APPENDIX II

Grain-Size Analysis Data

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1.0 INTRODUCTION

As of April 2000, GeoSea® is using a Malvern Mastersizer 2000 laser particle sizer for the grain-size analysis of sediments. This unit is state-of-the-art equipment. It is extremely accurate, the results are consistent, and it enables the determination of a large range of particle sizes using a single technique[1]. A laser particle sizer is also the most efficient way to analyze the large numbers of samples that are required in Sediment Trend Analysis. This Appendix describes the methodology used in our laboratory.

2.0 METHODOLOGY

2.1 Malvern Mastersizer 2000 laser particle sizer

The instrument is based on the principle of laser diffraction. Light from a low power helium-neon laser is used to form a collimated, monochromatic (red) beam of light which is the analyser beam. The unit also has a solid state blue light source. The shorter wavelength of the blue light allows for greater accuracy in the sub-micron range. Particles from sediment samples enter the beam via a dispersion tank that pumps the material, carried in water, through a sample cell. The resultant light scatter is incident onto the detector lens. The latter acts as a Fourier Transform Lens forming the far field diffraction patterns of the scattered light at its focal plane. Here a custom designed detector in the form of 52 concentric rings gathers the scattered light over a range of solid angles of scatter. When a particle is in the analyser beam its diffraction pattern is stationary and centred on the optical axis of the range lens. Un-scattered light is also focused onto an aperture on the detector. The total laser power exiting the optical system through this aperture enables measurement of the sample concentration.

In practice many particles are simultaneously present in the analyser beam and the scattered light measured on the detector is the sum of all individual patterns overlaid on the central axis. Our instrument is set to take 30,000 such measurements (snaps), which are then averaged to build up a light scattering characteristic for that sample based upon the population of individual particles. Applying the Mie theory of light scattering, the output from the detector is then processed by a computer, generating a final distribution.

Particles scatter light at angles related to their diameter (*i.e.*, the larger the particle, the smaller the angle of scatter and *vice versa*). Over the size range of interest, which is 0.02 micron (μ) and larger for this instrument, scattering is independent of the optical properties of the medium of suspension or the particles themselves. Through a process of constrained least squares fitting of theoretical scattering predictions to the observed data, the computer calculates a volume size distribution that would give rise to the observed scattering characteristics. No *a priori* information about the form of the size

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[1]Most techniques to measure grain-size distributions require sand to be separated from the finer fractions; different analytical methods are used for each split (e.g., settling tube and sedigraph) and the two distributions are then merged together to obtain a complete distribution. Laser analysis does not require such a split, except when very coarse materials are present (coarse sand to gravel-sized fractions).

distribution is assumed, allowing for the characterization of multi-modal distributions with high resolution.

2.2 Laboratory technique

GeoSea has developed a standard operating procedure (SOP) using the Malvern Mastersizer 2000 laser particle size analyser. This ensures that all parameters and variables will remain consistent throughout sample analysis. The methodology covers the range of sizes normally considered important in sediments, is relatively rapid and requires only small samples. No chemical pre-treatment of the samples is undertaken without prior request[2]. Our priority is to determine the size distribution of the naturally occurring sample.

Prior to every analysis, the Mastersizer 2000 automatically aligns the laser beam, and a background measurement of the suspension medium is taken. Samples are initially well mixed before obtaining a representative sub-sample for analysis. The amount of sediment required is about 2 to 4 grams for sands and 0.5 to 1 gram for silt and clay. Samples are introduced into the dispersion unit by wet sieving through a 1mm mesh, eliminating possible blockage of the pumping mechanism by particles that are too large. Disaggregation of the sample is achieved by both mechanical stirring and mild ultrasonic dispersion in the sample dispersion unit[3]. If material remains on the 1mm sieve then the weight percent for each of the coarse sizes (-2.0ϕ to 0.5ϕ[4]; 4.0mm to 0.7mm) is obtained by dry sieving at 0.5ϕ intervals.

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[2]Occasionally we are asked to remove organic matter by peroxide digestion, or carbonates by treatment with weak acid.

[3]GeoSea has conducted several experiments concerning the degree of ultrasonic dispersion that is desirable. If no ultrasonic dispersion is used, fine particles tend to remain as relatively large aggregates producing an erroneously coarse sediment distribution. With increasing ultrasonic disaggregation a distribution will tend to become increasingly finer as flocs become broken apart. Total disaggregation of the fine material may be desirable for some purposes, but for Sediment Trend Analysis we find that the flocs are best treated as part of the overall grain-size distribution. This is because flocs form particular sized particles that behave as separate entities in the transport regime, whereas total disaggregation would produce a grain-size distribution containing particle sizes that were not actually behaving independently during their transport and deposition. Although we find that increasing the degree of disaggregation changes the specific parameters of a grain-size distribution, it is insufficient to produce significant changes in the derived sediment trend statistics. The degree of ultrasonic dispersion presently used by GeoSea appears to be adequate to break apart the sediment into its component particle sizes without excessive damage to those sizes composed of flocculated material.

[4]ϕ (phi) is the unit of measure most commonly used in sediment size distributions where $\phi = -\frac{\log(mm)}{\log(2)}$.

Table 1: Grain-size scales for sediments.

U.S. Standard Sieve Mesh Number	Diameter (*mm*)	Diameter (*microns*)	Phi Value	Wentworth Size Class	Sediment Type
5	4.00		-2.00		
6	3.36		-1.75		
7	2.83		-1.50	Granule	GRAVEL
8	2.38		-1.25		
10	2.00		-1.00		
12	1.68		-0.75		
14	1.41		-0.50	Very Coarse	
16	1.19		-0.25	Sand	
18	1.00		0.00		
20	0.84	840	0.25		
25	0.71	710	0.50	Coarse	
30	0.59	590	0.75	Sand	
35	0.50	500	1.00		
40	0.42	420	1.25		
45	0.35	350	1.50	Medium	SAND
50	0.30	300	1.75	Sand	
60	0.25	250	2.00		
70	0.21	210	2.25		
80	0.177	177	2.50	Fine	
100	0.149	149	2.75	Sand	
120	0.125	125	3.00		
140	0.105	105	3.25		
170	0.088	88	3.50	Very Fine	
200	0.074	74	3.75	Sand	
230	0.0625	62.5	4.00		
270	0.053	53	4.25		
325	0.044	44	4.50	Coarse	
	0.037	37	4.75	Silt	
	0.031	31	5.00		
	0.0156	15.6	6.00	Medium Silt	
	0.0078	7.8	7.00	Fine Silt	
	0.0039	3.9	8.00	Very Fine Silt	MUD
	0.002	2	9.00		
	0.00098	0.98	10.00		
	0.00049	0.49	11.00	Clay*	
	0.00024	0.24	12.00		
	0.00012	0.12	13.00		
	0.00006	0.06	14.00		

(* The Clay/Silt boundary is sometimes taken at 2 microns, or 9 phi.)

2.3 Merge method

GeoSea has developed software that allows the dry-sieved weights and measurements from the laser unit to be merged into a final distribution within the range of -2.0ϕ to 15ϕ, in size bins of equal width (0.5 ϕ) in ϕ-space. The results from the Mastersizer 2000 consist of a set of 52 size bins, where the bin width is inversely proportional to the mean particle size in the bin, with the percentage of material in each bin. A summary of the merging process follows:

(1) Sieve data

Sieving is carried out at half-phi intervals from -2.0ϕ to 0.5ϕ. The weights are normalized and the percentage smaller than 0.5ϕ is used to renormalize the Malvern values using the methods described above. The portion of the lens data above 0.5ϕ is removed and replaced with sieve data.

2.4 Presentation of Results

Size distribution data are generally provided as both hard copy (Table 2) and as a PC computer file. The file format is as comma-separated ASCII values (*.csv) in which the data for each sample are contained in a single line. The first line in the file defines the variables and the phi scale, and is followed by the weight percentages for the samples. These files can be easily imported into a Microsoft Excel spreadsheet. The interpretation of the data is as follows: the weight percentage shown under a size heading is the amount of material found in a bin with size boundaries set by the previous size heading as the upper size limit and the current size heading as the lower limit. For example, the weight percent shown under the heading 1.5ϕ is the amount in the bin bounded by 1.0ϕ and 1.5ϕ. Because of the way the file is written the first size fraction in the list (-2.0ϕ) always has zero weight percent.

The hard copy (Table 2) consists of a printout of the data in spreadsheet form and, when requested, plots of each sample showing its histogram and cumulative curve in both phi and micron units.