A METHOD FOR COMPARING THE LISST 100 TO THE USGS PIPETTE METHOD FOR SUSPENDED SEDIMENT PARTICLE SIZE ANALYSIS IN THE MARINA SEDIMENT LAB, U.S. GEOLOGICAL SURVEY CALIFORNIA WATER SCIENCE CENTER

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Abstract: The Marina Sediment Lab of the USGS California Water Science Center has a need to automate and expand on the number of suspended sediment samples that can be processed for particle size analysis. Measuring the LASER scatter of sediment particles suspended in water is one method that shows promise for sediment lab application. The LISST 100 uses **L**ASER **I**n-**S**itu **S**cattering and **T**ransmissometry (LISST)[1] to determine particle size distribution in a water/sediment mixture sample volume. LISST is a Trademark of Sequoia Scientific, Inc. The technology represented by this instrument offers the potential to increase the number of samples that are submitted to the lab that can be processed for full particle size analysis, and at the same time, offers the potential for faster sample processing, along with improved data base entry. The LISST 100 is intended to operate as an in-situ time-series data collection instrument for monitoring suspended sediment concentration and particle size distribution in water bodies, Sequoia Scientific, Inc. (2002). A LISST 100 Type B was purchased by the Marina sediment lab.

INTRODUCTION

In order to broaden the range of samples that can be analyzed for particle size distribution, it is desirable to have the ability to accurately quantify the particle size distribution for samples with lower suspended sediment concentration than can be processed using the current USGS standard Pipette or Sedigraph methods. The LISST 100 is designed to determine particle size distribution in water having lower suspended sediment concentrations than either of these two methods. The Pipette method provides a theoretical particle size distribution with results for several size classes ranging from 2 to 62 microns in diameter. It requires a minimum dry weight of 0.8g of material for processing and provides a sediment concentration in terms of dry sediment weight in mg/l. The Sedigraph requires a 50 ml volume of condensed sediment mixture which is prepared by removing most of the native water from the sample, and then preparing the sub sample for analysis. The Sedigraph does not provide a sediment concentration value. The LISST 100 Type B produces results for 32 size ranges (bins). The 32 size ranges (fig. 1) are logarithmically incremented from approximately 1.25 – 250 microns in diameter. The particle size results for these increments are reported at the median point for each size class bin. These points range from 1.44 to 231 microns. Theoretically, the LISST 100 has no lower concentration limit. However, there may be a practical lower concentration threshold where a particle size distribution may have little meaning. The upper concentration limit is reported by the vendor to be approximately 500 mg/l, but higher concentration samples could be analyzed after careful splitting and/or dilution. The particle sizes are given as a volume percentage in terms of the total volume of particles in the sample. There is not a capability to produce a sediment concentration value that relates to a volume of water/sediment mixture. Testing will help determine the practical limits of using this technology in a production lab.

METHODS

Samples designated for full particle size analysis will first have the sand fraction removed for separate analysis using the wet sieve method. Once this sand fraction has been removed, a trial aliquot will be drawn from the fully mixed fines to ascertain if sufficient material is present in the sample to use the pipette procedure. A duplicate trial aliquot will be run through the LISST 100. A dispersing agent is used to assure separation of fine particles. A dissolved solids correction is required when a dispersing agent is used. The correction is needed to adjust both the Pipette and LISST results. The above procedures were modified from initial test attempts which had several procedural issues. Problems with the initial procedures may or may have not had an impact on the sample data comparison, but the potential existed.

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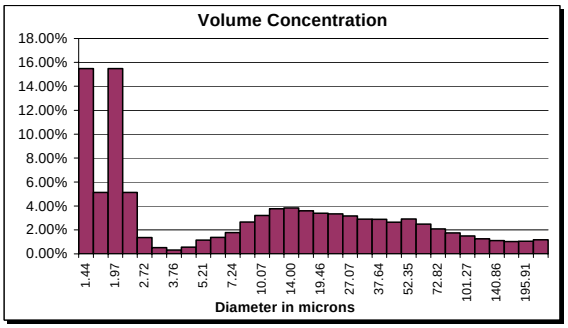
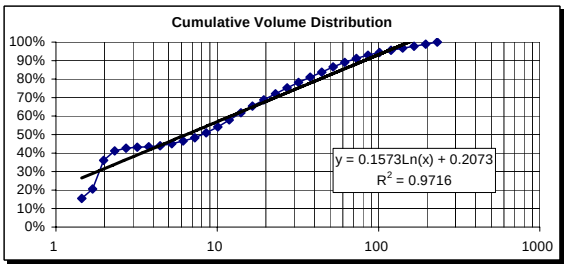
[1] Any use of trade, product, or firm names is for descriptive purposes only and does not imply endorsement by the U.S. Government.

Lab ID# C3503_lisst
Station number: SLQA
Collection Date: N/A
Technician: N/A
Sample Description: Full Size Particle Analysis

US Geological Survey
California District Sediment Lab
455 Reservation Rd. Suite F
Marina, Ca 93933
(831) 384 - 2143





Computed Statistics

D10 = #N/A
D50 = 7.24
D90 = 61.74
Dmean = 25.82

Specific Surface Area = 0.16 m^2/g

Total Instrument Results:

Size (microns)	Volume Conc (%)	Cumulative Volume
1.44	15.49%	15.49%
1.68	5.13%	20.62%
1.97	15.49%	36.11%
2.31	5.13%	41.24%
2.72	1.36%	42.60%
3.19	0.52%	43.12%
3.76	0.32%	43.44%
4.43	0.55%	43.98%
5.21	1.14%	45.12%
6.14	1.37%	46.50%
7.24	1.77%	48.27%
8.54	2.67%	50.93%
10.07	3.20%	54.14%
11.87	3.76%	57.90%
14.00	3.84%	61.74%
16.50	3.59%	65.33%
19.46	3.39%	68.72%
22.95	3.34%	72.06%
27.07	3.18%	75.24%
31.92	2.90%	78.13%
37.64	2.89%	81.02%
44.39	2.65%	83.67%
52.35	2.92%	86.59%
61.74	2.49%	89.08%
72.82	2.08%	91.16%
85.87	1.74%	92.90%
101.27	1.48%	94.38%
119.44	1.25%	95.64%
140.86	1.10%	96.74%
166.12	1.04%	97.78%
195.91	1.05%	98.83%
231.04	1.17%	100.00%

Comments: Dry sample was mixed with DI water and mixed in sample chamber. Background scatter included dispersing agent. Sample high concentration, was split twice (tag).

Analysis Performed by: TAG
Processing Date: 9/27/2005

Analysis performed using laser diffraction techniques as described in AWWA Standard No. 2560D. Instrumentation calibrated using NIST traceable standard particles.

USGS Standard Results

Size (microns)	Volume Conc (%)	Cumulative Volume
0.01 - 2.00	36.11%	36.11%
2.01 - 4.00	7.33%	43.44%
4.01 - 8.00	4.83%	48.27%
8.01 - 16.00	13.47%	61.74%
16.01 - 31.00	13.50%	75.24%
31.01 - 62.00	13.84%	89.08%
62.01 - 124	6.56%	95.64%
124.01 - 256	4.36%	100.00%

Figure 1 Example of LISST 100 data output format for one sample. Used by permission, Sequoia Scientific.

<u>The Pipette Method:</u> The Pipette procedure is the method used by the Marina sediment lab to determine full particle size. This procedure is based on Stokes Law which, when applied to the determination of sediment particle sizes, states that particles of different diameters fall through a given liquid at predictable rates (Guy 1969). The larger the size of a particle, the quicker its rate of fall through a fluid. A sample is stirred with a blender. The stirring is stopped and the settling process begins. Sample aliquots are taken using a pipette at specific depths and times during the duration of the settling process (which can last several days), theoretically capturing only specific particle sizes in each of the pipette withdrawals. Each withdrawal is dried and weighed to determine the percent of the total sample weight that is represented by each of these fall size classes.

The LISST 100 Method: A mixing chamber is attached to a flow through cell which is mounted to the optic head of the LISST 100. If concentration levels exceed the LISST 100 operational limits then the sample is accurately split, perhaps several times. Splitting is completed by using a standard laboratory silt/clay splitter. The final analysis begins with two instantaneous aliquot withdrawals made immediately at the starting time of the settling process. One aliquot is for the Pipette analysis, the other is used for the LISST 100. By performing the testing in this manner, all of the sediment material will be present in a fully dispersed state for both methods, thus, the direct comparison of sample results should be most valid.

Test Data Evaluation: Initial evaluation of results from the 2 methods was based on the analyses of 7 samples (Tables 1-7). The samples were provided to the Marina sediment lab by the USGS Sediment Lab Quality Assurance (SLQA) program. Each of the 7 samples was used twice. Each sample was processed using the Pipette method. After the drying and weighing the withdrawals, the sample was recovered, re-suspended in water and re-disbursed. Then a 20 ml aliquot was withdrawn. The LISST 100 analysis was performed on this aliquot. This was not an ideal method of comparison, as sample integrity and duplicity are not assured. The SLQA samples are composed of silica material (spark plug dust) with a specific gravity of 2.65, approximating that of quartz sediments.

Modified testing procedures described above have been implemented for a second round of testing currently in progress during December 2005. The second testing round uses duplicate sets of samples provided for each method by the SLQA program. One sample from the current round has been processed at the time of this writing. The Lab Chief has reported that the preliminary results still show a significant difference in the results of the two methods even when the revised procedure was used. The LISST 100 is still reporting a higher percentage of the finest and coarsest size material relative to the Pipette analysis. At this time, it is not clear what is causing the differences. More comparative testing is needed with SLQA samples and natural water samples. The revised methodology will also be reviewed and modified if necessary.

TABLES FOR RESULTS OF SLQA STUDY COMPLETED DECEMBER, 2004.

Table 1 Size distribution for SLQA sample ID C3503.

Particle size class range (microns)	Pipette results: Percent Finer Than	LISST 100 results: Percent Finer Than	SLQA Target Value
0.01-2.00	19.7	36.11	18
2.01-4.00	22.8	43.44	20
4.01-8.00	24.6	48.27	33
8.01-16.00	55.2	61.74	57
16.01-31.00	81.0	75.24	79
31.01-62.00	100	89.08	100
62.01-124		95.64	
124.01-256		100	

Table 2 Size distribution for SLQA sample ID C3504.

Particle size class range (microns)	Pipette results: Percent Finer Than	LISST 100 results: Percent Finer Than	SLQA Target Value
0.01-2.00	19.9	52.71	18
2.01-4.00	22.0	57.42	20
4.01-8.00	23.8	58.02	33
8.01-16.00	55.4	65.84	57
16.01-31.00	82.0	78.66	79
31.01-62.00	100	93.27	100
62.01-124		98.36	
124.01-256		100	

Table 3 Size distribution for SLQA sample ID C3505.

Particle size class range (microns)	Pipette results: Percent Finer Than	LISST 100 results: Percent Finer Than	SLQA Target Value
0.01-2.00	26.2	56.32	23
2.01-4.00	27.2	61.42	25
4.01-8.00	30.5	62.21	41
8.01-16.00	67.4	72.06	70
16.01-31.00	86.3	85.23	87
31.01-62.00	100	94.58	100
62.01-124		98.28	
124.01-256		100	

Table 4 Size distribution for SLQA sample ID C3506.

Particle size class range (microns)	Pipette results: Percent Finer Than	LISST 100 results: Percent Finer Than	SLQA Target Value
0.01-2.00	24.5	58.05	23
2.01-4.00	25.5	63.11	25
4.01-8.00	28.6	63.76	41
8.01-16.00	65.4	72.59	70
16.01-31.00	86.8	85.39	87
31.01-62.00	100	94.45	100
62.01-124		98.12	
124.01-256		100	

Table 5 Size distribution for SLQA sample ID C3507.

Particle size class range (microns)	Pipette results: Percent Finer Than	LISST 100 results: Percent Finer Than	SLQA Target Value
0.01-2.00	25.0	59.65	23
2.01-4.00	26.9	64.8	25
4.01-8.00	29.8	65.51	41
8.01-16.00	66.7	74.55	70
16.01-31.00	89.0	86.59	87
31.01-62.00	100	94.58	100
62.01-124		97.86	
124.01-256		100	

Table 6 Size distribution for SLQA sample ID C3508.

Particle size class range (microns)	Pipette results: Percent Finer Than	LISST 100 results: Percent Finer Than	SLQA Target Value
0.01-2.00	34.5	57.97	30
2.01-4.00	37.6	63.48	33
4.01-8.00	41.8	64.21	48
8.01-16.00	68.8	72.02	74
16.01-31.00	90.4	83.15	88
31.01-62.00	100	93.10	100
62.01-124		97.73	
124.01-256		100	

Table 7 Size distribution for SLQA sample ID C3509.

Particle size class range (microns)	Pipette results: Percent Finer Than	LISST 100 results: Percent Finer Than	SLQA Target Value
0.01-2.00	35.8	60.13	30
2.01-4.00	37.3	65.90	33
4.01-8.00	40.6	66.72	48
8.01-16.00	70.0	74.87	74
16.01-31.00	89.5	86.19	88
31.01-62.00	100	94.73	100
62.01-124		98.15	
124.01-256		100	

ISSUES TO CONSIDER FOR EVALUATION OF TEST RESULTS

1- The output of the LISST 100 does not provide particle size class information that corresponds directly with the class sizes historically used by the USGS.

2- A direct comparison of results for lower concentration samples (concentrations that fall in the LISST 100 operating range of <1 to ~500 mg/L) may not be possible. The Pipette method requires a substantial amount of sediment that is usually associated with samples of higher concentrations. The sediment concentrations of stream samples submitted to the lab for analysis that would characteristically fall within the operating range of the LISST 100 would most likely fall well below that of the samples that could be analyzed with the Pipette method. There may be little opportunity to compare samples having concentrations that overlap the limits of both methods.

3- The LISST 100 particle size determinations are based on 2 dimensional of the cross-sectional area of a particle. Measurements of the sediment particles are then converted mathematically to a 3 dimensional volume. The measurement is independent of the specific gravity of the particle. The Pipette method is based on theoretical fall rates of spherical particles through water. Thus, a large, but less dense particle could be classified by the Pipette method as having a size that is smaller than its true physical dimensions. The converse is true if the particle is composed of material with a high specific gravity.

4- Sediment concentration values are not generated by the LISST 100. The concentration of particles in a given class size is calculated as a volume percentage of the total of particle volume measurements of all the particles in the sample. This volumetric concentration is not related to the total particle volume in the water-sediment mixture of a sample. Determining a volumetric concentration (i.e. cc/ml) could be accomplished through use of a simple calculation by computing the ration of the total measured particle volume in cc versus the weight of the water/sediment mixture placed into the LISST 100 mixing chamber.

5- The Pipette method determines sediment concentration using the dry weight of all particles in a sample. If the additional analysis step in item 4 were used with the LISST 100 to determine sample concentration, it is probable that the sample concentration would be over estimated when samples contain organic materials or particles from sources where material is of low specific gravity. Conversely, there is also the potential for under estimating concentration where the sediment particles are composed of material with high specific gravity. Is this difference in determination of sediment concentration a significant problem? The implication of this difference has significant ramifications when computing suspended sediment transport. Calculations of suspended sediment transport are based on sediment concentrations expressed as dry sediment weight per unit volume of water, Porterfield (1972).

REFERENCES

Guy, H. P. (1969). “Laboratory Theory and Methods for Sediment Analysis” Techniques of Water Resources Investigations of the USGS, Book 5, Chapter C1, 89 pp.

Porterfield, G. (1972). “Computation of fluvial-sediment discharge” Techniques of Water Resources Investigations of the USGS, Book 3, Chapter C3, 66 pp.

Sequoia Scientific, Inc. (October 2002). Sequoia, LISST-100 Particle Size Analyzer, User’s Manual Version 4.2b, 75 pp.