Comparison of sediment grain size analysis among two methods and three instruments using environmental samples

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ABSTRACT

Sediment grain size is measured using a variety of methods, but comparisons of measurement methods on environmental samples are limited. Three instruments (Coulter LS230, Horiba LA900, and SediGraph 5100) utilizing two fundamentally different operating principles were employed to measure a single set of 20 different sediment samples collected at shelf depths from the Southern California Bight. Distribution estimates were compared using the Kolmogorov-Smirnov test, mean ϕ with analysis of variance (ANOVA), and the percent fines using Kruskall-Wallis tests. These instruments produced comparable results on all three types of grain size measurements. Within-instrument measurement variability was compared among instruments. The Coulter LS230 had the smallest measurement variance, the Horiba LA900 the next smallest variance, and the SediGraph 5100 the largest measurement variance. These results reflect improvements in technology over time.

INTRODUCTION

Measurements of sediment grain size are commonly employed in ecological investigations of the marine environment. Chemists use sediment grain size as a normalizing factor in determining the concentration of contaminants in sediments (Maurer *et al.* 1996, Schiff and Weisberg 1997, Schiff and Gossett 1998). Biologists recognize that benthic organisms partition their chosen habitat, in part, based upon sediment grain size, making this parameter important for assessing benthic communities (Bergen *et al.* 1998, Dorsey *et al.* 1995, Wu and Shin 1997, and Zmarzly *et al.* 1994). Similarly, microbiologists find that bacterial concentrations correlate to sediment grain size (Irvine and Pettibone 1993).

Several methods are currently used for measuring sediment grain size. Sediment grain size measurement methods for particles smaller than 63 μ are primarily based upon one of two principles regarding particle properties: Stokes' Law, the differential fall rate velocity of particles through a fluid (Guy 1969); or light-scattering diffraction. The sieving measurement method is used for particles larger than 63 μ. Historically, the most commonly used methods for sediment grain size analysis utilized a combination of mechanical methods (Snelgrove and Butman 1994), such as sieving for the coarse fraction (>63 μ), with measurement of fall velocity, by pipette or hydrometer analysis, for the fine fraction (<63 μ) (Galehouse 1971, Plumb 1981). Analytical instruments capable of rapidly producing more precise grain size measurements have replaced these mechanical methods (Syvitski *et al.* 1991b). The SediGraph 5100, which utilizes the principle of Stokes' Law, employs a collimated x-ray beam to measure particle concentration by capturing the fall rate electronically (Coakley and Syvitski 1991). As particles fall, changes in radiation pulses are converted into voltage and then compared to a background value (voltage from a pure suspending liquid) in order to produce a net signal that is proportional to the concentration of particles. Instruments utilizing light-scattering technology directly measure particle size utilizing the relationship among particle size, wavelength, and scattered light that is transmitted through an optical system to a series of silicon detectors (Xu 1996). By measuring a large number of particles, the instruments provide a particle size distribution of equivalent diameter calculated from the diffraction data of each light source or channel (Xu 1996; Marc Reyes, Coulter Instruments, oral communication).

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Critical evaluations of various grain size measurement methods (Singer *et al.* 1988, Merkus *et al.* 1995, Michoel *et al.* 1994, and Syvitski *et al.* 1991b) have focused on precision and accuracy relative to standards that are uniform and within narrowly selected size ranges. Syvitski *et al.* (1991b) found that a variety of analytical instruments were precise, but cautioned that laboratories should maintain records of their instruments' precision and accuracy. Merkus *et al.* (1995) cautioned that, in addition to methodological or instrument-related differences, analyst-induced bias could occur when multiple laboratories are used. These laboratory comparisons of standards cannot address the more complex question of suitability for field samples. The comparison of environmental samples is a critical factor in determining an instrument's accuracy since they contain a diverse array of grain size distributions.

In this study, we investigate comparability and variability of grain size distribution measurements from three instruments based upon two different principles, using a set of samples collected from the southern California coastal shelf. The goal of this comparison is to ascertain whether data obtained from different instruments can be combined, or compared across data sets.

METHODS

Three instruments were used in this study. The SediGraph method combines the SediGraph 5100 and settling tubes to measure a larger range of particle sizes. The SediGraph 5100 is used to measure the finer fraction (0.1-125 μ) while 10-ft settling tubes are used to measure the larger fraction (125-2000 μ). The Horiba LA900 and Coulter LS230 are both light-scattering instruments. The Horiba LA900 is equipped with 74 detectors and has the ability to measure a size range of 0.04-1019 μ. The Coulter LS230 incorporates both laser diffraction and polarization intensity differential scattering (PIDS for the sub micron, 0.04-0.8 μ, range), has 116 detectors, and calculates the results using the Fraunhofer theory of light-scattering relationships to measure a 0.04-2000 μ size range. All samples were screened over a 1 mm^2 screen to remove the 1000-2000 μ fraction prior to analysis in order to insure comparability between the light-scattering instruments, which measure different size ranges.

Twenty sediment samples were collected at shelf depths from 10 to 200 m within the Southern California Bight. All samples were divided into triplicates and analyzed using two methods with each of the three light-scattering instruments. An exception was the SediGraph, for which duplicates were performed for 14 of the 20 samples and single analysis was performed for the remaining 6 samples due to low sample volume.

Data were grouped into half-phi intervals to accommodate the different numbers of size categories from the three instruments. Phi (ϕ) intervals were calculated from the Wentworth scale of fixed geometric intervals converted into logarithm-transformed integer ϕ-intervals (Guy 1969). This method yielded 24 size intervals (0.5 - 12 ϕ) for the analysis ranging from coarse sand (1000 μ) to colloids (0.24 - 0.04 μ) (Table 1). The midpoint of each half-ϕ interval was used for all calculations.

A series of four statistical analyses were performed. First, the difference between each pair of instruments was compared by performing Kolmogorov-Smirnov (K-S) tests on the distribution of the half-ϕ data from each station. Pairwise differences between instruments were computed for each half-ϕ-interval value. The maximum difference was used to determine statistical significance. An overall significance level of 0.05 and comparison-wise significance level of 0.017 were used, based upon Bonferroni's method, since three pairs of instruments were compared. Second, the mean ϕ value among instruments was compared for each sample using an analysis of variance (ANOVA). Commonly, scientists use the mean ϕ in conjunction with standard deviation or skewness for simple characterization of grain size. Third, non-parametric Kruskal-Wallis tests were performed on percent fines—the combined silt, clay, and colloid fractions (< 63 μ)—to test for differences in large interval-grouped data. A statistical pairwise comparison of percent fines could not be achieved due to the small number of replicates obtained with the SediGraph. Finally, variability within instruments (measurement variance) and among instruments was calculated on the half ϕ data using a nested ANOVA model. The split sample data were used to provide an estimate of measurement variance of the instruments by pooling, or averaging, the variance estimate for each instrument over stations and ϕ-groups. The among-instrument variability was pooled over stations and ϕ-groups.

RESULTS

The grain size distribution estimates from the three instruments were nearly indistinguishable (Figure 1). The average of the maximum differences between pairs of estimated distributions was 20%. When differences in grain size distributions occurred, the differences were generally not significant within the lower ϕ ranges (larger particle sizes). Results were significantly different in only two of the 20 samples (Stations 2146 and 2171, Table 2) and the estimates at the maximum difference were higher in the Horiba LA900 than the Coulter LS230 or the SediGraph. For both stations, the maximum difference between the

TABLE 1. Wentworth scale based size intervals used for grouping SediGraph, Horiba LA900, and Coulter LS2300 data. Number of channels and size range (in microns) of channels (=detectors) provided for each class interval from Horiba LA900 and Coulter LS230.

Class	Size (microns)	Phi	SediGraph	Horiba	Coulter
Very Coarse Sand	1414-2000	-1		not available	3 channels: 1512-1822*
	1000-1414	-0.5		not available	4 channels: 1041-1377*
Coarse Sand	707-1000	0	Settling tubes	3 channels: 777.1-1019.5	4 channels: 716.9-948.2
	500-707	0.5		3 channels: 517.2-678.5	3 channels: 541.9-653.0
Medium Sand	354-500	1		2 channels: 394.2-451.5	4 channels: 373.1-493.6
	250-354	1.5		3 channels: 262.3-344.2	4 channels: 256.8-339.8
Fine Sand	177-250	2		2 channels: 200.0-229.0	4 channels: 176.8-234.1
	125-177	2.5		3 channels: 133.1-174.6	3 channels: 133.7-176.8
Very Fine Sand	88-125	3		3 channels: 88.58-116.2	4 channels: 92.09-121.8
	62-88	3.5		2 channels: 67.52-77.33	4 channels: 63.41-83.90
Coarse Silt	44-62	4		3 channels: 44.93-58.95	3 channels: 47.93-57.77
	31-44	4.5		2 channels: 34.25-39.23	4 channels: 33.00-43.66
Medium Silt	22-31	5		3 channels: 22.79-29.90	4 channels: 22.73-30.07
	16-22	5.5		2 channels: 17.37-19.9	3 channels: 17.18-20.70
Fine Silt	11-16	6		3 channels: 11.56-15.17	4 channels: 11.83-15.65
	7.8-11	6.5		2 channels: 8.815-10.09	4 channels: 8.147-10.78
Very Fine Silt	5.5-7.8	7		3 channels: 5.866-7.696	4 channels: 5.611-7.421
	3.9-5.5	7.5		2 channels: 4.472-5.122	3 channels: 4.241-5.111
Coarse Clay	2.8-3.9	8	SediGraph 5100	3 channels: 2.976-3.904	4 channels: 2.920-3.862
	2.0-2.8	8.5		3 channels: 1.980-2.598	4 channels: 2.010-2.660
Medium Clay	1.4-2.0	9		2 channels: 1.509-1.729	4 channels: 1.385-1.832
	0.97-1.4	9.5		3 channels: 1.004-1.318	3 channels: 1.047-1.261
Fine Clay	0.69-0.97	10		2 channels: 0.765-0.877	3 channels: 0.721-0.953
	0.49-0.69	10.5		3 channels: 0.509-0.668	4 channels: 0.496-0.657
Very Fine Clay	0.34-0.49	11		2 channels: 0.388-0.445	4 channels: 0.342-0.452
	0.24-0.34	11.5		3 channels: 0.258-0.339	5 channels: 0.214-0.311

Horiba LA900 and the Coulter LS230 and the SediGraph was approximately 54 and 62%, respectively. This maximum difference for Station 2146 occurred at 2.0 ϕ, corresponding to fine sand (177-250 μ). For Station 2171, the maximum difference occurred at 1.5 ϕ, corresponding to medium sand (250-354 μ).

Comparisons of the two summary measures of mean ϕ and percent fines resulted in different conclusions. The estimated mean ϕ was equivalent across instruments for all samples (Figure 2). In contrast, estimates of percent fines differed significantly among instruments at half of the stations (Table 3). The SediGraph displayed most of the significant differences, but all instruments differed from the other two for some samples. The differences in SediGraph results were inconsistent; the percent fines were sometimes higher and sometimes lower than the other instruments. The Horiba LA900 results were generally lower than the results for the other two instruments, while the Coulter LS230 results were generally higher.

The measurement variability was significantly less for the Coulter LS230 than both the Horiba LA900 and the SediGraph (Table 4, f-test p-value < 0.001). The SediGraph results were the most variable. The measurement variance differed over the range of half-ϕ intervals for all instruments (Table 5). The variance was smaller for the smaller particle ranges (larger ϕ values). The SediGraph results exhibited maximum variance at a slightly smaller particle range than did the light-scattering instruments.

The measurement variance was smaller (by 4 to 300 times) than the among-instrument variance (Table 4). The variance of the results combined across instruments was not significantly larger than the measurement variance for either the SediGraph or the Horiba LA900, but was significantly larger than the measurement variance for the Coulter LS230.

DISCUSSION

In this experiment, we found that the three instruments, the SediGraph, Horiba LA900, and Coulter LS230, produced sediment grain size distributions and mean ϕ estimates on marine sediment samples that were remarkably similar. Consequently, data from these instruments, which are based upon two fundamentally different principles, can be used interchangeably. Small differences were found in only two samples and at large particle size ranges, where all instruments exhibited greater variability.

FIGURE 1. Cumulative percent of median grain size distributions for the Coulter LS230, Horiba LA900, and SediGraph 5100 for all stations.

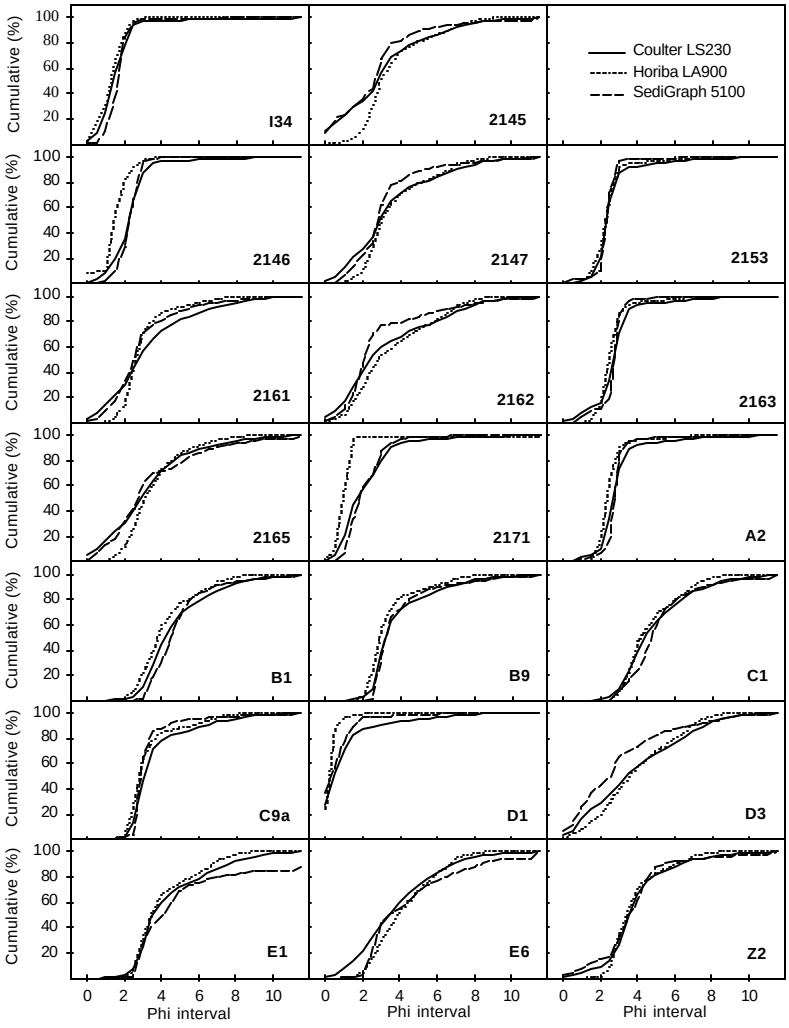

TABLE 2. Summary of Kolmogorov-Smirnov test results for three instrument pairings showing the maximum absolute difference in cumulative percent (d) and p-values. Boldface values indicate the difference between instruments is significant (p<-.017).

Station	Coulter - Horiba		Coulter - SediGraph		Horiba - SediGraph	
	d	p	d	p	d	p
I34	21	0.7000	19	0.8000	40	0.0500
2145	18	0.8000	9	0.9500	19	0.8500
2146	53	**0.0030**	9	0.9500	62	**0.0003**
2147	16	0.9000	10	0.9500	14	0.9500
2153	12	0.9500	11	0.9500	22	0.6000
2161	19	0.7500	13	0.9500	18	0.8500
2162	11	0.9500	18	0.8500	26	0.4000
2163	20	0.7500	15	0.9500	35	0.1000
2165	21	0.7000	8	0.9500	21	0.6500
2171	54	**0.0030**	13	0.9500	62	**0.0003**
A2	28	0.3000	12	0.9500	40	0.0400
B1	12	0.9500	17	0.9500	29	0.2500
B9	24	0.5000	10	0.9500	26	0.4000
C1	9	0.9500	12	0.9500	22	0.6500
C9a	19	0.8000	18	0.8500	24	0.5000
D1	31	0.2000	10	0.9500	25	0.5000
D3	6	0.9500	17	0.8500	14	0.9500
E1	7	0.9500	11	0.9500	7	0.9500
D6	18	0.8500	19	0.8000	15	0.9500
Z2	8	0.9500	7	0.9500	13	0.9500

FIGURE 2. Mean phi and simultaneous 95% confidence limits for the three instruments at each station.

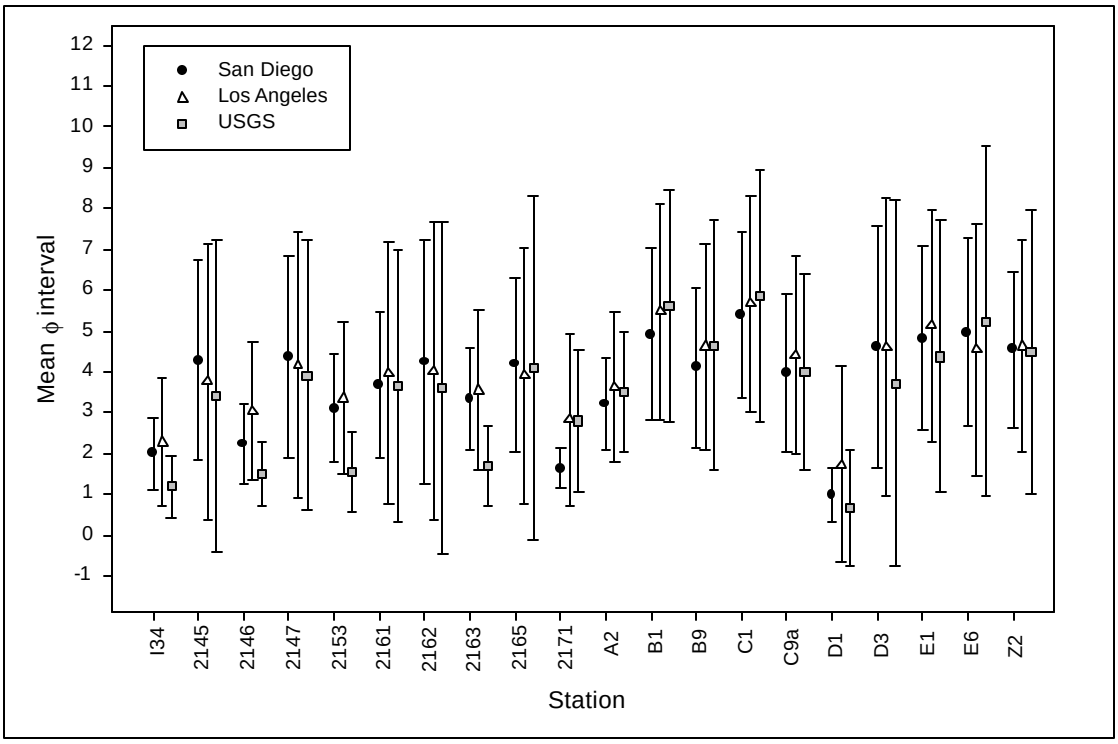

TABLE 3. Percent fines results for all instruments and Kruskal-Wallis p-values for test of significant difference among instruments. Bold-face values indicate the instruments are significantly different (p<0.05).

Station	Coulter	Horiba	SediGraph	p-value
I34	2%	0%	1%	0.06
2145	31%	37%	20%	**0.04**
2146	5%	1%	1%	0.10
2147	34%	38%	23%	**0.04**
2153	9%	5%	2%	0.08
2161	33%	19%	23%	**0.04**
2162	35%	41%	22%	**0.04**
2163	11%	7%	3%	0.08
2165	36%	40%	30%	**0.04**
2171	10%	0%	6%	**0.04**
A2	11%	5%	5%	0.08
B1	72%	62%	81%	**0.04**
B9	36%	27%	34%	0.32
C1	77%	78%	84%	0.07
C9a	28%	22%	15%	**0.04**
D1	8%	0%	3%	0.07
D3	48%	52%	31%	0.12
E1	52%	49%	46%	**0.04**
D6	48%	60%	49%	0.06
Z2	49%	47%	51%	**0.04**

TABLE 4. Variance estimates, of half phi data, within and among instruments.

		Variance
Within Instrument	Coulter	0.04
	Horiba	2.6
	SediGraph	3.2
	Pooled	1.8
Among Instrument		13
Combined Across Instruments		10

While the data were generally comparable, some differences are worth noting. The percent fines, which is a very common summary measure used in assessment investigations with chemical or biological data (Snelgrove and Butnam 1994), is statistically significantly different among instruments. This difference may be due to the collapse of the information into two categories at the 3.5 ϕ interval where the Horiba and SediGraph measurement variability is large, compounding the differing variability among instruments. Also, in using the cumulative distribution, variations in the larger particles affect the resulting percent fines calculation. The differences, although statistically significant, are not large. The difference in median values between any two instruments is only 6%, while the maximum difference in the median among all instruments is 10%. The maximum difference in median measurements among significantly different results is 13%. Our findings imply that this measure may not be the most effective for the comparison of samples from various areas analyzed by different instruments. Perhaps mean ϕ, which appears to be more stable across instruments, deserves consideration for use in ecological applications.

The largest difference among instruments was in measurement variance; this finding reflects technological advancements. The light-scattering instruments, which represent the most advanced technology, have smaller

TABLE 5. Measurement variance at each phi interval for each instrument.

Phi interval	Coulter	Horiba	SediGraph
0.5	0.365	9.422	0.150
1.0	0.090	5.661	1.084
1.5	0.086	9.612	0.091
2.0	0.061	8.646	1.081
2.5	0.041	11.145	0.802
3.0	0.081	9.754	3.754
3.5	0.056	6.880	17.523
4.0	0.062	0.583	44.223
4.5	0.015	0.367	1.478
5.0	0.008	0.068	3.195
5.5	0.006	0.147	1.307
6.0	0.004	0.029	0.362
6.5	0.009	0.041	0.304
7.0	0.009	0.016	0.237
7.5	0.007	0.049	0.140
8.0	0.003	0.010	0.116
8.5	0.003	0.008	0.101
9.0	0.002	0.004	0.098
9.5	0.001	0.005	0.111
10.0	0.001	0.001	0.104
10.5	0.000	0.000	0.000
11.0	0.000	0.000	0.000
11.5	0.000	0.000	0.000
12.0	0.001	0.000	0.536

variability. They measure grain size directly by computing volume from angular diffraction from individual particles (Xu 1996, Jonasz 1991). The Coulter LS230, the newest instrument in our study, possesses more detectors in a wider range of angles and has two independent light sources (laser and Tungsten-Halogen), giving it a technical advantage for characterizing particle sizes by virtue of averaging large numbers of measurements. The number of detectors is related to the resolution of the correct particle size distribution, where fewer detectors result in poorer resolution (Xu 1996). The SediGraph method measures particles indirectly by capturing the differential fall rate of particles (Syvitski *et al.* 1991a, Coakley and Syvitski 1991). Although the data were collapsed into phi intervals, the variability of the measuring mechanism within these intervals affects the resulting measurement variance.

Overall, the larger particle sizes (smaller phi values) were more variable, both within instruments and among instruments. The conversion of fixed geometric intervals into logarithm-transformed integer ϕ-intervals results in a larger range of particle sizes in the smaller ϕ intervals. Light-scattering instruments have fewer detectors, per μ, in the large size range, decreasing the precision of the laser instruments in this region. The addition of PIDS improves resolution in the smaller particle ranges for the Coulter LS230 (Xu 1996), which increases precision in this range. In the SediGraph method, the largest variability occurred in the 1.5–2.0 ϕ interval range, which is the upper end of the SediGraph 5100 measurement capability (McCave and Syvitsky 1991); this number is also within the range where data from the SediGraph 5100 were integrated with settling tube data. This integration from instruments at their limit may increase variability.

Inconsistency in the particle shape and composition of our samples contributed another source of variability in calculating sediment grain size. Principles for determining grain size, including both light scattering and Stokes' Law, assume particle uniformity that is rarely an attribute of environmental samples. For practical reasons, inconsistencies are ignored by using assumptions of equivalency for non-uniformity in particle shape. By virtue of the large number of measurements generated by light-scattering instruments, a closer approximation of mean particle size is achieved. While employing good techniques for sample introduction and particle detection with the SediGraph can control variability (Syvitski *et al.* 1991a), ultimately variability resulting from irregular particle shape and physical properties of our environmental samples cannot be controlled.

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