PARTICLE SIZE DISTRIBUTION OF GYPSEOUS SAMPLES

A Thesis

by

MORGAN PAIGE ARNETT

Submitted to the Office of Graduate Studies of
Texas A&M University
in partial fulfillment of the requirements for the degree of

MASTER OF SCIENCE

May 2009

Major Subject: Soil Science

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Approved by:

Chair of Committee,	Charles Hallmark
Committee Members,	Sam Feagley
	Robert Knight
Head of Department	David Baltensperger

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ABSTRACT

Particle Size Distribution of Gypseous Samples. (May 2009)

Morgan Paige Arnett, B.S., Texas A&M University

Chair of Advisory Committee: Dr. Charles Hallmark

Particle size distribution (PSD) of gypseous soils is important in the soil science community. When gypsum constitutes a major portion of the soil, its removal prior to PSD analysis distorts the results and may lead to textures that do not relate to conditions in the field. In order to understand the true characterization of the soil and the gypsum particles, the entire soil sample should be analyzed. Four different approaches to the $BaCl_2$ method presented in the literature (Hesse, 1976, Matar and Douleimy, 1978, Viellefon, 1979) were used to evaluate the use of $BaCl_2$ solution to reduce the solubility of gypsum by forming a protective coating of $BaSO_4$ around gypsum particles. Results showed that the $BaCl_2$ method was unsatisfactory, as dispersion of clays was not sufficient to allow particle size analysis using the pipette method. A procedure using a laser diffraction particle size analyzer (LPSA) was also evaluated. As gypsum is insoluble in methanol, methanol was selected as a possible solution, but it caused flocculation of clays and could not be used to analyze samples containing silicate clays. Gypsum saturated water containing Na hexametaphosphate was evaluated as a solution. First, 20 non-gypseous samples were analyzed on a sand-free basis using saturated gypsum water with Na hexametaphosphate. Results were used to establish a relationship comparing LPSA results and pipette results. An equation $y = 1.37x + 2.03$ was established relating LPSA clay percent by volume (x) to the pipette clay percent by weight (y). The equation had a R^2 value of 0.84 and was significant at the 1% level. From this equation a comparison of 21 gypseous samples was made, between clay percentages of the pipette method and the LPSA method. Results indicate that LPSA can be used to give a satisfactory particle size distribution of gypseous soils when coupled with sand analysis by sieving.

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CHAPTER I

INTRODUCTION

Particle size distribution (PSD) refers to the determination of the range of particles sizes that make up the soil (Zobeck, 2004). The PSD is usually expressed as a percentage of total mass and is one of the most fundamental physical properties of a soil, defining, for example, the soil's texture, and strongly affecting many physical and chemical soil properties (Eshel et al., 2004). The standard method for PSD of non gypseous samples is the pipette or sieve-pipette method. This is a lengthy procedure in which determines the fine fractions (clay and silt) by pipette method based on Stokes' Law and the coarse fractions (sand) by the sieve method. When performing this PSD method on gypseous samples, flocculation of clays occurs, making it impossible to obtain the true PSD of the sample. Pretreatment of gypseous soils is required to remove gypsum from the sample. However, when samples contain greater amounts of gypsum, removal of gypsum renders a residue of sand, silt and clay that may be greatly different from the initial sample, and results may have little interpretive meaning.

The overall goal of this research is to evaluate PSD methods for gypseous soils and to evaluate the feasibility of each procedure. Specifically, this research will address the following objectives: 1) evaluate the precision and accuracy of the $BaCl_2$ method for PSD of gypseous soils presented by Hesse (1976); 2) evaluate the precision, accuracy and reproducibility of the use of a laser particle size analyzer (LPSA) using

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different solutions; and 3) determine if a functional relationship exists between PSD results from the pipette method and the results obtained by the laser particle size analyzer (LPSA).

Literature Review

Particle Size Distribution Using $BaCl_2$

Particle size distribution is one of the most fundamental physical properties of a soil, defining, the soil's texture, and strongly affecting many physical and chemical soil properties (Eshel et al., 2004). Wen et al. (2002) states PSD relates to a soil's porosity, permeability, consolidation, shear and volume change behavior. It also reflects depositional history of transported soils and evolution of in situ (residual) soils. It is important to have an accurate PSD of a soil sample whether it has gypsum or not. The presence of gypsum in soils complicate the determination of PSD due to the difficulty in dispersion of the soils (Hesse, 1976). Hesse (1976) stated, during normal dispersion procedures, sufficient gypsum dissolves to reduce the zeta potential and cause flocculation of clay and silt. When flocculation occurs, it is virtually impossible to obtain an accurate PSD analysis. In cases where gypsum content is low, it is feasible to remove the gypsum with pretreatment and proceed with normal dispersion of the sample and the sieve pipette method as described in the Soil Survey Laboratory Methods Manual (Soil Survey Staff, 1996). As cited by FAO (1990), Vieillenfon (1977, 1978, 1979) performed comprehensive studies especially dealing with the effects of gypsum on PSD and concluded: 1) partial flocculation of the fine soil particles results in their underestimation; 2) gypsum has a density of 2.317 Mg m^{-3}, so methods using Stokes'

Law which assumes an average particle density of 2.65 Mg m^{-3} overestimate the clay and silt fractions of soils if not corrected; and 3) in the pipette method, during drying of each particle-size fraction, gypsum loses its water of crystallization and consequently, the fraction of soils containing the most gypsum is underestimated unless correction is made. Prior to the introduction of the $BaCl_2$ method, other procedures were investigated for PSD of gypsum-containing samples. It was recommended that gypsum be removed before adding Na hexametaphosphate as a dispersing agent as the phosphate will readily dissolve gypsum bringing calcium ions into solution (Tyner 1939). As cited by Hesse (1976), Vanlande (1953) boiled gypsic soils with ammonium oxalate solution before dispersing and Piper (1942) recommended that for soils containing more than 2 or 3 percent gypsum, the organic matter-free sample should be shaken with a large volume of dilute HCl for up to 16 h and then filter the soil until free of chloride. All known methods for analyzing gypsic soils for PSD are tedious, time-consuming and usually quite inaccurate; furthermore, it is actually the analysis of gypsum-free soil that is made and hence correlations with field data are impossible (Hesse, 1976). Hesse (1976) described a procedure for PSD in gypsic soils which depends upon a treatment of gypsum crystals with $BaCl_2$ solution to form a protective coating of $BaSO_4$ (barium sulfate) around gypsum. Garman and Hesse (1975) reported that treatment of a gypsic soil with $BaCl_2$ solution immobilizes the gypsum particles from further reaction in an alkaline medium by formation of a thin coating of $BaSO_4$. In theory this should reduce or eliminate the solubility of gypsum in water with a dispersing agent. Synthetic gypsic soils were prepared containing up to 90% gypsum using different particle sizes of gypsum and were analyzed using Hesse's (1976) $BaCl_2$ method. He reported satisfactory dispersion in every case, from a soil containing 1% coarse gypsum to one containing 90% very finely divided gypsum).

Matar and Douleimy (1978) found that the effectiveness of the Ba treatment depends on the gypsum content of the soil and the PSD of the gypsum particles. Matar and Douleimy (1978) and Vieillefon (1979) proposed procedures much like the one of Hesse, where results were comparable to that of Hesse. However, although these results showed promise, there is an apparent lack of acceptance of a $BaCl_2$ procedure as indicated by no follow-up studies and no evidence of use of the procedure subsequent to publication of these studies.

Laser Particle Size Analyzer

Numerous articles can be found in the literature pertaining to laser particle size analysis (LPSA), but there are no discussions relative to determine PSD of gypseous soils. For several decades, classic sedimentation methods (hydrometer and pipette) combined with sieving have been adopted as the international standard to determine PSD of soils (Cooper et al., 1984). As previously stated it is not feasible to use these methods for samples containing gypsum. The pipette and hydrometer methods give comparable results (Liu et al., 1966; Walter et al., 1978) provided similar pretreatment techniques are used (Eshel et al., 2004). The main limitations of the sieve-pipette method include the difficulty of setting up the equipment properly, time of analysis and differences in operators' skills (Indorante et al., 1990). The pipette method is time consuming in comparison to the LPSA. There are advantages to the sieve-pipette method such as the relatively low cost of the equipment, high precision and reproducibility (Arriaga et al., 2006). Recent advances in instrumentation have led to the development of devices that measure the distribution of particles using laser diffraction (Zobeck, 2004). Zobeck (2004) states this device uses a small sample and can provide a relatively easy, rapid, and highly reproducible way of determining the fraction of total volume or weight fractions of particles for a large number of size classes. The main advantage of laser light methods is speed of analysis (Arriaga et al., 2006). Laser diffraction size analysis is based on the principle that particles of a given size diffract light through a given angle, the angle increasing with decreasing size (McCave et al., 1986). The intensity of the diffracted beam at any angle is a measure of the number of particles with a specific cross-section in the beam's path (Eshel et al., 2004). Zobeck (2004) observed 43 surface soil or sediment samples from the Texas Southern High Plains. The samples were air dried and passed through a 2-mm sieve. No pretreatment for organic matter was necessary because all samples contained less than 1%. Textures ranging from loamy fine sand (lfs) to clay (c) were examined. Sample size ranged from 100 to 450 mg depending on the affect on the light obscuration. The samples were dispersed and shaken overnight. Zobeck (2004) concluded the total time for analysis and flushing or cleaning by an experienced user was approximately 10 min. This takes much less time than does the pipette method that is the national standard for PSD. Three replications were performed for each sample and in almost every sample tested, differences among replicated subsamples were negligible (Zobeck, 2004). A range of refractive indices was also tested for different soil materials. Deer et al. (1962) suggested the following refractive indices for selected soil material: illites 1.54-1.57; smectites 1.48-1.61; kaolinites 1.55-1.56; and quartz 1.54. Zobeck (2004) concluded measurements of PSD using laser diffraction technology provides a relatively easy and rapid method to determine the volume or weight of particles for a large number of size classes with a great degree of reproducibility on a small sample.

Wen et al. (2002) compared result of the LPSA to the sieve-hydrometer method. The LPSA (Coulter® LS-100) produced continuous and smooth PSD curves for the eight samples evaluated. It was concluded that both methods were reproducible but the LPSA produced almost replicate curves (Wen et al., 2002). To confirm the conclusion, Wen et al. (2002) quantified the degree of interrelationship between the two methods by linear regressions. The correlation coefficient R for all samples was greater than 0.99, indicating an almost perfect agreement. This may be compared to results one might see from the pipette method.

Wen et al. (2002) also concluded there are many advantages to using the LPSA in order to determine PSD. It is rapid, precise and provides more detailed information. Also, it has much better reproducibility, requires only a small amount of sample, and the data are provided in digital format. Eshel et al. (2004) found it extremely important that an entire range of particle sizes can be divided into a wide range of size fractions. This has extreme importance because the availability of a continuous PSD, rather than an arbitrary division of the particles among a limited number of size fraction, enables a more detailed data analysis and a simultaneous use of the same data sets for classification of the analyzed samples under different national classification systems (Eshel et al., 2004).

Eshel et al. (2004) performed a critical evaluation of the LPSA and the combined sieve-pipette method for determining PSD of soils and assessed 1) if a functional relationship existed between the two types of methods for determining PSD; and 2) the suitability of LPSA as a routine procedure for PSD determination in soil science. Forty-two samples were observed in the study. They found that for all but two of the 42 samples tested, the LPSA yielded a smaller clay fraction and a higher silt fraction, than did the pipette method. In the case of the sand fraction, a trend was noted whereby at sand fractions <35% the LPSA yielded a higher proportion of sand than the pipette method, but the opposite was true for sand fractions >55%. They concluded from their study that observations suggest that in individual studies LPSA data could at times be satisfactorily correlated with pipette data for a given size fraction, but no universal relation between PSD obtained by LPSA and that obtained by the pipette method can be formulated for the entire PSD range. Another conclusion drawn from their research was two major disadvantages: the high cost of the instrument and the lack of a database that correlates LPSA derived PSDs with soil properties similar to the very extensive database existing for pipette derived PSDs. Beuselinck et al. (1998) found the reproducibility of the Coulter LS-100 to be better than that of the sieve-pipette method, except for the sand fraction. There was also an underestimation of the clay content of the silty soil samples, much like Eshel et al. (2004). It may be stated that LPSA determination will always differ from sieve and pipette analysis when particle forms deviate from the idealized sphere and particle density is heterogeneous (Konert and Vandenberghe, 1997). As in much of the literature, Beuselinck et al. (1998) found many advantages of the LPSA, such as rapid, good reproducibility, a single analysis gives a wide range of particle sizes, small amounts of sample are required, detailed information is obtained, and again the information is in digital format and easy to work with. The LPSA is almost 50 times faster than the sieve-pipette, increasing the number of samples that can be analyzed (Arriaga et al., 2006). Arriaga and coworkers, (2006) found that operator error and differences seem to be minimal with the LPSA and that sonication alone seemed to work as well as chemical dispersion. Sonication is a function to insure minimal flocculation occurs in the sample. Arriaga et al. (2006) also believes that discrepancies between the light diffraction and sieve-pipette method are a concern, but with the advent of new instrumentation and data analysis software, these differences could be reduced further. Literature shows if appropriate sample preparation and handling procedures are employed, laser diffraction provides a precise method for the analysis and comparison of sediments, soils and similar materials samples (Blott et al., 2004; Pye and Blott, 2004).

CHAPTER II

EVALUATION OF THE $BaCl_2$ METHOD

Introduction

Particle size distribution (PSD) of soils is important and fundamental when defining properties of the soil. Textural classes of soils affect both the chemical and physical properties, such as infiltration of water into the surface soil, permeability of the subsoil, available water holding capacity, porosity and soil structure. Chemical properties of the soil affected by texture may include pH, cation exchange capacity, and base saturation. Large amounts of gypsum inhibit the ability to perform PSD and obtain a representative analysis of the soil sample. The presence of gypsum complicates the PSD determination due to an inability to disperse the sample in an aqueous solution as gypsum dissolves, and the high soluble Ca levels cause flocculation of clay-sized particles. All known methods for determining PSD of gypseous samples are time consuming and lengthy. The current, widely accepted procedures for PSD of gypseous samples require a pretreatment of the sample to remove all gypsum, either by dialysis (Rivers et al., 1982) or by repeated washings (Lovelady, 1974). Once the pretreatment is complete, PSD is completed as described in procedure 3A1 in the Soil Survey Laboratory Methods Manual (1996) or as described by Bouyoucos (1927). Although removal of lesser quantities of gypsum (i.e., less than 10%) may have little influence on the overall PSD results, removal of large quantities of gypsum may result in PSD data that are unrelated to field observation and accessory soil properties as only the non-gypseous portions of the sample is reported. Therefore, it is important to develop a procedure that determines the PSD of the soil sample without removal of gypsum particles. Hesse (1976) recognized this dilemma and noted that proportions of different sized gypsum crystals contribute largely to the texture and influence other soil properties dependent upon soil texture. For example, coarse gypsum crystals tend to make a soil behave as if it were sandy and influenced permeability. Therefore, determining PSD of the entire sample could be important in characterizing the behavior of the soil. In an attempt to perform PSD analysis without removal of gypsum, Hesse (1976) recommends the treatment of gypsum crystals with $BaCl_2$ solution to form a protective coating of $BaSO_4$, which isolates the gypsum from further reaction in an alkaline medium. Hesse (1976) reported satisfactory results for soils containing as much as 90% gypsum, even if present as particles of less than 0.05-mm diameter. The overall goal of this research was to evaluate the suitability and functionality of the $BaCl_2$ method for PSD of gypseous soils as presented in the literature by Hesse (1976), Matar and Douleimy (1978), and Vieillefon (1979).

Materials and Methods

Soil Sample

The soil sample used in this study was the 2Btk3 horizon of the Colmena series (S06TX273-002) taken from a depth 155-203 cm from Kleberg County, Texas. Colmena is a fine-loamy, mixed, superactive, hyperthermic Typic Argiustoll. A prior particle size distribution was determined by the pipette method in the Texas AgriLife Research Soil Characterization Laboratory following Hallmark et al. (1986). It was a sandy clay loam with 62% sand, 21% clay, and 17% silt. As the sample was also non-gypseous, gypsic material of approximately 90% gypsum content from a gypsic horizon was added in various amounts to the soil sample to give 10g samples. Table 2.1 gives the composition of each prepared sample used to test the $BaCl_2$ method.

Table 2.1. Air-dried weights of soil and gypsic material mixed to give 10-g samples for evaluation of $BaCl_2$ procedures.

Sample Number	Soil Sample (g)	Gypsic Material (g)
1	10	0
2	9	1
3	8	2
4	6	4
5	4	6

$BaCl_2$ Solution

The purpose of the $BaCl_2$ solution is to form a protective coating of $BaSO_4$ around gypsum particles to reduce the solubility of gypsum. As $BaSO_4$ is extremely insoluble in water in comparison to the gypsum, (0.00222 g/L vs. 2.41 g/L, respectively; Handbook of Chemistry and Physics, 1968), an adequate coating of $BaSO_4$ on gypsum crystal surfaces should reduce its solubility in an aqueous solution and allow the dispersion of clay. The $BaCl_2$ solution was made by dissolving 50 g of $BaCl_2 \cdot 2H_2O$ in distilled water and bringing to a volume of 1 liter with deionized water (0.20 M).

BaCl$_2$ Procedure

Four different procedures were evaluated in relation to the BaCl$_2$ method. In the first procedure five 10-g samples of varying gypsum content (Table 2.1) were transferred to 100-mL centrifuge tubes. Forty mL of BaCl$_2$ solution were added to each centrifuge tube, and a stopper was placed in the top to assure no solution was lost. The tubes were shaken gently for 1 h using a reciprocating shaker. Once removed from the shaker, stoppers were removed, and the samples were centrifuged for 8 min at 2000 rpm. The solution was then decanted and 40 mL of distilled water were added to the sample. A stopper was once again put in place to assure no sample was lost. The tube was shaken by hand until the sample was loosened and mixed well. Again, the sample was centrifuged for 8 min at 2000 rpm and the solution decanted. The washing step was preformed once more to remove excess Ba^{+2} from the solution. The absence of Ba^{+2} was confirmed with a drop of potassium chromate. If Ba^{+2} is present a white precipitant will occur. The sample was transferred into a 400-mL square bottle and filled approximately half full with distilled water. Five mL of 10% Na hexametaphosphate were added to the sample to serve as a dispersing agent. Stoppers were applied to the bottles and shaken for 2 h in the reciprocating shaker. Afterwards, the bottles were removed from the shaker, and PSD analysis was initiated following the pipette method of Kilmer and Alexander (1949).

In the second procedure five 10-g samples (Table 2.1) were placed into 100-mL centrifuge tubes. Forty mL of BaCl$_2$ solution were added to each centrifuge tube, and a stopper was placed in the top to assure no solution was lost. The tubes were shaken gently for 5 min using a reciprocating shaker. Once removed from the shaker, stoppers were removed, and the samples were centrifuged for 8 min at 2000 rpm. The solution was decanted, and the previous step was repeated twice more. After the third decanting, 40 mL of distilled water were added to the sample. A stopper was once again put in place to assure no sample was lost. The tube was shaken by hand until the sample was loosened and mixed well. Again, the sample was centrifuged for 8 min at 2000 rpm and the solution decanted. The distilled washing was preformed once more to remove excess Ba^{+2} from the solution. The absence of Ba^{+2} was confirmed with a drop of potassium chromate. The sample was transferred in a 400-mL square bottle and filled to about half full with distilled water. Five mL of 10% Na hexametaphosphate were added to each sample to serve as a dispersing agent. Stoppers were applied to the bottles and shaken for 10 min (shorter time than the first procedure) in the reciprocating shaker. After 10 min the bottles were removed from the shaker, and the pipette method was initiated following Kilmer and Alexander (1949).

In the third procedure, five 10 g samples (Table 2.1) were transferred to 100-mL centrifuge tubes. Forty mL of $BaCl_2$ solution were added to each centrifuge tube, and a stopper was placed in the top to assure no solution was lost. The tubes were shaken gently for 5 min using a reciprocating shaker. Once removed from the shaker, stoppers were removed. and the samples were centrifuged for 8 min at 2000 rpm. The solution of each sample was then decanted and collected in separate containers after centrifugation. The previous step was repeated a total of five times. After the fifth decantate was collected, 40 mL of distilled water were added to the sample. A stopper was once again put in place to assure no sample was lost. The tube was shaken by hand until the sample was loosened and mixed well. Again, the sample was centrifuged for 8 min at 2000 rpm and the solution decanted. The previous step was preformed once more to remove any excess Ba^{+2} from the solution. The absence of Ba^{+2} was confirmed with a drop of potassium chromate. The sample was transferred into a 400-mL bottle and filled to approximately half full with distilled water. Five mL of 10% Na hexametaphosphate were added to each sample to serve as a dispersing agent. Stoppers were applied to the bottles and shaken for 10 min (shorter time than the first procedure) in the reciprocating shaker. After 10 min the bottles were removed from the shaker, and the pipette method was initiated according to Kilmer and Alexander (1949). The Ca content was determined on each decantate of the five washings using a nitrous oxide-acetylene flame by atomic absorption spectroscopy.

In the fourth procedure, the $BaCl_2$ solution used differed from that in the other three procedures. It was prepared by dissolving 50 g of $BaCl_2 \cdot 2H_2O$ in 500 mL of deionized water, adding 20 mL of triethanolamine (TEA) and bringing to a volume of 1 L. Hesse (1976) suggested the use of TEA. Five 10 g samples (Table 2.1) were transferred in 100-mL centrifuge tubes. Forty mL of $BaCl_2$ solution were added to each centrifuge tube, and a stopper was placed in the top to assure no solution was lost. The tubes were shaken gently for 5 min using a reciprocating shaker. Once removed from the shaker, stoppers were removed, and the samples were centrifuged for 8 min at 2000 rpm. The solution was then decanted, and the previous step was repeated twice more. After the third decanting, 40 mL of distilled water were added to the sample. A stopper was once again put in place to assure no sample was lost. The tube was shaken by hand until the sample was loosened and mixed well. Again, the sample was centrifuged for 8 min at 2000 rpm and the solution decanted. The previous step was preformed once more to remove any excess Ba^{+2} from the solution. The absence of Ba^{+2} was confirmed with a drop of potassium chromate. The sample was transferred into a 400-mL square bottle and filled partially with distilled water. Five mL of 10% Na hexametaphosphate were added to the sample to serve as a dispersing agent. Stoppers were applied to the bottles and shaken for 10 min (shorter time than the first procedure) in the reciprocating shaker. After 10 min the bottles were removed from the shaker, and the pipette method was initiated according to Kilmer and Alexander (1949).

Results and Discussion

Procedure One

It was imperative to obtain results that showed good dispersion of clays in the samples that contained gypsum. This was not evident in the first procedure. The first pipette aliquot (<20 µ fraction) was taken at a settling time of approximately 2 min depending on the temperature of the sample, and flocculation of clays was no evident. However, at the time for the second pipetting for the clay fraction (approximately 3.5 h), flocculation was evident in the four samples that contained gypsum. It was concluded than an adequate coating of $BaSO_4$ around the gypsum crystals was not achieved. Since obvious flocculation occurred before the second pipetting, it was decided that further pipette aliquots were not needed. Figure 2.1 shows the obvious flocculated state of the clays at the time of the <2 µ clay fraction pipetting. Table 2.1 provides the

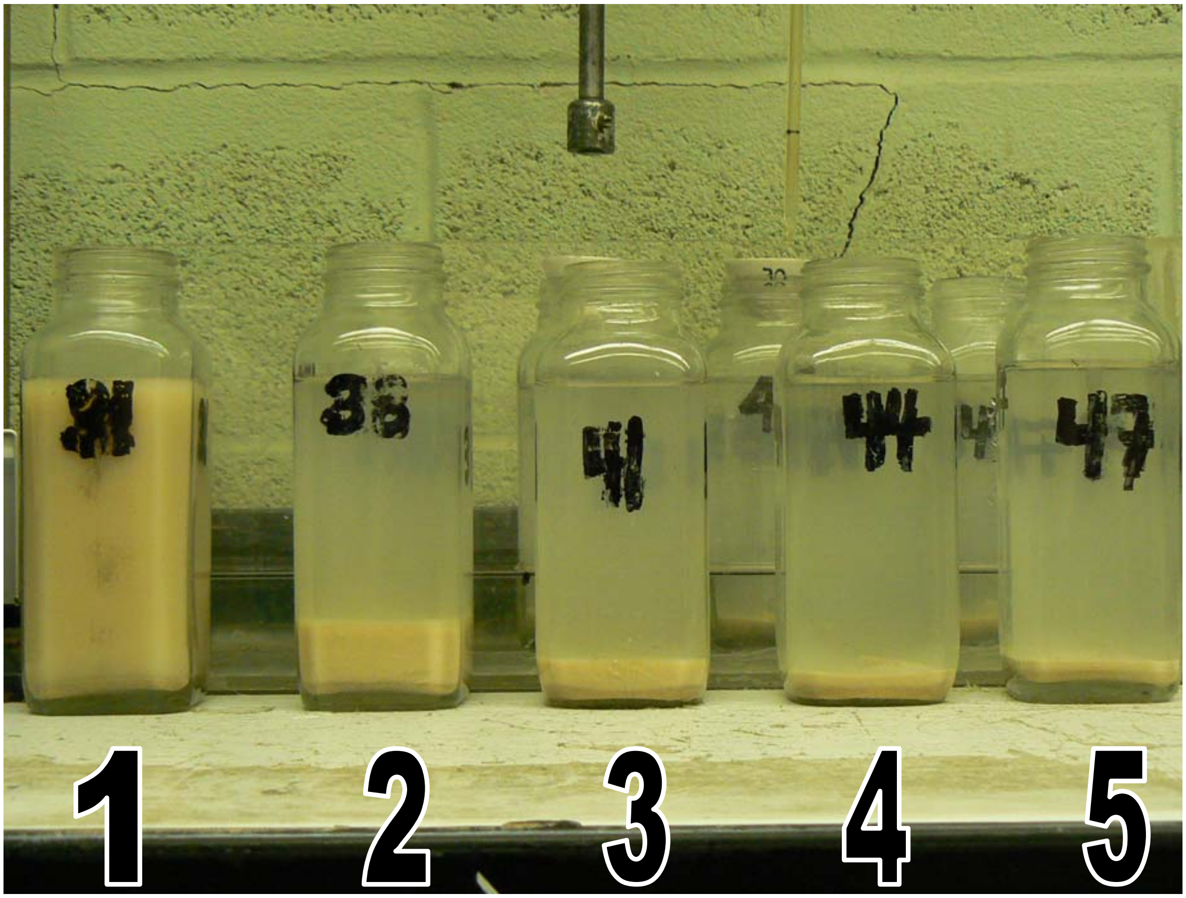


Fig. 2.1. Photograph of sample “suspensions” at the time to withdraw the <2 µ fraction by pipette. Sample treatment followed Procedure One. Prepared samples 1 through 5 are given in Table 2.1.

corresponding numbers and sample information that is represented in Fig. 2.1. Flocculation was slightly less in the second sample containing only 1 g of gypsum, but still substantial flocculation was evident. Also, the $BaCl_2$ solution did not have an effect on prepared sample 1 that contained no gypsum as the sample seemed to disperse properly. In procedure one the samples were washed in the $BaCl_2$ solution only once, but for 1 h duration. As flocculation was evident with one washing, it was hypothesized that multiple washings for shorter periods of time might result in a better coating of $BaSO_4$ around gypsum particles.

Procedure Two

Results of this method were much like procedure one in respect to clay flocculation of the samples that contained gypsum. Once again, the first pipette aliquot was taken (<20µ fraction), but the second pipetting (<2 µ fraction) was not. Like the previous result, flocculation was evident at the time for the second pipetting. There was slight dispersion of the second sample containing only 1 g of gypsic material. Figure 2.2 shows flocculation of the samples at the time for obtaining the second pipette aliquot (<2 µ fraction), and Table 2.1 provides the corresponding numbers and information for each sample. Because there was evidence of slight dispersion of the sample containing 1 g of gypsic material, it was hypothesized that an increase in the number of $BaCl_2$ washings and a decrease in the amount of time of each could possibly help with the dispersion of the samples containing gypsum.

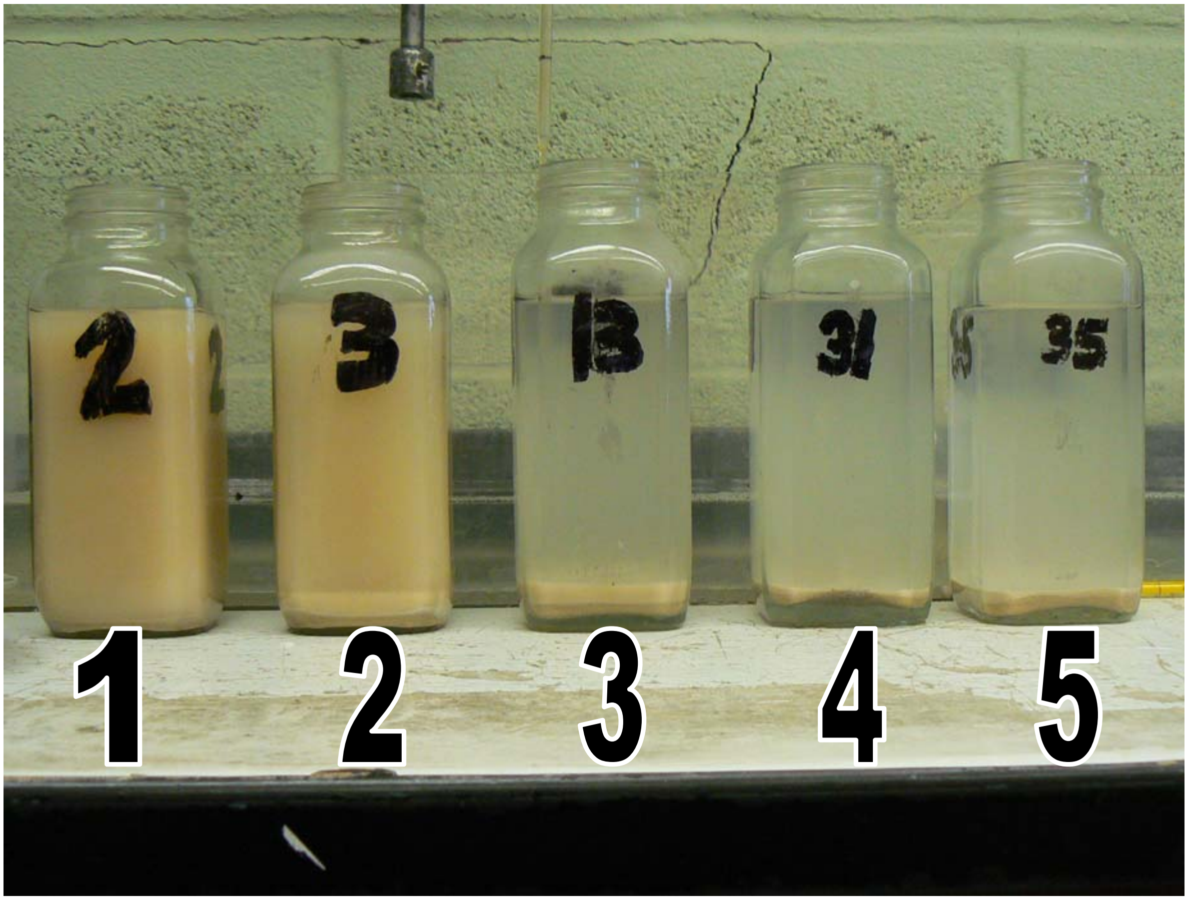


Fig. 2.2. Photograph of sample “suspensions” at the time to withdraw the <2 μ fraction. Sample treatment followed Procedure Two. Composition of prepared samples 1 through 5 are given in Table 2.1.

Procedure Three

Procedure three showed visual evidence of flocculation much like the previous procedures. Sample two showed a small reduction in flocculation, as it only contained 1g of gypsic material. This procedure, unlike the last, had five $BaCl_2$ washings. It was believed that if three washings aided in the reduction in flocculation of the sample containing 1 g of gypsic material, then an increase in the number of washing might reduce the flocculation of samples containing more gypsic material by forming a better protective coating around the gypsum crystals. The additional washings did not reduce flocculation, and results were similar to those of procedure two where only 3 washings were conducted. Like the other procedures, the first pipette aliquot was taken (<20 µ fraction), but the second (<2 µ fraction) was not, due to the obvious flocculation. Figure 2.3 is provided to show flocculation of the samples at the time to obtain the second pipette aliquot (<2 µ fraction). Again, Table 2.1 gives the corresponding sample numbers and sample information. Unlike the previous two procedures, the decantate of each of the five washings for each sample was collected, and the Ca content was determined by atomic absorption spectroscopy. Theoretically, the Ca content of each decantate should decrease and approach zero if adequate coatings of $BaSO_4$ were forming on the gypsum particle surfaces. Results from the decantate are given in Table 2.2 and graphed in Fig. 2.4. Figure 2.4 clearly shows that washings for samples 1 and 2 reach the same level of Ca in the fourth and fifth washings and this is why some dispersion of these samples was successful.

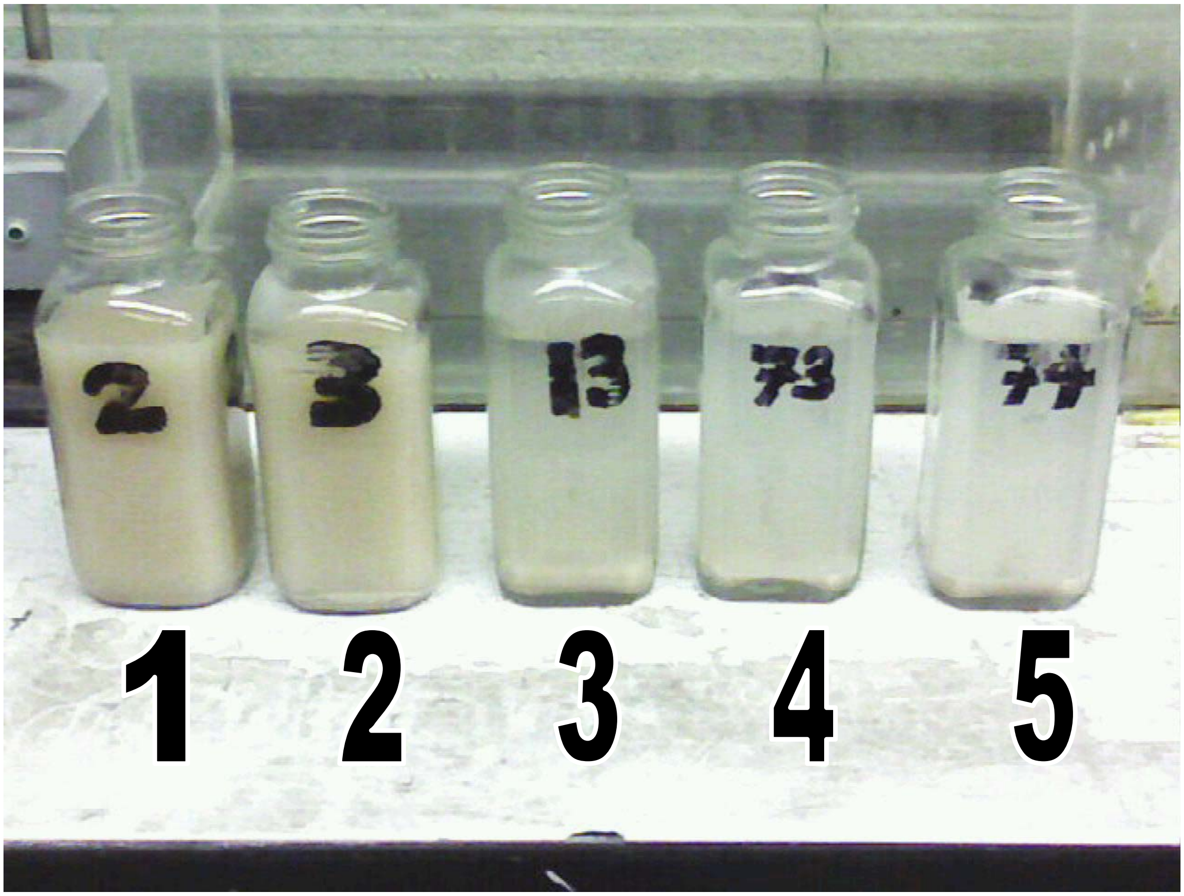


Fig. 2.3. Photograph of sample “suspensions” at the time to withdraw the <2 µ fraction pipetting. Sample treatment followed Procedure Three. Composition of prepared samples 1 through 5 are given in Table 2.1.

Table 2.2. Ca^{+2} concentration in decantates of each $BaCl_2$ washing.

Sample Number	Sample (g)	Gypsic Material (g)	Number of Washings with BaCl2	Ca^{+2} Content (mmol(+)/40 ml)	Maximum * Gypsum Removed (g)
1	10	0	1	0.82	0.071
			2	0.20	0.017
			3	0.10	0.009
			4	0.24	0.021
			5	0.22	0.019
2	9	1	1	1.60	0.138
			2	0.58	0.050
			3	0.28	0.024
			4	0.22	0.019
			5	0.22	0.019
3	8	2	1	2.60	0.224
			2	0.98	0.084
			3	0.52	0.045
			4	0.40	0.034
			5	0.34	0.029
4	6	4	1	4.20	0.361
			2	1.40	0.120
			3	1.02	0.088
			4	0.74	0.064
			5	0.64	0.055
5	4	6	1	5.40	0.464
			2	2.40	0.206
			3	1.60	0.138
			4	1.36	0.117
			5	1.02	0.088

* Assuming all Ca^{+2} is from gypsum.

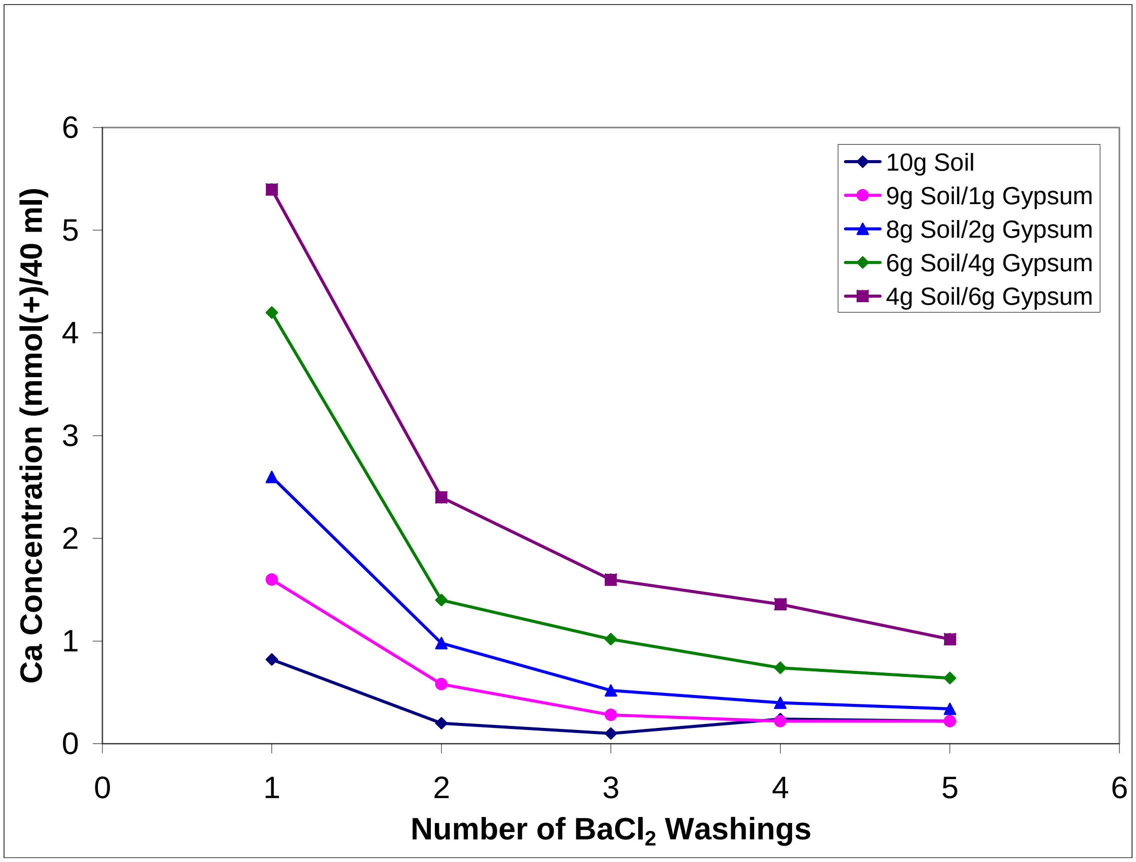


Fig. 2.4. Ca^{+2} content of the decantate of each $BaCl_2$ washing for the five prepared samples studied.

If one assumed a gypsum content of 1g in sample 2, the Ca in the gypsum would be 11.6 mmol (+). Complete dispersion of sample 2 does not occur, but the $BaSO_4$ coatings around gypsum particles reduces the rate of dissolution. There are sufficient coatings of gypsum particles at the time of the first pipetting to increase the dispersion of clays, but at the time for withdrawing the second aliquot (approximately 4 h) there is enough Ca^{+2} in solution to cause flocculation of clay-sized particles. Although sample 2 had increased dispersion, sample 3 through 5 did not disperse therefore, the $BaCl_2$ washings did not aide in the reduction of flocculation of samples containing large amounts of gypsum.

Procedure Four

Procedure four was much like procedure two except that the buffering agent TEA was added to the $BaCl_2$ solution used to wash the samples. In procedure four the samples were washed three times with the TEA- $BaCl_2$ solution as procedure three showed an increase in the number of washings with the $BaCl_2$ solution did not result in a decrease in flocculation of the samples containing gypsum. The TEA-$BaCl_2$ solution did not decrease flocculation of sample two, which only contained 1 g of gypsum. Like all other procedures evaluated, the first pipette aliquot was taken (<20 µ fraction), but the second aliquot (<2 µ fraction) was not due to flocculation of clays. Figure 2.5 is shows the flocculation of the samples at the time to withdraw the second aliquot (<2 µ fraction). Again Table 2.1 provides the corresponding sample numbers and sample information.

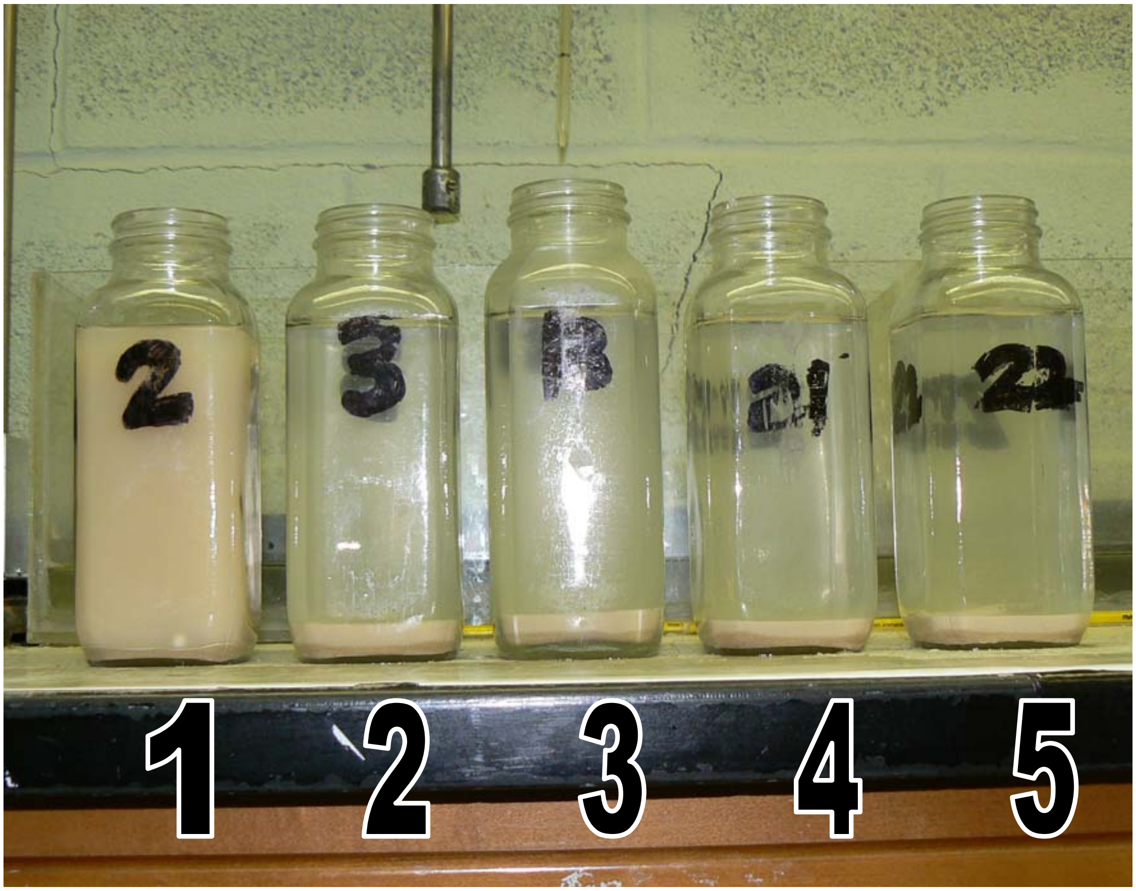


Fig. 2.5. Photograph of sample "suspensions" at the time to withdraw the <2 µ fraction pipetting. Sample treatment followed Procedure Four. Composition of prepared samples 1 through 5 are given in Table 2.1.

Conclusions

The $BaCl_2$ method suggested in the literature by Hesse (1976) showed little promise for obtaining a particle size analysis in the presence of gypsum. Of the four procedures evaluated, procedure two and procedure three reduced flocculation in only sample two, the sample that contained 1 g of gypsic material. There was no visual evidence of reduction in flocculation in any of the other procedures evaluated. Length of shaking with the $BaCl_2$ solution did not significantly change dispersion of samples. Similarly, three $BaCl_2$ washings instead of one proved to marginally increase dispersion of only sample two. Five $BaCl_2$ washings proved to be no better than three as the additional two washings in procedure three did not reduce flocculation of samples 3 through 5. In procedure four the addition of TEA to the $BaCl_2$ solution did not result in a visual reduction in flocculation of any samples containing gypsum. It is evident in all procedures evaluated that adequate coatings of $BaSO_4$ around the gypsum particles were not obtained in samples containing greater than 1 g (>10%) of gypsic material. This is apparent in Fig. 2.4 where Ca^{+2} content in decantates of each sample does not approach that of sample 1 in subsequent washings of $BaCl_2$. All four procedures evaluated produced unsatisfactory results and do not support the findings of Hesse (1976).

CHAPTER III

LASER DIFFRACTION PARTICLE SIZE ANALYZER

Introduction

The classical techniques for determining PSD in soils include sieving and procedures based on sedimentation, such as the pipette and hydrometer methods (Gee and Bauder, 1986). The pipette and hydrometer methods give comparable results (Liu et al., 1966, Walter et al., 1978) which are typically presented as percentage of the total mass of soil occupied by a given fraction (Eshel et al., 2004). Both pipette and hydrometer methods are lengthy and time consuming. As presented in Chapter II, when analyzing gypseous soils by sedimentation, the gypsum must first be removed by pretreatment either by dialysis (Rivers et al., 1982) or repeated washings (Lovelady, 1974). Since many soils contain high quantities of gypsum, it is imperative to develop a procedure which analyzes the entire sample, not just the non-gypseous fraction. For that reason, the laser diffraction particle size analyzer (LPSA) was evaluated as a way to determine PSD of gypseous soils.

The LPSA is a rapid way of determining PSD of samples and presents the PSD results on a percent volume basis. There are many advantages to using the LPSA to determine PSD of soils samples such as rapid analysis, high reproducibility, small sample size needed, and a wide range of size fractions into which the entire range of particle sizes can be divided (Eshel et al., 2004). The LPSA provides a continuous curve in which the user is provided a wide range of results that may be broken into many size fractions. The LPSA has been evaluated in numerous studies by researchers such as

Zobeck (2004), Eshel et al. (2004), Beuselinck et al. (1999), and Wen et al. (2002), but none evaluated the LPSA for gypseous soils.

The overall goal of this research was to evaluate the LPSA as a means of determining PSD of gypseous samples to include its precision, accuracy and reproducibility using methanol and gypsum saturated water with Na hexametaphosphate as solutions and to determine if a functional relationship exists between PSD results from the pipette method and results from the LPSA.

Materials and Methods

Laser Diffraction Particle Size Analyzer

The LPSA evaluated in this study was the Beckman-Coulter LS-230. This apparatus has a 750-nm laser beam that measures particles ranging in size 0.04 to 2000 µm in diameter. The laser beam accurately measures particles of an apparent cross-sectional diameter >0.4 µm (Buurman et al., 1997). For particles with diameters of <0.4 µm, the LS-230 uses the polarization intensity differential of scattered light system (PIDS), which uses polarized beams. The PIDS allows PSD analysis of smaller clay-sized particles. The Mie theory was used for PSD calculations. Eshel et al. (2004) states the Mie theory is a solution of the Maxwell equations (a set of four fundamental equations governing the behavior of electric and magnetic fields) describing propagation of the electromagnetic wave of light in space. The theory provides a solution for the case of a plane wave on a homogeneous sphere of any size (Jonasz, 1991). The Mie theory model requires the refractive index (RI) as an input parameter, which is a complex number comprised of a real part, which represents the change in the velocity of light through the test material compared with the velocity of light in vacuum and an imaginary term which represents the transparency and absorptivity of the material (Eshel et al., 2004). There is a real part for both the solution and the material being tested. In addition to the LPSA, there is a Sonics Vibra Cell ultrasonic processor that provides sonication to the samples. The device has three settings; amplitude, time and pulse. These settings were tested and selected to give the highest clay percentage of each sample. The sample was 0.2 g clay combined with 5 mL of methanol in a 40 mL centrifuge tube, shaken by hand, and added drop by drop to the LPSA. The sample was replicated 18 times first without sonication and then with sonication. Different sonication settings were used to determine the greatest increase in clay. The settings used were amplitude of 70, time of 1 min, and a pulse of 8 sec and used for each soil sample evaluated. Beckman Coulter LS version 3.19 software was used for the calculation of the PSD (Beckman Coulter Manual, 1994).

Methanol as Solution

Methanol was chosen as a solution for the LPSA because gypsum is not soluble in methanol. For this study, separate fractions were obtained from non-gypseous soil samples using sieving for sands and sand-subfractions, and sedimentation for silts and clays. For gypsum particles, a gypsic horizon of about 90% gypsum was separated into sand-subfractions by dry sieving. Fractionated sand, silt, and clay and sand-sized gypsum particles were combined to make 0.2 g samples of a known particle size class and analyzed using the LPSA. Two mL of methanol were added to each sample, the sample was then shaken by hand, and added drop by drop into the LPSA chamber for analysis. The amount of sample used to obtain a reading was dependent on the amount of each fraction in the sample. A reading without sonication was obtained and then sonication was applied using an amplitude of 70, time of 1 min, and a pulse of 8 sec. These sonication settings were used on all samples in this study. Once sonication was complete, the sample was analyzed again. Each sample was analyzed twice to observe reproducibility and the mean was taken of the two results. The real refractive index used for soil was 1.62 and the imaginary refractive index used was 0.1. The 0.1 was chosen because the samples had little pigmentation. The real refractive index for methanol as solution was 1.326 (Beckman-Coulter Manual, 1994). These manufactured samples results allowed an evaluation of both precision and accuracy of the LPSA.

Gypsum Saturated Water with Na Hexametaphosphate as Solution

The second solution evaluated using the LPSA was gypsum saturated water with Na hexametaphosphate. This solution was chosen as it should minimize dissolution of gypsum contained in soil samples as would methanol. Theoretically, if water is saturated with gypsum, gypsum in the sample should not dissolve in solution. Five g of analytical grade gypsum were equilibrated at least 4 h in 1 L of deionized water. The excess gypsum was removed by centrifugation to ensure that no gypsum particles were left in solution. Fifty g of Na hexametaphosphate were dissolved in the 1 L of gypsum saturated water to act as a dispersing agent in the solution. This solution was used throughout the procedure.

Both gypseous and non-gypseous samples were evaluated using gypsum saturated water. In the beginning, fractionated sand, silt, and clay were tested to determine if gypsum saturated water with Na hexametaphosphate was a feasible approach to PSD analysis of both gypseous and non-gypseous samples. The fractionated soil separates were combined to form 0.2-g samples and added to 2-mL of gypsum saturated water, shaken overnight on a reciprocating shaker, and added drop by drop to the LPSA. Results of each sample were obtained with and without sonication. Once the gypsum saturated water was determined to be feasible and provided promising results, 20 non-gypseous samples were evaluated. The 20 samples had previous PSD determined by the sieve-pipette method (Hallmark et al. 1986). Table 3.1 shows the origin and soil classification of each sample and Table 3.2 gives the PSD results determined by pipette of each sample evaluated. Ten g of each sample combined with 200 mL of gypsum saturated water were placed in a 400-mL square bottle, a stopper was placed in the top to assure no sample was lost, and shaken overnight on a reciprocating shaker. Once removed from the shaker, each sample was washed through a 300-mesh sieve with gypsum saturated water and the silt and clay fractions were collected. From the collected solution, two 10-mL aliquots were taken and placed in a 40 mL centrifuge tube and a cap was placed on each sample. Following hand shaking, samples were then added drop by drop to the LPSA. All samples analyzed using the LPSA for this procedure were analyzed on a sand-free basis. The first reading was analyzed without sonication, then sonication was applied to the sample and a second reading was taken. Each aliquot had two readings with and two readings without sonication. The results where sonication was performed were used when comparing the pipette method and the LPSA. The parameters used for the LPSA were a real refractive index for the solid

Table 3.1. Non-gypseous soil samples used to evaluate the LPSA.

Sample Number	Soil Horizon	Soil Series	Family
6421	Bt/E1	Kurth	Fine-loamy, siliceous, semiactive, thermic Oxyaquic Glossudalfs
6439	Bk3	Topsey	Fine-loamy, carbonatic, thermic Udic Calciustolls
6468	Bk1	Brackett	Loamy, carbonatic, thermic, shallow Typic Haplustepts
6474	Btk	Evant	Clayey, smectitic, thermic, shallow Petrocalcic Paleustolls
6481	Bk1	Krum	Fine, smectitic, thermic Udertic Haplustolls
6492	2Akb	Lewisville	Fine-silty, mixed, active, thermic Udic Calciustolls
6496	4BCkb1	Lewisville	Fine-silty, mixed, active, thermic Udic Calciustolls
6510	2Akb	Lewisville	Fine-silty, mixed, active, thermic Udic Calciustolls
6515	2Bkb2	Lewisville	Fine-silty, mixed, active, thermic Udic Calciustolls
6589	Bg2	Nona	Fine-silty, siliceous, active, thermic Natric Vermaqualfs
6590	Btg/Eg1	Nona	Fine-silty, siliceous, active, thermic Natric Vermaqualfs
6598	EB	Kenefick	Fine-loamy, siliceous, active, thermic Ultic Hapludalfs
6601	Btg3	Kenefick	Fine-loamy, siliceous, active, thermic Ultic Hapludalfs
6609	Btg/Eg1	Sorter	Coarse-loamy, siliceous, superactive, thermic Natric Vermaqualfs
6613	B't/E'g	Sorter	Coarse-loamy, siliceous, superactive, thermic Natric Vermaqualfs
6617	Bt/E1	Kirbyville	Fine-loamy, siliceous, semiactive, thermic Oxyaquic Paleudults
6628	Bt/Eg2	Waller	Fine-loamy, siliceous, semiactive, thermic Typic Glossaqualfs
6635	Btvc2	Hockley	Fine-loamy, siliceous, semiactive, hyperthermic Plinthic Paleudalfs
6639	Bt1	Prairieview	Fine-loamy, siliceous, semiactive, hyperthermic Oxyaquic Paleudalfs
6651	Bt	Snakecreek	Fine-loamy, siliceous, semiactive, hyperthermic Aquic Glossudalfs

Table 3.2. Texture of non-gypseous soil samples used to evaluate the LPSA. Analysis was performed by sieve-pipette method.

Lab Number	Texture	Clay	Silt	Sand
			----------------%-----------------	
6421	Sandy Clay Loam	24.6	14.7	60.7
6439	Silty Clay Loam	27.5	53.1	19.4
6468	Silty Clay	41.6	46.9	11.5
6474	Clay	47.5	21.7	30.8
6481	Clay Loam	37.3	29.5	33.2
6492	Clay	49.5	38.8	11.7
6496	Silt Loam	23.5	64.8	11.7
6510	Silty Clay	47.1	41.3	11.6
6515	Silty Clay Loam	36.5	46.5	17
6589	Silt Loam	7.3	52.7	40
6590	Loam	17.6	49.4	33
6598	Very Fine Sandy Loam	10.1	21.6	68.3
6601	Sandy Clay Loam	26.6	15.6	57.8
6609	Fine Sandy Loam	5.5	43.8	50.7
6613	Loam	10.8	40.5	48.7
6617	Loam	21.7	30.9	47.4
6628	Clay Loam	28.9	31.3	39.8
6635	Sandy Clay	35	18.4	46.6
6639	Fine Sandy Loam	18.6	20.9	60.5
6651	Sandy Clay	38.4	7.4	54.2

fraction of 1.54, real refractive index of solution of 1.33 for water (Beckman Coulter Manual, 1994) and an imaginary refractive index of 0.1 because of little pigmentation. Results of each sample were established from a continuous curve and broken into size classes primarily focusing on percent clay. The results from the LPSA were then compared to those of the pipette method expressed on a sand-free basis using regression analysis. From the results, an equation was developed to express the relationship of percent clay on a weight basis to the LPSA percent clay on a volume basis. The root mean squared deviation (RMSD), ratio of standard deviation (SD) to RMSD (RPD), and bias were calculated to compare the accuracy of the LPSA percent clay to pipette percent clay. Statistical formulas to calculate RMSD, RPD, and bias follow Gauch et al. (2003), Brown et al.(2005), and Chang et al. (2005:

$$\text{RMSD} = \sqrt{\sum_n \left(Y_{pred} - Y_{meas} \right)^2 / N}\ , \qquad [1]$$

$$\text{RPD} = \text{SD/RMSD, and} \qquad [2]$$

$$\text{Bias} = \sum_n \left(Y_{pred} - Y_{meas}\right) / N\ ; \qquad [3]$$

where Y_{pred} are predicted percent clay values from LPSA using Equation [4], Y_{meas} are percent clay values from the pipette method and N is the total number of samples analyzed.

Next, 21 gypseous samples were evaluated using the LPSA. Again, gypsum saturated water with Na hexametaphosphate was used as solution. Seventeen of the 21 samples had previous PSD analysis determined by the sieve-pipette method after dialysis to remove the gypsum (Rivers et al.,1982). The four samples without previous PSD were samples containing greater than 70% gypsum. Table 3.3 shows the gypseous soil samples that were used and their classification. Table 3.4 shows the PSD analysis by the pipette method and the gypsum content of each sample. Gypsum content was determined as described in procedure 6F1a in the Soil Survey Laboratory Methods Manual (1996). Ten g of each sample combined with 200 mL of gypsum saturated water were placed in a 400-mL square bottle, a stopper was placed in the top to assure no sample was lost, and shaken overnight in a reciprocating shaker. Once removed from the shaker, each sample was washed through a 300-mesh sieve with gypsum saturated water, and the silt and clay suspensions were collected. The sand fraction was washed again, this time with methanol, set aside to allow the methanol to evaporate, and then the sands were weighed. From the collected suspension, two 10-mL aliquots were taken and placed in a 40-mL centrifuge tube, and a cap was placed on each sample. The freshly hand shaken-suspensions were then added drop by drop to the LPSA chamber. All samples analyzed using the LPSA for this procedure were sand-free. The first reading was obtained without sonication, then sonication was applied to the sample, and a second reading was taken. Each aliquot had two readings with and without sonication. The results where sonication was performed were used when comparing the pipette method and the LPSA. The parameters used for the LPSA were a real refractive index for the solid fraction of 1.54, real refractive index of solution of 1.33 for water (Beckman Coulter Manual, 1994) and an imaginary refractive index of 0.1 for the lightly pigmented soil.

Table 3.3. Gypseous soil samples selected for evaluation of LPSA.

Gypsum Samples	Horizon	Soil Series	Family
4260	Bky1	Monahans	Coarse-loamy, mixed, superactive, thermic Typic Calcigypsids
4261	Bky2	Monahans	Coarse-loamy, mixed, superactive, thermic Typic Calcigypsids
4898	BCkssy	Houston Black	Fine, smectitic, thermic Udic Haplusterts
4899	BCy	Houston Black	Fine, smectitic, thermic Udic Haplusterts
4904	Bkss3	Houston Black	Fine, smectitic, thermic Udic Haplusterts
4905	Bssy	Houston Black	Fine, smectitic, thermic Udic Haplusterts
4906	BCssy	Houston Black	Fine, smectitic, thermic Udic Haplusterts
5020	Bsskn2	Cedarlake	Fine-loamy, mixed, superactve, calcareous, thermic Typic Halaquepts
6168	By1	Cepgenli (Turkey)	Fine, smectitic, thermic Aridic Haploxererts
6326	Btny1	Quiteria	Coarse-loamy, mixed, active, hyperthermic Typic Natrustalfs
6762	Bssy	Zilaboy	Fine, smectitic, thermic Oxyaquic Hapluderts
6850	Bz3	Melado *	Fine, smectitic, hyperthermic Typic Haplosalids
6864	Byz2	Changas *	Fine, smectitic, hyperthermic Leptic Haplogypsids
6868	Cydss1	Changas *	Fine, smectitic, hyperthermic Leptic Haplogypsids
6874	Bkyz1	Corrientes *	Fine, smectitic, hyperthermic Vertic Natrigypsids
6876	CBy	Corrientes *	Fine, smectitic, hyperthermic Vertic Natrigypsids
6891	Bkssy	Victoria	Fine, smectitic, hyperthermic Sodic Haplusterts
7171	By1	Pokorny	Gypsic, thermic Ustic Petrogypsids
7179	By2	Orla-like	Gypsic, thermic, shallow Ustic Petrogypsids
7181	By4	Orla-like	Gypsic, thermic, shallow Ustic Petrogypsids
7182	By5	Orla-like	Gypsic, thermic, shallow Ustic Petrogypsids

* Proposed series names

Table 3.4. Particle size and gypsum content of gypseous soil samples used to evaluate the LPSA.

Gypsum Samples	Texture	Sand	Silt	Clay	Gypsum
		-----------------------	%	------------------------	
4260	Loam	40.8	42.6	16.6	24.7
4261	Very Fine Sandy Loam	64.1	28	7.9	26.7
4898	Clay	2.7	28.5	68.8	13.5
4899	Clay	0.9	30.1	69	6.6
4904	Clay	3.85	31.8	64.4	9.3
4905	Clay	2.9	21.8	75.3	24.7
4906	Clay	1.47	23.8	74.8	23.7
5020	Silty Clay	7.9	44.2	47.9	3.2
6168	Silty Clay	6	41.5	52.8	8.9
6326	Sandy Clay Loam	71.2	3.3	25.5	0.5
6762	Clay	15.8	35.8	48.4	4.6
6850	Clay Loam	27.9	32.5	39.6	1.3
6864	Clay	18.7	27.6	53.7	5.4
6868	Clay	5.6	28	66.4	2
6874	Clay	17.3	32	50.7	11
6876	Clay	10.7	21.5	67.8	3.2
6891	Clay	14.9	22	63.1	6.2
7171	*	-	-	-	82
7179	*	-	-	-	83.7
7181	*	-	-	-	84.9
7182	*	-	-	-	78.4

* Samples contained high quanities of gypsum and were not subjected to dialysis and sieve-pipette analysis.

Results of each sample were established from a continuous curve and broken into size classes. The equation established from non-gypseous samples to relate LPSA volume % clay to pipette weight % clay was used to compare the expected percent clay on a sand-free basis from the pipette method to that of the LPSA, by entering the LPSA volume percent into the equation and establishing percent clay on a weight basis. With the weight of sand, results of PSD by LPSA for each sample was expressed on a total soil fines basis (<2 mm).

Results and Discussion

Methanol as Solution

The LPSA was extremely fast in producing results for PSD of each sample. The average processing time for a sample, including sonication and cleaning the chamber was approximately 15 min. This is significantly less time than required for the pipette method. The success of methanol as the solution phase depended upon the dispersability of silicate clay in methanol so samples containing 100% clay were evaluated to select the parameters that would give the greatest clay percentage. Initial results showed that even with sonication, methanol caused flocculation of clay, and results did not reflect 100% clay. Table 3.5 shows the results of the sonication experiment. The instruments settings to produce the greatest percentage of clay was determined to be an amplitude of 70, time of 1 min, and a pulse of 8 sec. These specific settings provided the greatest increase in clay content in comparison to the results of the sample without sonication, even though the target percentage of 100 was not reached.

Figure 3.1 is the continuous PSD curve provided by the Beckman Coulter software and shows the clay percentage under the curve in the <2 µm fraction to be 7.8% by volume which should be 100% for the clay fraction separated from sample 7062.

Table 3.5. Comparison of percent clay in methanol with and without sonication. The same 0.2 g of clay in 5 mL of methanol was used through out the experiment.

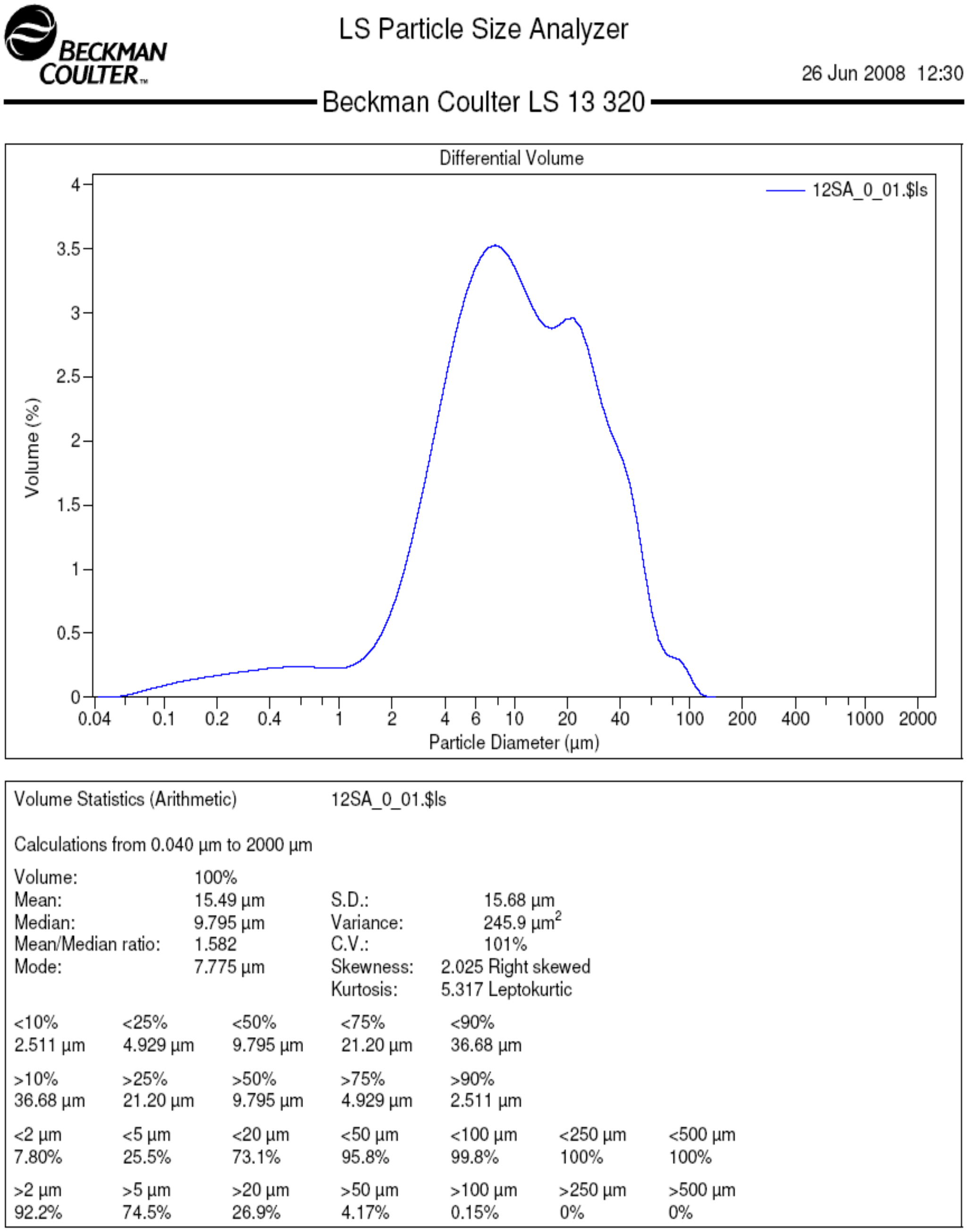
Lab Number	**Sample Run**	**Sonication**	**Amplitude**	**Time**	**Pulser**	**Clay Volume Basis**	*** Difference**
				min	**sec**	**%**	
7062	a	no				7.8	
7062	b	yes	40	1	2	12.3	4.5
7062	c	no				7.6	
7062	d	yes	50	1	2	15	7.4
7062	e	no				7.7	
7062	f	yes	60	1	2	17.9	10.2
7062	g	no				7.9	
7062	h	yes	65	1	3	17	9.1
7062	i	no				7.9	
7062	j	yes	70	1	4	19.8	11.9
7062	k	no				8.4	
7062	l	yes	70	1	6	20.5	12.1
7062	m	no				8.5	
7062	n	yes	70	1	8	23.3	14.8
7062	o	no				8.1	
7062	p	yes	70	1	10	18.5	10.4
7062	q	no				8.3	
7062	r	yes	70	2	10	20.2	11.9

* Percent clay with sonication minus percent clay without sonication.

As any PSD procedure for gypseous sample would necessitate analysis of clay-sized layer silicate particles, it was concluded that methanol was a poor liquid for dispersion of clays, even with sonication. However, in working with other samples fabricated with mixtures of sands and silts, difficulty was noted that in keeping the sand-sized fraction dispersed in the sample while adding drop by drop to the LPSA. Consequently, the decision was made to work only with sand-free samples to ensure that representative samples entered the chamber.

Gypsum Saturated Water with Na Hexametaphosphate as Solution

When evaluating gypsum saturated water containing Na hexametaphosphate, it was obvious that drawing and delivering a sample containing sands resulted in a non-representative sample as the sand particles would not stay in suspension. To illustrate this point, Fig. 3.2 shows the results of a sample containing sand-sized particles (0.06-g clay, 0.06-g silt and 0.07-g fine sand). The resulting PSD showed 98.2 % by volume of silt and clay-sized particles, although sands comprised 37% by weight of the sample. Again, it was concluded that sands must be removed before PSD analysis of the silt and clay fractions. Figure 3.3 is the PSD for the clay fraction of sample 7075 in gypsum saturated water. The sample containing 100% clay showed a 93.3% by volume result in gypsum saturated water compared to only 23% using methanol.



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LS Particle Size Analyzer
                                                        26 Jun 2008  12:30
Beckman Coulter LS 13 320

Volume Statistics (Arithmetic)          12SA_0_01.$ls

Calculations from 0.040 µm to 2000 µm

Volume:              100%
Mean:                15.49 µm       S.D.:           15.68 µm
Median:              9.795 µm       Variance:       245.9 µm²
Mean/Median ratio:   1.582          C.V.:           101%
Mode:                7.775 µm       Skewness:   2.025 Right skewed
                                    Kurtosis:   5.317 Leptokurtic

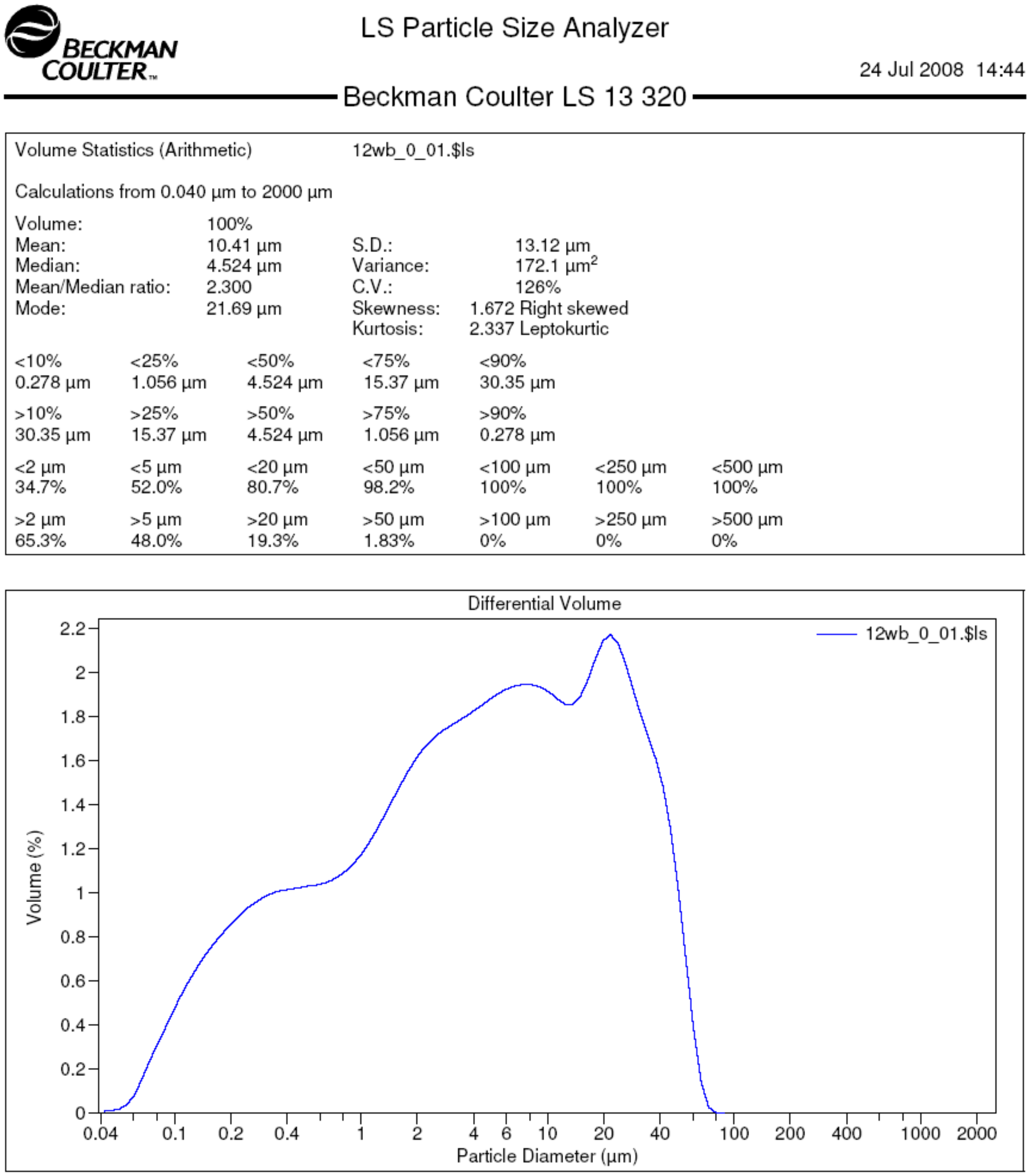
<10%       <25%       <50%       <75%       <90%
2.511 µm   4.929 µm   9.795 µm   21.20 µm   36.68 µm

>10%       >25%       >50%       >75%       >90%
36.68 µm   21.20 µm   9.795 µm   4.929 µm   2.511 µm

<2 µm      <5 µm      <20 µm     <50 µm     <100 µm    <250 µm    <500 µm
7.80%      25.5%      73.1%      95.8%      99.8%      100%       100%

>2 µm      >5 µm      >20 µm     >50 µm     >100 µm    >250 µm    >500 µm
92.2%      74.5%      26.9%      4.17%      0.15%      0%         0%
```

Fig. 3.1. Particle size distribution of the clay fraction of sample 7062 using methanol as the solution phase. Sonication was performed with an amplitude of 70, time of 1 min, and pulse of 8 sec.



LS Particle Size Analyzer

24 Jul 2008 14:44

Beckman Coulter LS 13 320

Volume Statistics (Arithmetic) 12wb_0_01.$ls

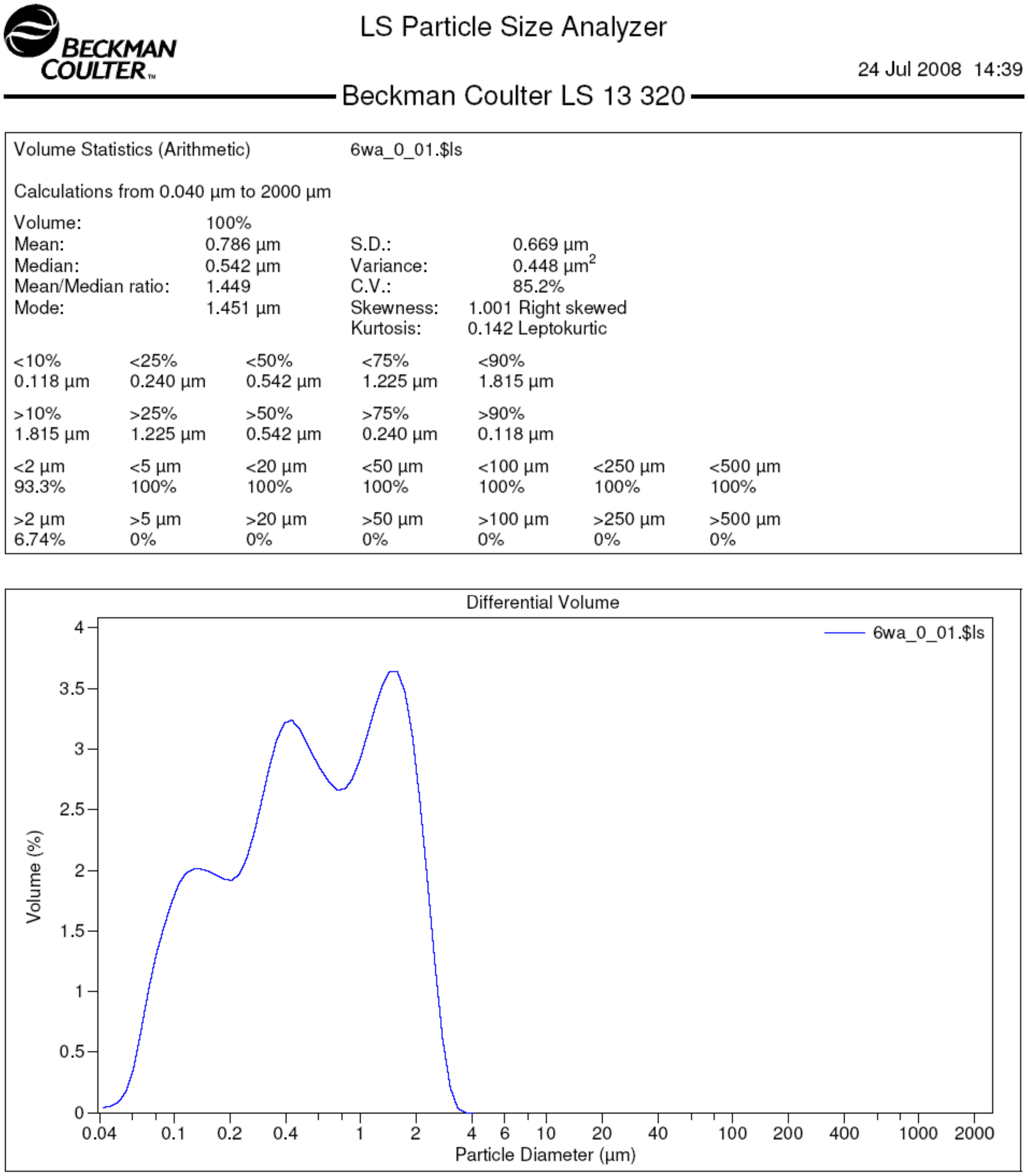
Calculations from 0.040 µm to 2000 µm

Volume:	100%		
Mean:	10.41 µm	S.D.:	13.12 µm
Median:	4.524 µm	Variance:	172.1 µm^2
Mean/Median ratio:	2.300	C.V.:	126%
Mode:	21.69 µm	Skewness:	1.672 Right skewed
		Kurtosis:	2.337 Leptokurtic

<10%	<25%	<50%	<75%	<90%
0.278 µm	1.056 µm	4.524 µm	15.37 µm	30.35 µm
>10%	>25%	>50%	>75%	>90%
30.35 µm	15.37 µm	4.524 µm	1.056 µm	0.278 µm

<2 µm	<5 µm	<20 µm	<50 µm	<100 µm	<250 µm	<500 µm
34.7%	52.0%	80.7%	98.2%	100%	100%	100%
>2 µm	>5 µm	>20 µm	>50 µm	>100 µm	>250 µm	>500 µm
65.3%	48.0%	19.3%	1.83%	0%	0%	0%

Fig. 3.2. Particle size distribution for a fabricated sample containing 0.06-g clay, 0.06-g silt, and 0.07-g fine sand. This sample was analyzed using the LPSA with sonication and gypsum saturated water with Na hexametaphosphate as solution.



LS Particle Size Analyzer

24 Jul 2008 14:39

Beckman Coulter LS 13 320

Volume Statistics (Arithmetic) 6wa_0_01.$ls

Calculations from 0.040 µm to 2000 µm

Volume:	100%		
Mean:	0.786 µm	S.D.:	0.669 µm
Median:	0.542 µm	Variance:	0.448 μm^2
Mean/Median ratio:	1.449	C.V.:	85.2%
Mode:	1.451 µm	Skewness:	1.001 Right skewed
		Kurtosis:	0.142 Leptokurtic

<10%	<25%	<50%	<75%	<90%
0.118 µm	0.240 µm	0.542 µm	1.225 µm	1.815 µm

>10%	>25%	>50%	>75%	>90%
1.815 µm	1.225 µm	0.542 µm	0.240 µm	0.118 µm

<2 µm	<5 µm	<20 µm	<50 µm	<100 µm	<250 µm	<500 µm
93.3%	100%	100%	100%	100%	100%	100%

>2 µm	>5 µm	>20 µm	>50 µm	>100 µm	>250 µm	>500 µm
6.74%	0%	0%	0%	0%	0%	0%

Fig. 3.3. Particle size distribution for sample 7075 containing 0.2-g clay. This sample was analyzed using the LPSA with sonication and gypsum saturated water with Na hexametaphosphate as solution.

The < 50 µm fraction (silt and clay) of twenty non-gypseous samples were analyzed using the LPSA and gypsum saturated water. Particle size distributions from the silts and clays collected after washing through a 300-mesh sieve are given in Appendix A, and the clay volume percentages were used to establish a relationship with the clay percentage by weight on a sand-free basis from the pipette method (Fig. 3.4). Data in Fig. 3.4 shows the relationship between the pipette weight percent clay and the LPSA volume percent clay is linear and can be expressed as

$$y = 1.37x + 2.03 \qquad [4]$$

where x is the LPSA clay fraction in volume % and y is the pipette clay fraction as weight %. The equation has a R^2 value of 0.84 at a significance level of 0.01.

Different refractive indices (RI) for the imaginary part were evaluated in relation to the color of the samples using the moist color value of the Munsell Soil Color Charts. As the sample color became darker (lower value), the imaginary RI was assigned a greater value up to a maximum value of 0.4. After evaluating the relationship of the clay percentage by the pipette method on a sand-free weight basis and the LPSA clay percentage sand-free volume basis using different RI values, it was concluded that 0.1 for the imaginary RI resulted in the best linear relationship and gave the greatest R^2 value. Therefore, equation [4] established from the relationship of the pipette and LPSA methods was used to convert the percent clay on a volume basis from the LPSA to percent clay on a weight basis. This allowed LPSA results to be compared to those results of the pipette method, all on a sand-free basis (Table 3.6) and as weight %. The expected percent clay on a sand-free basis is the percent clay by pipette on a weight basis calculated without the sand fraction. The predicted clay percentage by weight was obtained using Equation [4] from the percent clay by volume from the LPSA. The RMSD of percent clay on a sand-free basis was 7.59% with a bias of 0 and RPD value of 2.57%. From the RMSD it is concluded that 2/3 of the time, LPSA will to predict clay within 8% of the clay percentage determined by the pipette method.

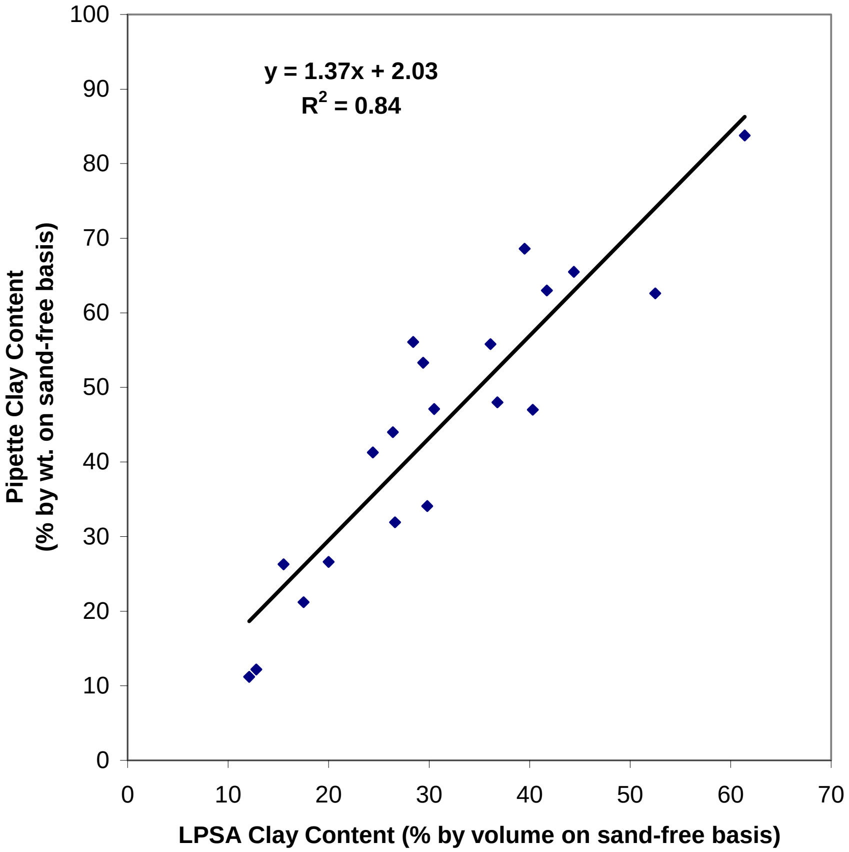


Fig. 3.4. The relationship of clay content by LPSA and pipette, both on a sand-free basis using selected soil samples that are non-gypseous.

Table 3.6. Comparison of clay percentage results of the pipette method and the clay percentage results of the LPSA.

Sample Number	**Clay by Pipette by Weight**	**Clay from LPSA by Volume**	**Expected Clay on Sand-Free Basis**	**Predicted Clay by Weight ***	**** Difference**
			%		
6421	24.6	52.5	62.6	74.1	-11.5
6439	27.5	29.8	34.1	42.9	-8.8
6468	41.6	40.3	47.0	57.3	-10.3
6474	47.5	39.5	68.6	56.2	12.4
6481	37.3	36.1	55.8	51.6	4.2
6492	49.5	28.4	56.1	41.0	15.1
6496	23.5	20.0	26.6	29.5	-2.9
6510	47.1	29.4	53.3	42.4	10.9
6515	36.5	26.4	44.0	38.3	5.7
6589	7.3	12.8	12.2	19.6	-7.4
6590	17.6	15.5	26.3	23.3	3.0
6598	10.1	26.6	31.9	38.5	-6.6
6601	26.6	41.7	63.0	59.2	3.8
6609	5.5	12.1	11.2	18.6	-7.4
6613	10.8	17.5	21.2	26.0	-4.8
6617	21.7	24.4	41.3	35.5	5.8
6628	28.9	36.8	48.0	52.5	-4.5
6635	35.0	44.4	65.5	62.9	2.6
6639	18.6	30.5	47.1	43.9	3.2
6651	38.4	61.4	83.8	86.3	-2.5

* By equation [4]

** Expected % clay on a sand-free basis minus predicted clay % by weight.

Twenty-one gypseous samples were subjected to PSD analysis by LPSA using gypsum saturated water with Na hexametaphosphate. Although the samples were analyzed on a sand-free basis, results are expressed on a total soil fines basis. The percent clay by volume from the LPSA was converted to percent clay by weight using equation [1] established in the evaluation of non-gypseous samples. Table 3.7 shows the comparison of percent sand from the pipette method and the percent sand washed with gypsum saturated water and then methanol on a total weight basis. It is important to note that the results from the pipette method are after dialysis and removal of gypsum particles, so it is expected that the sand percentages should not be identical as gypsum could be in the sand, the silt fraction, or clay, or all. Table 3.7 suggests the difference in sand percentages washed in methanol and the pipette sand percentages are greatest in samples with higher gypsum content. This suggests the fraction (sand or silt) that gypsum was dominant. It is expected that samples with larger amounts of gypsum would differ the greatest in percent sand if the most of the gypsum particles were sand-sized. This is because sand percentages from the pipette method are evaluated after all gypsum is removed from the sample by dialysis.

Table 3.8 presents the comparison of percent silt from the pipette method and the LPSA method on a total weight basis. Again, the gypsum was removed by dialysis (Rivers et al.,1982) in the pipette method and percentages are not expected to be the same in the two procedures evaluated. If samples contain larger quantities of gypsum and the gypsum particles are primarily silt-sized, then it is expected that silt content would be greatest in the LPSA silts as compared to those of the pipette method.

Table 3.7. Comparison of percent sand from the LPSA and pipette methods on a total weight basis.

Gypsum Samples	**Gypsum Content**	**LPSA Sand**	**Pipette Sand**	*** Difference**
	----------------------------------	------------%	------------	----------------------------------
4260	24.7	30.6	40.8	-10.2
4261	26.7	46.0	64.1	-18.1
4898	13.5	2.1	2.7	-0.6
4899	6.6	1.0	0.9	0.1
4904	9.3	2.9	3.9	-1.0
4905	24.7	1.6	2.9	-1.3
4906	23.7	5.9	1.5	4.4
5020	3.2	7.0	7.9	-0.9
6168	8.9	5.7	6.0	-0.3
6326	0.5	69.6	71.2	-1.6
6762	4.6	14.1	15.8	-1.7
6850	1.3	25.6	27.9	-2.3
6864	5.4	14.2	18.7	-4.5
6868	2	4.5	5.6	-1.1
6874	11	14.2	17.3	-3.1
6876	3.2	8.7	10.7	-2.0
6891	6.2	12.1	14.9	-2.8
7171	82	7.2	-	-
7179	83.7	9.5	-	-
7181	84.9	15.5	-	-
7182	78.4	35.1	-	-

* LPSA sand minus pipette sand

Table 3.8. Comparison of percent silt from the LPSA and pipette methods on a total weight basis.

Gypsum Samples	LPSA Silt	Pipette Silt	* Difference
	------------------------	%	--------------------------
4260	36.3	42.6	-6.3
4261	39.2	28	11.2
4898	33.4	28.5	4.9
4899	27.5	30.1	-2.6
4904	43.3	31.8	11.5
4905	40.1	21.8	18.3
4906	34.5	23.8	10.7
5020	38.8	44.2	-5.4
6168	40.8	41.5	-0.7
6326	-8.5	3.3	-11.8
6762	47.0	35.8	11.2
6850	30.6	32.5	-1.9
6864	26.3	27.6	-1.3
6868	21.7	28	-6.3
6874	20.9	32	-11.1
6876	16.0	21.5	-5.5
6891	20.9	22	-1.1
7171	73.0	-	-
7179	76.5	-	-
7181	71.4	-	-
7182	41.3	-	-

*LPSA silt minus pipette silt.

Table 3.9 compares of results of percent clay on a total weight basis of the pipette method and the LPSA method. It should be noted that samples 7171, 7179, 7181 and 7182 did not have clay percentages from the pipette method because of the high gypsum content in the sample. It was not meaningful to conduct PSD analysis on these samples after the pretreatment process as the undissolved residue would not be representative of the properties of the sample. Therefore the only results presented are from the evaluation of the LPSA.

Conclusions

The use of the laser diffraction particle size analyzer can be beneficial in many ways. It is produces rapid results, that can be expressed as many different size classes, and a very small sample is required for analysis. This small sample necessitates that a representative sample is analyzed. This was accomplished in the study with gypsum saturated water as a 10-g sample was first taken and then a 10-mL aliquot was taken from that sample after sands had been removed. This was also accomplished in the methanol study because samples were already fractionated and a sample of known composition fabricated.

It was concluded from the procedure utilizing methanol that samples should be analyzed on a sand-free basis because sand-sized particles did not remain suspended in solution when transferring the sample drop by drop into the LPSA chamber. The use of methanol as a solution was good for samples that contained gypsum as gypsum is insoluble in methanol, but in the case of samples containing silicate clay, flocculation occurred.

Table 3.9. Comparison of pipette percent clay and LPSA percent clay on a total weight basis. LPSA percent clay was converted from percent volume by using the equation established in the non-gypseous soil study.

Gypsum Samples	LPSA Clay	Pipette Clay	* Difference
	---------------------	%	---------------------
4260	33.1	16.6	16.5
4261	14.8	7.9	6.9
4898	64.5	68.8	-4.3
4899	71.5	69.0	2.5
4904	53.8	64.4	-10.6
4905	58.3	75.3	-17.0
4906	59.6	74.8	-15.2
5020	54.2	47.9	6.3
6168	53.5	52.8	0.7
6326	38.9	25.5	13.4
6762	38.9	48.4	-9.5
6850	43.8	39.6	4.2
6864	59.5	53.7	5.8
6868	73.8	66.4	7.4
6874	64.9	50.7	14.2
6876	75.3	67.8	7.5
6891	67.0	63.1	3.9
7171	19.8	-	-
7179	14.0	-	-
7181	13.1	-	-
7182	23.6	-	-

* LPSA clay minus pipette clay.

Thus, methanol showed little promise for samples that contain gypsum and silicate clay so efforts were focused on gypsum saturated water containing Na hexametaphosphate as the solution phase.

In the evaluation of gypsum saturated water containing Na hexametaphosphate, samples should be analyzed on a sand-free basis as separation by settling occurred when transferring the sample to the LPSA chamber. An acceptable R^2 value (0.84) was established relating clay percentages on a sand-free basis of non-gypseous samples from the pipette method (weight %) and from the LPSA method (volume %). With this equation a comparison between pipette analysis and LPSA clay percentages of gypseous soils was made. It should be noted that the clay percentages from the pipette method were performed on samples in which a pretreatment process had occurred and all gypsum was removed from the sample prior to PSD analysis. Results show that a difference in percent sand, silt and clay does occur. This difference is more prominent in different size classes depending on where gypsum particles dominated.

CHAPTER IV

SUMMARY AND CONCLUSIONS

Particle size distribution of gypseous soils is important in the soil science community. Many of the soil areas remaining to be mapped in the U.S. are in the southwest where gypsum is abundant and gypsum particles in the soil will affect the chemical and physical properties of the soil. When gypsum constitutes a major portion of the soil, its removal prior to PSD analysis distorts the results and may lead to textures that do not relate well to conditions in the field. In order to understand the true characterization of the soil and the gypsum particles, the entire soil sample should be analyzed.

The $BaCl_2$ method presented in the literature by Hesse (1976), Matar and Douleimy (1978), and Vieillefon (1979) was evaluated. Results showed that the $BaCl_2$ method was unsatisfactory as dispersion of clays was not accomplished. Length of shaking with $BaCl_2$ solution did not significantly increase dispersion of samples. Also, increasing the number of washings with $BaCl_2$ from one to three increased the dispersion of the sample with low quantities (10%) of gypsum marginally, but did not have an affect on samples containing greater quantities (>10%) of gypsum. Five $BaCl_2$ washings provided the same results as three washings. One may conclude the additional two washings did not increase the effectiveness of the $BaSO_4$ coating around the gypsum particle. Adding TEA to the $BaCl_2$ solution did not increase dispersion as judged by visual flocculation of samples. All $BaCl_2$ procedures evaluated resulted in insufficient coatings of $BaSO_4$ leading to the conclusion that the $BaCl_2$ method should not be used to determine particle size distribution of gypseous samples.

The laser diffraction particle size analyzer has numerous advantages as it produces results rapidly. The average time to run a sample including sonication and cleaning of the chamber was about 15 min. This is a significantly shorter time than needed for results using the pipette or hydrometer methods. It also requires a very small amount of sample compared to pipette and hydrometer methods which require from 10 to 100-g samples. Particle sizes can be broken into numerous precise classes, over a range of 0.04 µm to 2000 µm. Disadvantages of a LPSA include the cost of the instrument and the uncertainty in selection of input parameters that are associated with obtaining the best results of a sample.

In the evaluation of methanol as solution, it was observed that methanol causes flocculation of clays, and flocculation could not be controlled through sonication. Therefore, methanol should not be used to analyze soil samples containing silicate clays using the LPSA. The use of sonication increased the clay percentage in samples containing 100 % clay, although flocculation still occurred. The best settings found for the sonication were an amplitude of 70, a time of 1 min, and a pulse every 8 sec. It was important in this study that sonication be administered to all samples because clay percentage was an important aspect of each sample analyzed.

It was concluded while evaluating the gypsum saturated water containing Na hexametaphosphate, that samples should be analyzed on a sand-free basis. An equation $y = 1.37 x + 2.03$ was established relating the LPSA clay percent by volume (x) to the pipette clay percentage by weight (y). The equation had a R^2 of 0.84 and was significant at the 1% level. From this equation a comparison could be made between clay percentages of the pipette method and the LPSA method for non-gypseous and gypseous soils.

As the LPSA is an emerging application in soil science, it is important for more comparisons of the LPSA procedure to be made with both the pipette method and the hydrometer method using a wide variety of soils. It appears from this study that some promise exists for application to highly gypseous soils that will give information on PSD that is more meaningful than PSD on residue after gypsum is removed.

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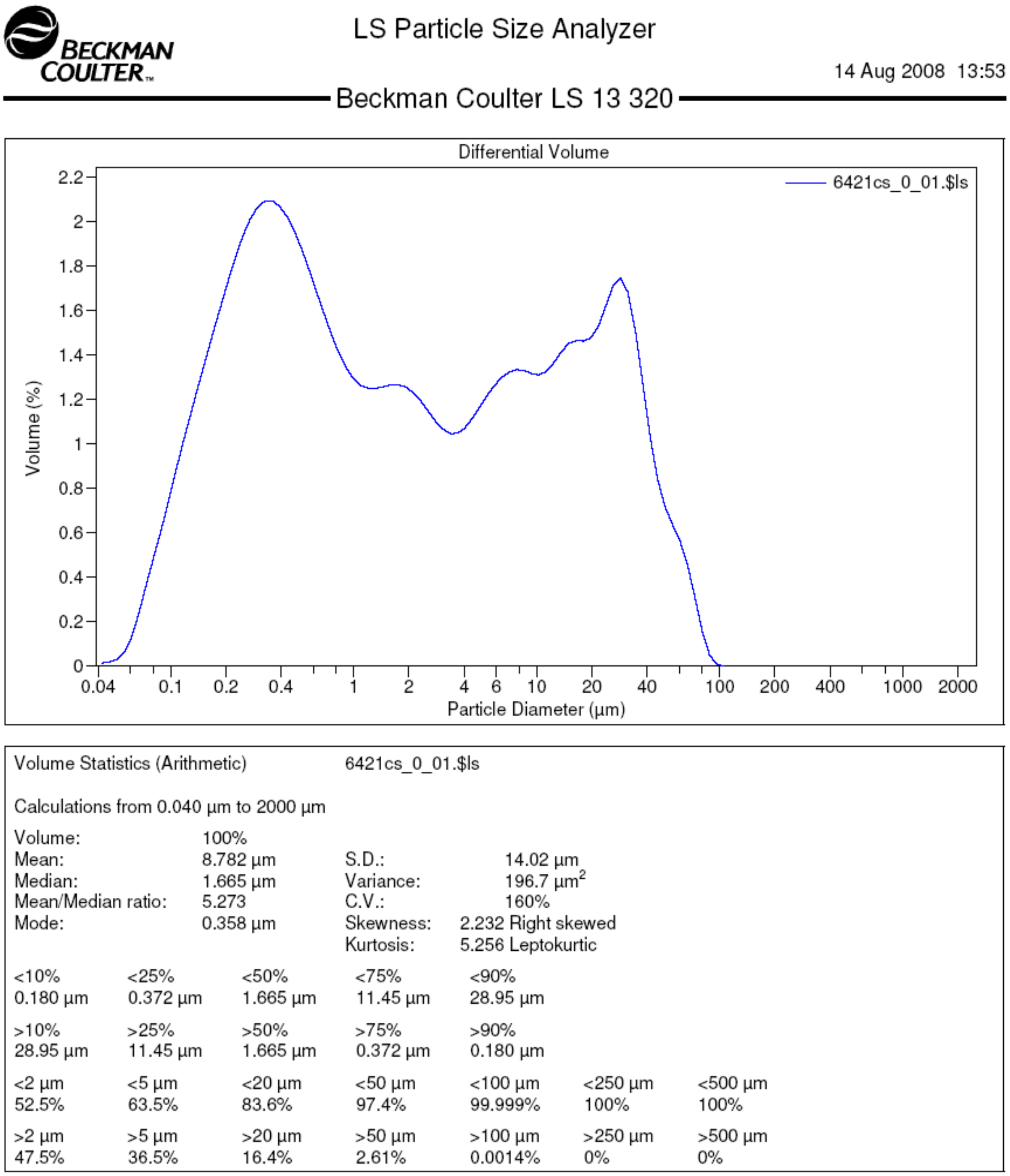
APPENDIX A

BECKMAN COULTER LASER PARTICLE SIZE ANALYZER GRAPHS

(NON-GYPSEOUS SAMPLES)



BECKMAN COULTER

LS Particle Size Analyzer

14 Aug 2008 13:53

Beckman Coulter LS 13 320

Volume Statistics (Arithmetic) 6421cs_0_01.$ls

Calculations from 0.040 µm to 2000 µm

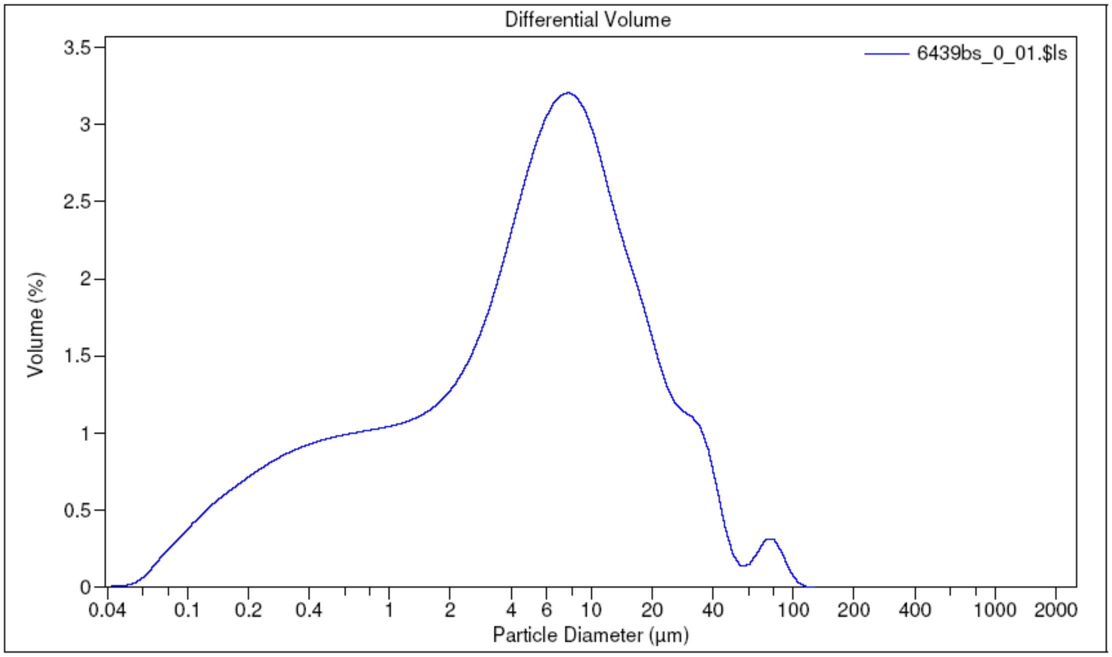
Volume:	100%		
Mean:	8.782 µm	S.D.:	14.02 µm
Median:	1.665 µm	Variance:	196.7 µm^2
Mean/Median ratio:	5.273	C.V.:	160%
Mode:	0.358 µm	Skewness:	2.232 Right skewed
		Kurtosis:	5.256 Leptokurtic

<10%	<25%	<50%	<75%	<90%		
0.180 µm	0.372 µm	1.665 µm	11.45 µm	28.95 µm		
>10%	>25%	>50%	>75%	>90%		
28.95 µm	11.45 µm	1.665 µm	0.372 µm	0.180 µm		
<2 µm	<5 µm	<20 µm	<50 µm	<100 µm	<250 µm	<500 µm
52.5%	63.5%	83.6%	97.4%	99.999%	100%	100%
>2 µm	>5 µm	>20 µm	>50 µm	>100 µm	>250 µm	>500 µm
47.5%	36.5%	16.4%	2.61%	0.0014%	0%	0%

LS Particle Size Analyzer

14 Aug 2008 15:34

Beckman Coulter LS 13 320



Volume Statistics (Arithmetic) 6439bs_0_01.$ls

Calculations from 0.040 µm to 2000 µm

Volume:	100%		
Mean:	9.042 µm	S.D.:	12.33 µm
Median:	5.262 µm	Variance:	152.0 μm^2
Mean/Median ratio:	1.718	C.V.:	136%
Mode:	7.775 µm	Skewness:	3.220 Right skewed
		Kurtosis:	14.23 Leptokurtic

<10%	<25%	<50%	<75%	<90%
0.337 µm	1.366 µm	5.262 µm	11.25 µm	21.99 µm

>10%	>25%	>50%	>75%	>90%
21.99 µm	11.25 µm	5.262 µm	1.366 µm	0.337 µm

<2 µm	<5 µm	<20 µm	<50 µm	<100 µm	<250 µm	<500 µm
29.8%	48.5%	88.4%	98.4%	99.95%	100%	100%

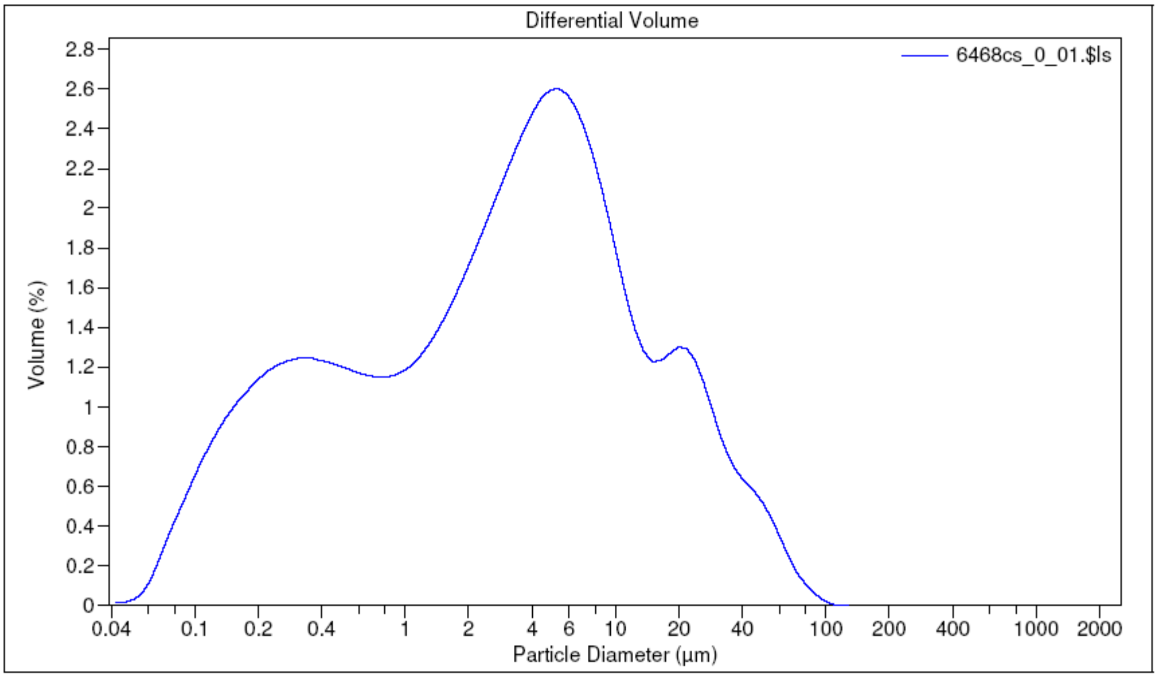
>2 µm	>5 µm	>20 µm	>50 µm	>100 µm	>250 µm	>500 µm
70.2%	51.5%	11.6%	1.63%	0.049%	0%	0%

BECKMAN COULTER

LS Particle Size Analyzer

14 Aug 2008 15:49

Beckman Coulter LS 13 320



Volume Statistics (Arithmetic) 6468cs_0_01.$ls

Calculations from 0.040 µm to 2000 µm

Volume: 100%
Mean: 7.395 µm
Median: 3.175 µm
Mean/Median ratio: 2.329
Mode: 5.354 µm

S.D.: 11.55 µm
Variance: 133.4 µm^2
C.V.: 156%
Skewness: 2.997 Right skewed
Kurtosis: 11.12 Leptokurtic

<10%	<25%	<50%	<75%	<90%
0.214 µm	0.679 µm	3.175 µm	8.172 µm	20.78 µm

>10%	>25%	>50%	>75%	>90%
20.78 µm	8.172 µm	3.175 µm	0.679 µm	0.214 µm

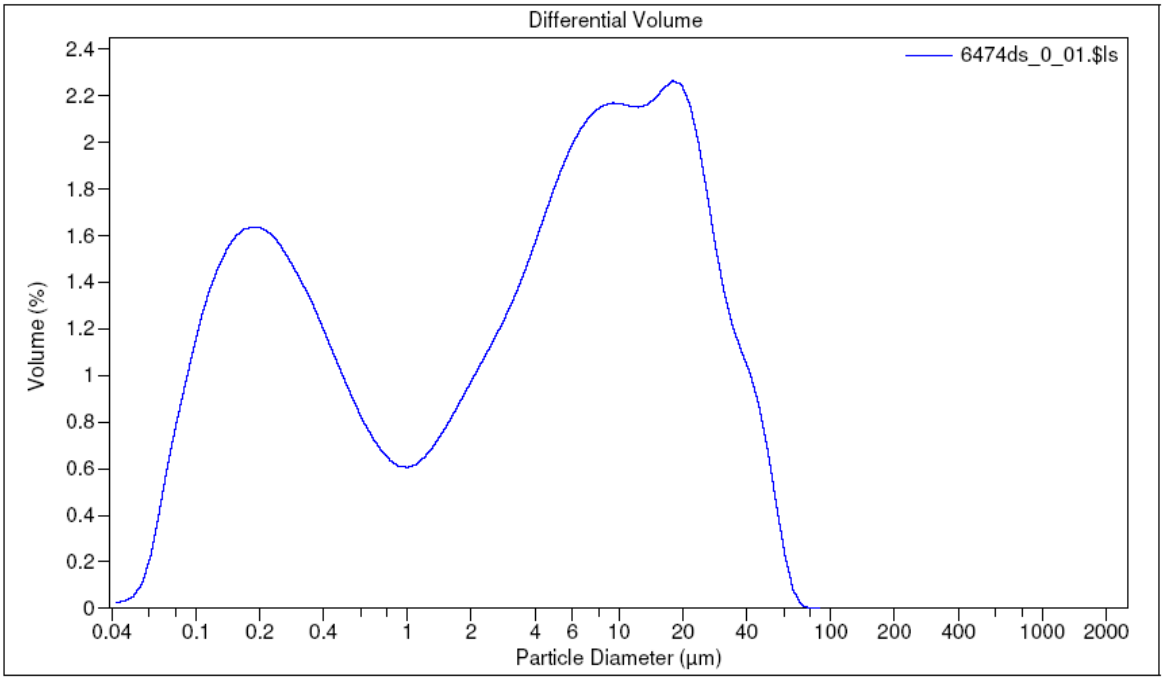
<2 µm	<5 µm	<20 µm	<50 µm	<100 µm	<250 µm	<500 µm
40.2%	61.9%	89.5%	98.3%	99.99%	100%	100%

>2 µm	>5 µm	>20 µm	>50 µm	>100 µm	>250 µm	>500 µm
59.8%	38.1%	10.5%	1.69%	0.011%	0%	0%

LS Particle Size Analyzer

14 Aug 2008 15:50

Beckman Coulter LS 13 320



```
Volume Statistics (Arithmetic)              6474ds_0_01.$ls

Calculations from 0.040 µm to 2000 µm

Volume:              100%
Mean:                9.018 µm       S.D.:             11.74 µm
Median:              4.240 µm       Variance:         137.9 µm²
Mean/Median ratio:   2.127          C.V.:             130%
Mode:                18.00 µm       Skewness:     1.820 Right skewed
                                    Kurtosis:     3.322 Leptokurtic

<10%        <25%        <50%        <75%        <90%
0.148 µm    0.370 µm    4.240 µm    13.33 µm    25.41 µm

>10%        >25%        >50%        >75%        >90%
25.41 µm    13.33 µm    4.240 µm    0.370 µm    0.148 µm

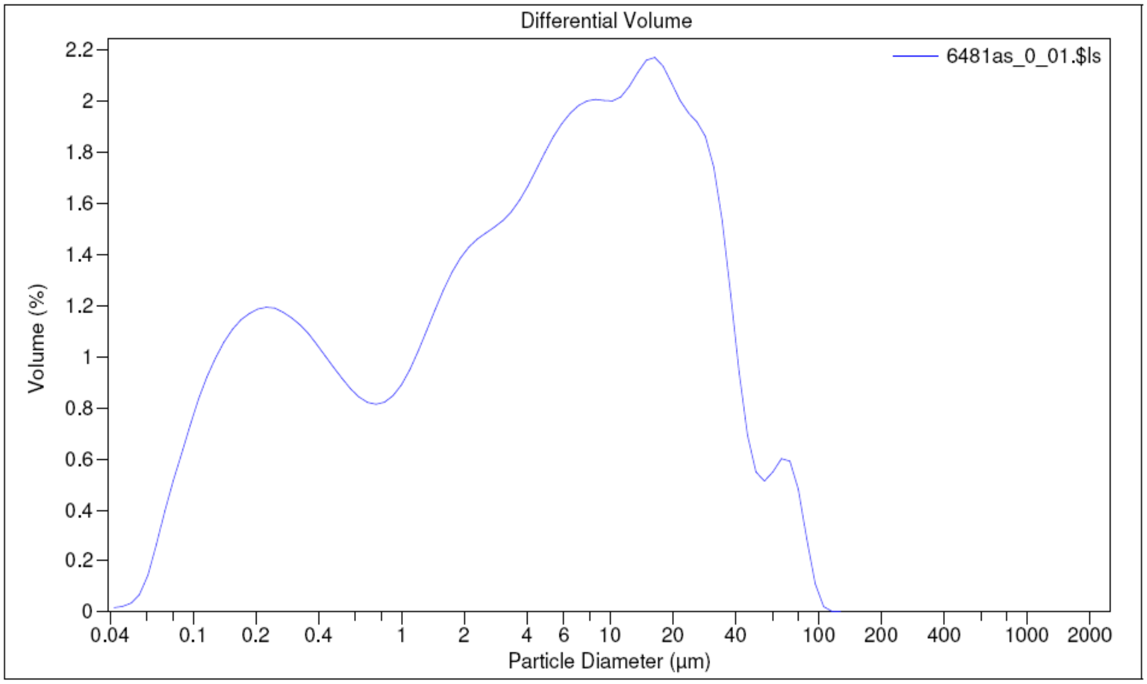
<2 µm       <5 µm       <20 µm      <50 µm      <100 µm     <250 µm     <500 µm
39.8%       53.0%       84.7%       98.8%       100%        100%        100%

>2 µm       >5 µm       >20 µm      >50 µm      >100 µm     >250 µm     >500 µm
60.2%       47.0%       15.3%       1.17%       0%          0%          0%
```

LS Particle Size Analyzer

4 Sep 2008 11:35

Beckman Coulter LS 13 320



Volume Statistics (Arithmetic)		6481as_0_01.$ls		
Calculations from 0.040 µm to 2000 µm				
Volume:	100%			
Mean:	10.88 µm	S.D.:		15.33 µm
Median:	4.610 µm	Variance:		235.1 μm^2
Mean/Median ratio:	2.360	C.V.:		141%
Mode:	16.40 µm	Skewness:	2.397 Right skewed	
		Kurtosis:	6.793 Leptokurtic	

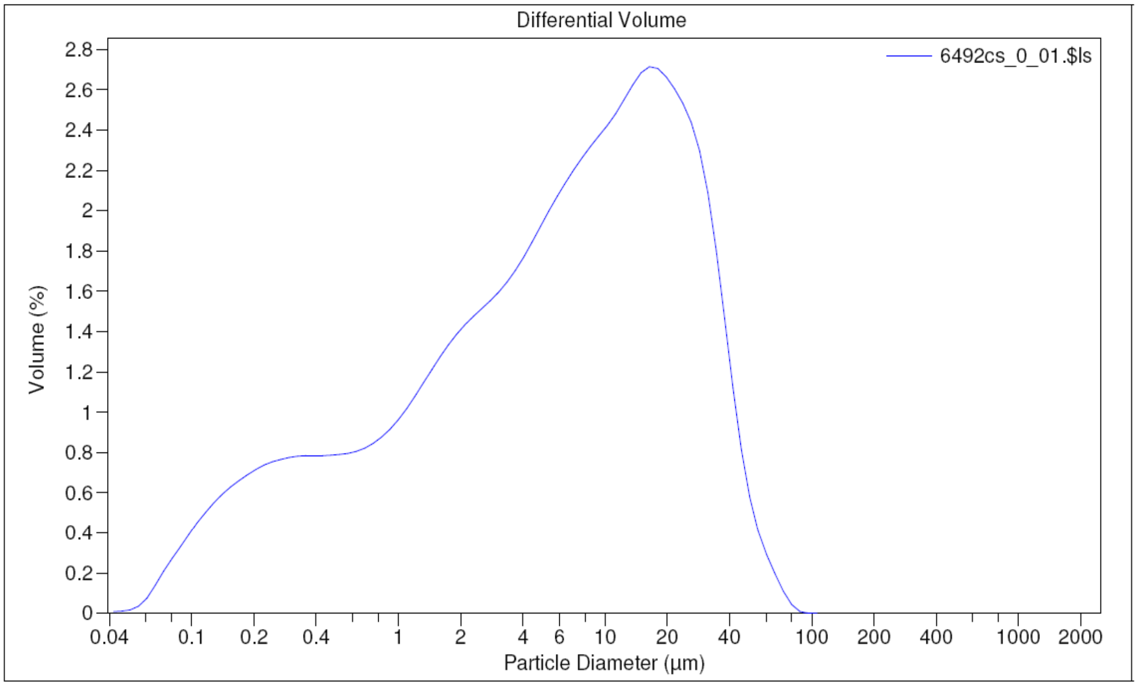
<10%	<25%	<50%	<75%	<90%		
0.195 µm	0.755 µm	4.610 µm	14.98 µm	29.85 µm		
>10%	>25%	>50%	>75%	>90%		
29.85 µm	14.98 µm	4.610 µm	0.755 µm	0.195 µm		
<2 µm	<5 µm	<20 µm	<50 µm	<100 µm	<200 µm	<250 µm
36.1%	51.6%	81.6%	96.5%	99.96%	100%	100%
>2 µm	>5 µm	>20 µm	>50 µm	>100 µm	>200 µm	>250 µm
63.9%	48.4%	18.4%	3.46%	0.036%	0%	0%

BECKMAN COULTER

LS Particle Size Analyzer

4 Sep 2008 11:43

Beckman Coulter LS 13 320



Volume Statistics (Arithmetic) 6492cs_0_01.$ls

Calculations from 0.040 µm to 2000 µm

Volume:	100%		
Mean:	11.04 µm	S.D.:	12.40 µm
Median:	6.427 µm	Variance:	153.7 µm^2
Mean/Median ratio:	1.718	C.V.:	112%
Mode:	16.40 µm	Skewness:	1.640 Right skewed
		Kurtosis:	3.010 Leptokurtic

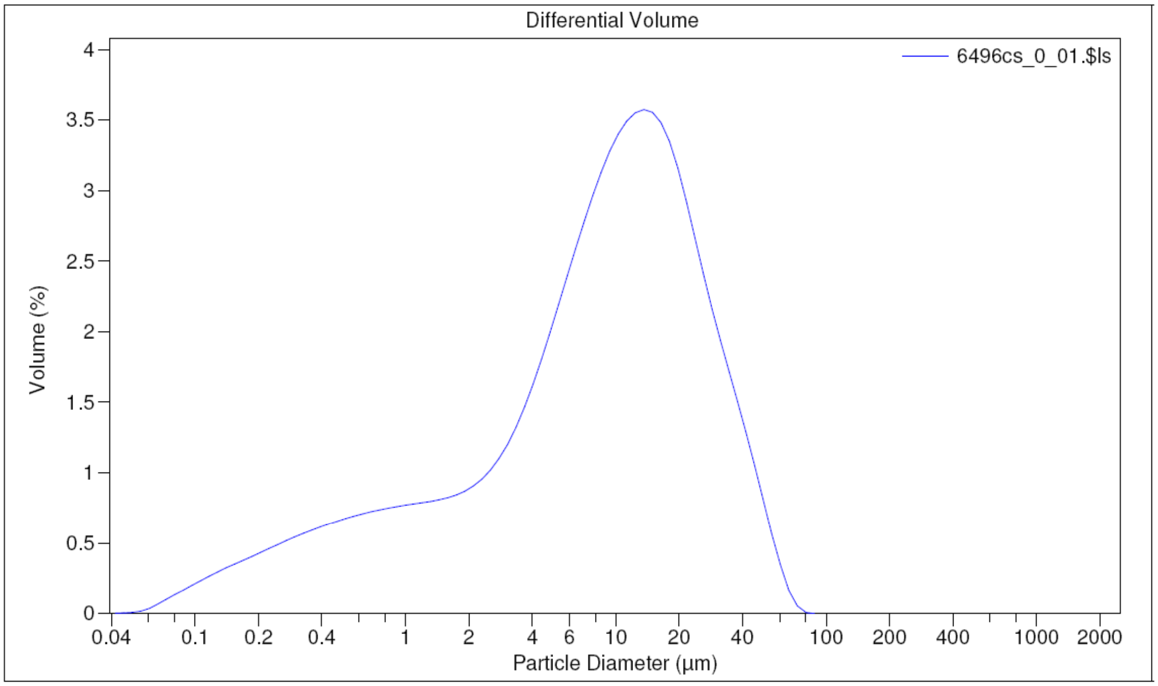
<10%	<25%	<50%	<75%	<90%		
0.337 µm	1.576 µm	6.427 µm	16.72 µm	28.82 µm		
>10%	>25%	>50%	>75%	>90%		
28.82 µm	16.72 µm	6.427 µm	1.576 µm	0.337 µm		
<2 µm	<5 µm	<20 µm	<50 µm	<100 µm	<200 µm	<250 µm
28.4%	44.5%	80.2%	98.6%	100%	100%	100%
>2 µm	>5 µm	>20 µm	>50 µm	>100 µm	>200 µm	>250 µm
71.6%	55.5%	19.8%	1.39%	0.000092%	0%	0%

BECKMAN COULTER

LS Particle Size Analyzer

4 Sep 2008 11:46

Beckman Coulter LS 13 320



Volume Statistics (Arithmetic) 6496cs_0_01.$ls

Calculations from 0.040 µm to 2000 µm

Volume:	100%		
Mean:	12.46 µm	S.D.:	12.19 µm
Median:	9.070 µm	Variance:	148.7 µm^2
Mean/Median ratio:	1.374	C.V.:	97.9%
Mode:	13.61 µm	Skewness:	1.513 Right skewed
		Kurtosis:	2.465 Leptokurtic

<10%	<25%	<50%	<75%	<90%
0.608 µm	3.150 µm	9.070 µm	17.75 µm	29.41 µm

>10%	>25%	>50%	>75%	>90%
29.41 µm	17.75 µm	9.070 µm	3.150 µm	0.608 µm

<2 µm	<5 µm	<20 µm	<50 µm	<100 µm	<200 µm	<250 µm
20.0%	33.0%	79.2%	98.4%	100%	100%	100%

>2 µm	>5 µm	>20 µm	>50 µm	>100 µm	>200 µm	>250 µm
80.0%	67.0%	20.8%	1.60%	0%	0%	0%

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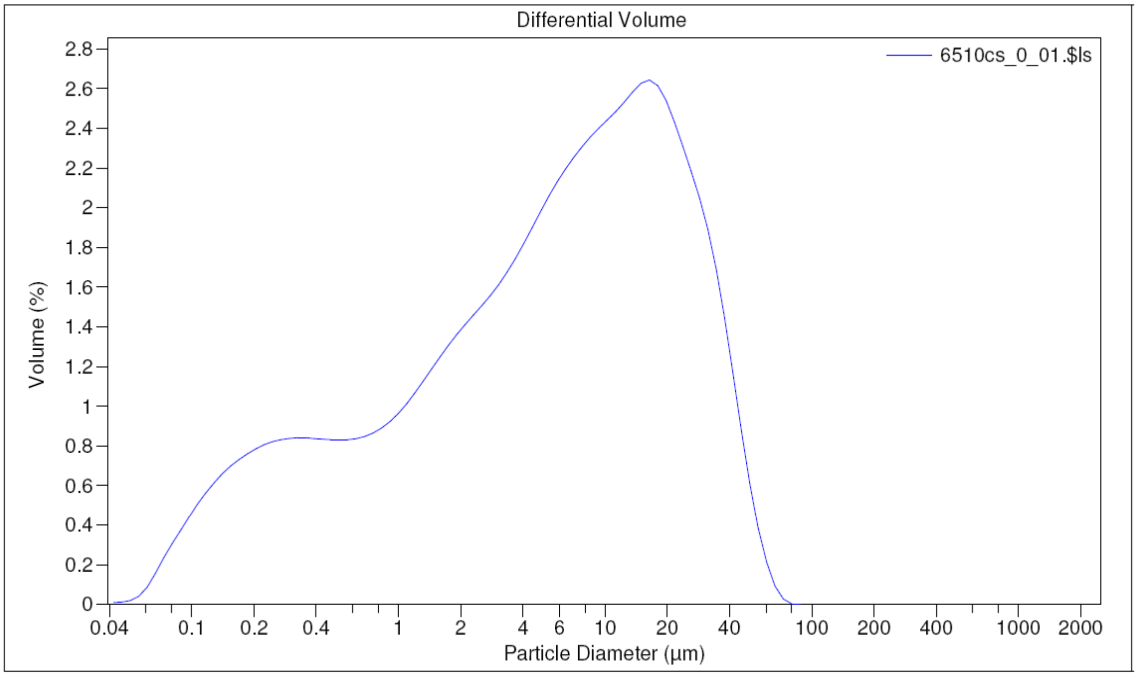
LS Particle Size Analyzer

4 Sep 2008 11:48

Beckman Coulter LS 13 320



Volume Statistics (Arithmetic) 6510cs_0_01.$ls

Calculations from 0.040 µm to 2000 µm

Volume:	100%		
Mean:	10.51 µm	S.D.:	11.91 µm
Median:	5.994 µm	Variance:	141.8 µm^2
Mean/Median ratio:	1.753	C.V.:	113%
Mode:	16.40 µm	Skewness:	1.577 Right skewed
		Kurtosis:	2.388 Leptokurtic

<10%	<25%	<50%	<75%	<90%
0.302 µm	1.422 µm	5.994 µm	15.75 µm	27.98 µm

>10%	>25%	>50%	>75%	>90%
27.98 µm	15.75 µm	5.994 µm	1.422 µm	0.302 µm

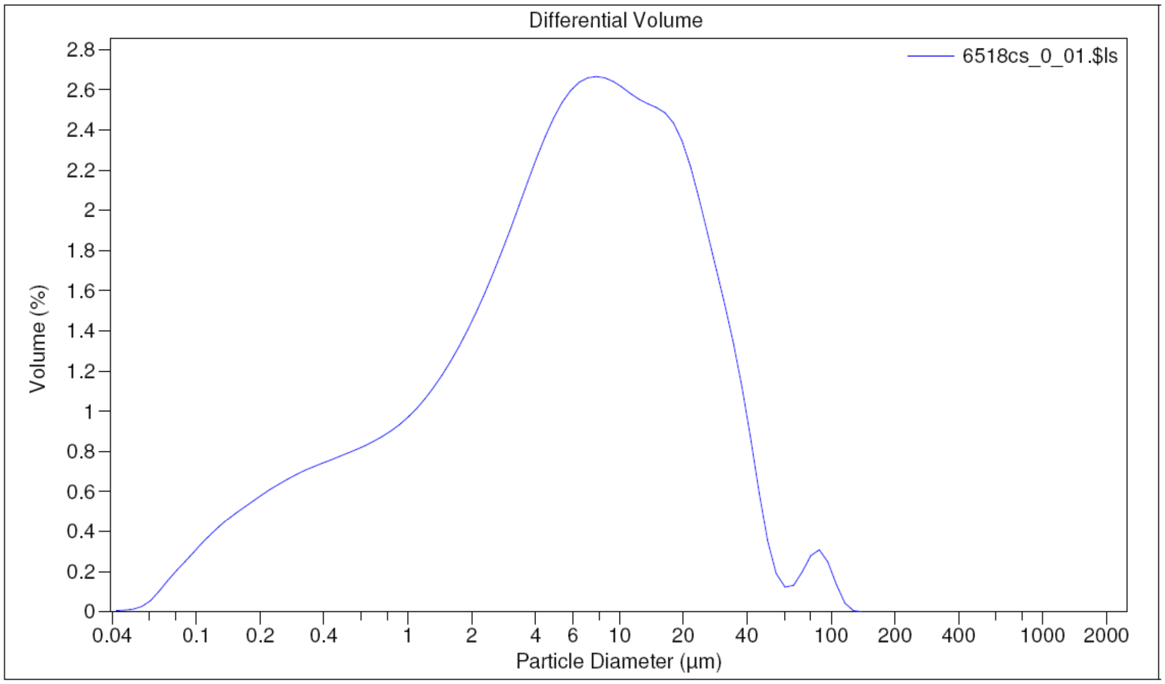
<2 µm	<5 µm	<20 µm	<50 µm	<100 µm	<200 µm	<250 µm
29.7%	46.0%	81.7%	98.9%	100%	100%	100%

>2 µm	>5 µm	>20 µm	>50 µm	>100 µm	>200 µm	>250 µm
70.3%	54.0%	18.3%	1.06%	0%	0%	0%

LS Particle Size Analyzer

9 Sep 2008 15:25

Beckman Coulter LS 13 320



Volume Statistics (Arithmetic)   6518cs_0_01.$ls

Calculations from 0.040 µm to 2000 µm

Volume:	100%		
Mean:	10.61 µm	S.D.:	13.81 µm
Median:	5.872 µm	Variance:	190.8 µm^2
Mean/Median ratio:	1.806	C.V.:	130%
Mode:	7.775 µm	Skewness:	3.038 Right skewed
		Kurtosis:	13.34 Leptokurtic

<10%	<25%	<50%	<75%	<90%
0.430 µm	1.821 µm	5.872 µm	14.32 µm	26.33 µm

>10%	>25%	>50%	>75%	>90%
26.33 µm	14.32 µm	5.872 µm	1.821 µm	0.430 µm

<2 µm	<5 µm	<20 µm	<50 µm	<100 µm	<200 µm	<250 µm
26.4%	45.6%	83.8%	98.1%	99.8%	100%	100%

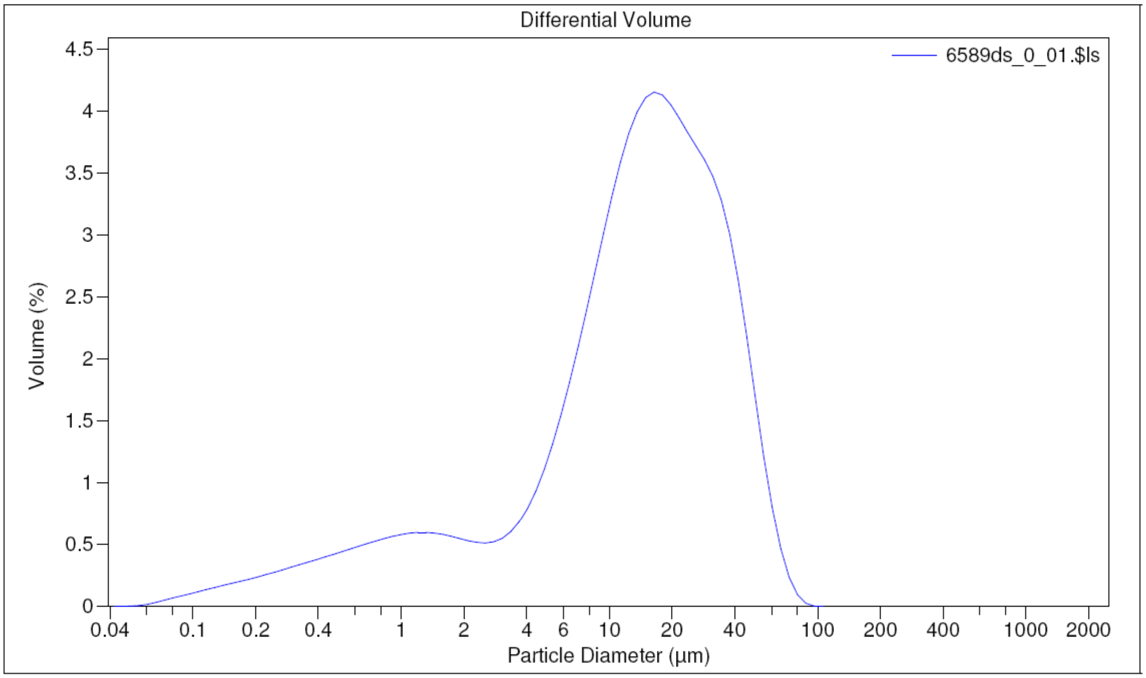
>2 µm	>5 µm	>20 µm	>50 µm	>100 µm	>200 µm	>250 µm
73.6%	54.4%	16.2%	1.86%	0.22%	0%	0%

BECKMAN
COULTER

LS Particle Size Analyzer

9 Sep 2008 15:31

Beckman Coulter LS 13 320



Volume Statistics (Arithmetic) 6589ds_0_01.$ls

Calculations from 0.040 µm to 2000 µm

Volume:	100%		
Mean:	17.90 µm	S.D.:	14.67 µm
Median:	14.47 µm	Variance:	215.1 μm^2
Mean/Median ratio:	1.237	C.V.:	81.9%
Mode:	16.40 µm	Skewness:	1.084 Right skewed
		Kurtosis:	1.016 Leptokurtic

<10%	<25%	<50%	<75%	<90%
1.231 µm	6.923 µm	14.47 µm	25.89 µm	39.03 µm

>10%	>25%	>50%	>75%	>90%
39.03 µm	25.89 µm	14.47 µm	6.923 µm	1.231 µm

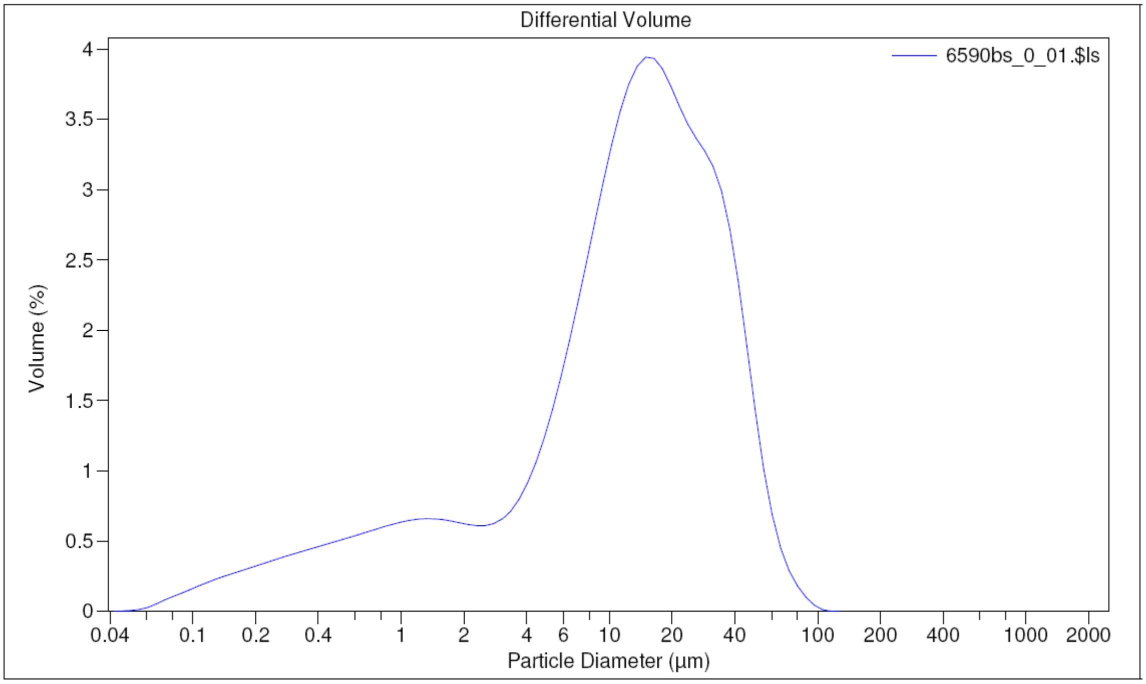
<2 µm	<5 µm	<20 µm	<50 µm	<100 µm	<200 µm	<250 µm
13.0%	19.5%	64.3%	96.2%	99.999%	100%	100%

>2 µm	>5 µm	>20 µm	>50 µm	>100 µm	>200 µm	>250 µm
87.0%	80.5%	35.7%	3.81%	0.00068%	0%	0%

LS Particle Size Analyzer

9 Sep 2008 15:34

Beckman Coulter LS 13 320



Volume Statistics (Arithmetic) 6590bs_0_01.$ls

Calculations from 0.040 µm to 2000 µm

Volume:	100%		
Mean:	16.81 µm	S.D.:	14.88 µm
Median:	13.12 µm	Variance:	221.6 µm^2
Mean/Median ratio:	1.281	C.V.:	88.5%
Mode:	14.94 µm	Skewness:	1.324 Right skewed
		Kurtosis:	2.122 Leptokurtic

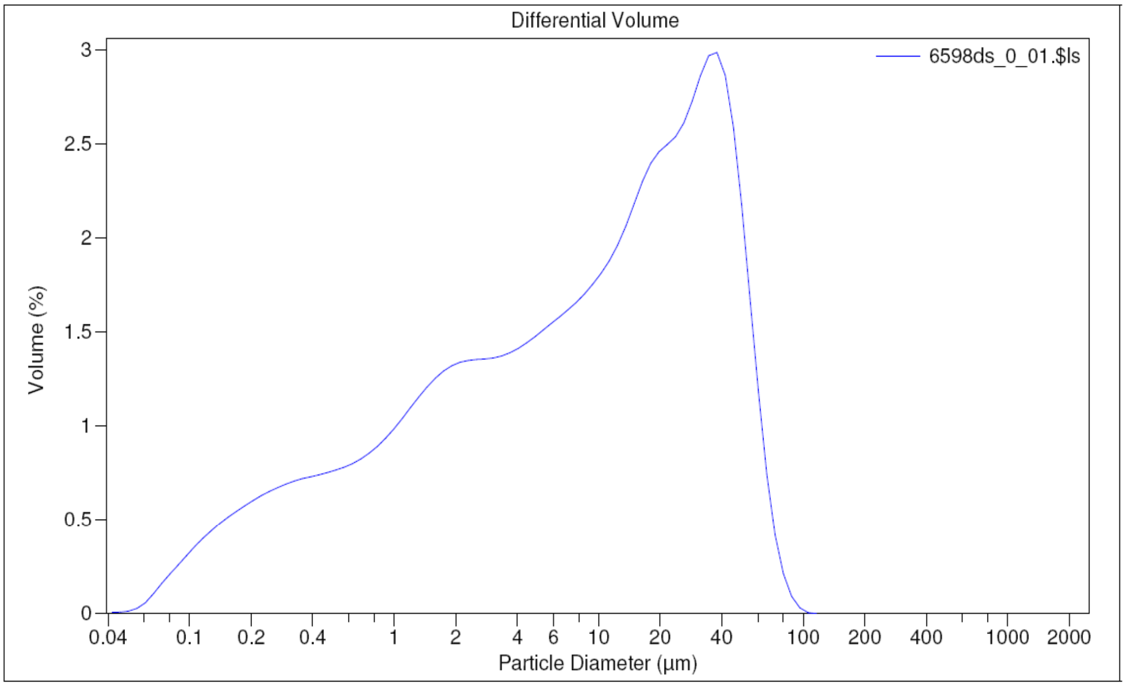
<10%	<25%	<50%	<75%	<90%		
0.903 µm	5.662 µm	13.12 µm	24.30 µm	37.78 µm		
>10%	>25%	>50%	>75%	>90%		
37.78 µm	24.30 µm	13.12 µm	5.662 µm	0.903 µm		
<2 µm	<5 µm	<20 µm	<50 µm	<100 µm	<200 µm	<250 µm
15.5%	23.1%	67.5%	96.4%	99.98%	100%	100%
>2 µm	>5 µm	>20 µm	>50 µm	>100 µm	>200 µm	>250 µm
84.5%	76.9%	32.5%	3.59%	0.020%	0%	0%

BECKMAN COULTER

LS Particle Size Analyzer

10 Sep 2008 10:58

Beckman Coulter LS 13 320



Volume Statistics (Arithmetic) 6598ds_0_01.$ls

Calculations from 0.040 µm to 2000 µm

Volume:	100%		
Mean:	15.90 µm	S.D.:	17.33 µm
Median:	8.925 µm	Variance:	300.4 μm^2
Mean/Median ratio:	1.781	C.V.:	109%
Mode:	37.97 µm	Skewness:	1.231 Right skewed
		Kurtosis:	0.906 Leptokurtic

<10%	<25%	<50%	<75%	<90%
0.418 µm	1.798 µm	8.925 µm	26.01 µm	42.46 µm

>10%	>25%	>50%	>75%	>90%
42.46 µm	26.01 µm	8.925 µm	1.798 µm	0.418 µm

<2 µm	<5 µm	<20 µm	<50 µm	<100 µm	<200 µm	<250 µm
26.5%	40.1%	67.9%	94.4%	99.99%	100%	100%

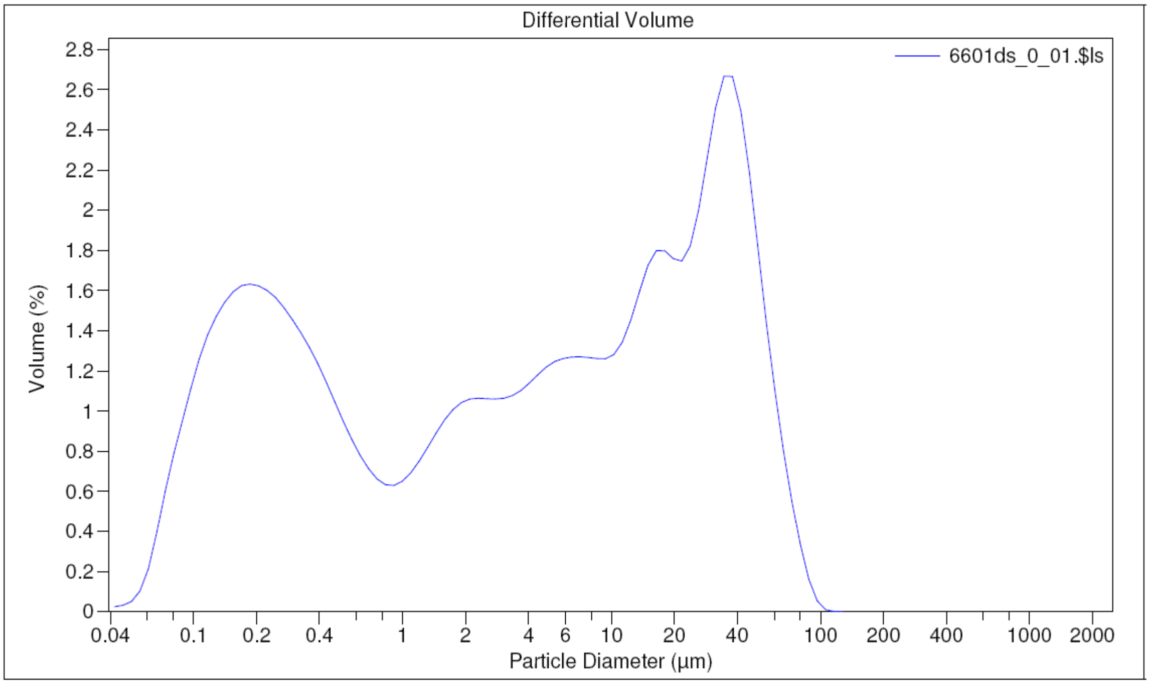
>2 µm	>5 µm	>20 µm	>50 µm	>100 µm	>200 µm	>250 µm
73.5%	59.9%	32.1%	5.57%	0.0098%	0%	0%

BECKMAN COULTER

LS Particle Size Analyzer

10 Sep 2008 11:02

Beckman Coulter LS 13 320



Volume Statistics (Arithmetic) 6601ds_0_01.$ls

Calculations from 0.040 µm to 2000 µm

Volume: 100%
Mean: 13.65 µm S.D.: 17.90 µm
Median: 4.464 µm Variance: 320.3 µm^2
Mean/Median ratio: 3.059 C.V.: 131%
Mode: 34.58 µm Skewness: 1.491 Right skewed
Kurtosis: 1.671 Leptokurtic

<10%	<25%	<50%	<75%	<90%
0.148 µm	0.369 µm	4.464 µm	22.61 µm	41.30 µm

>10%	>25%	>50%	>75%	>90%
41.30 µm	22.61 µm	4.464 µm	0.369 µm	0.148 µm

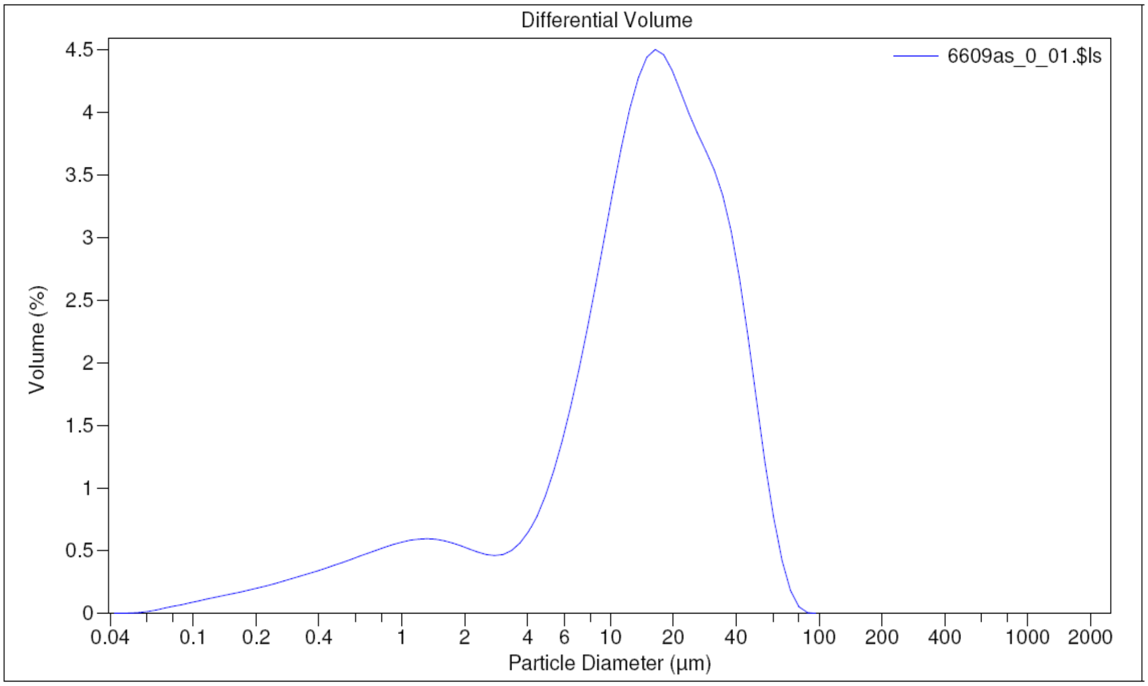
<2 µm	<5 µm	<20 µm	<50 µm	<100 µm	<200 µm	<250 µm
40.7%	51.5%	72.7%	94.5%	99.98%	100%	100%

>2 µm	>5 µm	>20 µm	>50 µm	>100 µm	>200 µm	>250 µm
59.3%	48.5%	27.3%	5.49%	0.017%	0%	0%

LS Particle Size Analyzer

10 Sep 2008 11:04

Beckman Coulter LS 13 320



Volume Statistics (Arithmetic) 6609as_0_01.$ls

Calculations from 0.040 µm to 2000 µm

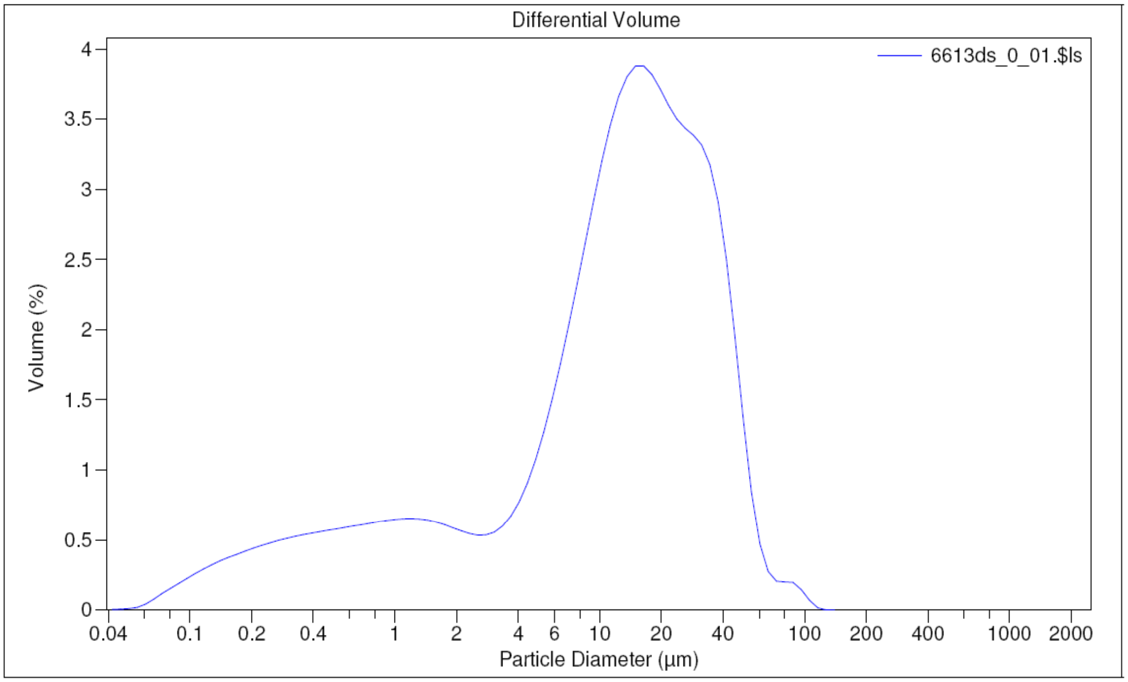
Volume:	100%		
Mean:	18.21 µm	S.D.:	14.31 µm
Median:	15.07 µm	Variance:	204.8 µm^2
Mean/Median ratio:	1.208	C.V.:	78.6%
Mode:	16.40 µm	Skewness:	1.019 Right skewed
		Kurtosis:	0.827 Leptokurtic

<10%	<25%	<50%	<75%	<90%		
1.406 µm	7.697 µm	15.07 µm	26.03 µm	38.89 µm		
>10%	>25%	>50%	>75%	>90%		
38.89 µm	26.03 µm	15.07 µm	7.697 µm	1.406 µm		
<2 µm	<5 µm	<20 µm	<50 µm	<100 µm	<200 µm	<250 µm
12.1%	17.8%	63.5%	96.4%	100%	100%	100%
>2 µm	>5 µm	>20 µm	>50 µm	>100 µm	>200 µm	>250 µm
87.9%	82.2%	36.5%	3.61%	0.000061%	0%	0%

LS Particle Size Analyzer

17 Sep 2008 11:09

Beckman Coulter LS 13 320



Volume Statistics (Arithmetic) 6613ds_0_01.$ls

Calculations from 0.040 µm to 2000 µm

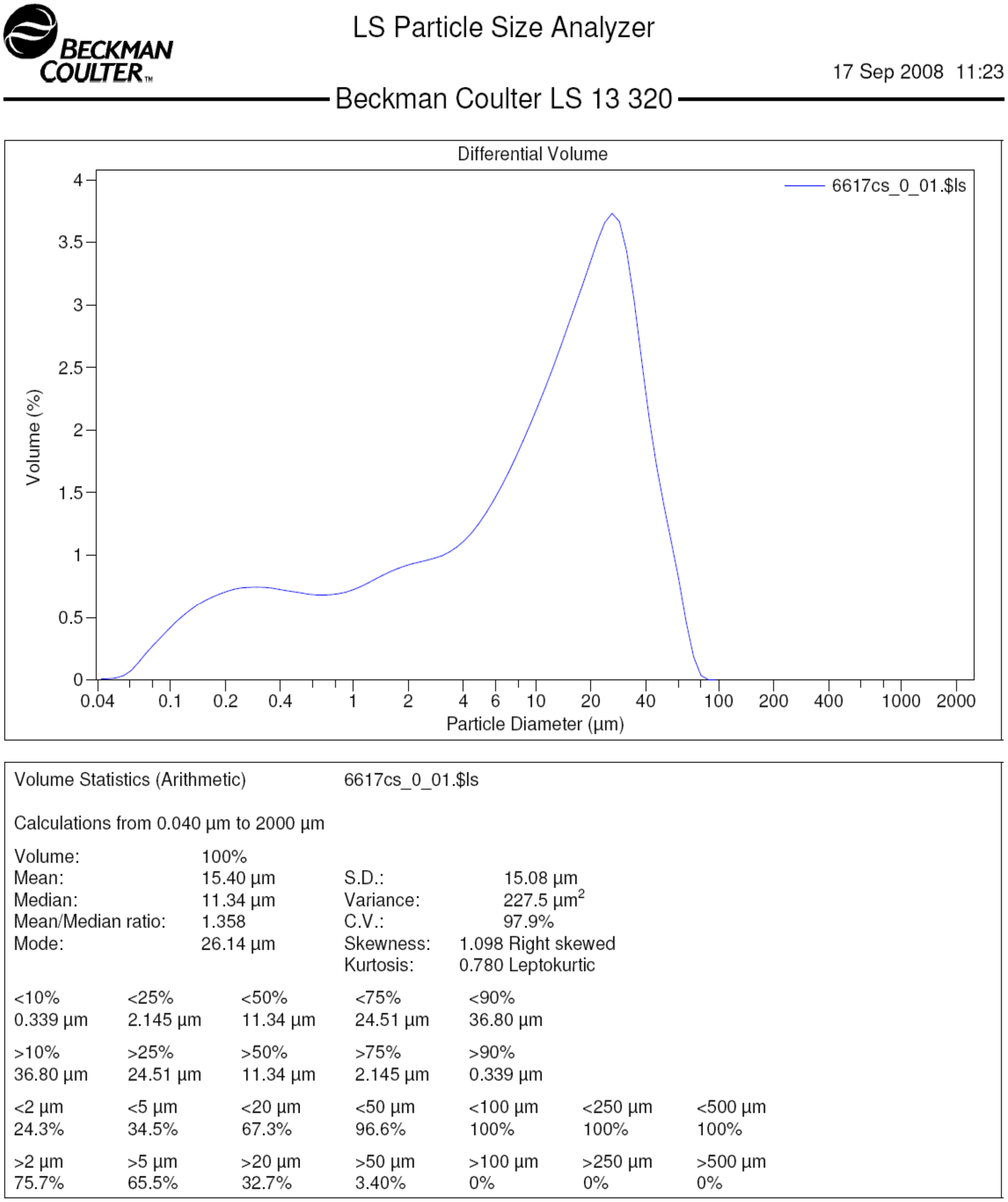
Volume:	100%		
Mean:	16.80 µm	S.D.:	15.22 µm
Median:	13.22 µm	Variance:	231.7 µm^2
Mean/Median ratio:	1.271	C.V.:	90.6%
Mode:	16.40 µm	Skewness:	1.466 Right skewed
		Kurtosis:	3.369 Leptokurtic

<10%	<25%	<50%	<75%	<90%
0.639 µm	5.296 µm	13.22 µm	24.63 µm	37.66 µm

>10%	>25%	>50%	>75%	>90%
37.66 µm	24.63 µm	13.22 µm	5.296 µm	0.639 µm

<2 µm	<5 µm	<20 µm	<50 µm	<100 µm	<250 µm	<500 µm
17.7%	24.3%	67.0%	96.8%	99.9%	100%	100%

>2 µm	>5 µm	>20 µm	>50 µm	>100 µm	>250 µm	>500 µm
82.3%	75.7%	33.0%	3.20%	0.10%	0%	0%



LS Particle Size Analyzer

17 Sep 2008 11:23

Beckman Coulter LS 13 320

Volume Statistics (Arithmetic) 6617cs_0_01.$ls

Calculations from 0.040 µm to 2000 µm

Volume:	100%		
Mean:	15.40 µm	S.D.:	15.08 µm
Median:	11.34 µm	Variance:	227.5 µm^2
Mean/Median ratio:	1.358	C.V.:	97.9%
Mode:	26.14 µm	Skewness:	1.098 Right skewed
		Kurtosis:	0.780 Leptokurtic

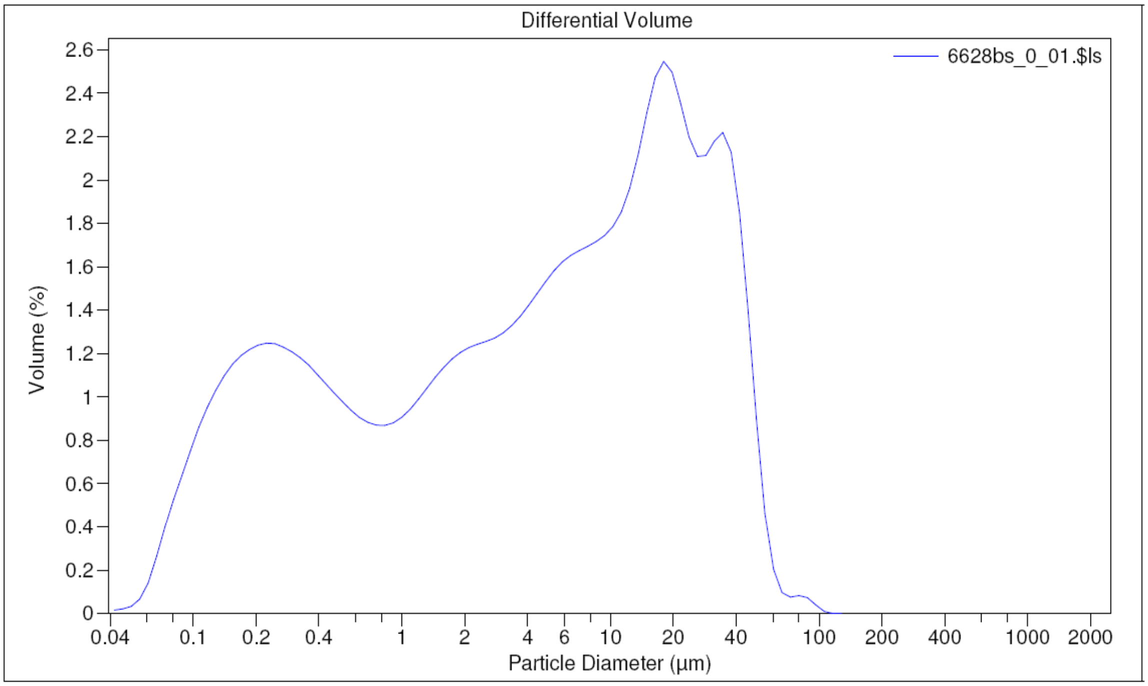
<10%	<25%	<50%	<75%	<90%		
0.339 µm	2.145 µm	11.34 µm	24.51 µm	36.80 µm		
>10%	>25%	>50%	>75%	>90%		
36.80 µm	24.51 µm	11.34 µm	2.145 µm	0.339 µm		
<2 µm	<5 µm	<20 µm	<50 µm	<100 µm	<250 µm	<500 µm
24.3%	34.5%	67.3%	96.6%	100%	100%	100%
>2 µm	>5 µm	>20 µm	>50 µm	>100 µm	>250 µm	>500 µm
75.7%	65.5%	32.7%	3.40%	0%	0%	0%

BECKMAN COULTER™

LS Particle Size Analyzer

17 Sep 2008 11:28

Beckman Coulter LS 13 320



Volume Statistics (Arithmetic) 6628bs_0_01.$ls

Calculations from 0.040 µm to 2000 µm

Volume: 100%
Mean: 11.14 µm
Median: 5.025 µm
Mean/Median ratio: 2.217
Mode: 18.00 µm

S.D.: 13.76 µm
Variance: 189.3 μm^2
C.V.: 124%
Skewness: 1.628 Right skewed
Kurtosis: 2.941 Leptokurtic

<10%	<25%	<50%	<75%	<90%
0.190 µm	0.666 µm	5.025 µm	17.53 µm	32.39 µm

>10%	>25%	>50%	>75%	>90%
32.39 µm	17.53 µm	5.025 µm	0.666 µm	0.190 µm

<2 µm	<5 µm	<20 µm	<50 µm	<100 µm	<250 µm	<500 µm
36.8%	49.9%	78.6%	98.4%	99.98%	100%	100%

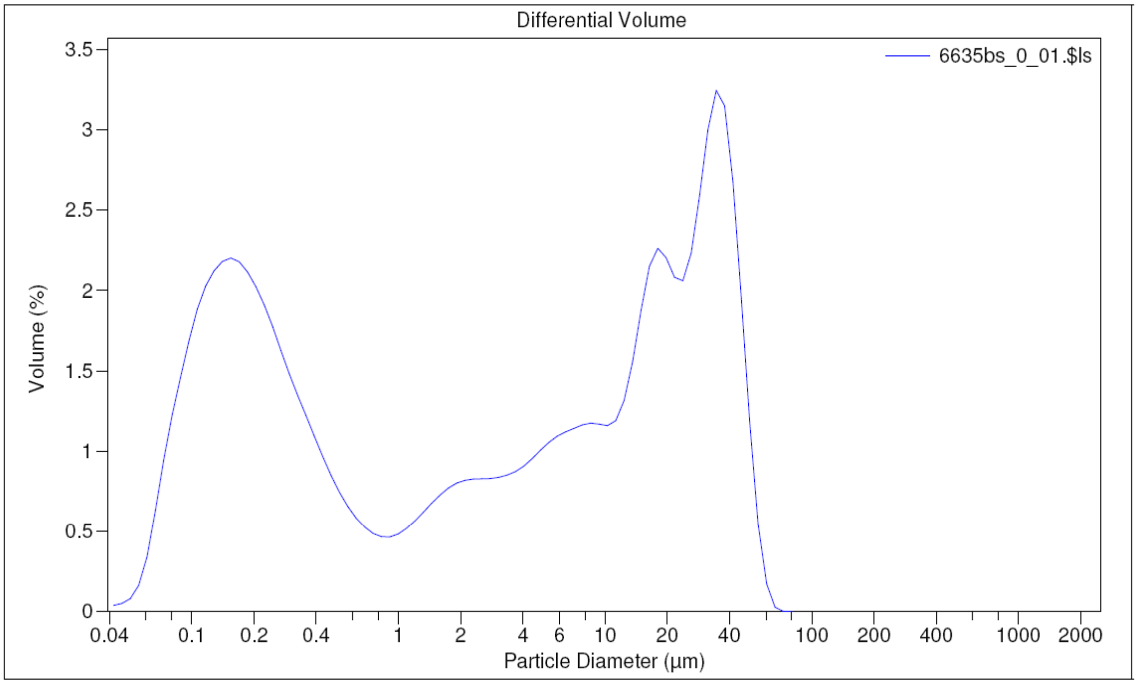
>2 µm	>5 µm	>20 µm	>50 µm	>100 µm	>250 µm	>500 µm
63.2%	50.1%	21.4%	1.55%	0.016%	0%	0%

BECKMAN COULTER

LS Particle Size Analyzer

18 Sep 2008 12:54

Beckman Coulter LS 13 320



Volume Statistics (Arithmetic) 6635bs_0_01.$ls

Calculations from 0.040 µm to 2000 µm

Volume:	100%		
Mean:	11.71 µm	S.D.:	14.68 µm
Median:	3.637 µm	Variance:	215.5 µm^2
Mean/Median ratio:	3.219	C.V.:	125%
Mode:	34.58 µm	Skewness:	1.143 Right skewed
		Kurtosis:	0.147 Leptokurtic

<10%	<25%	<50%	<75%	<90%
0.120 µm	0.232 µm	3.637 µm	20.64 µm	35.94 µm

>10%	>25%	>50%	>75%	>90%
35.94 µm	20.64 µm	3.637 µm	0.232 µm	0.120 µm

<2 µm	<5 µm	<20 µm	<50 µm	<100 µm	<250 µm	<500 µm
44.7%	53.2%	74.2%	98.6%	100%	100%	100%

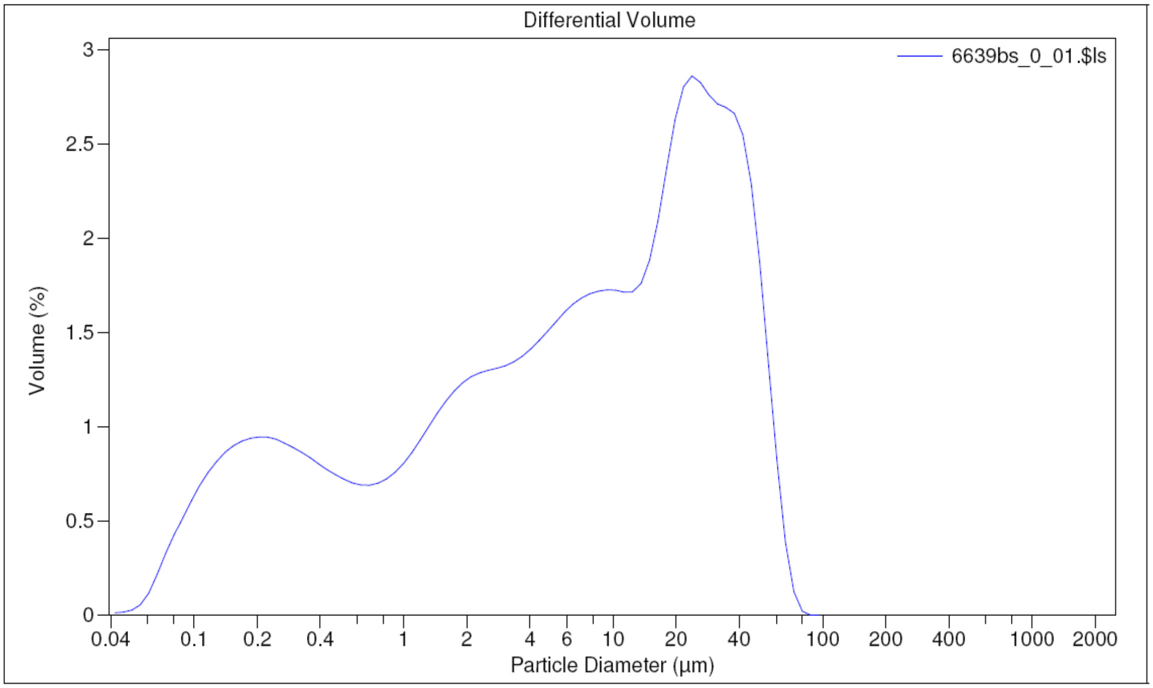
>2 µm	>5 µm	>20 µm	>50 µm	>100 µm	>250 µm	>500 µm
55.3%	46.8%	25.8%	1.41%	0%	0%	0%

BECKMAN COULTER

LS Particle Size Analyzer

18 Sep 2008 12:51

Beckman Coulter LS 13 320



Volume Statistics (Arithmetic) 6639bs_0_01.$ls

Calculations from 0.040 µm to 2000 µm

Volume:	100%		
Mean:	14.16 µm	S.D.:	15.81 µm
Median:	7.332 µm	Variance:	249.9 µm^2
Mean/Median ratio:	1.932	C.V.:	112%
Mode:	23.81 µm	Skewness:	1.187 Right skewed
		Kurtosis:	0.578 Leptokurtic

<10%	<25%	<50%	<75%	<90%
0.232 µm	1.323 µm	7.332 µm	23.45 µm	38.99 µm

>10%	>25%	>50%	>75%	>90%
38.99 µm	23.45 µm	7.332 µm	1.323 µm	0.232 µm

<2 µm	<5 µm	<20 µm	<50 µm	<100 µm	<250 µm	<500 µm
30.1%	43.4%	70.3%	96.2%	100%	100%	100%

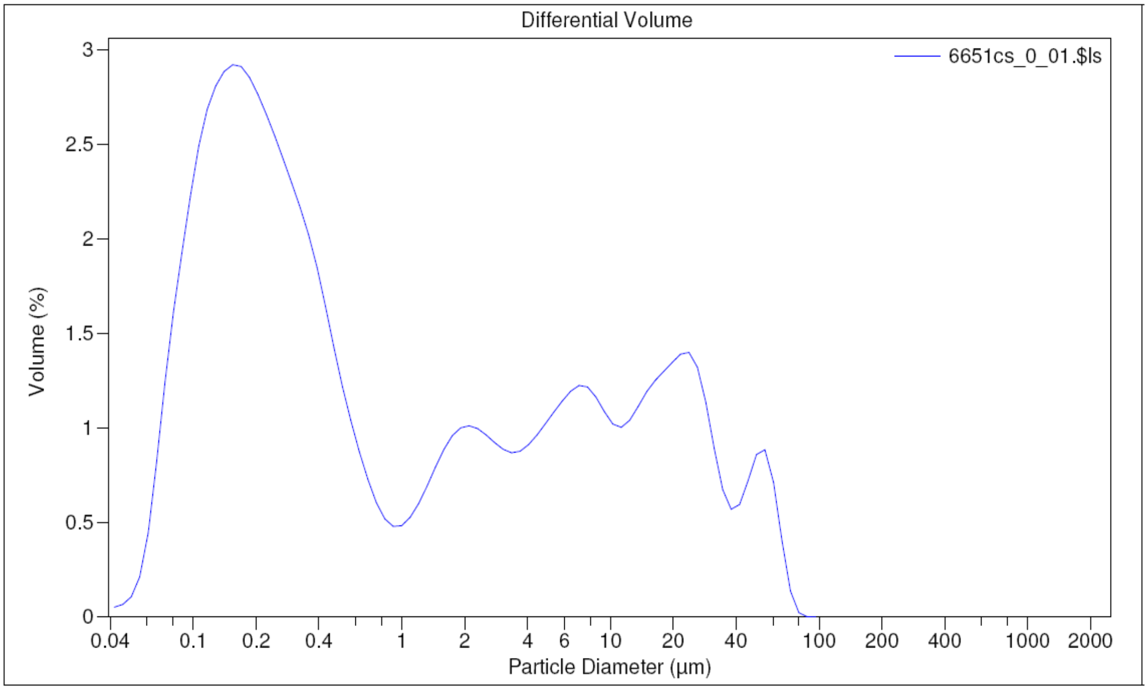
>2 µm	>5 µm	>20 µm	>50 µm	>100 µm	>250 µm	>500 µm
69.9%	56.6%	29.7%	3.75%	0%	0%	0%

BECKMAN COULTER™

LS Particle Size Analyzer

18 Sep 2008 12:59

Beckman Coulter LS 13 320



Volume Statistics (Arithmetic) 6651cs_0_01.$ls

Calculations from 0.040 µm to 2000 µm

Volume:	100%		
Mean:	6.863 µm	S.D.:	12.84 µm
Median:	0.496 µm	Variance:	165.0 µm^2
Mean/Median ratio:	13.85	C.V.:	187%
Mode:	0.155 µm	Skewness:	2.591 Right skewed
		Kurtosis:	6.834 Leptokurtic

<10%	<25%	<50%	<75%	<90%
0.107 µm	0.176 µm	0.496 µm	7.136 µm	23.26 µm

>10%	>25%	>50%	>75%	>90%
23.26 µm	7.136 µm	0.496 µm	0.176 µm	0.107 µm

<2 µm	<5 µm	<20 µm	<50 µm	<100 µm	<250 µm	<500 µm
61.4%	70.6%	87.8%	97.4%	100%	100%	100%

>2 µm	>5 µm	>20 µm	>50 µm	>100 µm	>250 µm	>500 µm
38.6%	29.4%	12.2%	2.64%	0%	0%	0%

APPENDIX B

BECKMAN COULTER LASER PARTICLE SIZE ANALYZER GRAPHS

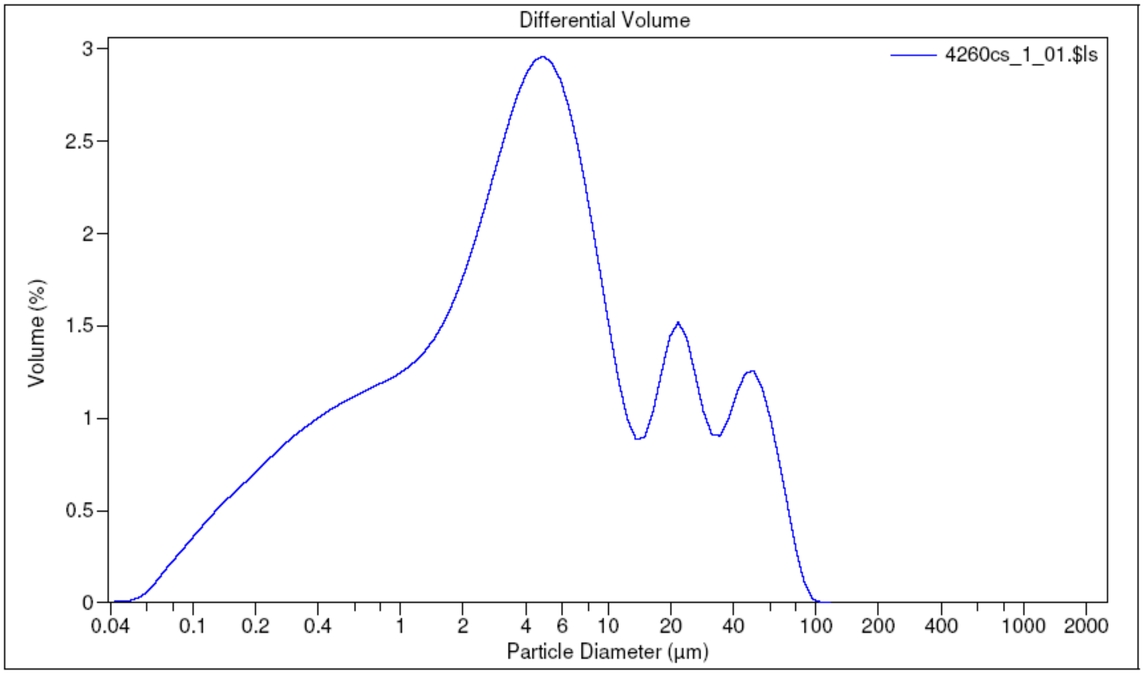
(GYPSEOUS SAMPLES)

BECKMAN COULTER™

LS Particle Size Analyzer

11 Dec 2008 14:23

Beckman Coulter LS 13 320



Volume Statistics (Arithmetic) 4260cs_1_01.$ls

Calculations from 0.040 µm to 2000 µm

Volume:	100%		
Mean:	10.07 µm	S.D.:	15.43 µm
Median:	3.910 µm	Variance:	238.1 μm^2
Mean/Median ratio:	2.575	C.V.:	153%
Mode:	4.877 µm	Skewness:	2.370 Right skewed
		Kurtosis:	5.405 Leptokurtic

<10%	<25%	<50%	<75%	<90%
0.342 µm	1.182 µm	3.910 µm	9.657 µm	31.10 µm

>10%	>25%	>50%	>75%	>90%
31.10 µm	9.657 µm	3.910 µm	1.182 µm	0.342 µm

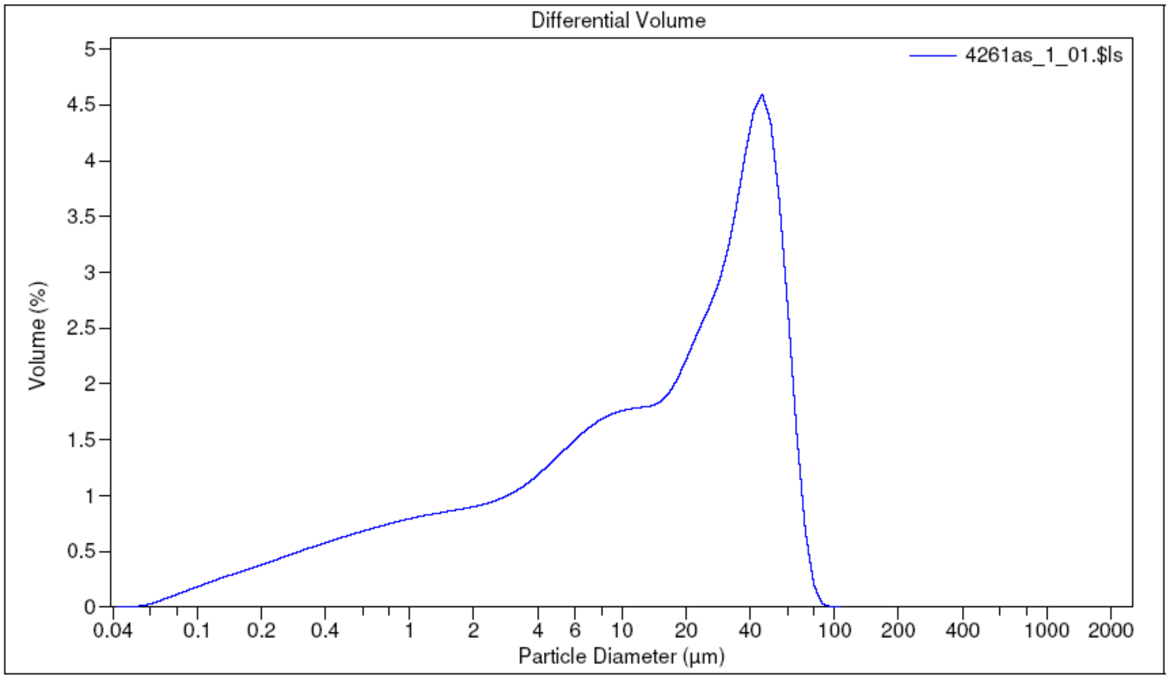
<2 µm	<5 µm	<20 µm	<50 µm	<100 µm	<250 µm	<500 µm
33.4%	57.7%	83.9%	95.4%	99.996%	100%	100%

>2 µm	>5 µm	>20 µm	>50 µm	>100 µm	>250 µm	>500 µm
66.6%	42.3%	16.1%	4.58%	0.0041%	0%	0%

LS Particle Size Analyzer

11 Dec 2008 14:24

Beckman Coulter LS 13 320



Volume Statistics (Arithmetic) 4261as_1_01.$ls

Calculations from 0.040 μm to 2000 μm

Volume:	100%		
Mean:	21.46 μm	S.D.:	19.78 μm
Median:	15.36 μm	Variance:	391.4 μm^2
Mean/Median ratio:	1.397	C.V.:	92.2%
Mode:	45.75 μm	Skewness:	0.693 Right skewed
		Kurtosis:	-0.668 Platykurtic

<10%	<25%	<50%	<75%	<90%
0.686 μm	3.433 μm	15.36 μm	37.23 μm	51.22 μm

>10%	>25%	>50%	>75%	>90%
51.22 μm	37.23 μm	15.36 μm	3.433 μm	0.686 μm

<2 μm	<5 μm	<20 μm	<50 μm	<100 μm	<250 μm	<500 μm
19.4%	29.9%	55.7%	88.9%	100%	100%	100%

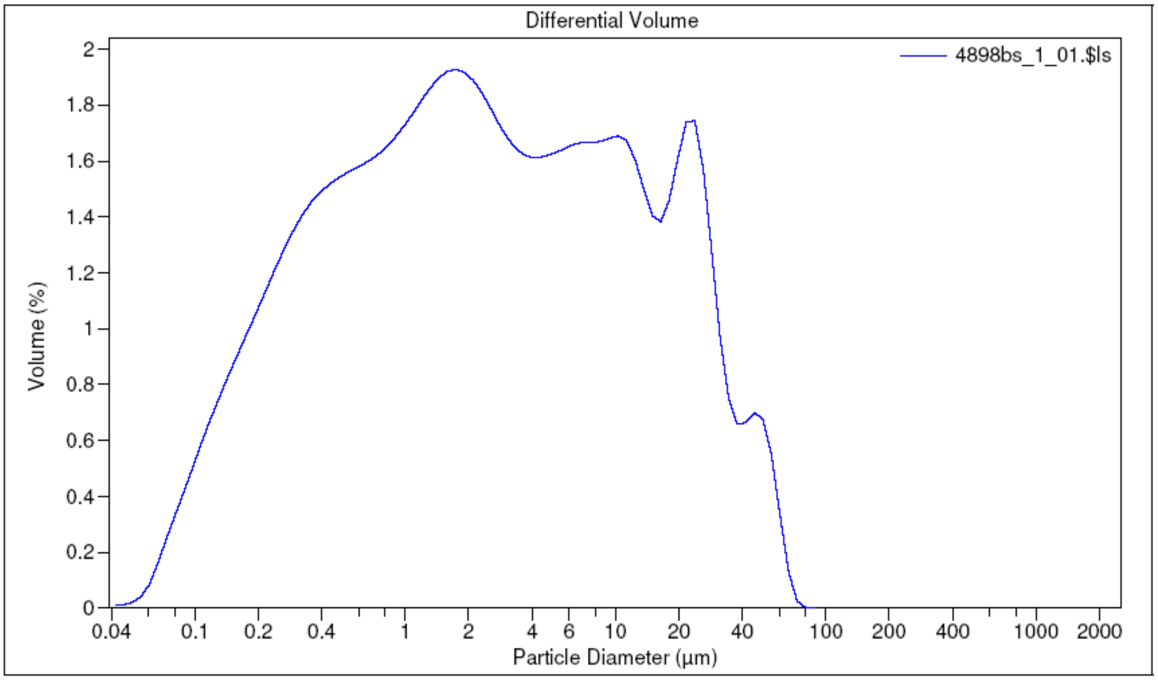
>2 μm	>5 μm	>20 μm	>50 μm	>100 μm	>250 μm	>500 μm
80.6%	70.1%	44.3%	11.1%	0.00017%	0%	0%

BECKMAN COULTER™

LS Particle Size Analyzer

11 Dec 2008 14:27

Beckman Coulter LS 13 320



Volume Statistics (Arithmetic) 4898bs_1_01.$ls

Calculations from 0.040 µm to 2000 µm

Volume:	100%		
Mean:	7.459 µm	S.D.:	11.26 µm
Median:	2.314 µm	Variance:	126.8 µm^2
Mean/Median ratio:	3.223	C.V.:	151%
Mode:	1.748 µm	Skewness:	2.354 Right skewed
		Kurtosis:	5.953 Leptokurtic

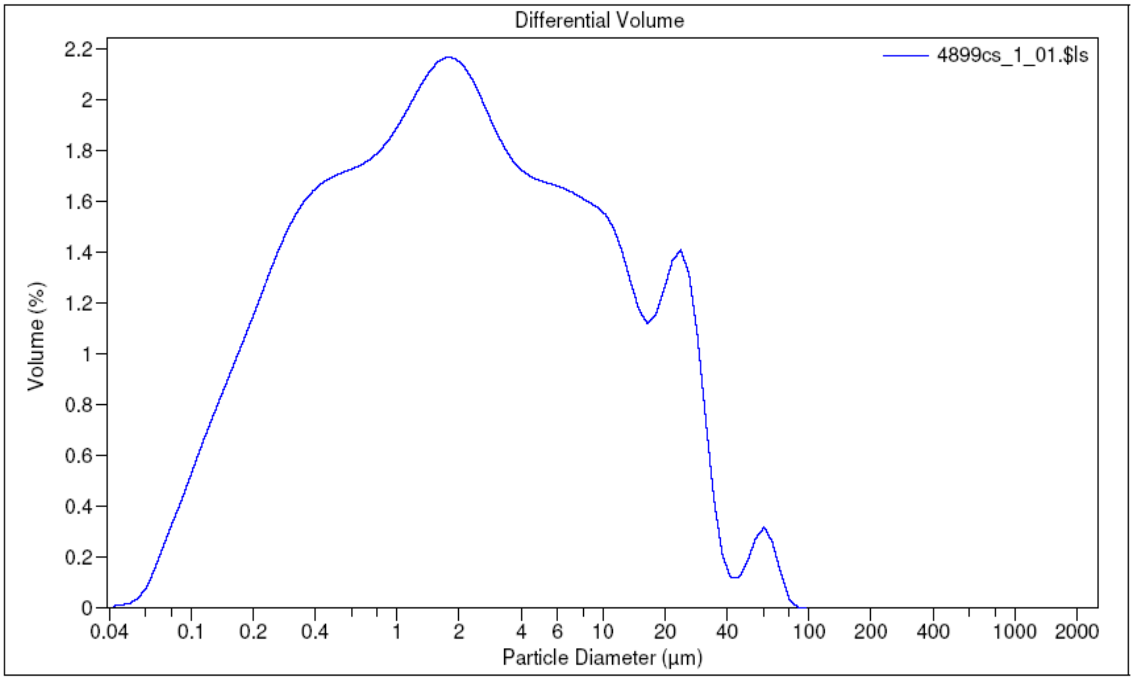
<10%	<25%	<50%	<75%	<90%		
0.240 µm	0.629 µm	2.314 µm	9.355 µm	22.78 µm		
>10%	>25%	>50%	>75%	>90%		
22.78 µm	9.355 µm	2.314 µm	0.629 µm	0.240 µm		
<2 µm	<5 µm	<20 µm	<50 µm	<100 µm	<250 µm	<500 µm
47.1%	63.9%	87.6%	98.6%	100%	100%	100%
>2 µm	>5 µm	>20 µm	>50 µm	>100 µm	>250 µm	>500 µm
52.9%	36.1%	12.4%	1.42%	0%	0%	0%

BECKMAN COULTER

LS Particle Size Analyzer

11 Dec 2008 14:46

Beckman Coulter LS 13 320



Volume Statistics (Arithmetic) 4899cs_1_01.$ls

Calculations from 0.040 µm to 2000 µm

Volume:	100%		
Mean:	5.905 µm	S.D.:	9.746 µm
Median:	1.895 µm	Variance:	94.98 µm^2
Mean/Median ratio:	3.117	C.V.:	165%
Mode:	1.748 µm	Skewness:	3.232 Right skewed
		Kurtosis:	13.64 Leptokurtic

<10%	<25%	<50%	<75%	<90%
0.233 µm	0.568 µm	1.895 µm	6.722 µm	17.81 µm

>10%	>25%	>50%	>75%	>90%
17.81 µm	6.722 µm	1.895 µm	0.568 µm	0.233 µm

<2 µm	<5 µm	<20 µm	<50 µm	<100 µm	<250 µm	<500 µm
51.3%	69.7%	91.5%	98.9%	100%	100%	100%

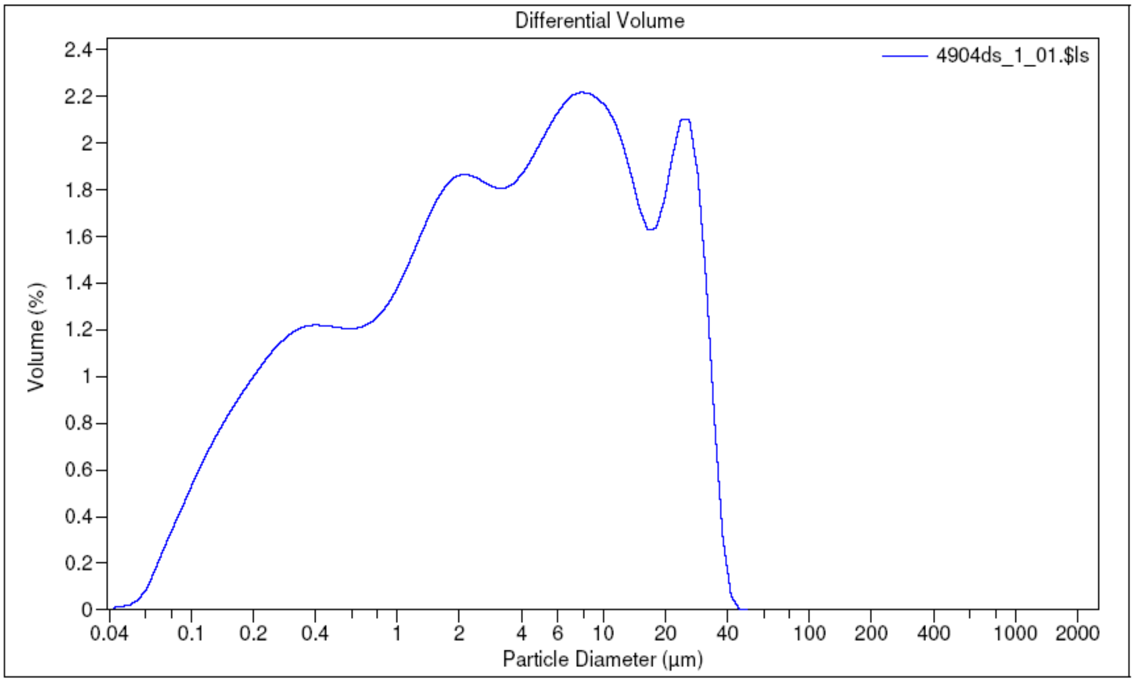
>2 µm	>5 µm	>20 µm	>50 µm	>100 µm	>250 µm	>500 µm
48.7%	30.3%	8.51%	1.14%	0.000008%	0%	0%

BECKMAN COULTER

LS Particle Size Analyzer

11 Dec 2008 14:49

Beckman Coulter LS 13 320



Volume Statistics (Arithmetic) 4904ds_1_01.$ls

Calculations from 0.040 µm to 2000 µm

Volume:	100%		
Mean:	7.034 µm	S.D.:	8.587 µm
Median:	3.223 µm	Variance:	73.74 µm^2
Mean/Median ratio:	2.182	C.V.:	122%
Mode:	7.775 µm	Skewness:	1.541 Right skewed
		Kurtosis:	1.580 Leptokurtic

<10%	<25%	<50%	<75%	<90%
0.248 µm	0.793 µm	3.223 µm	10.05 µm	21.39 µm

>10%	>25%	>50%	>75%	>90%
21.39 µm	10.05 µm	3.223 µm	0.793 µm	0.248 µm

<2 µm	<5 µm	<20 µm	<50 µm	<100 µm	<250 µm	<500 µm
40.6%	58.8%	88.7%	100%	100%	100%	100%

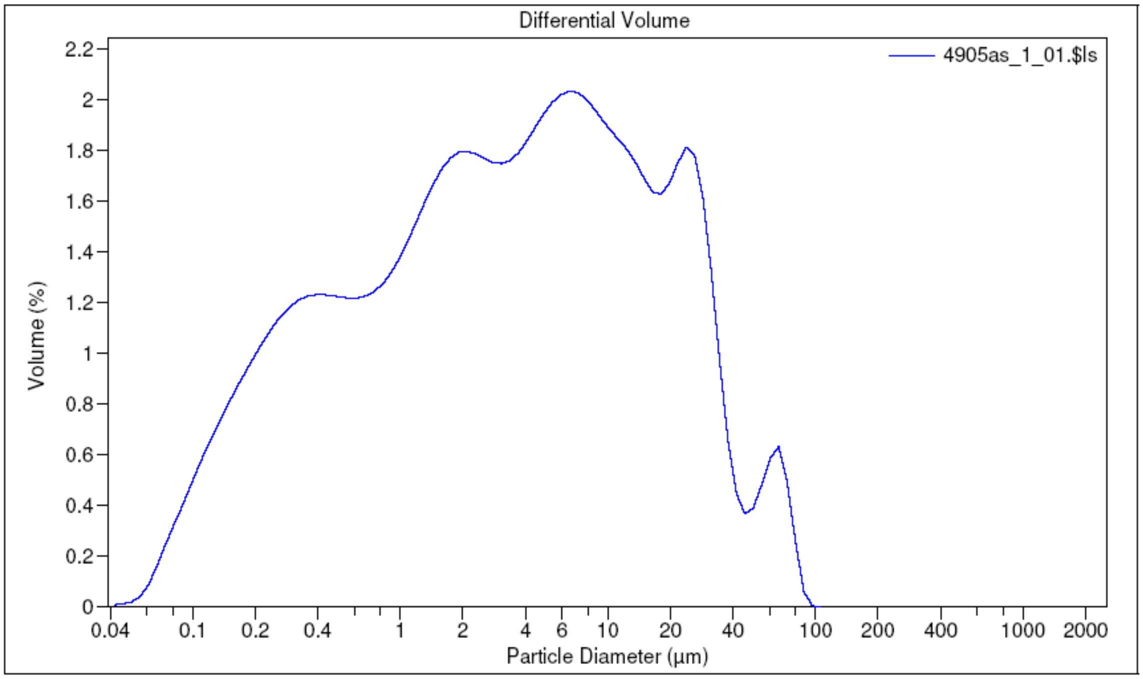
>2 µm	>5 µm	>20 µm	>50 µm	>100 µm	>250 µm	>500 µm
59.4%	41.2%	11.3%	0%	0%	0%	0%

BECKMAN COULTER™

LS Particle Size Analyzer

11 Dec 2008 14:50

Beckman Coulter LS 13 320


Differential Volume

Volume Statistics (Arithmetic) 4905as_1_01.$ls

Calculations from 0.040 µm to 2000 µm

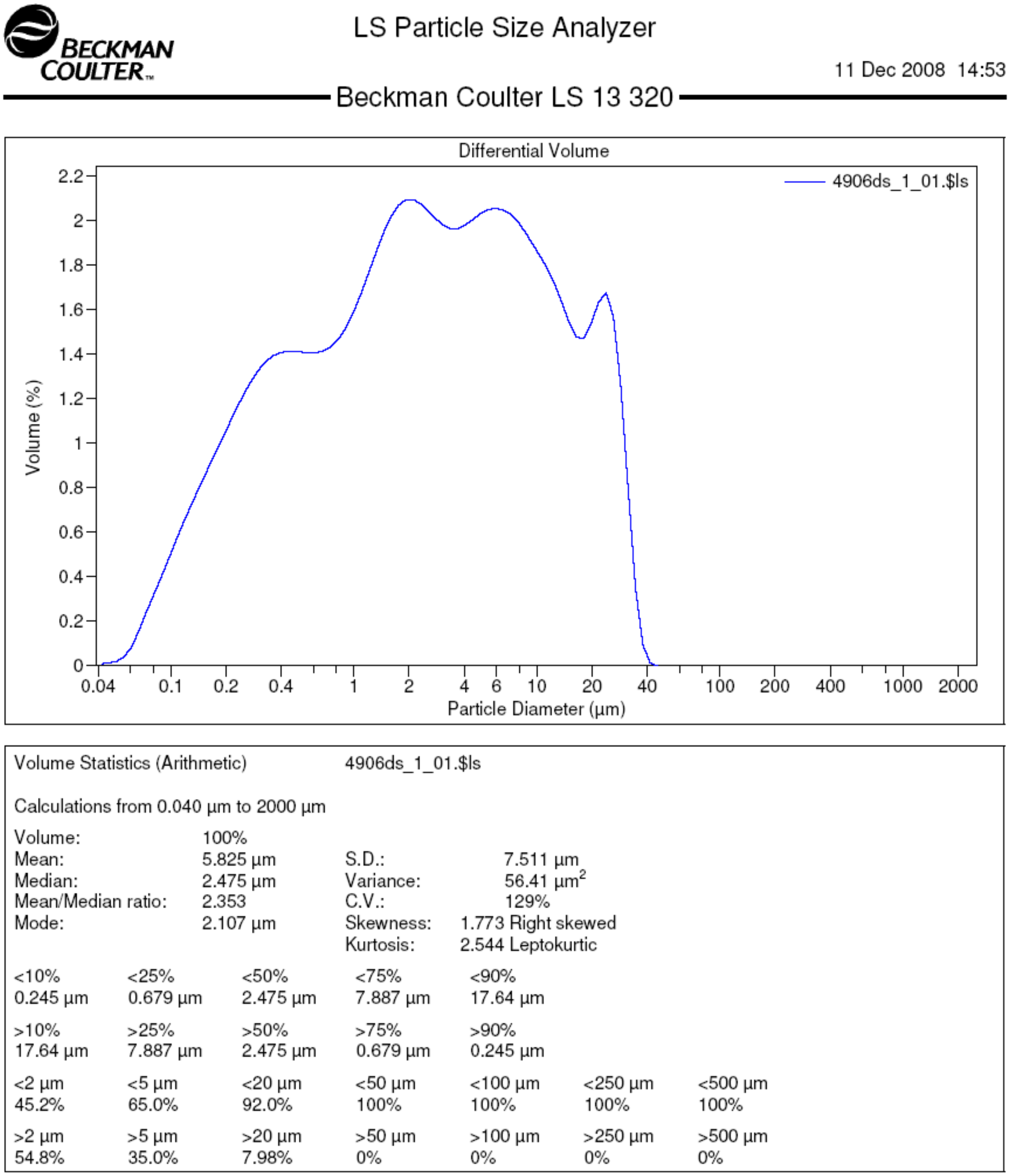
Volume:	100%		
Mean:	8.875 µm	S.D.:	13.32 µm
Median:	3.336 µm	Variance:	177.4 µm^2
Mean/Median ratio:	2.660	C.V.:	150%
Mode:	6.452 µm	Skewness:	2.657 Right skewed
		Kurtosis:	8.265 Leptokurtic

<10%	<25%	<50%	<75%	<90%
0.253 µm	0.799 µm	3.336 µm	11.16 µm	25.05 µm

>10%	>25%	>50%	>75%	>90%
25.05 µm	11.16 µm	3.336 µm	0.799 µm	0.253 µm

<2 µm	<5 µm	<20 µm	<50 µm	<100 µm	<250 µm	<500 µm
40.3%	58.0%	85.7%	97.3%	99.999%	100%	100%

>2 µm	>5 µm	>20 µm	>50 µm	>100 µm	>250 µm	>500 µm
59.7%	42.0%	14.3%	2.74%	0.00076%	0%	0%



BECKMAN COULTER

LS Particle Size Analyzer

11 Dec 2008 14:53

Beckman Coulter LS 13 320

Volume Statistics (Arithmetic) 4906ds_1_01.$ls

Calculations from 0.040 µm to 2000 µm

Volume:	100%		
Mean:	5.825 µm	S.D.:	7.511 µm
Median:	2.475 µm	Variance:	56.41 µm^2
Mean/Median ratio:	2.353	C.V.:	129%
Mode:	2.107 µm	Skewness:	1.773 Right skewed
		Kurtosis:	2.544 Leptokurtic

<10%	<25%	<50%	<75%	<90%
0.245 µm	0.679 µm	2.475 µm	7.887 µm	17.64 µm

>10%	>25%	>50%	>75%	>90%
17.64 µm	7.887 µm	2.475 µm	0.679 µm	0.245 µm

<2 µm	<5 µm	<20 µm	<50 µm	<100 µm	<250 µm	<500 µm
45.2%	65.0%	92.0%	100%	100%	100%	100%

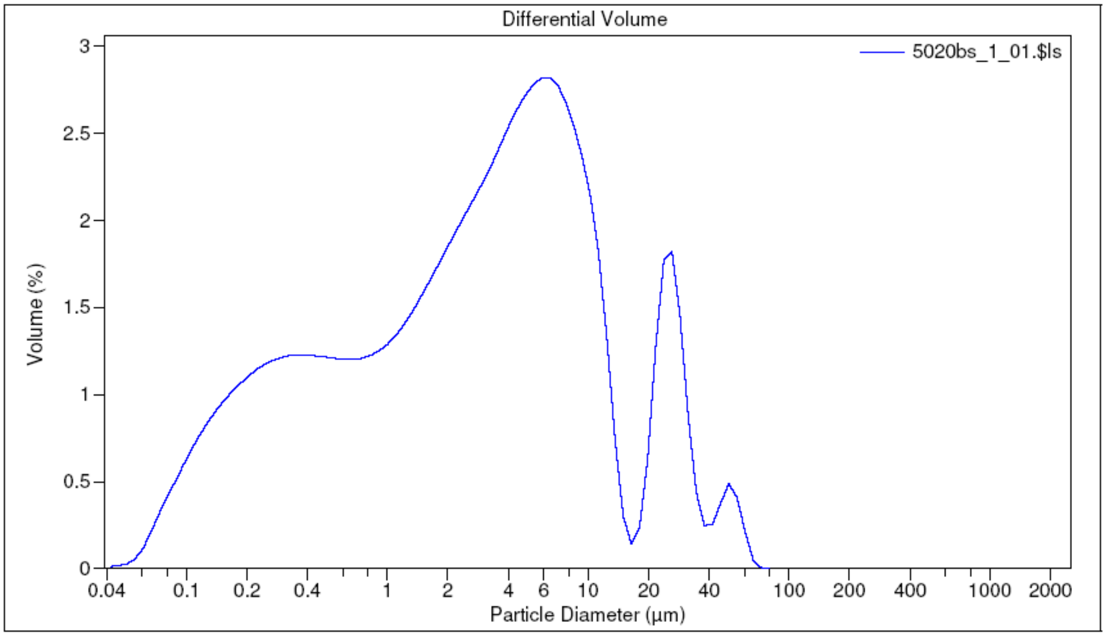
>2 µm	>5 µm	>20 µm	>50 µm	>100 µm	>250 µm	>500 µm
54.8%	35.0%	7.98%	0%	0%	0%	0%

BECKMAN COULTER™

LS Particle Size Analyzer

11 Dec 2008 14:55

Beckman Coulter LS 13 320



Volume Statistics (Arithmetic) 5020bs_1_01.$ls

Calculations from 0.040 µm to 2000 µm

Volume:	100%		
Mean:	6.363 µm	S.D.:	9.486 µm
Median:	3.007 µm	Variance:	89.99 µm^2
Mean/Median ratio:	2.116	C.V.:	149%
Mode:	6.452 µm	Skewness:	2.799 Right skewed
		Kurtosis:	9.092 Leptokurtic

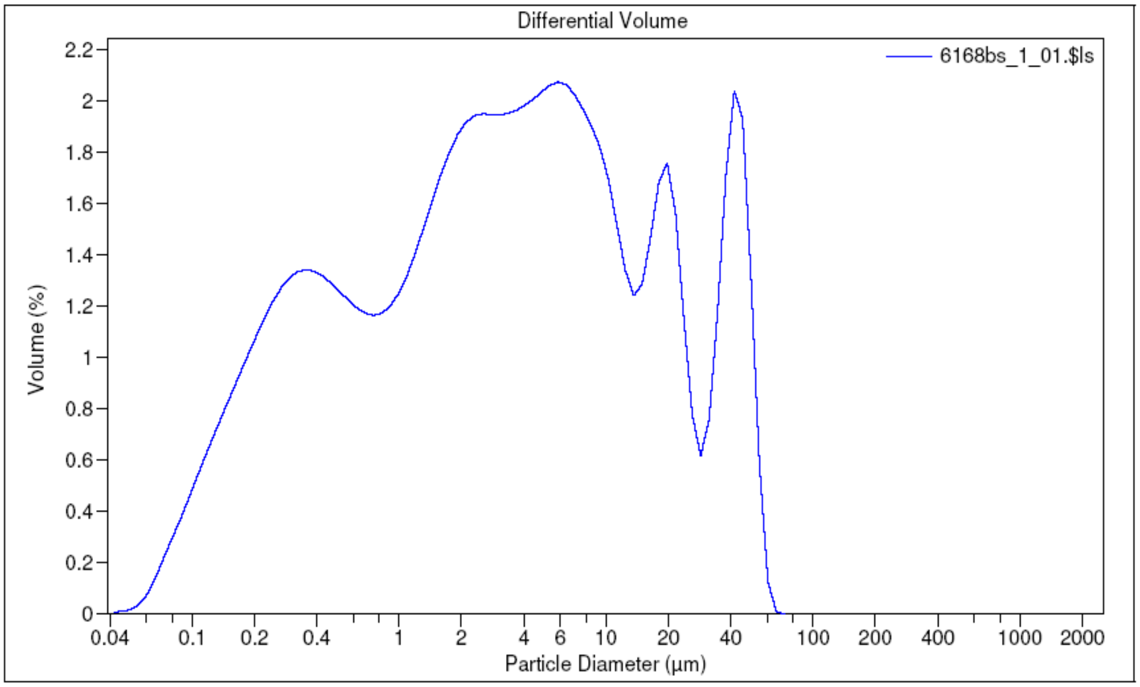
<10%	<25%	<50%	<75%	<90%		
0.220 µm	0.703 µm	3.007 µm	7.317 µm	19.74 µm		
>10%	>25%	>50%	>75%	>90%		
19.74 µm	7.317 µm	3.007 µm	0.703 µm	0.220 µm		
<2 µm	<5 µm	<20 µm	<50 µm	<100 µm	<250 µm	<500 µm
41.1%	63.6%	90.1%	99.1%	100%	100%	100%
>2 µm	>5 µm	>20 µm	>50 µm	>100 µm	>250 µm	>500 µm
58.9%	36.4%	9.91%	0.95%	0%	0%	0%

BECKMAN COULTER

LS Particle Size Analyzer

29 Oct 2008 16:35

Beckman Coulter LS 13 320



Volume Statistics (Arithmetic) 6168bs_1_01.$ls

Calculations from 0.040 µm to 2000 µm

Volume: 100%
Mean: 8.795 µm
Median: 3.148 µm
Mean/Median ratio: 2.794
Mode: 5.878 µm

S.D.: 12.74 µm
Variance: 162.4 µm^2
C.V.: 145%
Skewness: 1.975 Right skewed
Kurtosis: 3.094 Leptokurtic

<10%	<25%	<50%	<75%	<90%
0.247 µm	0.742 µm	3.148 µm	10.24 µm	28.58 µm

>10%	>25%	>50%	>75%	>90%
28.58 µm	10.24 µm	3.148 µm	0.742 µm	0.247 µm

<2 µm	<5 µm	<20 µm	<50 µm	<100 µm	<250 µm	<500 µm
40.6%	59.8%	85.6%	98.5%	100%	100%	100%

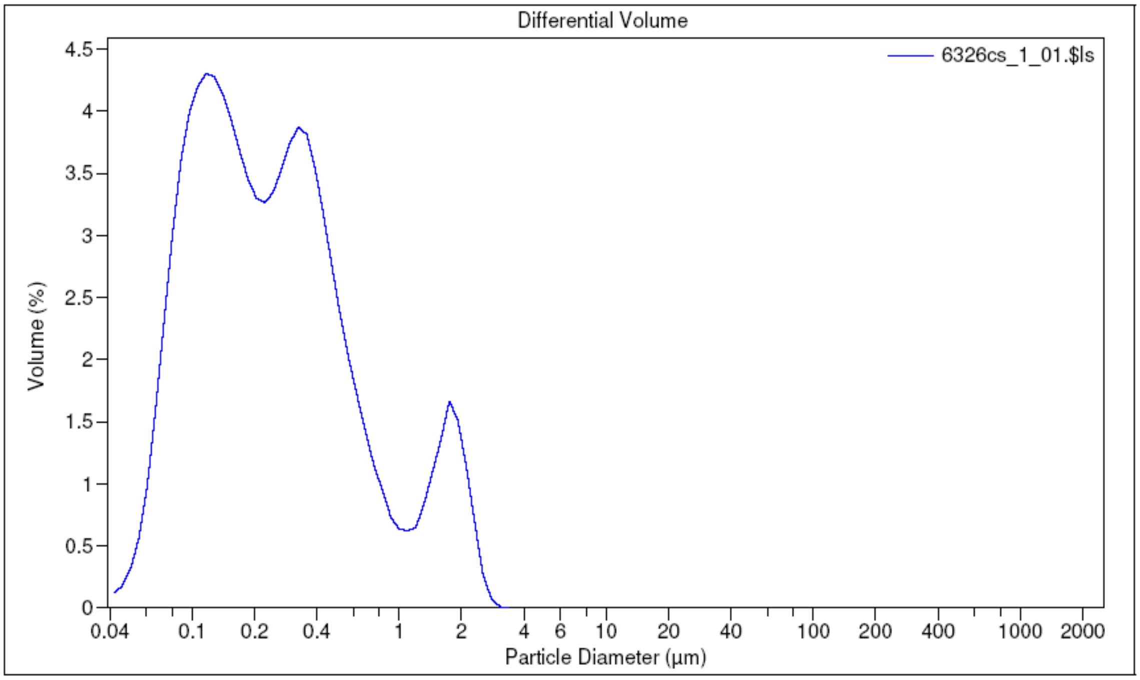
>2 µm	>5 µm	>20 µm	>50 µm	>100 µm	>250 µm	>500 µm
59.4%	40.2%	14.4%	1.46%	0%	0%	0%

BECKMAN COULTER

LS Particle Size Analyzer

29 Oct 2008 16:42

Beckman Coulter LS 13 320



Volume Statistics (Arithmetic) 6326cs_1_01.$ls

Calculations from 0.040 µm to 2000 µm

Volume:	100%		
Mean:	0.409 µm	S.D.:	0.489 µm
Median:	0.227 µm	Variance:	0.239 µm^2
Mean/Median ratio:	1.798	C.V.:	120%
Mode:	0.117 µm	Skewness:	2.356 Right skewed
		Kurtosis:	5.118 Leptokurtic

<10%	<25%	<50%	<75%	<90%
0.086 µm	0.122 µm	0.227 µm	0.436 µm	1.043 µm

>10%	>25%	>50%	>75%	>90%
1.043 µm	0.436 µm	0.227 µm	0.122 µm	0.086 µm

<2 µm	<5 µm	<20 µm	<50 µm	<100 µm	<250 µm	<500 µm
97.7%	100%	100%	100%	100%	100%	100%

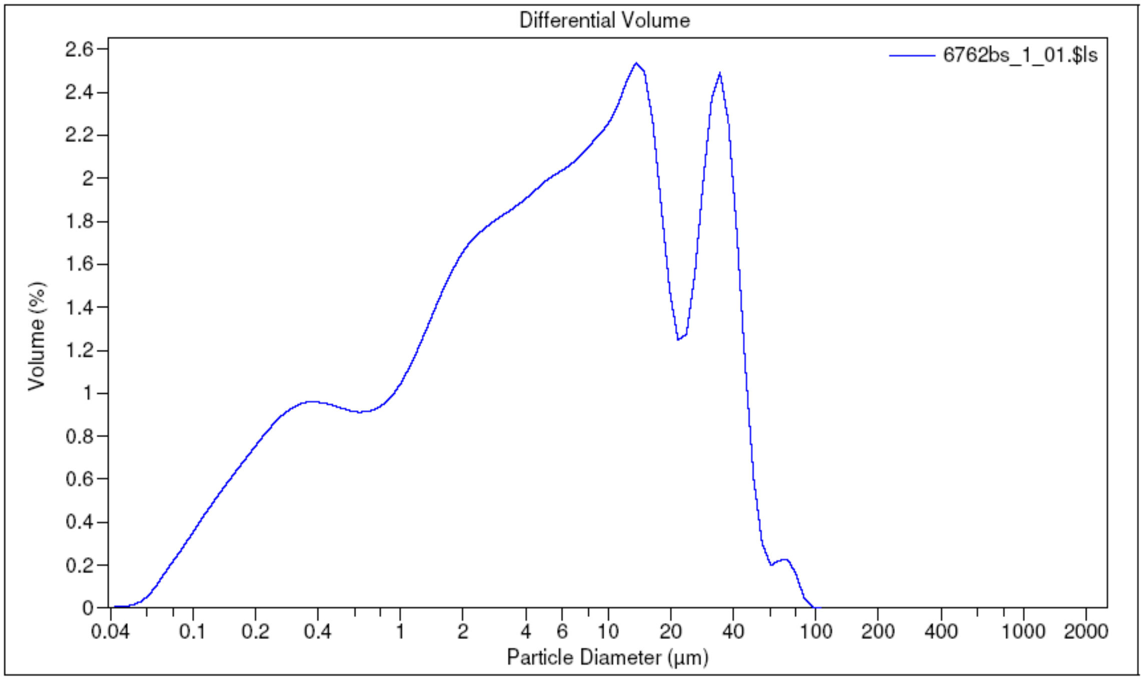
>2 µm	>5 µm	>20 µm	>50 µm	>100 µm	>250 µm	>500 µm
2.32%	0%	0%	0%	0%	0%	0%

BECKMAN COULTER

LS Particle Size Analyzer

29 Oct 2008 16:38

Beckman Coulter LS 13 320

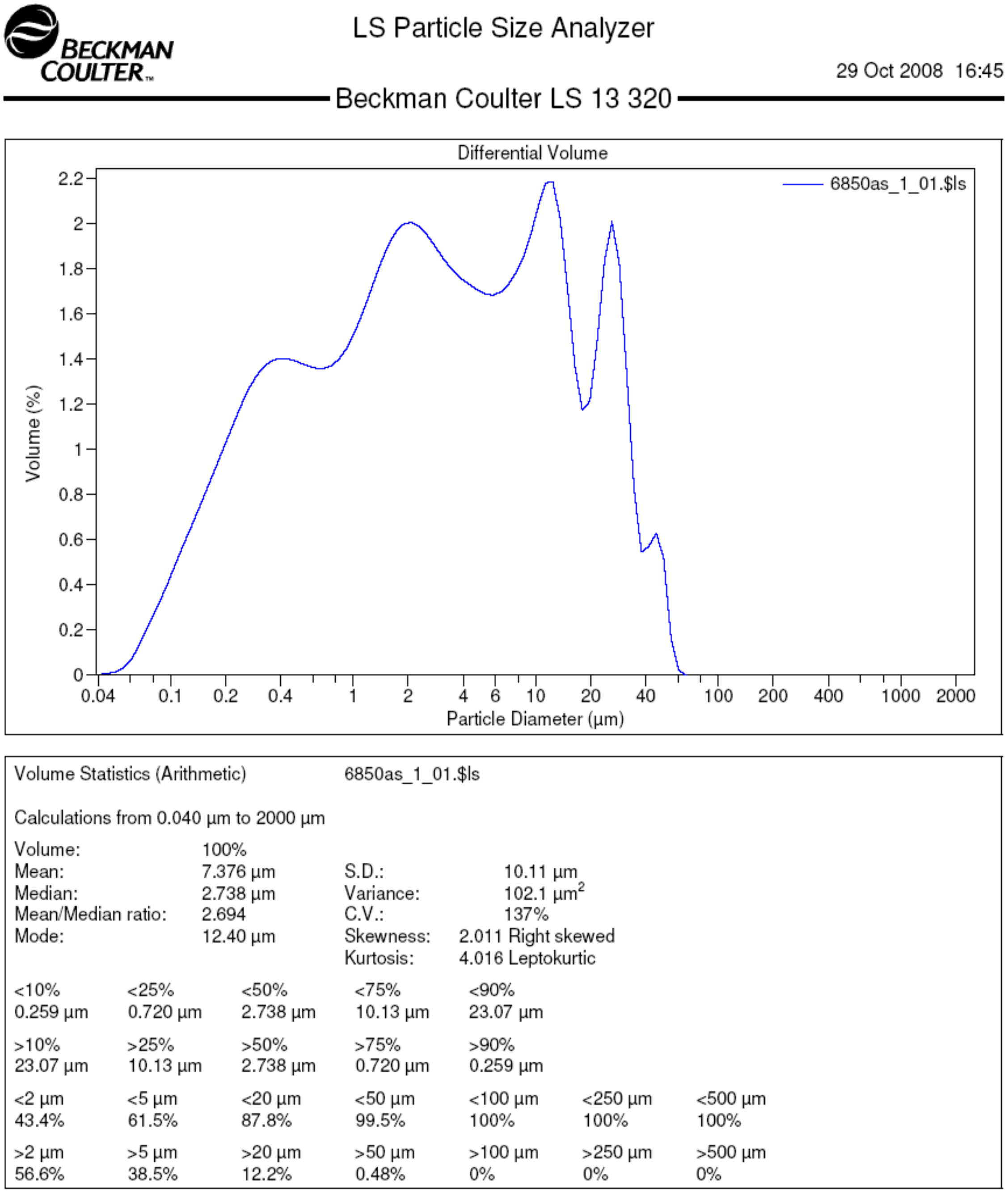


Volume Statistics (Arithmetic) 6762bs_1_01.$ls

Calculations from 0.040 µm to 2000 µm

Volume:	100%		
Mean:	10.78 µm	S.D.:	13.39 µm
Median:	5.173 µm	Variance:	179.4 µm^2
Mean/Median ratio:	2.084	C.V.:	124%
Mode:	13.61 µm	Skewness:	1.837 Right skewed
		Kurtosis:	3.666 Leptokurtic

<10%	<25%	<50%	<75%	<90%		
0.331 µm	1.352 µm	5.173 µm	14.77 µm	32.25 µm		
>10%	>25%	>50%	>75%	>90%		
32.25 µm	14.77 µm	5.173 µm	1.352 µm	0.331 µm		
<2 µm	<5 µm	<20 µm	<50 µm	<100 µm	<250 µm	<500 µm
31.3%	49.3%	81.6%	98.5%	99.999%	100%	100%
>2 µm	>5 µm	>20 µm	>50 µm	>100 µm	>250 µm	>500 µm
68.7%	50.7%	18.4%	1.50%	0.00066%	0%	0%



BECKMAN COULTER

LS Particle Size Analyzer

29 Oct 2008 16:45

Beckman Coulter LS 13 320

Volume Statistics (Arithmetic) 6850as_1_01.$ls

Calculations from 0.040 µm to 2000 µm

Volume:	100%		
Mean:	7.376 µm	S.D.:	10.11 µm
Median:	2.738 µm	Variance:	102.1 μm^2
Mean/Median ratio:	2.694	C.V.:	137%
Mode:	12.40 µm	Skewness:	2.011 Right skewed
		Kurtosis:	4.016 Leptokurtic

<10%	<25%	<50%	<75%	<90%
0.259 µm	0.720 µm	2.738 µm	10.13 µm	23.07 µm

>10%	>25%	>50%	>75%	>90%
23.07 µm	10.13 µm	2.738 µm	0.720 µm	0.259 µm

<2 µm	<5 µm	<20 µm	<50 µm	<100 µm	<250 µm	<500 µm
43.4%	61.5%	87.8%	99.5%	100%	100%	100%

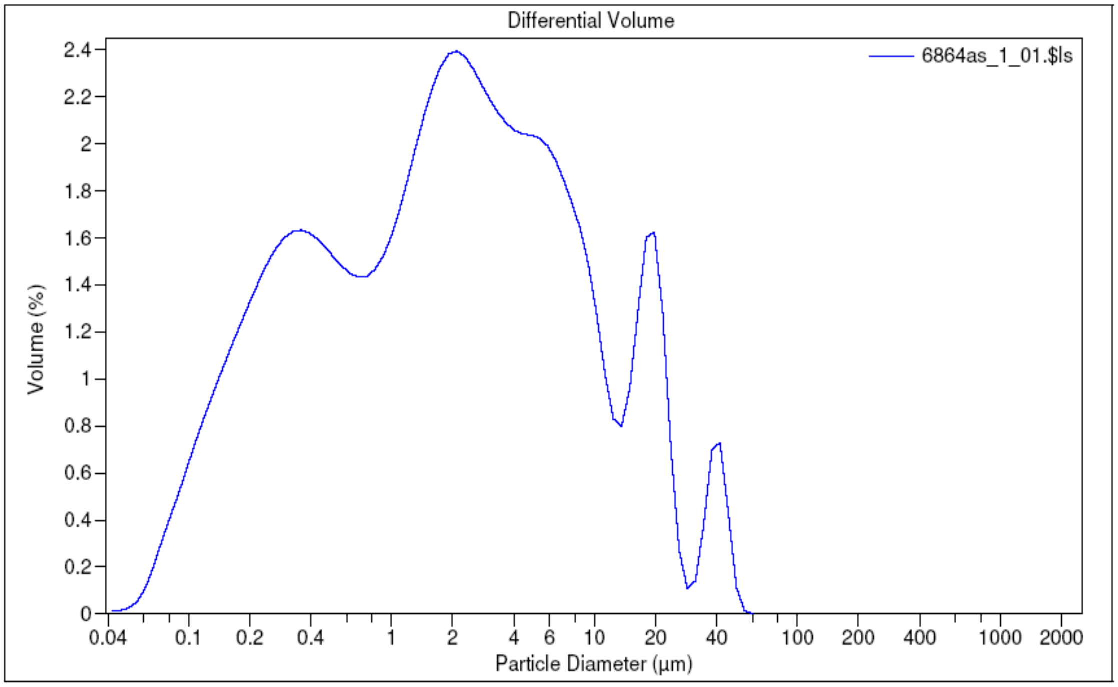
>2 µm	>5 µm	>20 µm	>50 µm	>100 µm	>250 µm	>500 µm
56.6%	38.5%	12.2%	0.48%	0%	0%	0%

BECKMAN COULTER

LS Particle Size Analyzer

19 Nov 2008 15:00

Beckman Coulter LS 13 320



Volume Statistics (Arithmetic) 6864as_1_01.$ls

Calculations from 0.040 µm to 2000 µm

Volume: 100%
Mean: 4.958 µm
Median: 1.920 µm
Mean/Median ratio: 2.582
Mode: 2.107 µm
S.D.: 7.820 µm
Variance: 61.16 µm^2
C.V.: 158%
Skewness: 2.900 Right skewed
Kurtosis: 9.619 Leptokurtic

<10%	<25%	<50%	<75%	<90%
0.207 µm	0.507 µm	1.920 µm	5.586 µm	14.78 µm

>10%	>25%	>50%	>75%	>90%
14.78 µm	5.586 µm	1.920 µm	0.507 µm	0.207 µm

<2 µm	<5 µm	<20 µm	<50 µm	<100 µm	<250 µm	<500 µm
51.1%	72.6%	94.5%	99.9%	100%	100%	100%

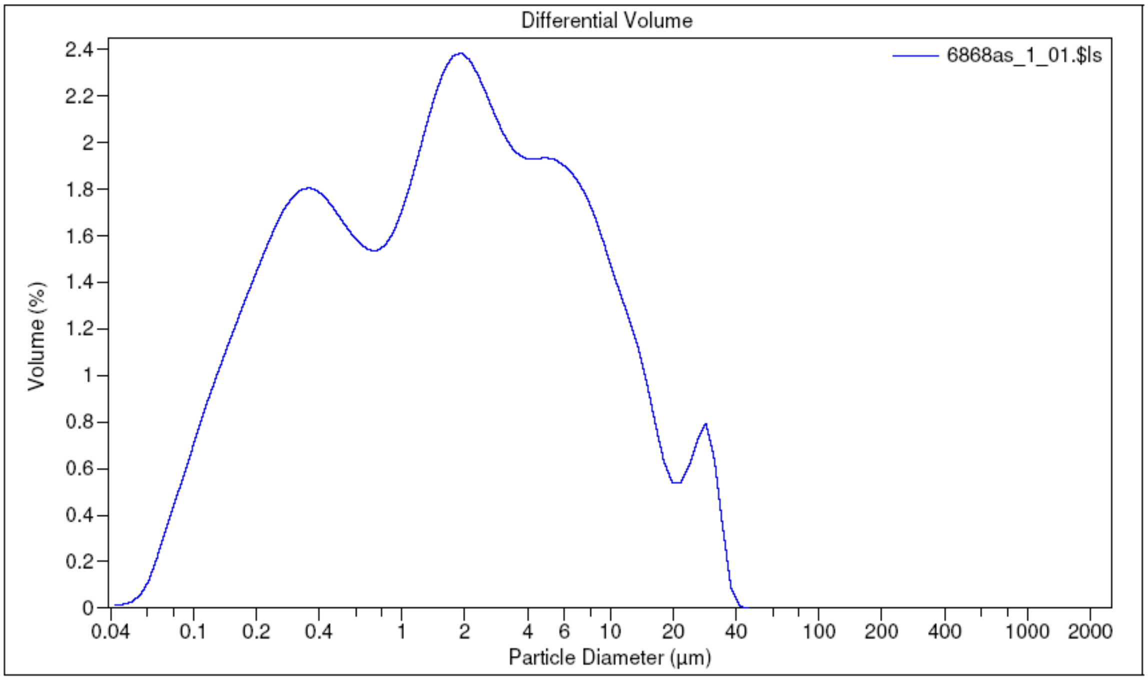
>2 µm	>5 µm	>20 µm	>50 µm	>100 µm	>250 µm	>500 µm
48.9%	27.4%	5.50%	0.074%	0%	0%	0%

BECKMAN COULTER

LS Particle Size Analyzer

29 Oct 2008 16:50

Beckman Coulter LS 13 320



Volume Statistics (Arithmetic) 6868as_1_01.$ls

Calculations from 0.040 µm to 2000 µm

Volume: 100%
Mean: 4.092 µm
Median: 1.656 µm
Mean/Median ratio: 2.471
Mode: 1.919 µm

S.D.: 6.131 µm
Variance: 37.59 µm^2
C.V.: 150%
Skewness: 2.652 Right skewed
Kurtosis: 7.711 Leptokurtic

<10%	<25%	<50%	<75%	<90%
0.195 µm	0.445 µm	1.656 µm	4.952 µm	11.06 µm

>10%	>25%	>50%	>75%	>90%
11.06 µm	4.952 µm	1.656 µm	0.445 µm	0.195 µm

<2 µm	<5 µm	<20 µm	<50 µm	<100 µm	<250 µm	<500 µm
54.8%	75.2%	96.1%	100%	100%	100%	100%

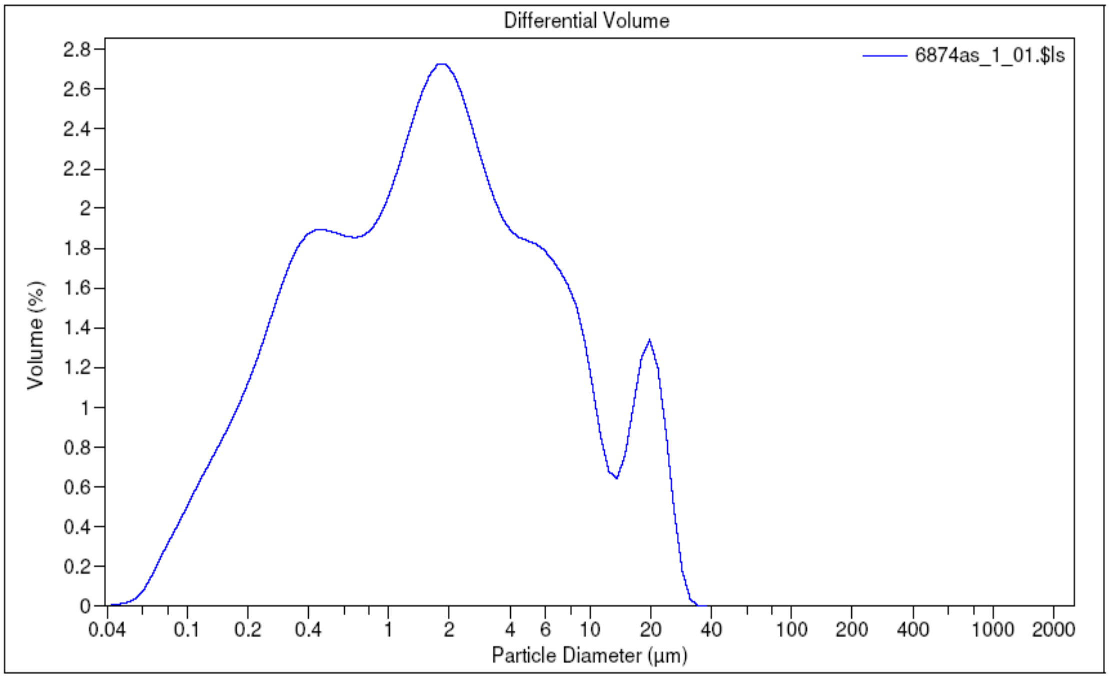
>2 µm	>5 µm	>20 µm	>50 µm	>100 µm	>250 µm	>500 µm
45.2%	24.8%	3.95%	0%	0%	0%	0%

BECKMAN COULTER

LS Particle Size Analyzer

19 Nov 2008 15:04

Beckman Coulter LS 13 320



Volume Statistics (Arithmetic) 6874as_1_01.$ls

Calculations from 0.040 µm to 2000 µm

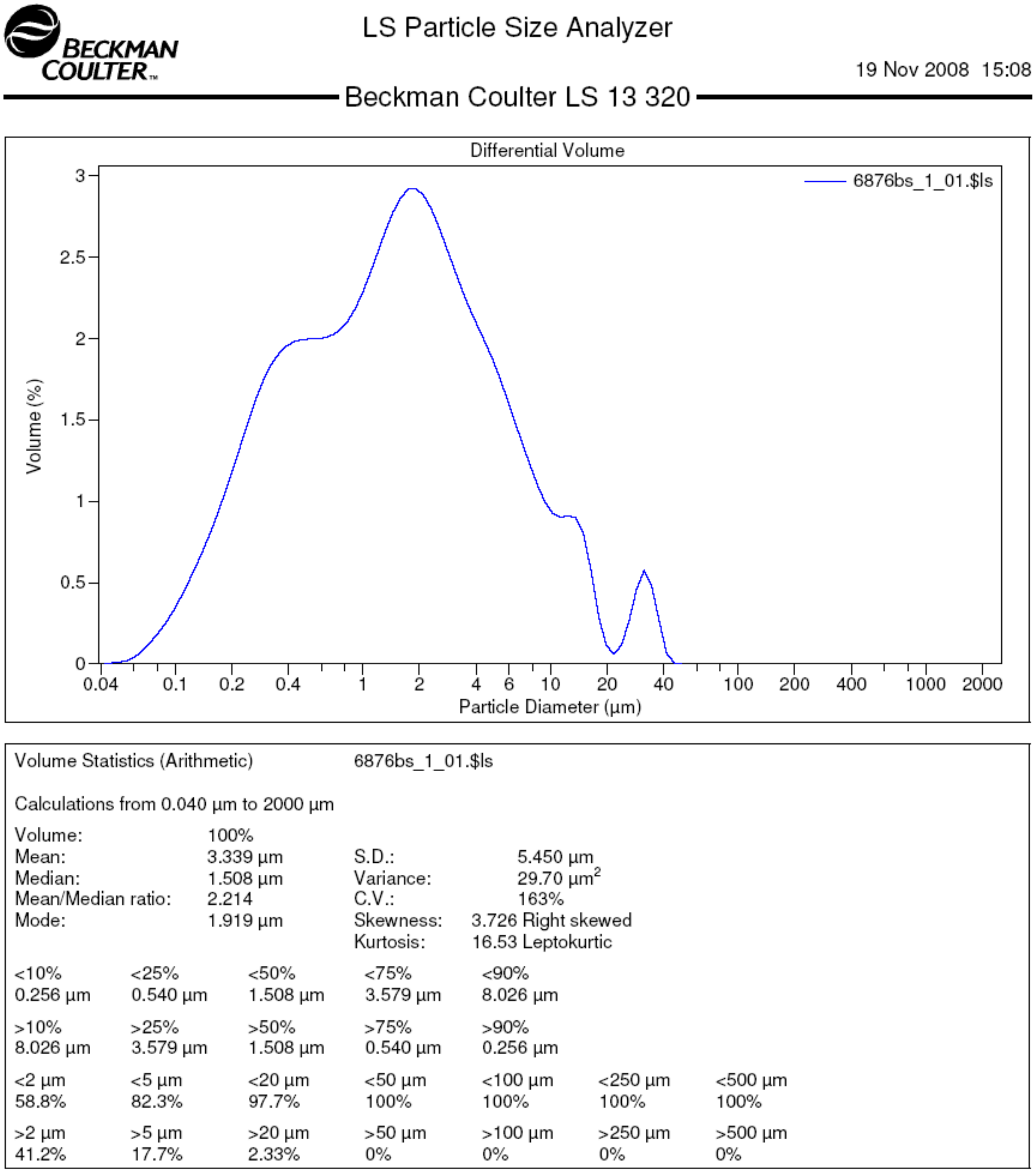
Volume:	100%		
Mean:	3.763 µm	S.D.:	5.354 µm
Median:	1.620 µm	Variance:	28.66 µm^2
Mean/Median ratio:	2.323	C.V.:	142%
Mode:	1.919 µm	Skewness:	2.332 Right skewed
		Kurtosis:	5.234 Leptokurtic

<10%	<25%	<50%	<75%	<90%
0.240 µm	0.536 µm	1.620 µm	4.384 µm	10.18 µm

>10%	>25%	>50%	>75%	>90%
10.18 µm	4.384 µm	1.620 µm	0.536 µm	0.240 µm

<2 µm	<5 µm	<20 µm	<50 µm	<100 µm	<250 µm	<500 µm
56.2%	77.6%	96.7%	100%	100%	100%	100%

>2 µm	>5 µm	>20 µm	>50 µm	>100 µm	>250 µm	>500 µm
43.8%	22.4%	3.28%	0%	0%	0%	0%



LS Particle Size Analyzer

19 Nov 2008 15:08

Beckman Coulter LS 13 320

Volume Statistics (Arithmetic) 6876bs_1_01.$ls

Calculations from 0.040 µm to 2000 µm

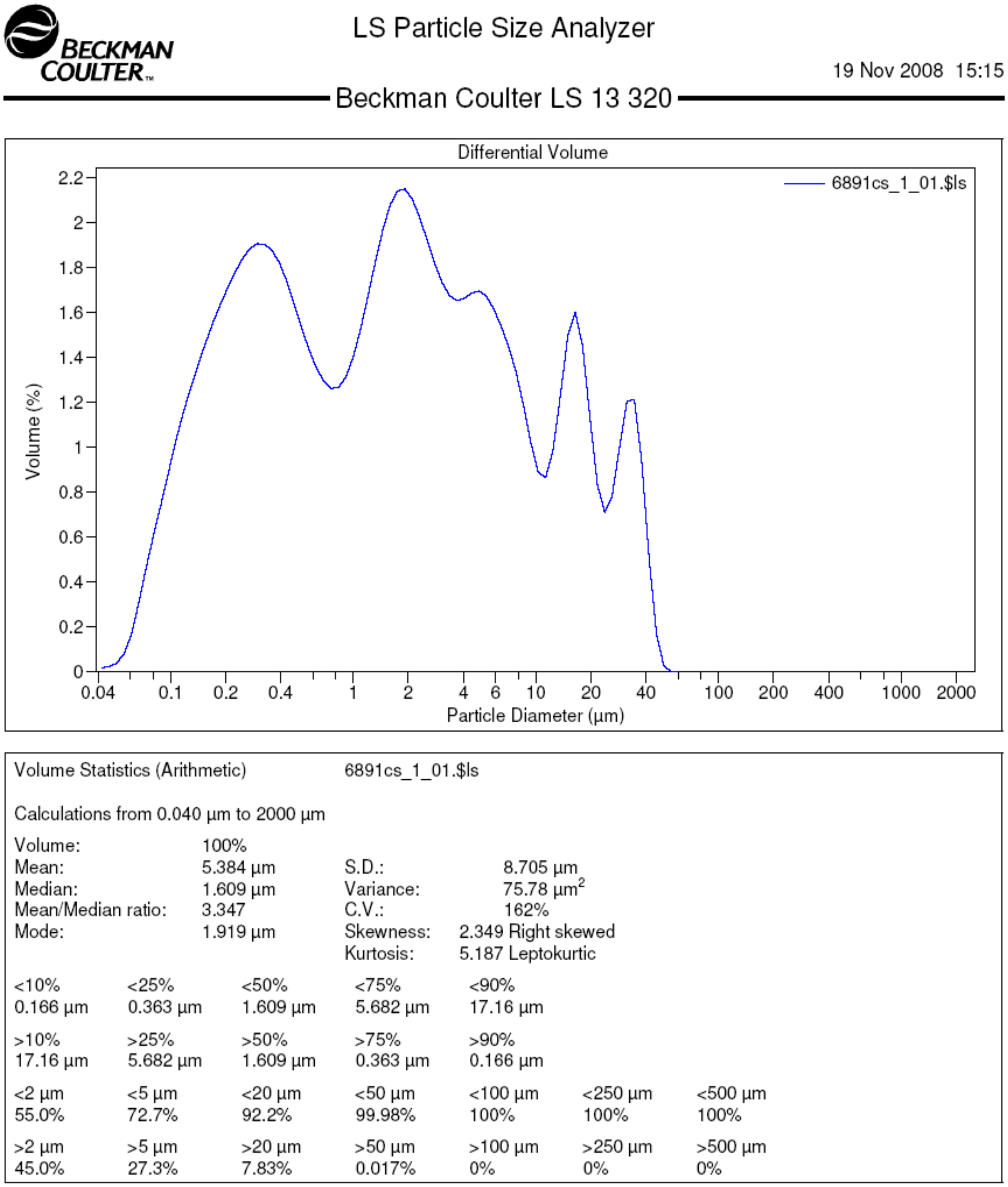
Volume:	100%		
Mean:	3.339 µm	S.D.:	5.450 µm
Median:	1.508 µm	Variance:	29.70 µm^2
Mean/Median ratio:	2.214	C.V.:	163%
Mode:	1.919 µm	Skewness:	3.726 Right skewed
		Kurtosis:	16.53 Leptokurtic

<10%	<25%	<50%	<75%	<90%
0.256 µm	0.540 µm	1.508 µm	3.579 µm	8.026 µm

>10%	>25%	>50%	>75%	>90%
8.026 µm	3.579 µm	1.508 µm	0.540 µm	0.256 µm

<2 µm	<5 µm	<20 µm	<50 µm	<100 µm	<250 µm	<500 µm
58.8%	82.3%	97.7%	100%	100%	100%	100%

>2 µm	>5 µm	>20 µm	>50 µm	>100 µm	>250 µm	>500 µm
41.2%	17.7%	2.33%	0%	0%	0%	0%



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LS Particle Size Analyzer

19 Nov 2008 15:15

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Volume Statistics (Arithmetic) 6891cs_1_01.$ls

Calculations from 0.040 µm to 2000 µm

Volume:	100%		
Mean:	5.384 µm	S.D.:	8.705 µm
Median:	1.609 µm	Variance:	75.78 µm^2
Mean/Median ratio:	3.347	C.V.:	162%
Mode:	1.919 µm	Skewness:	2.349 Right skewed
		Kurtosis:	5.187 Leptokurtic

<10%	<25%	<50%	<75%	<90%
0.166 µm	0.363 µm	1.609 µm	5.682 µm	17.16 µm

>10%	>25%	>50%	>75%	>90%
17.16 µm	5.682 µm	1.609 µm	0.363 µm	0.166 µm

<2 µm	<5 µm	<20 µm	<50 µm	<100 µm	<250 µm	<500 µm
55.0%	72.7%	92.2%	99.98%	100%	100%	100%

>2 µm	>5 µm	>20 µm	>50 µm	>100 µm	>250 µm	>500 µm
45.0%	27.3%	7.83%	0.017%	0%	0%	0%

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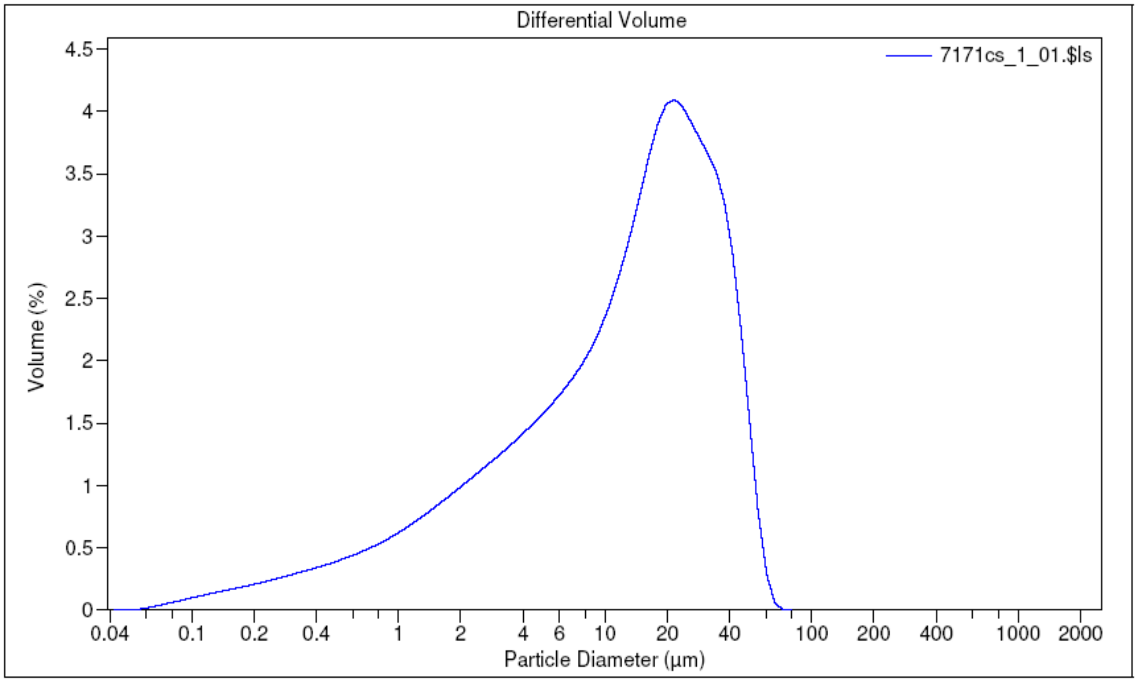

Volume Statistics (Arithmetic) 7171cs_1_01.$ls

Calculations from 0.040 µm to 2000 µm

Volume:	100%		
Mean:	16.63 µm	S.D.:	13.80 µm
Median:	13.83 µm	Variance:	190.6 µm^2
Mean/Median ratio:	1.203	C.V.:	83.0%
Mode:	21.69 µm	Skewness:	0.806 Right skewed
		Kurtosis:	-0.103 Platykurtic

<10%	<25%	<50%	<75%	<90%
1.305 µm	4.584 µm	13.83 µm	25.47 µm	37.27 µm

>10%	>25%	>50%	>75%	>90%
37.27 µm	25.47 µm	13.83 µm	4.584 µm	1.305 µm

<2 µm	<5 µm	<20 µm	<50 µm	<100 µm	<250 µm	<500 µm
14.0%	26.4%	64.5%	98.0%	100%	100%	100%

>2 µm	>5 µm	>20 µm	>50 µm	>100 µm	>250 µm	>500 µm
86.0%	73.6%	35.5%	1.97%	0%	0%	0%

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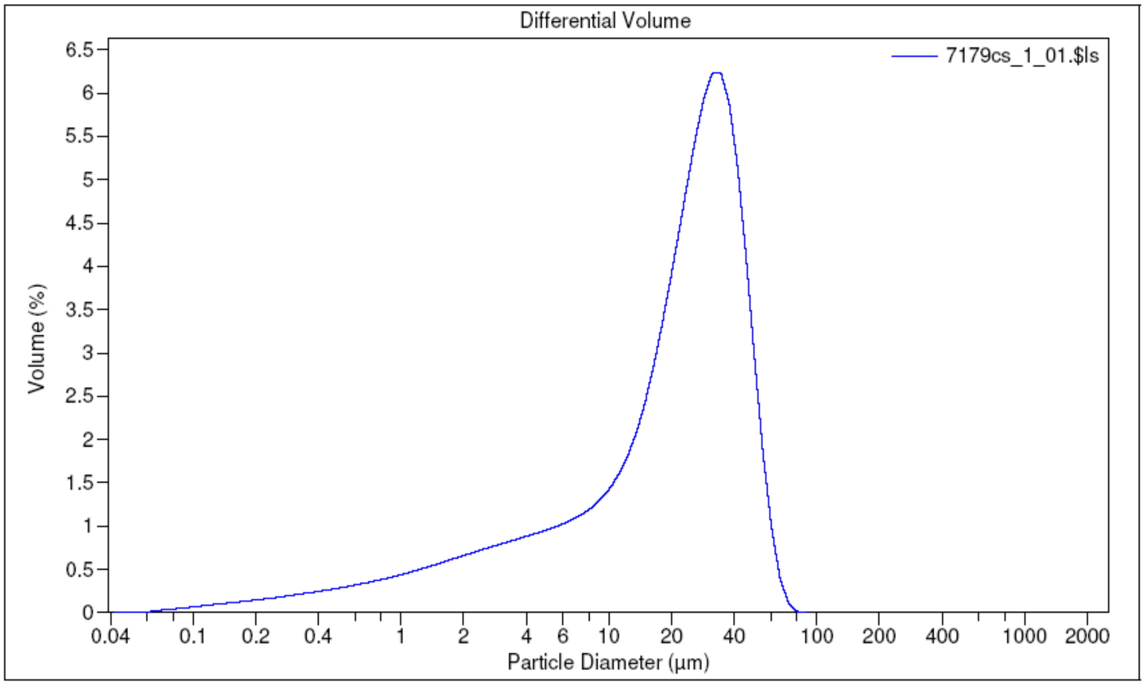

Volume Statistics (Arithmetic) 7179cs_1_01.$ls

Calculations from 0.040 µm to 2000 µm

Volume:	100%		
Mean:	23.08 µm	S.D.:	15.65 µm
Median:	22.86 µm	Variance:	244.9 µm^2
Mean/Median ratio:	1.010	C.V.:	67.8%
Mode:	34.58 µm	Skewness:	0.319 Right skewed
		Kurtosis:	-0.636 Platykurtic

<10%	<25%	<50%	<75%	<90%
2.053 µm	9.244 µm	22.86 µm	34.39 µm	44.10 µm

>10%	>25%	>50%	>75%	>90%
44.10 µm	34.39 µm	22.86 µm	9.244 µm	2.053 µm

<2 µm	<5 µm	<20 µm	<50 µm	<100 µm	<250 µm	<500 µm
9.82%	17.7%	43.9%	95.0%	100%	100%	100%

>2 µm	>5 µm	>20 µm	>50 µm	>100 µm	>250 µm	>500 µm
90.2%	82.3%	56.1%	5.01%	0%	0%	0%

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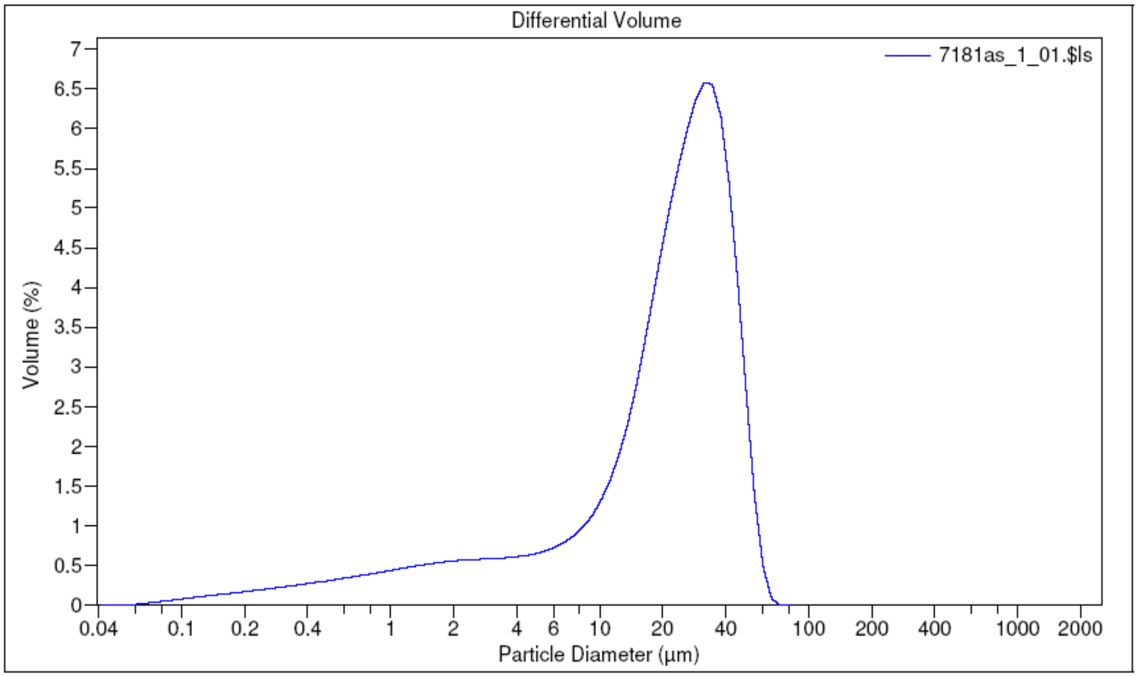

Volume Statistics (Arithmetic) 7181as_1_01.$ls

Calculations from 0.040 µm to 2000 µm

Volume:	100%		
Mean:	23.16 µm	S.D.:	14.63 µm
Median:	23.21 µm	Variance:	214.0 µm^2
Mean/Median ratio:	0.998	C.V.:	63.2%
Mode:	31.50 µm	Skewness:	0.161 Right skewed
		Kurtosis:	-0.743 Platykurtic

<10%	<25%	<50%	<75%	<90%
1.950 µm	11.70 µm	23.21 µm	33.88 µm	42.75 µm

>10%	>25%	>50%	>75%	>90%
42.75 µm	33.88 µm	23.21 µm	11.70 µm	1.950 µm

<2 µm	<5 µm	<20 µm	<50 µm	<100 µm	<250 µm	<500 µm
10.2%	16.0%	42.1%	96.5%	100%	100%	100%

>2 µm	>5 µm	>20 µm	>50 µm	>100 µm	>250 µm	>500 µm
89.8%	84.0%	57.9%	3.48%	0%	0%	0%

LS Particle Size Analyzer

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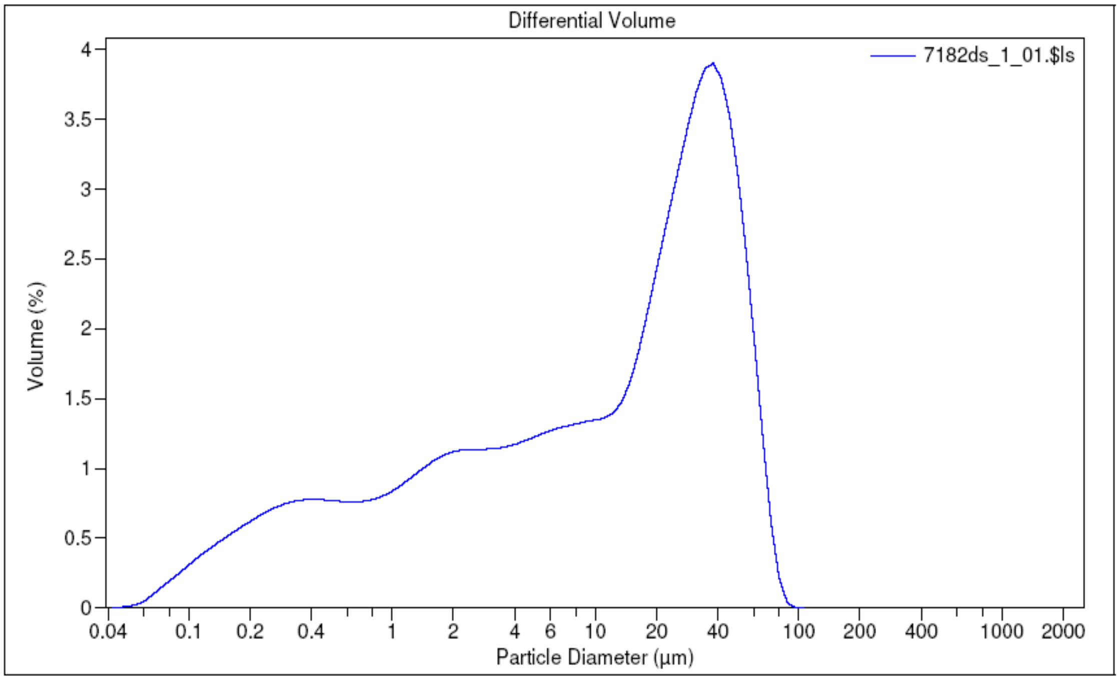

Volume Statistics (Arithmetic) 7182ds_1_01.$ls

Calculations from 0.040 µm to 2000 µm

Volume:	100%		
Mean:	19.19 µm	S.D.:	18.98 µm
Median:	13.19 µm	Variance:	360.2 µm^2
Mean/Median ratio:	1.455	C.V.:	98.9%
Mode:	37.97 µm	Skewness:	0.838 Right skewed
		Kurtosis:	-0.270 Platykurtic

<10%	<25%	<50%	<75%	<90%
0.404 µm	2.011 µm	13.19 µm	32.82 µm	47.55 µm

>10%	>25%	>50%	>75%	>90%
47.55 µm	32.82 µm	13.19 µm	2.011 µm	0.404 µm

<2 µm	<5 µm	<20 µm	<50 µm	<100 µm	<250 µm	<500 µm
24.9%	36.3%	58.3%	91.7%	100%	100%	100%

>2 µm	>5 µm	>20 µm	>50 µm	>100 µm	>250 µm	>500 µm
75.1%	63.7%	41.7%	8.31%	0.00034%	0%	0%

VITA

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