ml in volume. To augment this batch mode operation, a continuous flow mode will be desirable but not essential.

5.3 Sediment concentration ranges—The analyzer shall process samples ranging in concentration from 300 mg/L to 10,000 mg/L. Samples with concentrations falling outside the analyzer's range shall be detected automatically.

5.4 Particle-size ranges—The analyzer shall be capable of measuring particles in the fall diameter range from 2 microns to 62 microns. The relative amount of sediment finer than 2 micrometers shall be reported; however, a detailed size analysis below this limit is not required. It is desirable, but not essential, that the upper size limit exceed 62 micrometers. An upper limit of 1000 micrometers will greatly expand the instrument's range of application.

5.5 Dispersion equipment—Samples containing particles in the silt size and clay size range are usually treated with chemical dispersants prior to making a size analysis. The analyzer shall contain chemical metering (injection) equipment and ultrasonic transducers to aid in dispersing and deflocculating fine-grained suspensions.

5.6 Internal compensation—The analyzer shall be internally compensated to eliminate errors caused by chemical dispersants. The addition of dispersants alters fall velocities by changing fluid densities and fluid viscosities.

5.7 Internal diagnostics—Critical components in the analyzer shall be monitored automatically. Alarms shall warn operators if critical parameters shift outside permissible limits.

5.8 Data display—The instrument shall display particle-size data as a percent finer graph on either a CRT or on an inking type x-y plotter. The graph shall be displayed as it is being created by the addition of data points.

5.9 Data storage and transfer—Results of most analyzers must be transferred to ADP equipment to facilitate filing, searching, retrieving, and sorting the data. The size analyzer shall be equipped with a bar code reader and a keyboard for entering header data for each analysis. The header will include (a) a file number, (b) a date and time of sample collection, (c) a river name and location of the sampling station, (d) water temperature and gauge height at the time of sampling, and (e) about 500 characters of miscellaneous descriptive information. The analyzer shall be equipped with a solid state memory with enough capacity to store size data and header data for more than twenty runs. Under keyboard control, the analyzer shall transfer all stored files to an external device connected to a standard RS-232 serial output port on the analyzer. The analyzer shall also be capable of displaying all stored files selected by the operator. Three formats shall be available: distribution of “percent-finer-than” values, distribution of “percent-coarser-than” values and distributions of within class interval values. The default interval classes shall be ASTM sieve sizes and shall follow the fourth root of two progression. Default breakpoints of the size classes in micrometers shall be as follows: 37, 44, 53, 63, 74, 88, 105, 125, 149, 177, 210, 250, 297, 354, 420, 500, 595, 707, 841, and 1000. The analyzer shall accept by keyboard entry at least five size classes smaller than 37 micrometers. In addition, the analyzer shall accept any set of 25 sizes to override the default sizes.

5.10 Power—The analyzer shall operate on 120 vac, 50-60 Hz power lines and shall draw less than 1000 watts.

5.11 Warm-up time—Beginning from a cold start, the analyzer shall warm up and become fully operational in less than 30 minutes. As an option available to the operator, the analyzer shall also have a low-power, standby mode for instant-on operation.

5.12 Analysis time—Analysis time will likely depend upon the range of particle sizes in the sample. For worst case samples, probably those containing the bulk of material in the clay-size range, analysis time shall be less than 30 minutes.

5.13 Materials and cleaning—Some samples will be subjected to chemical analysis after removal from the size analyzer. It is therefore important that all wetted components in the size analyzer be made of inert fluorocarbons or type 316 stainless steel. Wetted components shall be designed for cleaning by simple rinsing operations.

5.14 Ambient temperature and humidity ranges—The analyzer shall operate through the temperature range of 10 °C to 30 °C and through the humidity range of 10% to 90%.

5.15 Instruction manuals and operator training—At least three copies of all operating manuals shall be provided with each analyzer. Factory-trained engineers shall be available for telephone consultation. If operator training is required, sessions shall be conducted at sites chosen by the user.

5.16 Safety—The analyzer shall conform with all Occupational Safety and Health Administration (OSHA) and National Bureau of Standards (NBS) safety standards.

5.17 Warranties and servicing—The analyzer shall carry one-year unconditional warranties to cover defects in material and workmanship. Servicing contracts shall be available to cover maintenance and servicing problems arising after the warranty period expires.

APPENDIX 2

TABLE 10. Cross Reference of Data.

The first number is the *data sheet* referenced by Hotchkiss (1994); the second is the *index number* in the database of this report.

1—*	2—6	3—*	4—*
5—*	6—7	7—*	8—8
9—*	10—9	11—*	12—10
13—*	14—11	15—*	16—12
17—13	18—14	19, 20, 21—15	22—16
23—17	24—18	25—19	26—20
27—21	28—22	29—23	30—24
31—25	32—26	33—27	34—28
35—29	36—30	37—31	38—32
39—33	40—34	41—35	42—36
43—37	44—38	45—39	46—40
47—41	48—42	49—43	50—44, 45, 46, 47
51—48, 49	52—50, 51	53—322, 323, 324	54—325, 326
55—54	56—55	57—56	58—57
59—58	60—59	61—52	62—53
63—60	64—61	65—62	66—63
67—***	68—***	69—***	70—***
71—***	72—***	73—***	74—***
75—***	76—74	77—65	78—66
79—67	80—68, 69	81—68, 69	82—70, 71
83—72, 73	84—74, 75	85, 86—76	87, 88, 89—77, 78
90, 91—79	90, 92—80	93—81	94—82
95—83	96—84	97—85	98—86
99—87	100—88	101—89	102—90
103—91	104—92	105—93	106—****
107—****	108—****	109—94	110—95
111—97	112—98	113—99	114—100
115—101	116—102	117—103	118—104
119—105	120—106	121—107	122—108
123—109	124—110	125—111, 112, 113	126—114, 115, 116
127—117, 118	128—119, 120	129—121, 122, 123, 124	130—125, 126
131—127, 128	132—129, 130	133—131, 132	134—133, 134
135—135, 136	136—137, 138	137—139, 140, 141	138—142, 143, 144
139—145, 146, 147	140—148, 149	141—150	142—151
143—152	144—153	145—154	146—155
147—156	148—157	149—158	150, 151—159, 160
152—161, 162, 163	153—164, 165	154—166, 167, 168	155—169, 170
156—171, 172	157—173, 174	158—175, 176	159—****
160—****	161—180, 181	162—****	163—****
164—****	165—****	166—****	167—183
168—184	169—185	170—186	171—187
172—188	173—189	174—190	175—191
176—192	177—193	178—194	179—195
180—196	181—197		

*Not recorded in this report. Hotchkiss notes Sedigraph data are missing.

** Not recorded in this report. Data sheets are missing in Hotchkiss file.

***Not recorded in this report. Data sheets are missing Hotchkiss notes pipet data.

**** Data recorded in comments file of this report. Data are not compatible with database format.

APPENDIX 3

TABLE 11. Supplementary Information for Database.

The leading number for each section refers to a Hotchkiss (1994) data sheet. Index numbers refer to data in this report.

50—Index numbers 44, 45, 46 and 47.

Data are for a special (handground) sediment tested at dispersant concentrations of 3 ml/g, 2 ml/g, 1 ml/g, and 0.5 ml/g. Units are in ml of dispersant per gram of sediment. Agricultural Research Service (ARS) practice for pipet analysis is about 1 ml of dispersant for each gram of sediment.

53-54—Index numbers 198-202.

All runs were made with Sedigraph: pipet data are missing. Samples were soaked for various time intervals before testing.

93-98—Four repeat runs made with pipet.

Means for the four-percent-less-than values are in the database of this report. (Refer to index numbers 81-86). The following table shows means and *standard deviation values* for the runs.

	Fall diameters in mm					
	63	31	16	8	4	2
	means of percent-less-than values followed by standard deviations					
Sheet 93	100 0	97.4 0.7	90.3 0.7	73.3 0.5	58.4 0.2	48.0 0.3
Sheet 94	100 0	83.2 0.6	73.2 0.7	61.3 0.5	51.3 0.3	43.3 0.6
Sheet 95	100 0.1	67.1 0.4	49.1 0.7	37.0 0.2	30.6 0.4	25.7 0.1
Sheet 96	100 0	99.0 0.3	97.4 0.4	86.2 0.8	71.5 0.4	60.2 0.5
Sheet 97	100 0	99.8 0.4	97.2 0.2	90.3 0.2	81.2 0.3	71.6 0.4
Sheet 98	100 0	98.1 0.5	89.5 0.3	71.7 0.5	57.2 0.6	47.2 0.4

99-104—Five repeat runs made with pipet,
Means for the-four percent-less-than values are in the database of this report. (Refer to index numbers 87-92). The following table shows means and *standard deviation values* for the runs.

	Fall diameter in mm					
	63	31	16	8	4	2
	means of percent-less-than values followed by standard deviations					
Sheet 99	99.8 0.1	69.4 1.0	48.2 0.6	34.7 0.3	27.7 0.2	23.4 0.6
Sheet 100	99.9 0.02	80.2 0.7	56.6 0.4	41.4 0.4	34.1 0.1	29.1 0.3
Sheet 101	99.9 0.04	74.8 1.2	43.3 0.2	28.6 0.5	23.2 0.4	20.8 0.8
Sheet 102	100 0	87.6 1.2	58.2 1.1	38.7 0.5	29.4 0.3	25.2 0.7
Sheet 103	100 0.02	81.0 0.8	39.3 1.0	21.3 0.1	15.2 0.3	12.5 0.5
Sheet 104	100 0	85.5 0.6	50.4 0.7	29.6 0.2	21.7 0.2	17.2 0.6

106—No entry in the database of this report.
 All runs were with pipet: no Sedigraph data. Comparison of U.S. Geological Survey round robin and single lab results on samples designated “FQ.” Data can be used for comparing means and standard deviations for pipet for *ten* labs and *one* lab.

	Fall diameter in mm					
	63	31	16	8	4	2
	mean percent-less-than values for <i>ten</i> labs					
	100	92	90	87	83	76
	standard deviation for <i>ten</i> laboratory analyses					
	0.9	2.7	2.3	3.3	6.3	6.6
	mean percent-less-than- values for <i>one</i> lab					
	100	93	88	88	86	81
	standard deviation for <i>one</i> laboratory analysis					
	0	1.2	3.0	1.1	0.9	1.8

107—No entry in the database of this report.

All runs were with the pipet: no Sedigraph data. Comparison of U.S. Geological Survey round robin and single laboratory results on samples designated “KC.” Data can be used for comparing mean and standard deviation of pipet between *nine* labs and *one* laboratory.

Fall diameter in mm					
63	31	16	8	4	2
mean percent-less-than values for <i>nine</i> labs					
100	61	40	28	20	15
standard deviation for <i>nine</i> labs					
0.9	3.2	1.1	1.0	3.1	4.4
mean percent-less-than values for <i>one</i> lab					
100	61	37	25	22	16
standard deviation for <i>one</i> lab					
0	1.1	1.4	1.1	0.8	1.0

108—No entry in the database of this report.

All runs were made with the pipet procedure: no Sedigraph data were collected. The file consists of comparisons of U.S. Geological Survey round robin and single lab results on samples labeled “SM.” Data can be used comparing mean and standard deviation for pipet between *ten* laboratories and *one* laboratory.

Fall diameter in mm					
63	31	16	8	4	2
mean percent-less-than values for <i>ten</i> laboratories					
100	73	52	38	32	28
standard deviation for <i>ten</i> laboratories					
0.4	4.4	2.6	4.6	5.0	5.2
mean for <i>one</i> laboratory					
100	75	50	37	34	29
standard deviation for <i>one</i> laboratory					
0	1.0	1.4	2.1	1.6	1.6

109—Sample from Fremont Lake, Wyoming, at outlet: depth of 5 meters: bed material.

110—Sample from Fremont Lake, Wyoming, at outlet: depth of 15 meters: bed material.

110A—Sample from Fremont Lake, Wyoming, at outlet: depth of 30 meters: bed material.

111—Sample from Fremont Lake, Wyoming, at outlet: depth of 45 meters: bed material.

112—Sample from Fremont Lake, Wyoming, at outlet: depth of 75 meters: bed material.

113—Sample from Fremont Lake, Wyoming, at outlet: depth of 105 meters: bed material.

114—Sample from Fremont Lake, Wyoming, at outlet: depth of 135 meters: bed material.

115—Sample from Fremont Lake, Wyoming, at outlet: depth of 185 meters: bed material.

116—Sample from Fremont Lake, Wyoming, at outlet: depth of 105 meters: bed material.

117—Sample from Fremont Lake, Wyoming, at outlet: depth unknown: bed material.

118—Sample from Fremont Lake, Wyoming, at outlet: depth of 90 meters: bed material.

119—Sample from Fremont Lake, Wyoming, at outlet: depth of 60 meters: total sample.

120—Sample from Fremont Lake, Wyoming, at outlet: depth of 45 meters: total sample.

121—Sample from Fremont Lake, Wyoming, at outlet: depth of 15 meters: bed material.

122—Sample from Fremont Lake, Wyoming, at inlet: depth of 5 meters: bed material.

123—Sample from Fremont Lake, Wyoming, at inlet: depth of 42 meters: bed material.

124—Sample from Fremont Lake, Wyoming, at inlet: depth of 60 meters: bed material.

125-130—These sheets correspond to index numbers 111-126 in this report.

All runs made at the same sediment concentration, slightly greater than 3% by volume. After each run, 10 ml of dispersing agent was added.

141-149—These sheets correspond to index numbers 150-158 in this report.

Percent-less-than values are recorded in database of this report. Following gives means and *standard deviation values* at each of the breakpoints.

141—Index number 150.

Sample from Rio Grande at El Paso, Texas.

	Fall diameter in mm					
	32	16	8	4	2	1
<i>means of 7 Sedigraph runs</i> at each fall diameter breakpoint						
	89.1	74.9	60.1	46.9	34.2	29.5
standard deviations of Sedigraph runs						
	1.93	1.54	2.21	1.07	1.78	3.04
<i>means of 10 pipet runs</i> at each fall diameter breakpoint						
	80.5	66.2	54.3	49.5	40.6	29.9
standard deviations of pipet runs						
	1.20	1.67	2.55	2.49	2.85	1.39

142—Index number 151.

Sample from Fort Quintan, Texas.

	Fall diameter in mm					
	32	16	8	4	2	1
<i>means of 25 Sedigraph runs</i> at each fall diameter breakpoint						
	96.3	92.4	90.4	86.8	76.9	63.7
standard deviations of Sedigraph runs						
	0.86	0.66	0.64	0.98	2.51	3.33
<i>means of 28 pipet runs</i> at each fall diameter breakpoint						
	92.6	89.7	87.6	84.4	79.3	65.4
standard deviations of pipet runs						
	1.97	1.74	2.40	4.74	5.81	1.60

143—Index number 152.

Sample from Kroto Creek near Willow, Alaska.

	Fall diameter in mm					
	32	16	8	4	2	1
<i>means of 14 Sedigraph runs</i> at each fall diameter breakpoint						
	77.1	49.3	37.1	27.0	19.2	12.7
standard deviations of Sedigraph runs						
	1.69	0.99	0.62	0.75	0.73	0.54
<i>means of 14 pipet runs</i> at each fall diameter breakpoint						
	61.6	38.3	27.2	23.1	18.2	11.4
standard deviations of pipet runs						
	2.02	2.44	3.37	3.33	3.51	1.30

144—Index number 153.

Sample from Los Padres Reservoir near Carmel, CA.

	Fall diameter in mm					
	32	16	8	4	2	1
<i>means of 9 Sedigraph runs</i> at each fall diameter breakpoint						
	93.2	68.1	41.6	23.6	13.3	6.8
standard deviations of Sedigraph runs						
	1.94	2.70	2.63	2.47	1.73	1.39
<i>means of 10 pipet runs</i> at each fall diameter breakpoint						
	87.8	58.8	35.1	23.4	13.8	7.0
standard deviations of pipet runs						
	2.84	2.49	6.30	8.07	7.40	0.93

145—Index number 154.

Sample from Rio Puerco near Bernardo, NM.

	Fall diameter in mm					
	32	16	8	4	2	1
<i>means of 20 Sedigraph runs at each fall diameter breakpoint</i>	99.2	98.0	91.5	76.3	62.6	50.6
standard deviations of Sedigraph runs	0.49	0.64	1.08	1.75	1.83	2.1
<i>means of 20 pipet runs at each fall diameter breakpoint</i>	99.6	97.1	88.5	81.8	69.4	55.1
standard deviations of pipet runs	0.81	0.56	2.35	2.62	5.21	2.02

146—Index number 155.

Sample from Ralston Creek at Iowa City, Iowa.

	Fall diameter in mm					
	32	16	8	4	2	1
<i>means of 10 Sedigraph runs at each fall diameter breakpoint</i>	78.8	39.0	28.0	24.0	21.4	18.4
standard deviations of Sedigraph runs	4.08	1.75	0.96	0.56	0.50	0.37
<i>means of 10 pipet runs at each fall diameter breakpoint</i>	66.8	27.8	22.0	19.2	17.1	14.1
standard deviations of pipet runs	1.01	1.92	1.61	1.63	1.61	0.83

147—Index number 156.

Sample from Rio Grande floodway at San Acacia, NM.

	Fall diameter in mm					
	32	16	8	4	2	1
<i>means of 19 Sedigraph runs at each fall diameter breakpoint</i>	99.8	98.6	95.8	82.3	65.3	49.4
standard deviations of Sedigraph runs	0.42	0.74	1.31	1.61	2.36	2.24
<i>means of 11 pipet runs at each fall diameter breakpoint</i>	99.4	98.3	93.3	82.5	67.4	48.6
standard deviations of pipet runs	0.80	0.82	0.80	2.53	3.48	2.37

148—Index number 157.

Sample from Tanana River above Moose Creek Dam, Alaska. Probably only one run on Sedigraph and pipet since no standard deviation values were recorded.

149—Index number 158.

Sample probably from Tanana River sample #2 but not specifically identified. Possibly only one run on Sedigraph and pipet since no standard deviation values were recorded.

152—Index number 161.

Sediment concentration about 5% by volume.

152—Index number 162.

Sediment concentration about 3.5% by volume.

152—Index number 163.

Sediment concentration about 2% by volume.

Data not logged by Hotchkiss (1994)—Index numbers 203, 204, and 205 in this report.

Three samples were prepared identically and dispersed in deionized water and 1% sodium hexametaphosphate. All runs were by same operator.

159—Data from Welch and others (1979).

Five samples: each was tested four times. Standard deviations are averages for the five samples. Prior to analysis, samples were soaked overnight. Dispersant concentration was 4.36%. Welch remarks the concentration was, "... considered to provide equal and complete dispersion." This is one of few tests in which most standard deviations for Sedigraph tests were larger than standard deviation for pipet tests. Sediment concentration in Sedigraph was 45,900 mg/L; pipet concentration was 2000 mg/L.

	Particle fall diameter in <i>mm</i>				
	31	16	8	4	2
	Standard deviation				
Sedigraph	0.7	0.8	0.7	0.7	0.1
Pipet	0.7	0.5	0.4	0.3	0.5

160—Data are in index 177, 178, and 179 of this report.

Data in index 177 are for Sedigraph at sediment concentration of 45,900 mg/L; index 178 is for 22,900 mg/L; index 179 is for 11,500 mg/L. All runs on same sample. Index 179 is pipet data.

Data from Welch and others (1979)

161—Data are in index numbers 180, 181, and 182 of this report.

Data in index 180 are for Sedigraph at sediment concentration of 45,900 mg/L; index 181 is for 91,800 mg/L; index 182 is for 137,700 mg/L. All runs on sample for data sheet 160. Index 182 in this report is pipet data.

162—Data from Wilson (1980) give size distribution of BCR 66 (natural quartz reference sample).

All measurements were by pipet.

	Fall diameter in <i>mm</i>										
	3.5	2.5	2.0	1.8	1.2	1.0	0.9	0.75	0.6	0.5	0.35

			Mean of percent finer values							
100	94	85	70	55	45	33	20	12	8	3
			Standards deviation of percent-finer values							
2	2	4	8	7	5	5	5	4	1.5	1.5

163—Data from Wilson (1980) gives size distribution of BCR 70 (natural quartz reference sample).

All measurements were by pipet.

			Fall diameter in mm										
20	11	9.5	8.0	7.0	6.0	4.8	3.5	3.0	2.5	2.0	1.8	1.5	1.2
			mean of percent-finer values										
100	97	95	92	88	83	77	60	48	37	27	22	13	10
			standard deviations of percent-finer values										
1	1	1	1	1	2	2	2.5	4.0	4.0	3	5	2	2

164—Data from Wilson (1980) gives size distribution of BCR 67 (natural quartz reference sample).

All measurements were by pipet.

			Fall diameter in mm					
32	22	17	11	8	5.8	4	3.5	2.5
			means of percent-finer values					
100	98	84	57	31	12	4	3	2
			standard deviations of percent-finer values					
1	1	2	4	5	3	1	1	1

165—Data from Wilson (1980) gives size distribution of BCR 69 (natural quartz reference sample).

All measurements were by pipet.

			Fall diameter in mm			
100	70	45	31	22	18	14
			means of percent-finer values			
97	82	63	40	20	5	3
			standard deviations of percent-finer values units			
2	3	3	3	3	2	1

166—Data from Wilson (1980) gives size distribution of BCR 66 (natural quartz reference sample).

Refer to sheet 162.

			Fall diameter in mm							
3.5	2.5	1.8	1.5	1.2	1.0	0.9	0.75	0.6	0.5	0.35
			mean of finer-than values by <i>pipet</i>							
100	94	85	73	55	45	33	20	12	8	3
			mean in finer-than values by <i>Sedigraph</i>							

97 93 84 73 57 47 36 25 17 12 5

167—Index number 183.

Data for a 0.20 m to 0.30 m deep core from center of Three Mile Lake, Sunflower Co., MS.

168—Index number 184.

Data for an 8-inch to 12-inch-deep core from Three Mile Lake, Sunflower Co., MS, station 8B.

169—Index number 185.

Data is for a 4-inch to 8-inch-deep core from Three Mile Lake, Sunflower Co., MS, station 8A.

170—Index number 186.

Data is for a 20-inch to 24-inch-deep core from Three Mile Lake, Sunflower Co, MS

171—Index number 187.

Data is for a 28-inch to 32-inch-deep core from Three Mile Lake, Sunflower Co. MS

APPENDIX 4

Pipet and X-RAY Basic Data Tables

In the tables, fields of data are identified by index numbers listed in the left column of each sheet. Data for each index is on two adjoining pages: the first gives the sample serial number, agency that performed the test, location of the testing laboratory, analysis date, whether or not a dispersant was used, and the source of the sample. The second page gives percent-less-than values at particle-size breakpoints ranging from 2 mm to 62 mm. The letter “p” indicates pipet data; the letter “s” indicates Sedigraph data. By way of example, sample index 10 was analyzed by the ARS (Agricultural Research Service) at Chickasha, Oklahoma. It contained 50.2% finer than 10 mm as analyzed by the pipet procedure. The following acronyms and abbreviations are used in the tables: PRS (Peoples Republic of China); YRCC (Yellow River Conservancy Commission); ARS (Agricultural Research Service); Chic., OK (Chickasha, Oklahoma); Cascades V.O. (Cascades Volcano Observatory); GS (U.S. Geological Survey); EPA (Environmental Protection Agency); Emmett (Dr. William Emmett, U.S. Geological Survey); Schiebe (Dr. Frank Schiebe, Agricultural Research Service); L (Lake); R (River) and Cr (Creek).

Index	Sample Serial #	Agency	Location	Analysis Date	Dispersant	Source of Sediment	2p	2s	4p	4s	8p	8s	10p	10s	16p	16s	20p	20s	32p	32s	50p	50s	62p	62s
1	1	PRC	YRCC	7/9/87		Cascades V. O.	8.3	10.7	13.4	16.9	19.9	25.6			27.6	34.6			37.3	48.1			56.2	56.2
2	2	PRC	YRCC	7/9/87		Cascades V. O.	12.8	14.3	19.2	19.5	27.4	27.4			36.7	37.6			49.9	54.1			75.1	75.1
3	3	PRC	YRCC	7/9/87		Cascades V. O.	12.2	12.3	19.2	19.7	27.9	29.3			39	40.1			52.7	60.2			77.2	77.2
4	4	PRC	YRCC	7/9/87		Cascades V. O.	10	11.7	16.7	18.3	25.9	28.2			35.7	38.4			48.5	57			73.1	73.1
5	48	ARS	Chic, OK	12/16/76	yes	Soil, Chic. Res. Plot	29	54	36.5	58.5	43.3	63.5			43.5	65.3	51	76	64.3	89.3	72.8	98.8		
6	70	ARS	Chic, OK	12/17/76	yes	Soil, Chic. Res. Plot	39	48	44	57	49.3	65	54.3	67			63.6	75	72.5	87	90.3	98		
7	66	ARS	Chic, OK	12/17/76	yes	Soil, Chic. Res. Plot	26.3	28	28.7	33	32.7	38	34.5	40			45.7	55	63.2	77	89.7	95		
8	62	ARS	Chic, OK	12/17/76	yes	Soil, Chic. Res. Plot	48.1	49	52.9	62	63.2	71	66	73			76.2	83	83.4	92	96.1	99		
9	60	ARS	Chic, OK	12/16/76	yes	Soil, Chic. Res. Plot	26.6	33	27	36	31.9	41	33.8	43			40.3	53	56.4	73	83.8	92		
10	59	ARS	Chic, OK	12/16/76	yes	Soil, Chic. Res. Plot	36.5	43	40	50	48.9	58	50.2	61			66	77	78	93	94	98		
11	56	ARS	Chic, OK	12/16/76	yes	Soil, Chic. Res. Plot	32.6	44	34.7	48	39.1	55	45.6	58			60.1	74	79.8	89	95.8	99.9		
12	54	ARS	Chic, OK	12/16/76	yes	Soil, Chic. Res. Plot	35.6	40	35.4	48	42.6	57	48.3	60			66	75	78.5	93	97.9	99		
13	BKR 129	ARS	Chic, OK	12/20/76	yes	Pond, E. Bitter 1	40.5	47.5	42.5	53.5	49	59	51	61			57.5	71.5	67.6	88	87	99.9		
14	BKR 132	ARS	Chic, OK	12/16/76	yes	East Bitter 2	50.5	53	58.5	61.5	66	73	68.5	76			76.5	87	86	97	96	99.9		
15	BKR 122	ARS	Chic, OK	12/20/76	yes	East Bitter 3	51	54	56.5	59	62.5	66	64	67.5			74	79	87	91	99	98		
16	BKR 130	ARS	Chic, OK	12/20/76	yes	East Bitter 3A	50	54	51	59	58	66	64.5	68			74	80	80	91.5	92.5	99		
17	BKR 135	ARS	Chic, OK	12/20/76	yes	East Bitter 4	48	46	51	54	57	62	59	64.5			71	77	83	90	98	98		
18	BKR 159	ARS	Chic, OK	12/20/76	yes	Pond, W. Bitter 1	50	57	54	64	60.5	71.5	64	74			72	84.5	84	93.5	95.5	99		
19	BKR 198	ARS	Chic, OK	12/20/76	yes	Pond, W. Bitter 2	36	43	37	47	44	54	46	56			55.5	70	67.5	87	86.5	98		
20	BKR 153	ARS	Chic, OK	12/20/76	yes	West Bitter 3	59	59	68.5	68.5	75	78	77	81			85.5	88.5	93	97	99.9	99.5		
21	BKR 57	ARS	Chic, OK	12/20/76	yes	Pond, Salt Creek	59	62	66	70.5	71.5	79	78	81			85	88	91	93	96	98		
22	BKR 101	ARS	Chic, OK	12/20/76	yes	Salt 2	41.5	44	49	50	51	58	53.5	62			69.5	78	85	94	98	99.5		
23	BKR 219	ARS	Chic, OK	12/20/76	yes	Salt 3	45	56	56	63	62.5	72	67.5	75			76.5	85	86	96	98	99.9		
24	BKR 113	ARS	Chic, OK	12/20/76	yes	Lake Chickasha	13.5	12	20.5	17	24	26	30	30			41	46	58.5	73	86	99		
25	BKR 113 Rerun	ARS	Chic, OK	12/20/76	yes	Lake Chickasha	14	16	20	22	24	33	30	37			41	56	58	83	86	99.9		
26	BKR 16	ARS	Chic, OK	1/7/77	yes	Soil, Chic. Res. Plot	30	52	37	57.5	43	64	44	65			51	76	64	89	73	98.8		
27	BKR 163 S51	ARS	Chic, OK	1/7/77	yes	Soil, Chic. Res. Plot	32.5	45	38	54	44	62	52	65			62	78.5	75	90	95.2	98		
28	BKR 39 S54	ARS	Chic, OK	1/16/77	yes	Soil, Chic. Res. Plot	35.5	40	35.5	47	42.5	57	48.5	60			66	75	78.5	93.5	98	99		
29	BKR 154 S55	ARS	Chic, OK	12/20/76	yes	Soil, Chic. Res. Plot	43.5	49	48.5	56	53.5	63	58.5	65.5			68.5	76	84	90	93.5	99		
30	BKR 208 S56	ARS	Chic, OK	12/16/76	yes	Soil, Chic. Res. Plot	32.5	44	34.5	48	39	55	45.5	57.5			60	74	80	89	96	99.9		
31	BKR 88 S58	ARS	Chic, OK	12/16/76	yes	Soil, Chic. Res. Plot	50	63.5	59	69.5	67.5	75.5	72.5	78			79.5	86	89.5	94	94.5	99.5		
32	BKR 886 S59	ARS	Chic, OK	12/16/76	yes	Soil, Chic. Res. Plot	36.5	43	40	50	48.5	58	50.5	61			66	77	78	93	94	98.5		
33	BKR 46 S60	ARS	Chic, OK	12/16/76	yes	Soil, Chic. Res. Plot	26	33	27	36	32	40.5	34	43			40.5	53.5	56.5	77	84	97		
34	BKR 213 S62	ARS	Chic, OK	12/17/76	yes	Soil, Chic. Res. Plot	48	49	53	60	63.5	70.5	66	72.5			76	83	83.5	92	96	99		
35	BKR 43 S63	ARS	Chic, OK	12/17/76	yes	Soil, Chic. Res. Plot	45	42	49	49	55.5	55	54	57			66	68	90	93	97	97		
36	BKR 197 S64	ARS	Chic, OK	12/17/76	yes	Soil, Chic. Res. Plot	44	41	50	49	55	55	57	57			68	69	84	84	99	97		
37	BKR 73 S65	ARS	Chic, OK	12/17/76	yes	Soil, Chic. Res. Plot	44	44	44.5	47.5	48	52	49	54			58.5	64.5	71.5	81.5	92	96		
38	BKR 254 S66	ARS	Chic, OK	12/17/76	yes	Soil, Chic. Res. Plot	26	28	28.5	32.5	32.5	38.5	34.5	40.5			45.5	55	63	78	89.5	95.5		
39	BKR 72 S67	ARS	Chic, OK	12/17/76	yes	Soil, Chic. Res. Plot	34.5	36	35	39	35	42	38.5	44.5			47	56	59	78.5	82.5	99.5		

Index	Sample Serial #	Agency	Location	Analysis Date	Dispersant	Source of Sediment	2p	2s	4p	4s	8p	8s	10p	10s	16p	16s	20p	20s	32p	32s	50p	50s	62p	62s
40	BKR 189 S68	ARS	Chic, OK	12/17/76	yes	Soil, Chic. Res. Plot	40.5	44	44	50	47.5	57	49.5	61			64	78	82	93	90	99.9		
41	BKR 204 S70	ARS	Chic, OK	12/17/76	yes	Soil, Chic. Res. Plot	39	49	44	57	49	65	54	67			63	75	72	87.5	90.5	97.5		
42	BKR 148 S71	ARS	Chic, OK	12/17/76	yes	Soil, Chic. Res. Plot	32	40	37.5	47	47	56.5	50	59.5			61.5	75	81	90	92.5	99		
43	BKR 249 S73	ARS	Chic, OK	12/17/76	yes	Soil, Chic. Res. Plot	37.5	46.5	41	51	45.5	57	49	58			56	71.5	71.5	91	92	99		
44	BKR 266 S73	ARS	Chic, OK	12/14/76	3 ml/g	Soil, Chic. Res. Plot	38	52	41	57	46	61	49	63			56	75	72	91	92	99		
45	BKR 266 S73	ARS	Chic, OK	12/14/76	2 ml/g	Soil, Chic. Res. Plot	38	49	41	54	46	59	49	62			56	75	72	91	92	99		
46	BKR 266 S73	ARS	Chic, OK	12/14/76	1ml/g	Soil, Chic. Res. Plot	38	46	41	52	46	58	49	60			56	75	72	91	92	99		
47	BKR 266 S73	ARS	Chic, OK	12/14/76	.5 ml/g	Soil, Chic. Res. Plot	38	24	41	32	46	41	49	45			56	64	72	86	92	98		
48	BKR 163 S51	ARS	Chic, OK	12/14/76	1 ml/g	Soil, Chic. Res. Plot	38	44	38	54	44	63	53	67			61	81	75	94	96	99.9		
49	BKR 250 S51	ARS	Chic, OK	12/14/76	.5 ml/g	Soil, Chic. Res. Plot	33	41	38	52	44	62	53	66			61	78	75	92	96	99		
50	BKR 250 S51	ARS	Chic, OK	12/14/76	1 ml/g	Soil, Chic. Res. Plot	32	46	38	55	44	65	53	68.5			61	81	75	94	95	99.5		
51	BKR 250 S51	ARS	Chic, OK	12/14/76	.5 ml/g	Soil, Chic. Res. Plot	32	42	38	51.5	44	63	53	66			61	79	75	91	95	99.5		
52	BKR 220 S1251	ARS-GS	Chic, OK	12/20/76		Callegwas Cr, CA	38	44	48	52	54	64		69	69	77		84	87	93		99		
53	BKR 238 S1252	ARS-GS	Chic, OK	12/20/76	1.3 ml/g	Arroyo Simi, CA	46	46	60	65	76	84		89	94	95		97	99	99		99.9		
54	BKR 221	ARS-GS	Chic, OK	12/14/76	yes	Santa Ana R, CA	44	50	52	63	66	76		80	81	86.5		91	92	95.5		99		
55	BKR 248	ARS-GS	Chic, OK	12/17/76	yes	San Diego Cr, CA	50	44	51	55	54	67		71	70	77		83	82	93		99.9		
56	BKR 227 S519	ARS-GS	Chic, OK	12/17/76	yes	San Diego Cr, CA	50	44	51	53	58	64		69	70	75		81	82	90		98		
57	BKR 232 S522	ARS-GS	Chic, OK	12/17/76	yes	San Diego Cr, CA	51	43	56	54	64	69		70	73	77		84	84	93		99		
58	BKR 233 S567	ARS-GS	Chic, OK	12/17/76	yes	Unknown	43	48	54	62	62	76		81	80	88		93	95	99		99.9		
59	BKR 236 S1226	ARS-GS	Chic, OK	12/17/76	yes	Rodero Cr, CA	34	23.5	45	36	57	57		59	71	71		80	82	93		99.9		
60	BKR 259 S1263	ARS-GS	Chic, OK	12/20/76	yes	Tijuana R, CA			97	89	99	94		95	99.9	96		97	99.9	99		99.9		
61	BKR 259 Dil 0.5	ARS-GS	Chic, OK	12/20/76	yes	Tijuana R, CA	87	78	96	88	98	94		94	99.9	98		98	99.9	98		99.9		
62	BKR 259 Dil 0.25	ARS-GS	Chic, OK		yes	Tijuana R, CA	86	72	96	84	98	90		92	99.9	92		96	99.9	98		99.9		
63	BKR 226 S1264	ARS-GS	Chic, OK	12/20/76	yes	Tijuana R, CA	82	77	97	86	98	90		92	98	93		95	99	97		99		
64	BKR 1 MS 26-30	ARS	Chic, OK	12/21/76	yes	Manufactured	26	22	30	31	41	46		52	62	64		74	89	93		99	99.9	99
65	BKR 2 MS 26-30	ARS	Chic, OK	12/21/76	yes	Manufactured	26	20	30	29	41	44		49	62	62		69	89	92		99	99.9	99
66	BKR 3 MS 26-30	ARS	Chic, OK	12/21/76	yes	Manufactured	26	19	30	28	41	43		47	62	60		69	89	93		99.9	99.9	99.9
67	BKR 4 MS 26-30	ARS	Chic, OK	12/21/76	yes	Manufactured	26	20	30	28	41	44		47	62	58		64	89	90		99.5	99.9	99.9
67A	Soil S. 2	ARS	Chic, OK	3/8/76	yes	Chick, Pip. Comp.	27.1		31.9		37.9		41.2				53.7		72.6		88.1			
67B	Soil S. 2	ARS	Chic, OK	3/13/76	yes	Chick, Pip. Comp.	26.5		31.7		33.7		37.8				53.1		68.9		87.1			
68	Soil S. 5	ARS	Chic, OK	3/8/76	yes	Chick, Pip. Comp.	18.1		24.3		28.2		32.9				55.1		76.4		89.5			
69	Soil S. 5	ARS	Chic, OK	3/13/76	yes	Chick, Pip. Comp.	21.7		24.1		29.9		34.1				53.4		73.1		88.6			
70	Soil S. 8	ARS	Chic, OK	3/8/76	yes	Chick, Pip. Comp.	29.3		36.6		42.5		47.1				63.5		79.1		88.6			
71	Soil S. 8	ARS	Chic, OK	3/13/76	yes	Chick, Pip. Comp.	33.4		36.1		44.1		47.2				60.9		79.3		92.9			
72	Soil S. 9	ARS	Chic, OK	3/8/76	yes	Chick, Pip. Comp.	39.2		48.7		57.6		61.5				75.2		87.2		92.1			
73	Soil S. 9	ARS	Chic, OK	3/13/76	yes	Chick, Pip. Comp.	39.9		48.5		54.8		59.4				71.7		83		94.5			
74	Soil S. 10	ARS	Chic, OK	3/8/76	yes	Chick, Pip. Comp.	51.7		58.7		68.4		71.4				87.1		94.4		96.9			
75	Soil S. 10	ARS	Chic, OK	3/13/76	yes	Chick, Pip. Comp.	49.6		56.2		64.9		70.6				82.8		92.6		98.2			

Index	Sample Serial #	Agency	Location	Analysis Date	Dispersant	Source of Sediment	2p	2s	4p	4s	8p	8s	10p	10s	16p	16s	20p	20s	32p	32s	50p	50s	62p	62s
76	C	GS	Iowa	11/19/86	yes	Unknown	20	18	29	31	42	52			59	73			82	95				
77	B	GS	Iowa	11/19/86	yes	Unknown	26	30	36	40	52	52			69	74			91	94				
78	B	GS	Iowa	11/19/86	no	Unknown	26	28	36	38	52	50			69	69			91	92				
79	A	GS	Iowa	11/19/86	yes	Unknown	25	22	32	30	42	41			54	61			75	85				
80	A	GS	Iowa	11/19/86	no	Unknown	25	22	32	30	42	41			54	61			75	85				
81	S. 731	ARS	Oxford	12/15/76	yes	Unknown	48	55	58.4	66	73.3	81		85	90.3			96	98	98		99	99.9	
82	S. 732	ARS	Oxford	12/15/76	yes	Unknown	43.3	48	51.3	58	61.3	69		73	73.2			83	83.2	92		98	99.9	
83	S. 733	ARS	Oxford	12/15/76	yes	Unknown	25.7	33	30.6	38	37	46		49	49.1			61	67.1	78		88	99.9	
84	S. 734	ARS	Oxford	12/15/76	yes	Unknown	60.2	65	71.5	78	86.2	90		93	97.4			97	99	98		99.9	99.9	
85	S. 735	ARS	Oxford	12/15/76	yes	Unknown	71.6	76	81.2	85	90.3	93		94	97.2			97.5	99.8	99		99.9	99.9	
86	S. 736	ARS	Oxford	12/15/76	yes	Unknown	47.2	54	57.2	65	71.7	80		85	89.5			97.5	98.1	99.5		99.9	99.9	
87	S. 737	ARS	Oxford	12/15/76	yes	Unknown	23.4	29	27.7	33.5	34.7	43		46	48.2			64	69.4	85		98.5	99.9	
88	S. 738	ARS	Oxford	12/15/76	yes	Unknown	29.1	36	34.1	41	41.4	50		54	56.6			73	80.2	91		98	99.9	98
89	S. 739	ARS	Oxford	12/15/76	yes	Unknown	20.8	26	23.2	30	28.6	36		40	43.3			64.5	74.8	89		98	99.9	99
90	S. 740	ARS	Oxford	12/15/76	yes	Unknown	25.2	29	29.4	36	38.7	48		53	58.2			79	87.6	95		99.5	99.9	99.9
91	S. 741	ARS	Oxford	12/15/76	yes	Unknown	12.5	18	15.2	20	21.3	30		35	39.3			67	81	94		99	99.9	
92	S. 742	ARS	Oxford	12/15/76	yes	Unknown	17.2	21	21.7	27	29.6	38		43	50.4			74	85.5	93		99	99.9	
93	S. 744	ARS	Oxford	12/15/76	yes	Unknown	20	24	21	27.5	27	36		40	46			70	81	92		99	99.9	99.9
94	BM 8501 Emmett	GS	Iowa	11/12/86	yes	Out, Fremont L	1.4	3.6	1.7	4.4		5.6			3.2	7					7.9			
95	BM 8502 Emmett	GS	Iowa	11/12/86	yes	Out, Fremont L	0.6	2.3	0.8	2.6		2.9			1.2	3.1					3.3			
96	BM 8505 Emmett	GS	Iowa	8/13/84	yes	Fremont L 30M S5	49.2	55.3	63.9	70.1	74.3	77.4			76.2	79.9			79.7	81.5				
97	BM 8508 Emmett	GS	Iowa	11/14/84	yes		21.5	24.7	28.1	32.9	37.7	42.4			50	53.1			59.4	68.7				
98	BM 8511 Emmett	GS	Iowa	11/14/84	yes	Fremont L 75M S11	28.1	30.7	33.1	38.7		42.8			46.1	54.3					77.8			
99	BM 8514 Emmett	GS	Iowa	11/14/84	yes	Fremont L 105M S14	42.9	46.7	51.7	60	69.9	73.8			79.3	84.1			92.1	94.4				
100	BM 8517 Emmett	GS	Iowa	8/13/84	yes	Fremont L 135M S17	41.4	48.3	52.1	59.6		69.8			75.9	81.1					91.3			
101	BM 8520 Emmett	GS	Iowa	10/15/84	yes	Fremont L 194M S20		49.6	45.8	61.3	56.8	70.5			72.2	78.8					92.8			
102	BM 8523 Emmett	GS	Iowa	8/15/84	yes	Fremont L 105M S23	47.9	50.8	57.9	61.2	73	74			81.5	84.9			91.5	95.7				
103	BM 8526 Emmett	GS	Iowa	10/13/84	yes	Fremont L	22.3	24.8	24.8	30.3	32.4	39			41.9	51.3			58.4	69				
104	BM 8529 Emmett	GS	Iowa	10/15/84	yes	Fremont L 90M S29	34.9	32.4	40.1	41.6	46.7	52.7			59.9	67.6			76.9	84.7				
105	BM 8532 Emmett	GS	Iowa	10/15/84	yes	Fremont L 60M S32	17.5	21.9	25.8	33.6	45.6	52.5			75.9	77.8			91.7	93.4				
106	BM 8535 Emmett	GS	Iowa	8/14/84	yes	Fremont L 45M S35	16.6	15.1	20.8	20.9	26.7	29.1			36.1	42.1			51.2	61.1				
107	BM 8538 Emmett	GS	Iowa	10/15/84	yes	Fremont L 15M S38	9.1	10.9	11.3	14.6		18.2			19.1	26.1					32.8			
108	BM 8541 Emmett	GS	Iowa	8/14/84	yes	Fremont L 5M Inlet	4.4	5.6	5.5	6.8		8.3			8.7	10.8					14.3			
109	BM 8542 Emmett	GS	Iowa	8/8/84	yes	Fremont L 42M S42	53	57.3	61.2	69	72.7	80.6			83.5	88.4			93.3	94.7				
110	BM 8543 Emmett	GS	Iowa	8/18/84	yes	Fremont L 60M S43	32.8	39.3	41.1	48.9	53.6	61.1			68.7	73.6			77.8	82				
111	1 of 8 PB 1007	GS	Iowa	1/1/79	yes, add inc	Unknown		62		72.5		88.8				96.5					98			
112	2 of 8 PB 1007	GS	Iowa	1/1/79	yes, add inc	Unknown		62.5		75.5		91.5				98					99.9			
113	3 of 8 PB 1007	GS	Iowa	1/1/79	yes, add inc	Unknown		62.5		75		91.5				98					99.5			

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114	4 of 8 PB 1007	GS	Iowa	1/1/79	yes, add inc	Unknown		61		76		91				98				99				
115	5 of 8 PB 1007	GS	Iowa	1/1/79	yes, add inc	Unknown		61		75.5		90.5				97.5				99				
116	6 of 8 PB 1007	GS	Iowa	1/1/79	yes, add inc	Unknown		62.5		77.5		92.5				99				99.9				
117	7 of 8 PB 1007	GS	Iowa	1/1/79	yes, add inc	Unknown		62		77		91.5				98.5				99				
118	8 of 8 PB 1007	GS	Iowa	1/1/79	yes, add inc	Unknown		61.5		77		92				99				99.9				
119	1 of 8 FQ 1605	GS	Iowa	1/1/79	yes, add inc	FQ 1605 MFG		79		86.5		90				91.5				96				
120	1 of 8 FQ 1605	GS	Iowa	1/1/79	yes, add inc	FQ 1605 MFG		77.5		86		90				92				96				
121	1 of 8 FQ 1605	GS	Iowa	1/1/79	yes, add inc	FQ 1605 MFG		77		86		89.5				92				96				
122	1 of 8 FQ 1605	GS	Iowa	1/1/79	yes, add inc	FQ 1605 MFG		76		87		90.5				92				96.5				
123	1 of 8 FQ 1605	GS	Iowa	1/1/79	yes, add inc	FQ 1605 MFG		77		87		91				92				96				
124	1 of 8 FQ 1605	GS	Iowa	1/1/79	yes, add inc	FQ 1605 MFG		77		87		91				93				97				
125	1 of 8 FQ 1605	GS	Iowa	1/1/79	yes, add inc	FQ 1605 MFG		78		86		90				92				94				
126	1 of 8 FQ 1605	GS	Iowa	1/1/79	yes, add inc	FQ 1605 MFG		80		87		90				92				95				
127	1 SM 126A-B Com	GS	Iowa	3/5/84	yes	Unknown		29.5		35		43.5				60				84				
128	1 SM 126A-B Com	GS	Iowa	3/5/84	yes	Unknown		28		34		44				61				85				
129	2 SM 1226A-B Co	GS	Iowa	3/5/84	yes	Unknown		29.5		34		43				59				84				
130	2 SM 1226A-B Co	GS	Iowa	3/5/84	yes	Unknown		28		34		44				61				85				
131	3 SM 1226A-B Co	GS	Iowa	3/5/84	yes	Unknown		29.5		35		44				60				84.5				
132	3 SM 1226A-B Co	GS	Iowa	3/5/84	yes	Unknown		28.5		35		45				62				85.5				
133	4 SM 1226A-B Co	GS	Iowa	3/5/84	yes	Unknown		29.5		35		43.5				60				85.5				
134	4 SM 1226A-B Co	GS	Iowa	3/5/84	yes	Unknown		28		35		45				62				85.5				
135	5 SM 1226A-B Co	GS	Iowa	3/5/84	yes	Unknown		29.5		35		43.5				60				85				
136	5 SM 1226A-B Co	GS	Iowa	3/5/84	yes	Unknown		29		35		45				62				86				
137	6 SM 1226A-B Co	GS	Iowa	3/5/84	yes	Unknown		29.5		35		43.5				60				85				
138	6 SM 1226A-B Co	GS	Iowa	3/5/84	yes	Unknown		28.5		34.5		45				63				86				
139	KC 1120 Cell B	GS	Iowa	3/5/84	yes	Unknown		20		28		38				51				79				
140	KC 1120 Cell B	GS	Iowa	3/5/84	yes	Unknown		20		28		37.5				50				78.5				
141	KC 1120 Cell B	GS	Iowa	3/5/84	yes	Unknown		19		27		37				50				78.5				
142	KC 1120 Cell A	GS	Iowa	3/5/84	yes	Unknown		18.5		26		36				47				77				
143	KC 1120 Cell A	GS	Iowa	3/5/84	yes	Unknown		18		26		37.5				48				76.5				
144	KC 1120 Cell A	GS	Iowa	3/5/84	yes	Unknown		18		26		36				49				77				
145	PB 1025 Cell B	GS	Iowa	3/5/84	yes	Unknown		65		78		92				98				99				
146	PB 1025 Cell B	GS	Iowa	3/5/84	yes	Unknown		66		79		93				98				99				
147	PB 1025 Cell B	GS	Iowa	3/5/84	yes	Unknown		65		77		92				98				99				
148	PB 1025 Cell A	GS	Iowa	3/5/84	yes	Unknown		63.5		77		91.5				98				99				
149	PB 1025 Cell A	GS	Iowa	3/5/84	yes	Unknown		63.5		77		91.5				99				99.9				
150	None	GS	Iowa	11/13/86	yes	Rio Grande, El Paso	40.6	34.2	49.5	46.9	54.3	60.1			66.2	74.9			80.5	89.1				
151	None	GS	Iowa	1/9/87	yes	Rio Grande, Ft Quin.	79.3	76.9	84.4	86.8	87.6	90.4			89.7	92.4			92.6	96.3				

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152	None	GS	Iowa	11/13/86	yes	Krato Cr Willow A.	18.2	19.2	23.1	27	27.2	37.1			38.3	49.3			61.6	77.1				
153	None	GS	Iowa	11/13/86	yes	Los Padres Carmel	13.8	13.3	23.4	23.6	35.1	41.6			58.8	68.1			87.8	93.2				
154	None	GS	Iowa	11/13/86	yes	Rio Puerco, Bern.	69.4	62.6	81.8	76.3	88.5	91.5			97.1	98			99.6	99.2				
155	None	GS	Iowa	11/13/86	yes	Ralston Cr, Iowa C.	17.1	21.4	19.2	24	22	28			27.8	39			66.8	78.8				
156	None	GS	Iowa	1/9/87	yes	Rio Grande, San AC	67.4	65.3	82.5	82.3	93.3	95.8			98.3	98.6			99.4	99.8				
157	None	GS	Iowa	11/13/86	yes	Tanana R Moose C	28.1	25.4	36.9	34.6	44	46.9			54.1	61.5			65.3	71.5				
158	None	GS	Iowa	11/13/86	yes	Tanana R S2?	29.2	28.8	34.1	34.7	37	44.1			50	60.8			74.9	85.2				
159	SRS Sed 3, B	GS	Denver	11/5/86	yes	Unknown	16.4	21.3	17.3	22.5	18.2	23.9			19.6	25			21	27.2				
160	SRS Sed 3, A	GS	Denver	11/5/86	yes	Unknown	16.4	19.6	17.3	21	18.2	22.3			19.6	23.6			21	25.7				
161	Weaver Grob	TEXT	?	?	yes	Unknown Var Conc		65		80		95		98				99.9		99.9				
162	Weaver Grob	TEXT	?	?	yes	Unknown Var Conc		61		77		93		96				98		99				
163	Weaver Grob	TEXT	?	?	yes	Unknown Var Conc		61		77		93		95				97		98				
164	Weaver Grob	TEXT	?	?	yes	Glass Beads						10		18				92		99.9				
165	Weaver Grob	TEXT	?	?	yes	Glass Beads						7		11				91		99.9				
166	Weaver Grob	TEXT	?	?	yes	Natural Silt Clay		46		58		68		71				80		85		92		
167	Weaver Grob	TEXT	?	?	yes	Natural Silt Clay		48		60		68		71				80		84		91		
168	Weaver Grob	TEXT	?	?	yes	Natural Silt Clay		43		57		67		69				78		81		89		
169	Weaver Grob	TEXT	?	?	yes	Natural Clay		87		92		99.9		99.9				99.9		99.9		99.9		
170	Weaver Grob	TEXT	?	?	yes	Natural Clay		86		91		99.9		99.9				99.9		99.9		99.9		
171	Weaver Grob	TEXT	?	?	yes	Made up Clay		97		99.9		99.9		99.9				99.9		99.9		99.9		
172	Weaver Grob	TEXT	?	?	yes	Made up Clay		96		99.9		99.9		99.9				99.9		99.9		99.9		
173	Weaver Grob	TEXT	?	?	yes	Unknown, Var Cell		56		65		75		79				87		92		98		
174	Weaver Grob	TEXT	?	?	yes	Unknown		53		62		73		78				86		92		98		
175	Weaver Grob	TEXT	?	?	yes	Natural Clay		94		99.9		99.9		99.9				99.9						
176	Weaver Grob	TEXT	?	?	yes	Natural Clay		86		94		98		99										
177	Welch	ARS	Oxford	1979	yes	Unknown, Var Cell		25		28		35				50			74		90			
178	Welch	ARS	Oxford	1979	yes	Unknown, Var Cell		22		22		36				44					84			
179	Welch	ARS	Oxford	1979	yes	Unknown, Var Cell	20	15	21	20	28	36			42	48					80			
180	Welch	ARS	Oxford	1979	yes	Unknown, Var C R2		22		26		35				52			74		91			
181	Welch	ARS	Oxford	1979	yes	Unknown, Var C R2		27		29		39				60					95			
182	Welch	ARS	Oxford	1979	yes	Unknown, Var C R2	20	30	21	35	28	47			42	74					99.9			
183	Schiebe	ARS	Oxford	?	yes	Core, Three Mile L	86	93	92	97	97													
184	Schiebe	ARS	Oxford	?	yes	Core, Three Mile L	87	92	93	96.5	97	98.5		10										
185	Schiebe NO 3	ARS	Oxford	?	yes	R Sta. Three Mile L	77	91	84	94	93	97		98										
186	Schiebe NO 4	ARS	Oxford	?	yes	20 In. Three Mile L	80	90	87	94	94	97		98										
187	Schiebe NO 5	ARS	Oxford	?	yes	28 In. Three Mile L	82	90	87	94	95	97		98										
188	Schiebe NO 6	ARS	Oxford	?	yes	11A, Wasp Lake	80	92	85	96	95	98.7		99										
189	Schiebe NO 7	ARS	Oxford	?	yes	11A, Wasp Lake	83	92	90	97	97	98.7												

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190	Schiebe NO 8	ARS	Oxford	?	yes	11A, Wasp Lake	79	91	87	96	97	98.5												
191	Schiebe NO 9	ARS	Oxford	?	yes	11B, Wasp Lake	71	91	81	94	93	98												
192	Schiebe NO 10	ARS	Oxford	?	yes	36 In. Wasp Lake	65	80	75	88	91	95												
193	Schiebe NO 11	ARS	Oxford	?	yes	7A Macon Lake	60	82	68	85	80	91												
194	Schiebe NO 12	ARS	Oxford	?	yes	6A Mossy Lake	78	89	88	96	97.5	98.5		99										
195	Schiebe NO 13	ARS	Oxford	?	yes	6A Mossy Lake	75	86	88	93	97.5	97		98										
196	Schiebe NO 14	ARS	Oxford	?	yes	4 In. Mossy Lake	77	88	87	93	95	97.5		98										
197	Schiebe NO 15	ARS	Oxford	?	yes	16 In. Wolf Lake	77	88	83	91	90	94		95										
198	BKR 23 S61	ARS	Chic, OK	12/16/76	yes	Unknown		33		42		53		56				68		83		99		
199	BKR 23 S61	ARS	Chic, OK	12/16/76	yes	Unknown		33		42		51		54				65		82		98		
200	BKR 23 S61	ARS	Chic, OK	12/16/76	yes	Unknown		30		42		50.5		53				64		81		97		
201	BKR 23 S61	ARS	Chic, OK	12/16/76	yes	Unknown		36		44		53		57				70		86		93		
202	BKR 23 S61	ARS	Chic, OK	12/16/76	yes	Unknown		37		45		54		56				68		85		99		
203	340-345	GS	CVO	6/24/90	yes	Blackberry Cr	21.2	21.2	34.7	34.7	39.7	47.4			60.3	68.1			85.5	91.6				
204	343-348	GS	CVO	6/21/90	yes	Nippersink	24.8	10.4	25.5	18	36.7	38.6			65	70.4			89.6	91.8				
205	344-347	GS	CVO	3/14/91	yes	Langan Creek	22.2	12.4	24.3	18.9	29.9	31.4			45.6	55.8			75.8	93				
206	EP 1201 PIP Only	GS	R. Robin	?	yes	Rio Grande, El Paso	38		46		52				66				81					
207	EP 1214 PIP Only	GS	R. Robin	?	yes	Rio Grande, El Paso	40		48		50				61				81					
208	EP 1227 PIP Only	GS	R. Robin	?	yes	Rio Grande, El Paso	38		50		52				67				79					
209	EP 1230 PIP Only	GS	R. Robin	?	yes	Rio Grande, El Paso	40		48		51				65				80					
210	EP 1302 PIP Only	GS	R. Robin	?	yes	Rio Grande, El Paso	42		50		56				66				81					
211	EP 1303 PIP Only	GS	R. Robin	?	yes	Rio Grande, El Paso	41		49		56				66				80					
212	EP 1307 PIP Only	GS	R. Robin	?	yes	Rio Grande, El Paso	39		50		55				65				78					
213	EP 1311 PIP Only	GS	R. Robin	?	yes	Rio Grande, El Paso	36		50		55				66				81					
214	EP 1313 PIP Only	GS	R. Robin	?	yes	Rio Grande, El Paso	41		50		53				66				80					
215	EP 1322 PIP Only	GS	R. Robin	?	yes	Rio Grande, El Paso	40		51		55				66				81					
216	EP 1324 PIP Only	GS	R. Robin	?	yes	Rio Grande, El Paso	40		50		53				66				79					
217	EP 1325 PIP Only	GS	R. Robin	?	yes	Rio Grande, El Paso	39		53		58				69				81					
218	EP 1328 PIP Only	GS	R. Robin	?	yes	Rio Grande, El Paso	37		45		56				67				81					
219	EP 1333 PIP Only	GS	R. Robin	?	yes	Rio Grande, El Paso	37		50		55				67				80					
220	EP 1335 PIP Only	GS	R. Robin	?	yes	Rio Grande, El Paso	42		45		53				66				80					
221	EP 1336 PIP Only	GS	R. Robin	?	yes	Rio Grande, El Paso	39		50		52				67				81					
222	EP 13012 PIP Only	GS	R. Robin	?	yes	Rio Grande, El Paso	44		54		59.9				68				83					
223	EP 13030 PIP Only	GS	R. Robin	?	yes	Rio Grande, El Paso	48.2		51.9		56.4				68				82					
224	FQ 1501 PIP Only	GS	R. Robin	?	yes	Rio Grande, Ft Quin.	82		86		88				90				93					
225	FQ 1514 PIP Only	GS	R. Robin	?	yes	Rio Grande, Ft Quin.	80		86		87				89				91					
226	FQ 1528 PIP Only	GS	R. Robin	?	yes	Rio Grande, Ft Quin.	79		85		87				89				93					
227	FQ 1541 PIP Only	GS	R. Robin	?	yes	Rio Grande, Ft Quin.	81		86		88				89				93					

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228	FQ 1601 PIP Only	GS	R. Robin	?	yes	Rio Grande, Ft Quin.	84		87		90				91				95					
229	FQ 1613 PIP Only	GS	R. Robin	?	yes	Rio Grande, Ft Quin.	83		86		89				90				93					
230	FQ 1627 PIP Only	GS	R. Robin	?	yes	Rio Grande, Ft Quin.	80		84		86				90				94					
231	FQ 1639 PIP Only	GS	R. Robin	?	yes	Rio Grande, Ft Quin.	80		85		88				89				91					
232	FQ 1616 PIP Only	GS	R. Robin	?	yes	Rio Grande, Ft Quin.			86		89				91				92					
233	FQ 1616 PIP Only	GS	R. Robin	?	yes	Rio Grande, Ft Quin.			84		88				89				92					
234	FQ 1605 PIP Only	GS	R. Robin	?	yes	Rio Grande, Ft Quin.	83.8		86.6		87				90				93					
235	FQ 1620 PIP Only	GS	R. Robin	?	yes	Rio Grande, Ft Quin.			86		89				91				92					
236	FQ 1637 PIP Only	GS	R. Robin	?	yes	Rio Grande, Ft Quin.	85.4		87.7		88.2				90.2				92.8					
237	FQ 1634 PIP Only	GS	R. Robin	?	yes	Rio Grande, Ft Quin.	88.4		86.2		88.7				90.1				92.8					
238	FQ 1623 PIP Only	GS	R. Robin	?	yes	Rio Grande, Ft Quin.	83.4		87		87.9				90.5				92.5					
239	FQ 1631 PIP Only	GS	R. Robin	?	yes	Rio Grande, Ft Quin.	84.6		86.2		89				90.8				92.9					
240	FQ 1610 PIP Only	GS	R. Robin	?	yes	Rio Grande, Ft Quin.	81		85		86				91				92					
241	FQ 1612 PIP Only	GS	R. Robin	?	yes	Rio Grande, Ft Quin.	79		84		86				91				92					
242	FQ 1609 PIP Only	GS	R. Robin	?	yes	Rio Grande, Ft Quin.	76		81		87				88									
243	FQ 1635 PIP Only	GS	R. Robin	?	yes	Rio Grande, Ft Quin.	77		83		87				89				92					
244	FQ 1508 PIP Only	GS	R. Robin	?	yes	Rio Grande, Ft Quin.	80		85						90									
245	FQ 1624 PIP Only	GS	R. Robin	?	yes	Rio Grande, Ft Quin.	76		86						88									
246	FQ 1617 PIP Only	GS	R. Robin	?	yes	Rio Grande, Ft Quin.	75		83		87				89				96					
247	FQ 1602 PIP Only	GS	R. Robin	?	yes	Rio Grande, Ft Quin.	78		85		89				91				94					
248	FQ 1614 PIP Only	GS	R. Robin	?	yes	Rio Grande, Ft Quin.	59		61		77				82				85					
249	FQ 1611 PIP Only	GS	R. Robin	?	yes	Rio Grande, Ft Quin.	72		84		88				90				92					
250	FQ 1615 PIP Only	GS	R. Robin	?	yes	Rio Grande, Ft Quin.	85		87						90									
251	FQ 1604 PIP Only	GS	R. Robin	?	yes	Rio Grande, Ft Quin.	77		84		89				91				95					
252	KC 1101 PIP Only	GS	R. Robin	?	yes	Kroto Cr Willow A.	16		21		23				36				60					
253	KC 1111 PIP Only	GS	R. Robin	?	yes	Kroto Cr Willow A.	15		22		24				35				61					
254	KC 1122 PIP Only	GS	R. Robin	?	yes	Kroto Cr Willow A.	16		21		24				36				59					
255	KC 1132 PIP Only	GS	R. Robin	?	yes	Kroto Cr Willow A.	16		21		25				38				61					
256	KC 1201 PIP Only	GS	R. Robin	?	yes	Kroto Cr Willow A.	15		21		24				35				62					
257	KC 1211 PIP Only	GS	R. Robin	?	yes	Kroto Cr Willow A.	17		23		25				36				62					
258	KC 1222 PIP Only	GS	R. Robin	?	yes	Kroto Cr Willow A.	18		21		27				38				61					
259	KC 1232 PIP Only	GS	R. Robin	?	yes	Kroto Cr Willow A.	17		22		25				39				63					
260	KC 1223 PIP Only	GS	R. Robin	?	yes	Kroto Cr Willow A.			20		29				39				58					
261	KC 1205 PIP Only	GS	R. Robin	?	yes	Kroto Cr Willow A.			23		29				40				61					
262	KC 1205 PIP Only	GS	R. Robin	?	yes	Kroto Cr Willow A.			21		29				40				61					
263	KC 1127 PIP Only	GS	R. Robin	?	yes	Kroto Cr Willow A.	23.7		28.8		32.4				42.8				64.6					
264	KC 1118 PIP Only	GS	R. Robin	?	yes	Kroto Cr Willow A.	22.8		28.9		31.7				40.1				64					
265	KC 1120 PIP Only	GS	R. Robin	?	yes	Kroto Cr Willow A.	24		29.4		32.5				41.4				64.8					

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266	LP 101 PIP Only	GS	R. Robin	?	yes	Carmel V Los. Pod.	13		19		31				57				86					
267	LP 101 PIP Only	GS	R. Robin	?	yes	Carmel V Los. Pod.	14		21		33				58				85					
268	LP 101 PIP Only	GS	R. Robin	?	yes	Carmel V Los. Pod.	16		22		34				59				87					
269	LP 101 PIP Only	GS	R. Robin	?	yes	Carmel V Los. Pod.	14		20		32				60				95					
270	LP 101 PIP Only	GS	R. Robin	?	yes	Carmel V Los. Pod.	15		19		29				57				86					
271	LP 101 PIP Only	GS	R. Robin	?	yes	Carmel V Los. Pod.	14		19		30				57				88					
272	LP 101 PIP Only	GS	R. Robin	?	yes	Carmel V Los. Pod.	14		20		31				57				87					
273	LP 101 PIP Only	GS	R. Robin	?	yes	Carmel V Los. Pod.	10		18		34				57				86					
274	LP 101 PIP Only	GS	R. Robin	?	yes	Carmel V Los. Pod.	31.7		39.1		47.5				63.7				89					
275	LP 101 PIP Only	GS	R. Robin	?	yes	Carmel V Los. Pod.	30.1		38.3		45.8				62.5				88.5					
276	PB 9002 PIP Only	GS	R. Robin	?	yes	Rio Puerco, Bern.	69		82		88				97				99					
277	PB 9003 PIP Only	GS	R. Robin	?	yes	Rio Puerco, Bern.	73		82		88				96				99.9					
278	PB 9005 PIP Only	GS	R. Robin	?	yes	Rio Puerco, Bern.	69		83		90				97				99.9					
279	PB 9012 PIP Only	GS	R. Robin	?	yes	Rio Puerco, Bern.	62		80		88				97				99.9					
280	PB 9017 PIP Only	GS	R. Robin	?	yes	Rio Puerco, Bern.	68		82		89				98				99.9					
281	PB 9021 PIP Only	GS	R. Robin	?	yes	Rio Puerco, Bern.	62		83		88				98				99					
282	PB 9022 PIP Only		R. Robin	?	yes	Rio Puerco, Bern.	65		84		89				98				99.9					
283	PB 9025 PIP Only		R. Robin	?	yes	Rio Puerco, Bern.	70		81		88				97				99.9					
284	PB 9030 PIP Only		R. Robin	?	yes	Rio Puerco, Bern.	69		81		89				97				99					
285	PB 9031 PIP Only		R. Robin	?	yes	Rio Puerco, Bern.	70		81		83				81				97					
286	PB 9034 PIP Only		R. Robin	?	yes	Rio Puerco, Bern.	70		85		90				97				99.9					
287	PB 9035 PIP Only		R. Robin	?	yes	Rio Puerco, Bern.	66		84		91				98				99.9					
288	PB 1001 PIP Only		R. Robin	?	yes	Rio Puerco, Bern.	61		74		85				96				99					
289	PB 1011 PIP Only		R. Robin	?	yes	Rio Puerco, Bern.	68		79		89				97				99.9					
290	PB 1022 PIP Only		R. Robin	?	yes	Rio Puerco, Bern.	69		79		87				97				99.9					
291	PB 1032 PIP Only		R. Robin	?	yes	Rio Puerco, Bern.	69		80		88				97				99.9					
292	PB 9033 PIP Only		R. Robin	?	yes	Rio Puerco, Bern.	74.1		86.1		90.8				97.3				98.4					
293	PB 9027 PIP Only		R. Robin	?	yes	Rio Puerco, Bern.	80.7		81.7		89.9				97.3				98.3					
294	PB 1025 PIP Only		R. Robin	?	yes	Rio Puerco, Bern.	80.1		82.1		91.7				97.2				99.9					
295	PB 1007 PIP Only		R. Robin	?	yes	Rio Puerco, Bern.	74		83.6		90.4				96.7				97.6					
296	RC 0101 PIP Only		R. Robin	?	yes	Ralston Cr, Iowa C.	15		17		19				27				65					
297	RC 0101 PIP Only		R. Robin	?	yes	Ralston Cr, Iowa C.	17		19		22				26				67					
298	RC 0101 PIP Only		R. Robin	?	yes	Ralston Cr, Iowa C.	16		19		22				27				67					
299	RC 0101 PIP Only		R. Robin	?	yes	Ralston Cr, Iowa C.	18		20		22				26				68					
300	RC 0101 PIP Only		R. Robin	?	yes	Ralston Cr, Iowa C.	17		19		21				28				67					
301	RC 0101 PIP Only		R. Robin	?	yes	Ralston Cr, Iowa C.	17		19		21				26				68					
302	RC 0101 PIP Only		R. Robin	?	yes	Ralston Cr, Iowa C.	17		19		24				29				68					
303	RC 0101 PIP Only		R. Robin	?	yes	Ralston Cr, Iowa C.	15		17		21				27				66					

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304	RC 0101 PIP Only		R. Robin	?	yes	Ralston Cr, Iowa C.	19.4		21.3		23.3				29.8				66.1					
305	RC 0101 PIP Only		R. Robin	?	yes	Ralston Cr, Iowa C.	19.8		22.2		24.4				31.8				66.2					
306	SA 1101 PIP Only		R. Robin	?	yes	Rio Grande, S. ACA	68		81		92				99				99.9					
307	SA 1114 PIP Only		R. Robin	?	yes	Rio Grande, S. ACA	65		82		94				99				99.9					
308	SA 1126 PIP Only		R. Robin	?	yes	Rio Grande, S. ACA	62		82		92				97				99.9					
309	SA 1201 PIP Only		R. Robin	?	yes	Rio Grande, S. ACA	65		80		92				98				99.9					
310	SA 1214 PIP Only		R. Robin	?	yes	Rio Grande, S. ACA	65		80		93				99				99.9					
311	SA 1227 PIP Only		R. Robin	?	yes	Rio Grande, S. ACA	65		80		93				99				99.9					
312	SA 1239 PIP Only		R. Robin	?	yes	Rio Grande, S. ACA	66		81		92				99				99					
313	SA 1105 PIP Only		R. Robin	?	yes	Rio Grande, S. ACA	70.2		86		94.6				98.2				99.3					
314	SA 1125 PIP Only		R. Robin	?	yes	Rio Grande, S. ACA	71.4		84.8		95.7				97.8				99.5					
315	SA 1113 PIP Only		R. Robin	?	yes	Rio Grande, S. ACA	70.2		83.4		94				98.1				98.9					
316	SA 1106 PIP Only		R. Robin	?	yes	Rio Grande, S. ACA	73.3		87.2		93.7				96.8				97.8					
317	732	EPA-ARS	Chic, OK	4/12/77	yes	Unknown	43	46	51	55	62	67			74	78			82.5	90			99	99.9
318	734	EPA-ARS	Chic, OK	4/12/77	yes	Unknown	61	65	72	79	85	91			97	97.5			98	98.5			99.9	99.9
319	735	EPA-ARS	Chic, OK	4/12/77	yes	Unknown	72	77	81	85	90	94			96	97			99	99.9			99	99.9
320	737	EPA-ARS	Chic, OK	4/12/77	yes	Unknown	23	28	27	34	35	43			48	57			69	85			99.9	99.9
321	742	EPA-ARS	Chic, OK	4/12/77	yes	Unknown	17	20	20	25	29	35			50	58			85	90			99.9	99.9
322	BKR 23 #61	ARS	Chic, OK	12/16/76	yes	Unknown		33		42		53		56				68		83		99		
323	BKR 23 #61	ARS	Chic, OK	12/17/76	yes	Unknown		33		42		51		54				65		82		98		
324	BKR 23 #61	ARS	Chic, OK	12/20/76	yes	Unknown		30		42		51		53				64		81		97		
325	BKR 23 #61	ARS	Chic, OK	12/21/76	yes	Unknown		36		44		53		57				70		86		98		
326	BKR 23 #61	ARS	Chic, OK	12/23/76	yes	Unknown		37		45		54		56				68		85		99		