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Compilation and Integration of K Basin Sludge Particle Size Analysis Data

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Summary and Conclusions

All existing particle size analysis data on the K Basin sludge were compiled and analyzed to develop a comprehensive picture of the particle size distributions (PSDs) for the various sludge types. This analysis will be used to update the design basis feed compositions for the baseline K East (KE) and K West (KW) Basin sludge process streams expected to be generated during Spent Nuclear Fuel (SNF) Project activities.

Particle size will dictate the makeup of most of the K Basin sludge process streams. For example, Stream KE2 will consist of 250 to 6350 µm sludge particles collected from the KE canister removal and fuel washing operations. Consequently, knowledge of the PSD is necessary to project the inventory of the process streams. Furthermore, the PSD of the sludge streams will affect the performance of all physical unit operations, including sludge retrieval, pumping/transport, and separations (settling, filtration, hydroclones). Particle size, along with uranium metal content of the sludge, largely determines the chemical reactivity of the sludge. Chemical reactivity will constrain how the sludge may be handled, transported, and stored. Additionally, some sludge disposition pathways being considered as alternative approaches to baseline chemical treatment include approaches in which the sludge may be segregated on the basis of particle size.

The PSD analyses were conducted during various sludge characterization efforts (from 1993 through 1998) and involved samples of sludge collected from the KE Basin floor, the KE Weasel Pit, the KE North Loadout Pit, the KE canisters, and the KW canisters. Also analyzed were residual sludge samples generated during the transport of KE and KW fuel elements. In performing the analysis of the data for this report, it became apparent that comprehensive PSD data (i.e., distribution from 0.12 to 6350 µm) are limited, and the existing data are not always fully representative of the individual sludge samples tested. Much of the data was collected from discrete fractions of individual sludge samples. In addition, several techniques were used to collect data that span the PSD range of interest. Because these techniques are not wholly compatible, assumptions were made to prepare the PSD curves for this document.

During future characterization efforts, PSD analyses should be performed on a larger fraction of the samples. The PSD analysis should be performed on representative subsamples of the discrete samples rather than on discrete sample layers. Alternatively, analyzing representative composite sludge samples would generate very useful PSD data.

As a part of the sludge characterization activities, wet and dry sieving with wire mesh screens was used to segregate and quantify the particle size distribution of larger particles (250 to 6350 µm). Smaller particles (below 710 µm) were analyzed using optical techniques (Leeds and Northrup Microtrac X100 Particle Size Analyzer and Brinkmann Model 2010 Analyzer). Some samples were analyzed by only one method; some were analyzed by both methods at the same time; and others were analyzed by both methods at different times (in some cases, samples dried out between the analyses). In addition to sludge samples, cold ion exchange resins (Purolite NRW 100 and NRW 400) were analyzed for comparison with sludge samples. For most of the optical analyses, the Microtrac X100 was used; the Brinkmann Analzer was used for a set of samples collected in 1993.

Since sieving uses mass and the optical analysis is based on volume, assumptions were used to combine the data sets. The approach used in this report was to assume the density of the particles was uniform from 0.12 to 6350 µm. With that assumption, the volume percent of particles in a given range equaled the mass percent in that range. From that key assumption and other minor assumptions, the information from the two techniques was combined to prepare PSD curves that span the range of 0.12 to 6350 µm.

Figures S.1 through S.4 are the integrated PDS curves for the following types of sludge: KE floor and Weasel Pit sludge, KE canister sludge, KW canister sludge, and residual sludge from fuel element transport. Figure S.5 compares the PSD for the five sludge types in a single plot. The error bars in these figures indicate the highest and lowest value for each discrete size range (high-low bars). These curves were prepared by averaging the PSD results from samples of the same sludge type. The data used for the lower particle size range in these figures (below 710 μm) were generated using the optical method after the sludge samples were sonicated to break up any particle agglomerates. A complete description of how the existing PSD data were manipulated to produce these comprehensive curves is provided in the main body of this report.

Comparing the PSD curves from the various sludges (Figure S.5) shows the KE canister sludge contains the greatest distribution of particles above 1000 μm. The PSD of the KE Weasel Pit sludge is comparable to that of the KE floor sludge below ~250 μm. The KE floor sludge is finer than both the KE Weasel Pit and the KE canister sludge. The residual sludge exhibits the finest PSD of the sludge types examined. Below 200 μm, the KW canister sludge is finer than all but the residual sludge. The bulk of the material above 200 μm in the KW canister sludge is the friable Grafoil fragments generated from the disintegration of the Grafoil seal between the canister and canister lids.

Through a series of assumptions and calculations, a diverse set of particle size data was integrated to provide particle size distribution curves that span a range of K Basin sludge. These curves can be used to update the design basis sludge feed compositions, provide input to sludge disposition alternative selection, and provide data for safety-related calculations. However, as shown in this report, the integrated curves were developed from limited samples and sample fractions. As new particle size data become available, these curves should be revised.

Figure S.1. Integrated Particle Size Distribution for K East Floor and Weasel Pit Sludge After Sonication (Microtrac X100 and Sieving, 0.12 to 6350 um)

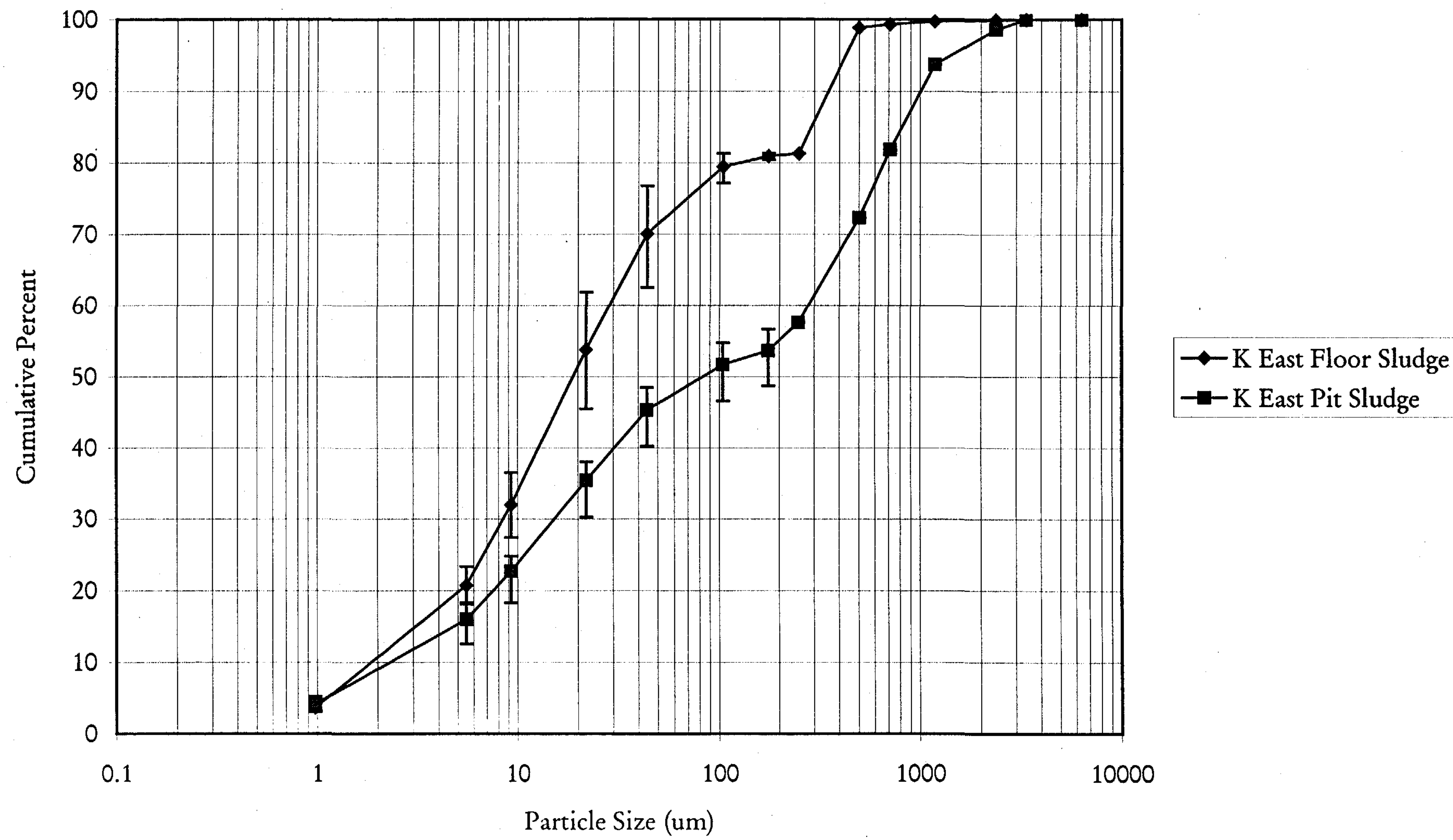


Figure S.2. Integrated Particle Size Distribution for K East Canister Sludge After Sonication (Microtrac X100 and Sieving, 0.12 to 6350 um)

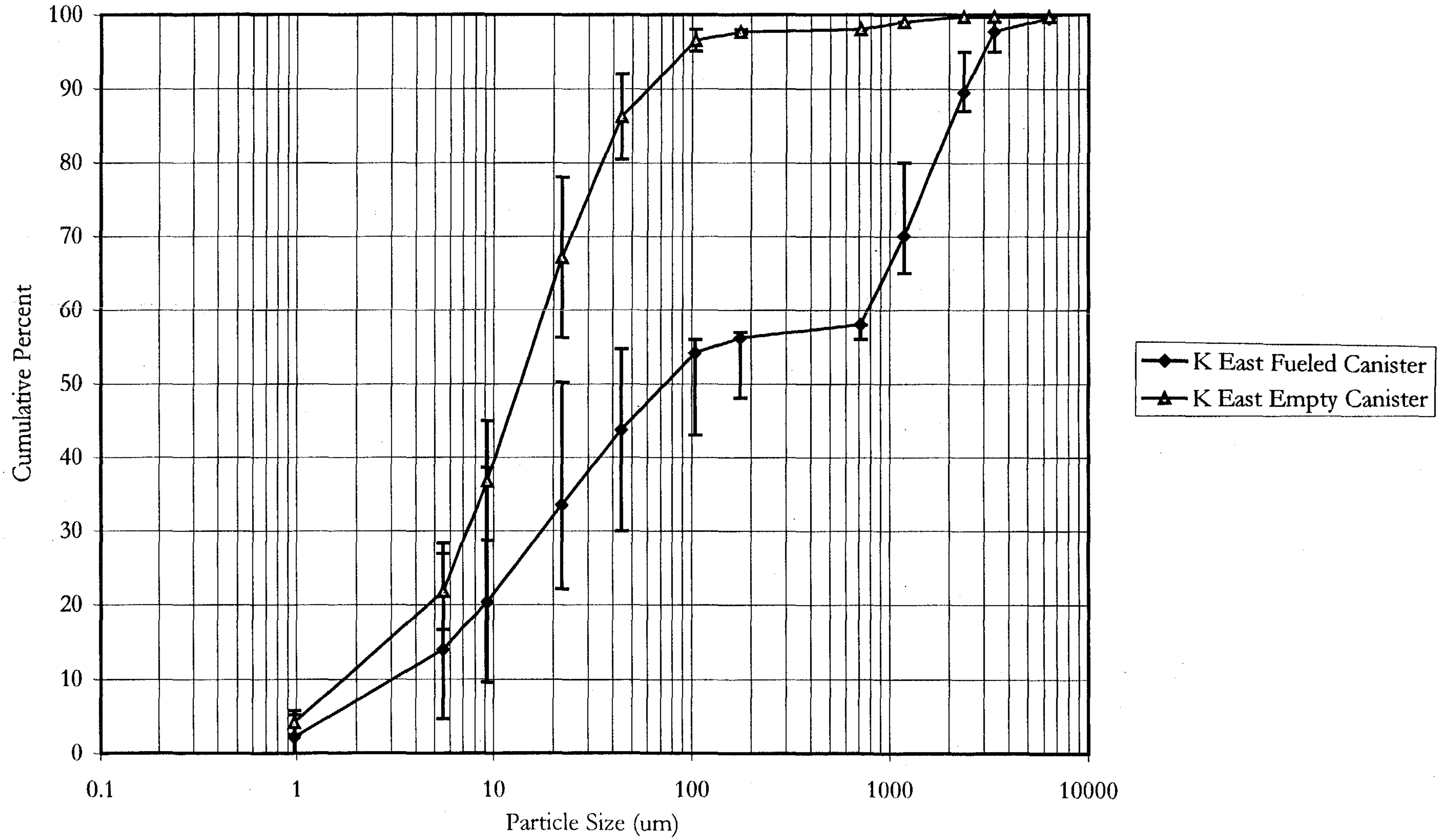


Figure S.3. Integrated Particle Size Distribution for K West Canister Sludge After Sonication (Microtrac X100 and Sieving, 0.12 to 6350 um)

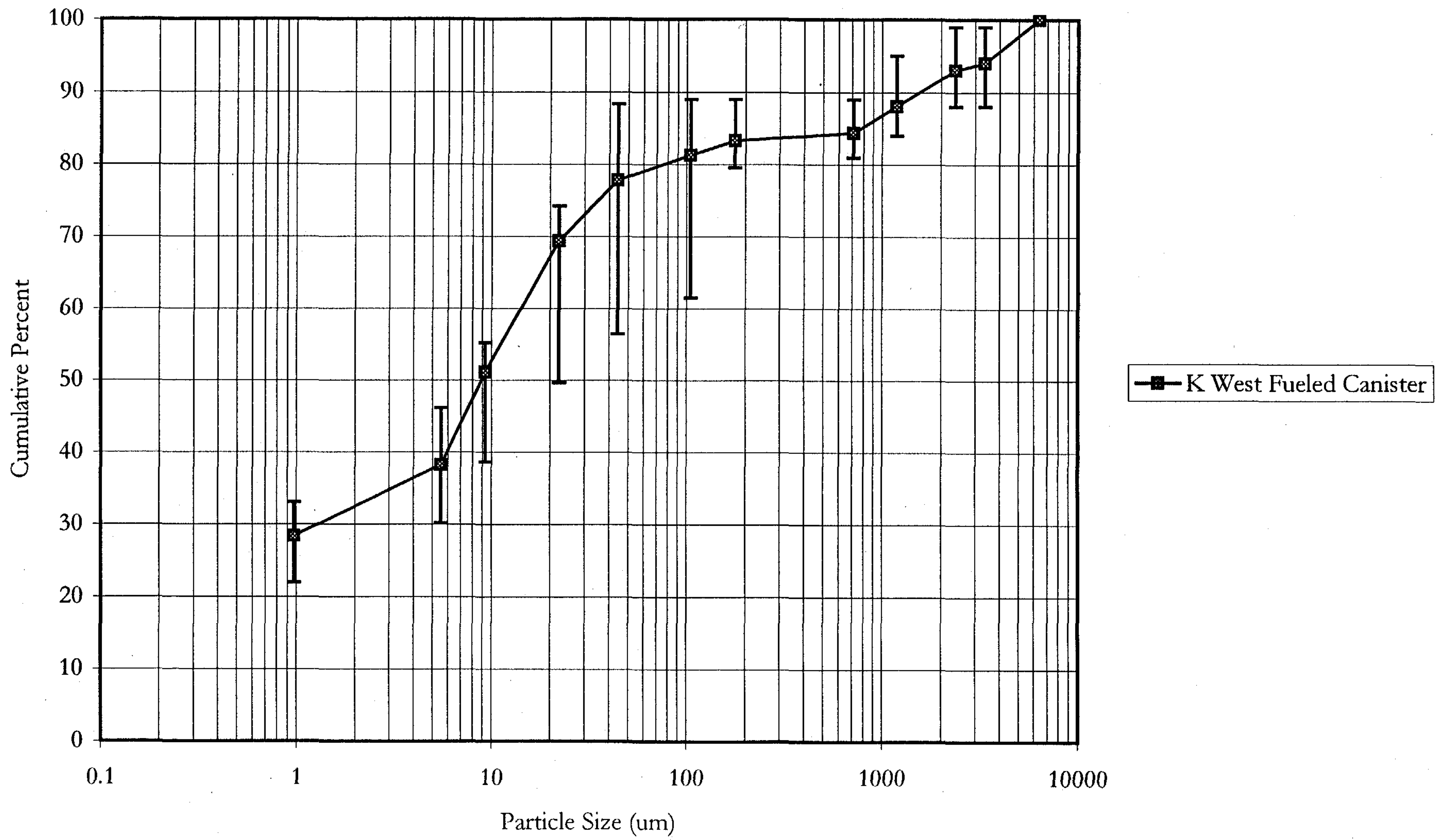


Figure S.4. Integrated Particle Size Distribution for Residual Sludge (from Fuel Element Transport) After Sonication (Microtrac X100 and Sieving, 0.12 to 6350 um)

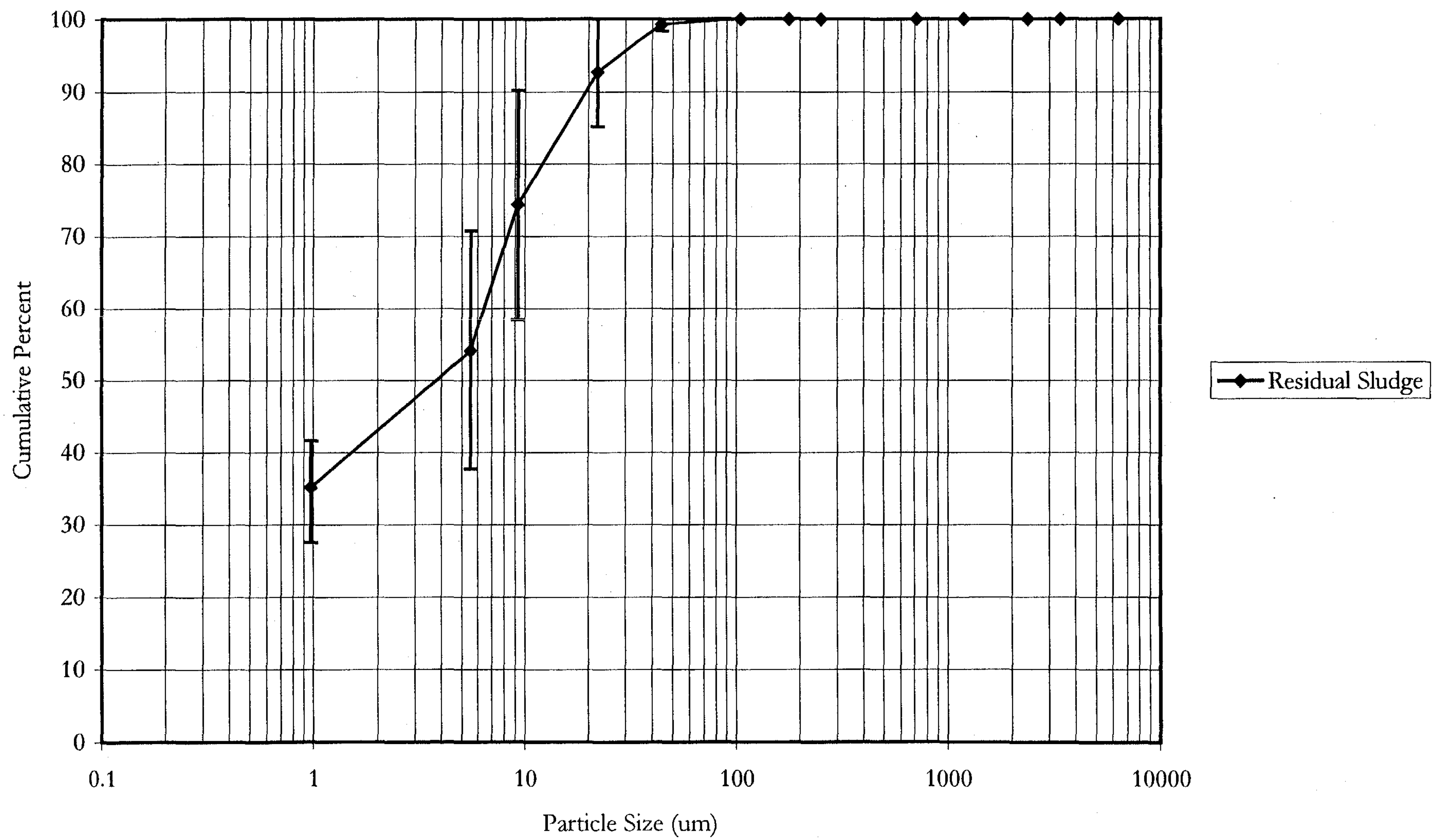
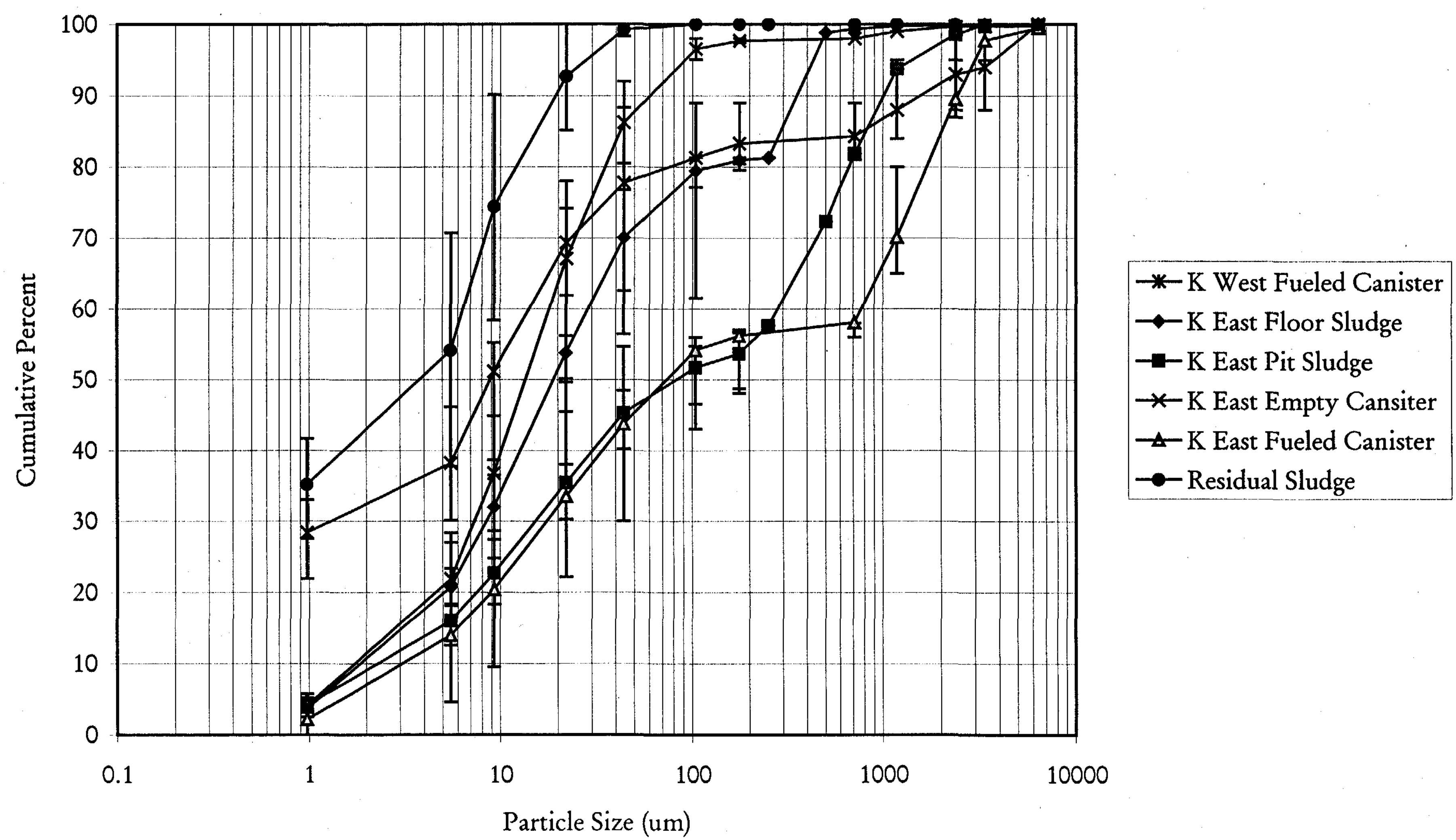


Figure S.5. Comparison of Integrated Particle Size Distributions for Sludge Types After Sonication (Microtrac X100 and Sieving, 0.12 to 6350 um)



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1.0 Introduction

Approximately 2100 metric tons of irradiated N Reactor fuel have been stored at two retention basins on the Hanford Site 100 K Area since the 1970s. During the time the waste has been stored, approximately 44 m^3 and 6.7 m^3 of sludge have accumulated in the K East (KE) Basin and K West (KW) Basin, respectively (Baker 1998). For the Spent Nuclear Fuel (SNF) Project, sludge is defined as any material in the K Basins less than or equal to 6350 μm (¼-in.). The sludge has been found within the spent fuel storage canisters, as well as on the basin floors and in associated pits. Characterization has shown the sludge is primarily uranium oxide, windblown sand, and aluminum and iron oxides and hydroxides. Minor components include organic and inorganic ion exchange materials, uranium metal particles, paint chips, and polychlorinated biphenyls. The concentrations of primary and minor components vary by location and depth.

The baseline plans are to remove the sludge, and then chemically treat it to meet Tank Waste Remediation Systems (TWRS) Waste Acceptance Criteria (Westra 1998). A critical component in developing the sludge retrieval and treatment process [led by Numatec Hanford Company (NHC)] is understanding the particle size distribution (PSD) of the sludge. This understanding is necessary to design the sludge retrieval and processing system and to address sludge transportation and storage safety issues due to the potential reactivity of sludge. Furthermore, other sludge disposition pathways are being considered as alternative approaches to chemical treatment. For some of these alternatives, including interim storage, the sludge may be segregated or processed on the basis of particle size. Particle size, along with uranium metal content of the sludge, largely determines the chemical reactivity of the sludge. Chemical reactivity will constrain how the sludge may be handled, transported, and stored.

The information provided in this report is a compilation of the particle size distribution data generated during the K Basin sludge characterization campaigns (1993 through 1998). The overall goal of this report is to consolidate and integrate the data to develop the best possible particle size distribution curves for the various sludge types. During the particle size distribution analyses, different measurement techniques were used to cover the large range of particles sizes in the K Basin sludge (i.e., submicron to 6350 μm). Specifically, sieving with wire mesh screens was used to segregate and quantify the particle size distribution of the larger particles (250 to 6350 μm), and optical techniques were used to analyze the particle size distribution of the smaller particles (below 710 μm). This report includes a description of samples analyzed, a description of the measurement techniques, and a presentation of the analyses that combines the two types of data gathered. Additional data tables and plots are included in the appendix.

The PSD analyses were previously conducted during various sludge characterization efforts and involved samples of sludge collected from the KE Basin floor, the KE Weasel Pit, the KE North Loadout Pit, the KE canisters, and the KW canisters. Also analyzed were residual sludge samples generated during the transport of KE and KW fuel elements. In performing the analysis of the data for this report, it became apparent that comprehensive PSD data (i.e., distribution from 0.12 to 6350 μm) are limited, and the existing data are not always fully representative of the individual sludge samples tested. Much of the data was collected from discrete fractions of individual sludge samples. In addition, several techniques were used to collect data that span the PSD range of interest. Because these techniques are not wholly compatible, assumptions were made to prepare the PSD curves for this document.

2.0 Sample History and Descriptions

Beginning in 1993, a systematic effort has been conducted to collect and characterize K Basin sludge samples. These overall characterization efforts have included particle size determinations on a portion of the sludge samples. This section describes the samples and subsamples from these characterization campaigns that were analyzed for particle size data.

2.1 1993 KE Basin Floor and Pit Sludge Sampling

In May 1993, four samples were collected from KE Basin and transferred to the 222-S Laboratory and analyzed for particle size distribution (Bechtold 1993). These samples consisted of clear water (i.e., no suspended fine particulates) and up to ~60 ml of sludge at the bottom of the sample vessels. The results of the initial analyses are reported in Bechtold (1993). Information provided by DESH (personal communication with Ron Baker, April 1999) indicates that one sample was collected from the west side of the Weasel Pit (S3-032-01), one from the east side of the Weasel Pit (S3-032-02), one from the KE Basin floor (S3-032-03), and one from the North Loadout Pit (S3-032-04).

2.2 1995 KE Basin Floor and Weasel Pit Sludge Sampling

Fifteen samples were collected from the KE Basin floor and five from the Weasel Pit in August and September 1995 (Makenas et al. 1996). Samples were collected by isolating a core of sludge with a metal tube. The isolated sludge cores were then collected in sample bottles by using a vacuum probe with a ¼-in. orifice (specifically designed to exclude particles greater than ¼-in. in diameter). Next, the samples were shipped to the 222-S Laboratory and were resettled using basin water as a suspension media to examine the settling rates and behavior. One sample from the KE Basin floor (KES-M-13) and one from the Weasel Pit (KES-T-20) were then split into research layer subsamples (i.e., distinct layers that formed during settling) for analyses. The four resulting research samples (KES-M-13 Top, KES-M-13 Bot, KES-T-20 Top, and KES-T-20 Bot) were shipped to the Radiochemical Processing Laboratory (RPL, 325 Building) for physical and rheological properties analyses, which included particle size distribution.

In 1997, sieving was performed on archived samples KES-H-08, KES-M-13 Top, KES-S-19, and KES-T-20 Bot (Makenas 1999). Sample KES-H-08, which consisted mainly of organic and inorganic ion exchange material, was wet. The other samples were dry and consequently were re-wet with K Basin supernatant. Additionally, these samples were sonicated at 40 W for 90 sec to break up agglomerates before wet sieving.

By June 1998, all remaining material from the 1995 sampling of the KE floor and Weasel Pit had been transferred to the RPL for archiving. With the exception of sample KES-D-14, all samples were dried prior to storage or they dried naturally during storage. In preparation for compositing sludge for process development testing, sample KES-D-14 was vacuum dried at room temperature for approximately 5 days. A significant portion of the archived sludge samples was then used to prepare two composites (Schmidt et al. 1999). One composite (KE Floor Comp) was prepared using material collected from the floor, and the other composite (KE Pit Comp) was prepared using material collected from the Weasel Pit. The composites were then dry sieved to gain information on particle size and the content of organic ion exchange resin.

Table 2.1 shows the KE Basin floor and Weasel Pit sludge samples (and subsamples) for which particle size analyses have been performed. In addition to the sludge samples, Table 2.1 also shows Purolite NRW 100 and 400, which are nonradioactive organic ion exchange resins. These samples were analyzed to provide data to compare against sample KES-H-08.

Table 2.1. KE Floor and Weasel Pit Sludge Samples (1995) Analyzed

Sample	Fraction of Original Sample in Layer
KE Floor Sludge	
KES-M-13 Top	50% (estimate)
KES-M-13 Bottom	50% (estimate)
KE Floor Comp	100%
KE Floor Ion Exchange Material	
KES-H-08	100%
Purolite NRW 100	NA
Purolite NRW 400	NA
KE Weasel Pit Sludge	
KES-S-19	100%
KES-T-20 Top	50% (estimate)
KES-T-20 Bottom	50% (estimate)
KE Pit Comp	100%

2.3 1996 KE Basin Canister Sludge Sampling

In April 1996, nine sludge samples were collected from different storage canisters located throughout the KE Basin (Makenas et al. 1997). Each canister sludge sample was collected using the same method as was used for the KE floor and Weasel Pit sludge samples in 1995. The samples were then transferred to the RPL.

The canister sludge samples were transferred to 2-L graduated cylinders, and settling studies were conducted. Additional liquid collected with the samples was transferred to 10-L glass carboys. The carboys containing the sample liquid were reexamined several weeks after sample transfer. A layer of fine solids had settled to the bottom of the carboys containing the liquid from samples 96-06 and 96-15. Particle size analyses were performed on the settled solids collected from the bottom of the 96-06 carboy (96-06 Carboy Solids).

Following settling studies, three samples, designated as research samples, were collected from the graduated cylinders in distinct layers that had formed during settling (see Table 2.2). The research samples were: sample 96-04 from a stainless steel canister; sample 96-06 from an aluminum canister; and sample 96-11 from a canister containing no fuel. Each recovered layer was treated as a unique sample. If enough sample (>100 ml) was present in the layer, the layer was collected by vacuum transfer. If the layer was less than 100 ml, a pipet was used to collect it.

Sample 96-04 was split at the interface between the upper smooth and lower granular layers. The upper layer (96-04 U) represented approximately 70% of the sample by volume, while the lower layer (96-04 L) represented approximately 30% of the volume. Sample 96-06 was split into three layers: a fluffy upper layer (96-06 U), a middle layer (96-06 M), and a lower layer (96-06 L). The upper layer represented approximately 5% of the original sample volume; the middle represented 53% of the volume; and the lower represented 42% of the volume. Sample 96-11 was split at the interface between the thin lighter-colored fluffy upper layer (96-11 U), and the darker-colored bulk of the sample (96-11 L). The upper layer represented approximately 7% of the sample volume, while the lower layer represented approximately 93% of the sample volume. To ensure the layers did not mix, the interfaces between them were collected as separate samples. These sample were then identified by the interface location (e.g., the sample collected between 96-04 U and 96-04 L was identified as 96-04 U/L). Particle size analyses were conducted on samples 96-04 U/L, 96-04 L, 96-06 M, 96-06 L, 96-06 Carboy Solids, and 96-11 L.

Table 2.2. KE Canister Sludge Layer Samples (1996)

Sample	Fraction of Original Sample in Layer	PSD Analyses Performed?
96-04 U	70%	No
96-04 U/L	NA	Yes
96-04 L	30%	Yes
96-06 U	5%	No
96-06 U/M	NA	No
96-06 M	53%	Yes
96-06 M/L	NA	No
96-06 L	42%	Yes
96-06 Carboy Solids	NA	Yes
96-11 U	7%	No
96-11 U/L	NA	No
96-11 L	93%	Yes

2.4 1996 KW Basin Canister Sludge Sampling

In December 1996, nine sludge samples were collected from different storage canisters located throughout the KW Basin (Makenas et al. 1998). Each sludge sample was collected in a sample container using specially designed sampling equipment similar to that used to collect KE floor and Weasel Pit (1995) and KE canister sludge samples (1996). The sample containers were removed from the basin and transferred to the RPL. The samples were then transferred to 2-L graduated cylinders, and settling tests were conducted. The volume of settled sludge in each sample was small (~10-30 ml) compared to the anticipated volume of ~500 ml.

Following settling studies, three of the samples (96-21, 96-23, and 96-24) were designated as research samples. Given the low sample volumes, it was not possible to recover the research samples in layers as was done for the KE canister samples; therefore, the entire samples were wet sieved.

2.5 1998 K Basin Residual Sludge Generated from Fuel Element Transport

Five fuel elements were sampled in 1998 to recover coatings on the element surfaces and sludge trapped under the cladding. While this work was primarily focused on the internal sludge and coatings samples, sludge (i.e., residual sludge) was observed in the shipping containers used to transport the fuel elements from the K Basins to the Postirradiation Testing Laboratory (PTL, 327 Building). This residual sludge was dislodged from the fuel elements during shipment, probably from the surface of the element, from the center coolant passage in the annular fuel slugs and/or from the damaged areas. During fuel element retrieval, the residual sludge would likely be removed by the fuel washing process and would become a part of the K Basin sludge inventory. Two of the residual sludge samples (R1 and R5) were analyzed at the RPL for selected physical properties, including particle size distribution. Sample R1 was collected from an element stored in an aluminum canister in KE Basin open to the basin water, while sample R5 was collected from an element stored in a sealed stainless steel canister in KW Basin. Therefore, in addition to being stored in canisters composed of different materials, one fuel element was exposed to intrusion of windblown sand and other basin debris, while the other was not.

Samples R1 and R5 dried prior to delivery to the RPL. The samples were resuspended in K Basin water and sonicated before the particle size analyses were performed. After the residual sludge samples were wet sieved, optical analyses were performed on certain sieved fractions. Sample numbers were assigned to these samples based on the applicable sieve. For example, the material from sample R1 retained in the Tyler 60 sieve was designated as R1-60; material that passed through all sieves and was captured by the bottom pan (also referred to as the receiver pan) was designated R1 REC.

3.0 Particle Size Measurement Methods

Measurement methods used during sludge sample analyses included wet and dry sieving and optical techniques (Leeds and Northrup Microtrac X100 Particle Size Analyzer and Brinkmann Model 2010 Analyzer). Table 3.1 lists the samples analyzed and the particle size analysis methods used. As illustrated in the table, some samples were analyzed by only one method; some were analyzed by both methods at the same time; and others were analyzed by both methods at different times (note: during storage between analyses, some samples dried out). Table 3.1 also lists ion exchange resins (Purolite NRW 100 and NRW 400) tested for comparison with sample KES-H-08, which contained a high concentration of organic ion exchange resin.

3.1 Sieving Methods

The sieving technique was used to separate and quantify the larger particles (250 to 6350 μm) of selected samples. Stainless steel sieves manufactured by W.S. Tyler in conformance to ASTM E11, ANSI, and ISO 565 3310-1 standards were used, with openings between 3350 μm and 250 μm. Table 3.2 lists the Tyler sieves used and their aperture sizes. The sieve sizes used varied, depending on the anticipated data needs for the individual samples at the time of the analysis. As a result, the same size information is not available for all samples.

3.1.1 Dry Sieving

Dry sieving was only performed on KE floor and Weasel Pit composite samples. During dry sieving, the Tyler 6 was place on top of the Tyler 8, with a bottom pan underneath. The entire mass of the dry KE Floor Comp or KE Pit Comp was measured and placed on top of the Tyler 6 sieve. The stacks were shaken gently until all the material appeared (by visual inspection) to be retained on the appropriate sieve or to have passed through to the bottom pan. The masses on each sieve and in the bottom pan were measured. Material on the sieves was transferred back to the original composite jar, and the material in the bottom pan was transferred to a new stack of finer sieves. This process was repeated until all sieves were used. The KE Pit Comp contained several large agglomerated chunks that would not pass through the Tyler 6 sieve. These chunks were easily broken up by applying light pressure. Laboratory records do not indicate agglomerated chunks were seen on any of the other sieves.

For dry sieving, wt% dry solids was calculated by dividing the mass of the material retained on each sieve by the total mass of material in each of the sieves and bottom pan. The original sample mass was not used in the dry sieving calculation, because some sample was lost as a fine aerosol during the sieving process. Approximately 5.2 wt% of the KE Pit Comp was lost during sieving; KE Floor Comp sample loss could not be calculated, but was estimated to be similar to the KE Pit Comp loss. The loss is likely associated with the smallest fraction. However, since definitive data are not available, the loss was not apportioned to any specific fraction within the calculations.

3.1.2 Wet Sieving

During wet sieving, the sieve set was stacked with the Tyler 6 on the top, followed by the other sieves in order of decreasing size (the sieve sizes varied among the various tests). A known amount of settled solids was slurried in basin water. In some cases, the slurry was either stirred or sonicated to break up agglomerates (see Table 3.1). The slurry was then poured through the stack of sieves. The same K Basin water used to prepare the slurry was used to wash the solids through each sieve. The accumulated slurry

Table 3.1. K Basin Samples Analyzed for Particle Size Distribution

Sample	Analyzed by Sieving	Analyzed by Optical Technique	Dry Prior to Analyses	Shearing prior to Analyses	Agglomerates Observed on Sieves
KE Samples from 1993 Sampling					
S3-032-01		X	No	No	NA
S3-032-02		X	No	No	NA
S3-032-03		X	No	No	NA
S3-032-04		X	No	No	NA
KE Floor Sludge					
KES-M-13 Top		X	No	No	NA
KES-M-13 Top	X		Yes	40W/90sec	Probable
KES-M-13 Bottom		X	No	No	NA
KE Floor Comp	X		Yes	No	No
KE Floor Ion Exchange Material					
KES-H-08	X		No	40W/90sec	No
Purolite NRW 100		X	Unknown	No	NA
Purolite NRW 400		X	Unknown	No	NA
KE Weasel Pit Sludge					
KES-S-19	X		Yes	40W/90sec	No
KES-T-20 Top		X	No	No	NA
KES-T-20 Bottom		X	No	No	NA
KES-T-20 Bottom	X		Yes	40W/90sec	No
KE Pit Comp	X		Yes	No	No
KE Canister Sludge					
96-04 U/L		X	Yes	Stirred	Yes, so data not used
96-04 L	X	X	No	Stirred	No
96-06 Carboy Solids		X	No	Stirred	NA
96-06 M	X	X	No	Stirred	Possible
96-06 L	X	X	No	Stirred	Possible
96-11 L	X	X	Yes	Stirred	No
KW Canister Sludge					
96-21	X	X	No	Stirred	No
96-23	X	X	No	Stirred	No
96-24	X	X	No	Stirred	No
Residual Sludge from Fuel Element Transport					
R1	X		Yes	40W/90sec	Yes
R1 REC	X	X	Yes	40W/90sec	Yes
R1 60	X	X	Yes	40W/90sec	Yes
R5	X		Yes	40W/90sec	No
R5 REC	X	X	Yes	40W/90sec	No

in the receiver pan was poured into a collection beaker. After the samples were washed through each sieve, the sieves were air-dried (typically 2 to 6 hr) to remove any free water retained and then weighed. While these solids were air-dried to remove free water, they were not dried at elevated temperatures or over an extended period to remove interstitial water and waters of hydration. Therefore, the weights measured after air-drying are referred to in this report as wet weights. A detailed discussion of the wet sieving procedure is given in the test instructions.

Table 3.2. Sieve Sizes Used for Wet and Dry Sieving

Tyler Size	US Mesh Equivalent	Sieve Aperture (mm)	Sieve Aperture (in.)
6	6	3.35	0.132
8	8	2.36	0.0937
14	16	1.18	0.0469
24	25	0.710	0.0278
32	35	0.500	0.0197
60	60	0.250	0.0098

For wet sieving, the wt% wet solids was calculated by dividing the mass of the material retained on each sieve by the mass of the original sample. The mass and the wt% of the sludge in the receiver pan were determined from a mass balance between the initial settled solids sample mass and the sum of the sludge masses retained on each sieve.

3.2 Optical Analyses

For most of the optical analyses, the Microtrac X100 was used; the Brinkmann Analyzer was used for the 1993 KE Basin sludge samples (S3-032-01 through -04).

3.2.1 Brinkmann Model 2010 Analyzer

Particle size analyses were conducted at the 222-S Laboratory on KE sludge samples collected in 1993 (samples S3-032-01, -02, -03, and -04) using a Brinkmann Model 2010 Analyzer. For these analyses, the samples were dispersed in their own associated liquid. The Model 2010 uses a laser eclipsing technique to measure the size of particles between 0.5 μm and 150 μm. The analyzer has a video camera for observing the circulating particles and checking for agglomeration or aspect ratio. The data are taken as maximum crossing diameters (i.e., signals with gradual rise and fall indicative of glancing eclipses are rejected). The volume distributions are derived by assuming spherical particles. The analyzer depends on the particles being adequately dispersed and stirred so that all sizes get a fair representation in the scanning beam. Large, heavy particles that are not lofted high enough in the cell are not counted. For very dense particles, this could occur at sizes below 150 μm.

3.2.2 Microtrac X100 Particle Size Analyzer

Particles between 0.12 μm and 710 μm were analyzed at the RPL using a Microtrac X100 instrument, which analyzes light scattered by the particles. For these analyses, a sample was added to a recirculating fluid so that a stream of well-dispersed particles passed through the sample cell for analysis. The Microtrac X100 instrument measures the diffraction pattern of the light scattered by the particles with an array of optical detectors to determine the size distribution. The flow rate of the recirculating fluid can be varied, and an ultrasonic probe can be used to induce shear forces on the sample to break up agglomerates. Both of these techniques were applied. During analysis of the KE canister sludge, a standard protocol was adopted, based on the effects of varying flow rate and applying shear forces, in which the particle size distributions of the samples were examined at 40 ml/sec flow (90 sec) before and after sonication at 40 W. A new sample was then introduced and examined at 70 ml/sec flow rate before and after sonication. Since this protocol was adopted after the KE floor and Weasel Pit sludge characterization campaigns (Makenas et al. 1996), it was not used for all sludge types. However, all samples were analyzed before and after application of some level of sonication.

Table 3.3 lists the flow conditions and sonication power applied to each analysis. "As-Received" in Table 3.3. indicates a new subsample was placed in the instrument. Analyses were conducted for each sample sequentially. For example, sample R5 REC, a new sample, was placed in the instrument and analyzed at a flow rate of 40 ml/sec (analysis 1). The sample was then sonicated at 40 W for 90 sec and analyzed again (analysis 2). A new sample was then placed in the instrument and analyzed (analysis 3). The resulting data, both tabular and plotted, are presented in the appendix.

As will be discussed in Section 4.0, calculations were performed to determine an average particle size distribution for each sample. The average particle size distributions for the samples were then used to calculate an average particle size distribution for the different sludge types. Table 3.3 also indicates which analyses were used in the calculations for average particle size distributions.

Table 3.3. Samples Analyzed Using the Microtrac X100, Including Conditions and Analyses Used in Calculations

Sample	Analysis Number	Flow Rate (ml/sec)	Sonication (power/duration)	Analyses Averaged in Before Sonication Calculations	Analyses Averaged in After Sonication Calculations	Comments
KE Floor Sludge						
KES-M-13 Top	1	60	As-Received[a]	1,4	2,5	
	2	60	25 W/120 s			
	3	60	40 W/300 s			
	4	60	As-Received			
	5	60	25 W/120 s			
	6	60	40 W/300 s			
KES-M-13 Bottom	1	60	As-Received	1,4	2,5	
	2	60	25 W/120 s			
	3	60	40 W/300 s			
	4	60	As-Received			
	5	60	25 W/120 s			
	6	60	40 W/300 s			
KE Weasel Pit Sludge						
KES-T-20 Top	1	60	As-Received	1,4	2,5	
	2	60	25 W/120 s			
	3	60	40 W/300 s			
	4	60	As-Received			
	5	60	25 W/120 s			
	6	60	40 W/300 s			
KES-T-20 Bottom	1	60	As-Received	1,4	2,5	
	2	60	25 W/120 s			
	3	60	40 W/300 s			
	4	60	As-Received			
	5	60	25 W/120 s			
	6	60	40 W/300 s			

Table 3.3. (Continued) Samples Analyzed Using the Microtrac X100, Including Conditions and Analyses Used in Calculations

Sample	Analysis Number	Flow Rate (ml/sec)	Sonication (power/duration)	Analyses Averaged in Before Sonication Calculations	Analyses Averaged in After Sonication Calculations	Comments
KE Canister Sludge						
96-04 U/L	1	60	As-Received	1,4,7	3,6,8	This sample was not sieved, so 96-04 L sieving results were used for calculations.
	2	60	25 W/125 s			
	3	60	40 W/300 s			
	4	60	As-Received			
	5	60				
	6	60	25 W/90 s			
	7	40	As-Received			
	8	40	40 W/90 s			
	9	70				
	10	70	40 W/90 s			
96-04 L	1	60	As-Received	1,4	2,5	Analysis 7 shows large particles, probably bubbles generated by sonication entrained by the system at this high flow rate.
	2	60	25 W/120 s			
	3	60	40 W/300 s			
	4	40	As-Received			
	5	40	40 W/90 sec			
	6	70				
	7	70	40 W/90 sec			
96-06 Carboy	1	60	As-Received			No significant size reduction with sonication
	2	60	25 W/120 s			
	3	60	40 W/300 s			
96-06 M	1	60	As-Received	1,2	3	
	2	40	As-Received			
	3	40	40 W/90 s			
	4	70				
	5	70	40 W/90 s			
96-06 L	1	60	As-Received			
	2	60	25 W/120 s			
	3	60	40 W/300 s			
96-11 L	1	60	As-Received	1,4	2,5	
	2	60	25 W/120 s			
	3	60	40 W/300 s			
	4	40	As-Received			
	5	40	40 W/90 s			
	6	70				
	7	70	40 W/90 s			

Table 3.3. (Continued) Samples Analyzed Using the Microtrac X100, Including Conditions and Analyses Used in Calculations

Sample	Analysis Number	Flow Rate (ml/sec)	Sonication (power/duration)	Analyses Averaged in Before Sonication Calculations	Analyses Averaged in After Sonication Calculations	Comments
KW Canister Sludge						
96-21 REC	1	70	As-Received	1,5,8	3,6,9	Particles above 100 μm appear to be real, but not seen in as high ratios in analysis 5. This could be Grafoil that should have been removed during sieving. Analyses 2 and 3 recalculated to exclude particles above 200 μm, and analyses 6 and 9 recalculated to exclude particles above 300 μm. Following recalculation, all as-received samples have similar PSD.
	2	70	40 W/90 s			
	3	40				
	4	40	40 W/90 s			
	5	70	As-Received			
	6	70	40 W/90 s			
	7	40				
	8	40	As-Received			
	9	40	40 W/90 s			
96-23 REC	1	70	As-Received	1	2	Dissolution or particle size reduction below detection limits was observed following sonication in analysis 4.
	2	70	40 W/90 s			
	3	40				
	4	40	40 W/90 s			
96-24 REC	1		As-Received	1	2	
	2		40 W/90 s			
	3					
	4		40 W/90 s			
Residual Sludge from Fuel Element Transport						
R1-60	1	40	As-Received	NA[(b)]	NA	High flow rates were required to keep larger particles suspended. Dissolution or particle size reduction below detection limits was observed following sonication in analyses 6 and 8.
	2	40	40 W/90 s			
	3	70				
	4	70	40 W/90 s			
	5	40	As-Received			
	6	40	40 W/90 s			
	7	70	As-Received			
	8	70	40 W/90 s			

Table 3.3. (Continued) Samples Analyzed Using the Microtrac X100, Including Conditions and Analyses Used in Calculations

Sample	Analysis Number	Flow Rate (ml/sec)	Sonication (power/duration)	Analyses Averaged in Before Sonication Calculations	Analyses Averaged in After Sonication Calculations	Comments
R1 REC	1	40	As-Received	1,5,7	2,6,8	
	2	40	40 W/90 s			
	3	70				
	4	70	40 W/90 s			
	5	40	As-Received			
	6	40	40 W/90 s			
	7	70	As-Received			
	8	70	40 W/90 s			
R5 REC	1	40	As-Received	1,3,5,7	2,4,6,8	Dissolution or particle size reduction below detection limits was observed following sonication in analyses 2, 4, 6, and 8.
	2	40	40 W/90 s			
	3	70	As-Received			
	4	70	40 W/90 s			
	5	40	As-Received			
	6	40	40 W/90 s			
	7	70	As-Received			
	8	70	40 W/90 s			

(a) As-Received under Sonication indicates a new subsample was placed in the instrument for this and subsequent analyses.
(b) NA, sample R1-60 not included in averages.

4.0 Results

In the analyses of particle size distribution for K Basin sludge samples, the results from sieving provided a distribution based on the mass of particles in different size ranges, while the results from optical techniques provided a distribution based on the volume of particles in different size ranges. Since one technique uses mass and the other volume, caution must be used when trying to combine the data sets. The approach employed in this report to integrate the two data types was to assume the density of the particles was uniform from 0.12 to 6350 μm. With that assumption, the volume percent of particles in a given range equals the mass percent in that range. The results from individual data sets are given in Sections 4.1 through 4.5, then the integrated PSD results are presented in Section 4.6.

4.1 Results from Sieving

The wet and dry sieving results are presented in Table 4.1. Sieving results for KE Basin samples show comparable results for sludge samples from similar areas of the basin. The KE floor samples are primarily fine particles (below 710 μm); the Weasel Pit samples have a higher wt% of larger particles; and the canister samples have the largest content of particles above 710 μm. These results are presented in Figure 4.1. The KE canister sludges (96-04 L, 96-06 M, and 96-06 L) average 57 wt% below 710 μm, 10 to 20 wt% between 710 μm and 1180 μm, and 6 to 12 wt% between 2360 μm and 6350 μm. Sample 96-11 L is from a KE canister containing no fuel, and compares well with the KE Floor Comp sample. Samples 96-11 L and KE Comp are composed primarily (99%) of particles below 710 μm. Sample KES-H-08 does not compare well with the other floor samples due to its high ion exchange material content. The integrity of sample KES-M-13 Top was compromised due to a high number of agglomerates that formed during the drying of the sample that occurred during archiving. The Weasel Pit samples (KES-S-19, KES-T-20 Bot, and KE Pit Comp) all contain 82 to 87 wt% particles below 710 μm, 8 to 12 wt% particles between 710 μm and 1180 μm, and 4 to 5 wt% between 1180 μm and 2360 μm.

Results of sieving tests also showed the individual KW canister sludge samples (96-21, 96-23, and 96-24) have very similar particle size distributions. The samples all contained 81 to 89 wt% particles below 710 μm. Visual inspection during the sieving indicated that most of the material above 710 μm was Grafoil (a graphite gasketing material) used to seal the lids on the fuel storage canisters.

Sieving results suggest that residual sludge samples (R1 and R5) were the finest material in the basins. The results for sample R5 indicate that all the solid particles or agglomerates were less than 500 μm in diameter, whereas 49 wt% of solids in sample R1 were above 250 μm. The large chunks in R1 could be crushed by applying moderate pressure with a pair of tongs. These observations suggest that the large particles were agglomerates most likely generated by partial drying of R1 before the samples were received at the RPL. Thus, it is speculated that the R1 and R5 samples may have had similar wet sieving distributions if they had not dried prior to this testing. Based on these results, all of the particles in the residual sludge are anticipated to be below 500 μm.

As described above, K Basin water was used as the rinsing fluid during the wet sieving. The as-received K Basin water used for sieving the R1 and R5 samples was clear and colorless. Following the rewetting and sieving work, the rinsing fluid was clear and green. The green color suggests that either some of the solids in the R1 and R5 samples have dissolved or some suspended particles are present that are too fine to settle by gravity.

Table 4.1. Wet and Dry Sieving Results on a Wt% Solids Basis

Sample	Sieving Method	Tyler 6 (3350 µm)	Tyler 8 (2360 µm)	Tyler 14 (1180 µm)	Tyler 24 (710 µm)	Tyler 32 (500 µm)	Tyler 42 (335 µm)	Tyler 60 (250 µm)	Receiver Pan
KE Floor Sludge									
KES-M-13 Top[(a)]	Wet	NA[(b)]	NA	NA	41	NA	24	NA	35
KE Floor Comp	Dry	0[(c)]	0.1	0.2	0.4	0.5	NA	18	81
KES-H-08[(d)]	Wet	0	0	3	66	NA	23	NA	8
KE Weasel Pit									
KES-S-19	Wet	1	1	4	8	NA	22	NA	65
KES-T-20 Bottom	Wet	1	1	5	12	NA	24	NA	57
KE Pit Comp	Dry	0[(c)]	1.4	5	12	10	NA	15	58
KE Canister Sludge									
96-04 L	Wet	0	12	22	9	NA	NA	NA	56
96-06 M	Wet	5	6	20	11	NA	NA	NA	58
96-06 L	Wet	3	3	15	22	NA	NA	NA	58
96-11 L	Wet	0	0.1	0.7	1	NA	NA	NA	98
KW Canister Sludge									
96-21	Wet	1	0	4	6	NA	NA	NA	89
96-23	Wet	6	3	8	3	NA	NA	NA	81
96-24	Wet	12	0	3	2	NA	NA	NA	83
Residual Sludge from Fuel Element Transport									
R1	Wet	30	5	4	3	NA	NA	6	51
R5	Wet	0	0	0	0	0	NA	NA	100

(a) Sample KES-M-13 Top contained a large number of agglomerates formed by drying during the archiving process; therefore sieving results are suspect.
(b) NA-Not Applicable, not all sieve sizes used for all samples.
(c) A Tyler 5 screen (3960 µm) was used in place of the Tyler 6 screen.
(d) Sample KES-H-08 is primarily ion exchange material and therefore is not representative of the other floor samples.

4.2 Brinkmann Analyzer Results

The shape of the number distribution histograms suggest a significant number of particles existed below 0.5 µm that could not be quantified. One sample (S3-032-02) indicated significant volumes greater than 150 µm existed. Results are plotted in Figure 4.2.

4.3 Microtrac X100 Results on Individual Samples Before Sonication (0.12 to 710 µm)

Sample analyses conducted by laser scattering (Microtrac X100) were averaged to generate an average particle size distribution for each sample before sonication. These plots are presented in Figures 4.3 through 4.8. The error bars in these figures indicate the highest and lowest value for each range (high-low bars). A line is used to connect the points, but this line only provides direction to the next point and should not be used to interpolate values between points.

For samples that were split into research layers (96-04, 96-06, 96-11, KES-M-13, and KES-T-20), the average curves for each research layer were combined in proportion to the estimated volume percent in each layer to calculate the original sample average. The data in Tables 2.1 and 2.2 were used in this calculation. Because no information was available on the relative volumes of the top and bottom layers of

KES-M-13 and KES-T-20, it was assumed that these samples were evenly divided. This approach seems reasonable since there was very little difference between the top and bottom layers.

No particle size distribution data were available for the upper layers of the KE canister research samples; therefore, for sample 96-04, the 96-04 U/L layer was assumed to be representative of the upper layer of 96-04. For sample 96-06, the 96-06 Carboy sample was assumed to be representative of the upper layer of 96-06.

4.4 Microtrac X100 Results on Individual Sludge Samples After Sonication (0.12 to 710 μm)

The calculations described in Section 4.3 were repeated using the laser scattering particle size distribution data following sonication. These data are presented in Figures 4.9 through 4.14. Comparing figures for the same samples before and after sonication (for example, Figures 4.4 and 4.10, respectively) shows, in general, sonication shifts the curves to the left, (i.e., to smaller particle sizes). In addition, sonication reduces the error bars. Therefore, sonication both reduces the particle size and produces a more uniform particle size distribution between duplicate sludge types.

4.5 Microtrac X100 Results for Sludge Types Before and After Sonication (0.12 to 710 μm)

Average particle sludge distributions were calculated for the different sludge types (KE canister sludge, KW canister sludge, residual sludge, KE floor sludge, and KE Weasel Pit sludge) using the average of the results from the individual samples (Figures 4.3 through 4.14). Because KES-M-13 and KES-T-20 were the only KE floor and Weasel Pit samples analyzed, they were assumed to be representative of the KE floor and KE Weasel Pit averages, respectively. The particle size distributions for samples 96-04 and 96-06 were averaged to generate an average particle size distribution for KE canister sludge from canisters containing fuel elements. Sample 96-11 was collected from a KE canister that did not contain fuel; thus, its particle size distribution was very different from 96-04 and 96-06 and was not averaged with 96-04 and 96-06. Data from samples 96-21, 96-23, and 96-24 were combined to generate the average for KW canister sludge, and sample R1 and R5 data were combined to calculate the average for residual sludge.

The resulting particle size distributions for the different sludge types before and after sonication are plotted in Figures 4.15 and 4.16 respectively. These figures are complicated by overlap in the error bars. Based on the overlap in the high-low bars in Figure 4.15, there is no significant difference in particle size distributions between any of the sludge types below 100 μm prior to sonication. It appears that above 100 μm, the KE canister and KW canister sludges have a higher percentage of large particles.

Figure 4.16 shows that after sonication the residual sludge is the finest material, followed by the KW canister sludge. The KE canister sludge, KE Weasel Pit sludge, and KE floor sludge are all very similar. It appears the KE floor sludge and sludge from KE canister 96-11 (without fuel) may be slightly finer with a very tight distribution of particles centered around 10 μm. It is interesting to note that the particle size distributions of sample 96-11 and the KE floor sludge are similar. This observation is also valid for the sieve data. The similarity is likely because the bottom of the KE canister is open to the KE floor.

4.6 Combined PSD Results from Sieving and Microtrac X100 Data (0.12 to 6350 μm)

The average particle size distribution calculations were repeated factoring the sieving data with the laser scattering data. This calculation was performed by multiplying the volume percent data reported from the Microtrac X100 by the mass fraction of material passing through the finest sieve. For example, sample 96-06 M contained 58 wt% (0.58 mass fraction) particles below 710 μm, according to the sieve data. Based on the Microtrac X100 data (analysis 1), the sample also contained 4.88 vol% particles below 0.972 μm. Therefore, combining the two data sets, the percentage of particles below 0.972 μm is 2.83% (4.88 x 0.58 = 2.83). As stated previously, this calculation is only valid by assuming the density of the particles above 710 μm is the same as the density of the particles below 710 μm. Since wt% and vol% are the same for a sample with uniform density, the resulting plots are then both wt% and vol%.

In some cases, sieving data were not available for all the samples analyzed using the Microtrac X100, and in some cases sieving data were not valid because the samples had dried. Sample 96-04 U/L was not sieved, so the 96-04 L sieving results were used for calculations involving this sample. Sample KES-M-13 was sieved, but very little material was observed on the sieves. Large agglomerates that broke up easily after the material was dried were also observed. As a result, the KE Floor Comp sieving results were used for calculations involving KES-M-13. Use of the KE Floor Comp sieving results should also reduce errors in sample inhomogeneity, since the KE Floor Comp represents a much larger material mass (~120 g for KE Floor Comp versus 1.5 g of KES-M-13 sieved). While sample KES-T-20 was sieved, the results were very similar to those for the KE Pit Comp. Since the KE Pit Comp represents a much greater fraction of Weasel Pit material than KES-T-20 alone (~280 g for KE Pit Comp versus 27 g of KES-T-20 sieved), the KE Pit Comp sieving data were used for calculations involving KES-T-20.

The sample R1 sieving results suggest that agglomerates were formed during the drying of the sample prior to delivery to the RPL and that the agglomerates formed in R1 were not broken up during the sieving process. As a result, the R1 sieving results were not used in these calculations. The R5 sieving results were used for calculations of the R1 particle size distribution.

The results of the calculations combining the sieving and laser scattering (following sonication) data are shown in Figures 4.17 through 4.22. Average particle size distributions were then calculated for the different sludge types (KE floor sludge, KE Weasel Pit sludge, KE canister sludge, KW canister sludge, and residual sludge,) using the average sample plots (Figures 4.17 through 4.22). The resulting average sludge types are presented in Figures 4.23 through 4.26 and compared in Figure 4.27.

As shown in Figure 4.1, sieving results indicate that the KE canister sludge (96-04 L, 96-06 M, 96-06 L) has the greatest distribution of larger particles in sludges examined from the KE Basin. The Weasel Pit sludge is finer than the canister sludge, and the floor sludge is finer than the Weasel Pit sludge. Figure 4.16 shows that using the Microtrac X100 data alone, the KE canister sludge, KE Weasel Pit sludge, and KE floor sludge are all very similar. However, adding the sieving data forces the trend on the new calculated data set presented in Figure 4.27.

Figure 4.27 suggests that after sonication, the KE canister sludge contains the greatest distribution of large particles above 1000 μm. The particle size distribution of the KE Weasel Pit sludge is comparable to that of the floor sludge below ~250 μm. The KE floor sludge is finer than both the KE Weasel Pit sludge and the KE canister sludge. The residual sludge remains the finest material of those examined. The particle size distribution for the residual sludge is not affected by the addition of sieving data since R1 and R5 were calculated using the R5 sieving data, and all material in R5 was below 500 μm. Comparing the plots for the KW canister sludge in Figures 4.16 and 4.27 shows that below 200 μm this sludge is finer than all but the residual sludge. However, adding the sieving data to the KW canister plot results in a very broad particle size distribution. This wide distribution is the result of friable Grafoil present in the samples. Removing the Grafoil would result in a particle size distribution similar to that found by laser scattering as shown in Figure 4.16.

Figure 4.1. Comparison of Sieving Results for KE Basin Sludges

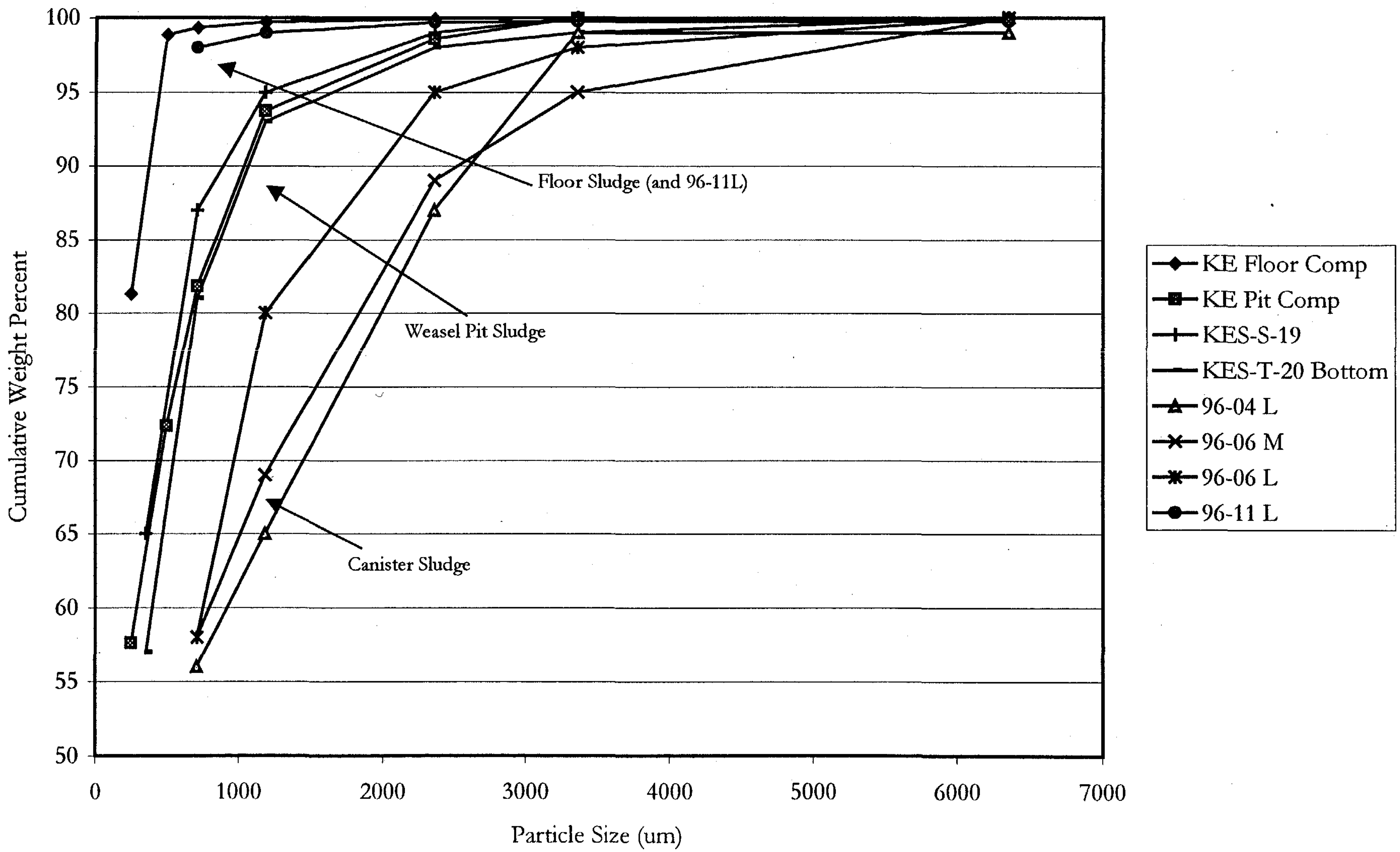


Figure 4.2. Particle Size Distribution Results for KE Basin 1993 Sampling (Brinkmann Model 2010, 0.5 to 150 um)

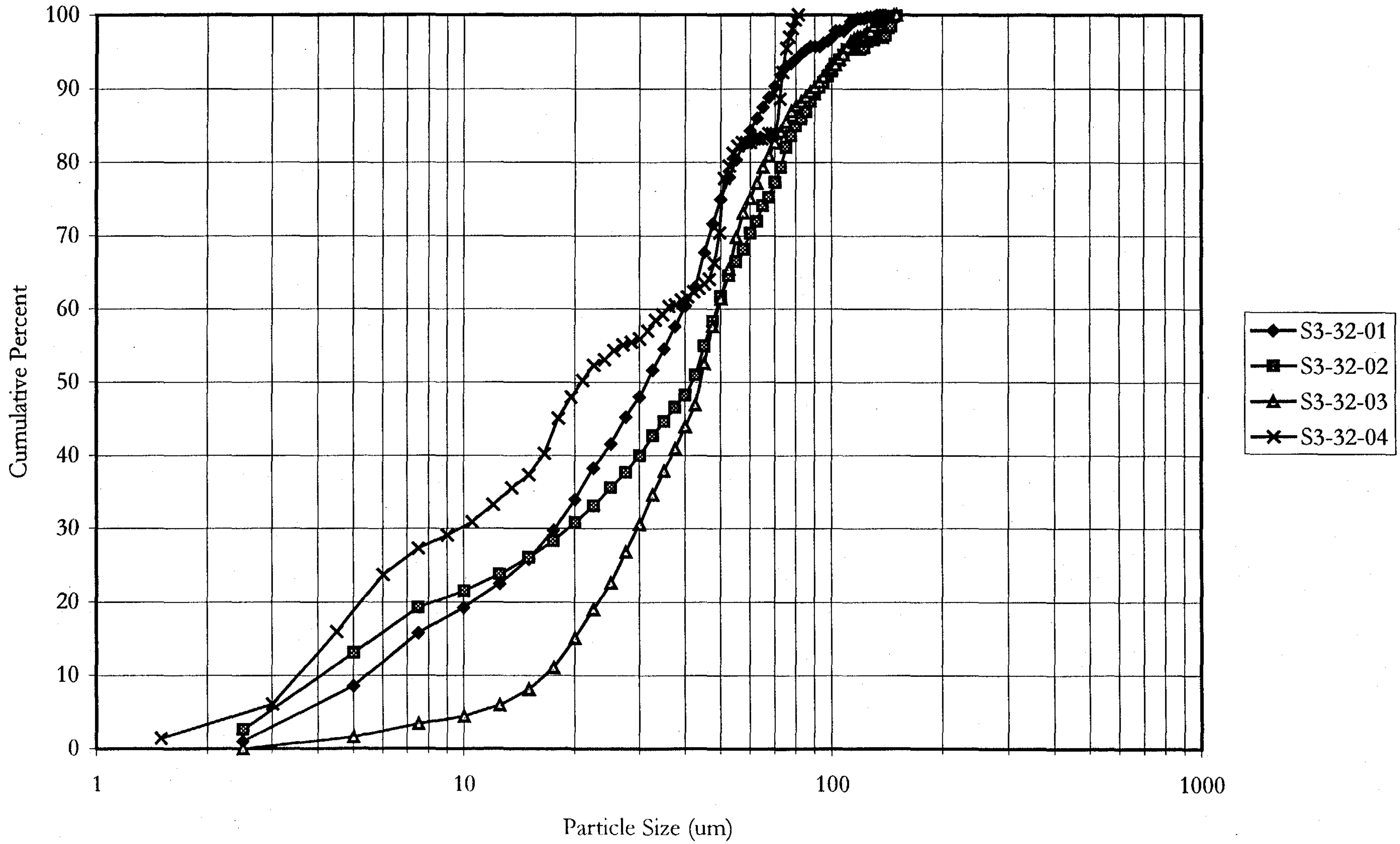


Figure 4.3. Particle Size Distribution Data from Samples KES-M-13 (KE Floor Sludge) and KES-T-20 (KE Weasel Pit) Before Sonication Without Sieving (Microtrac X100, 0.12 to 710 um)

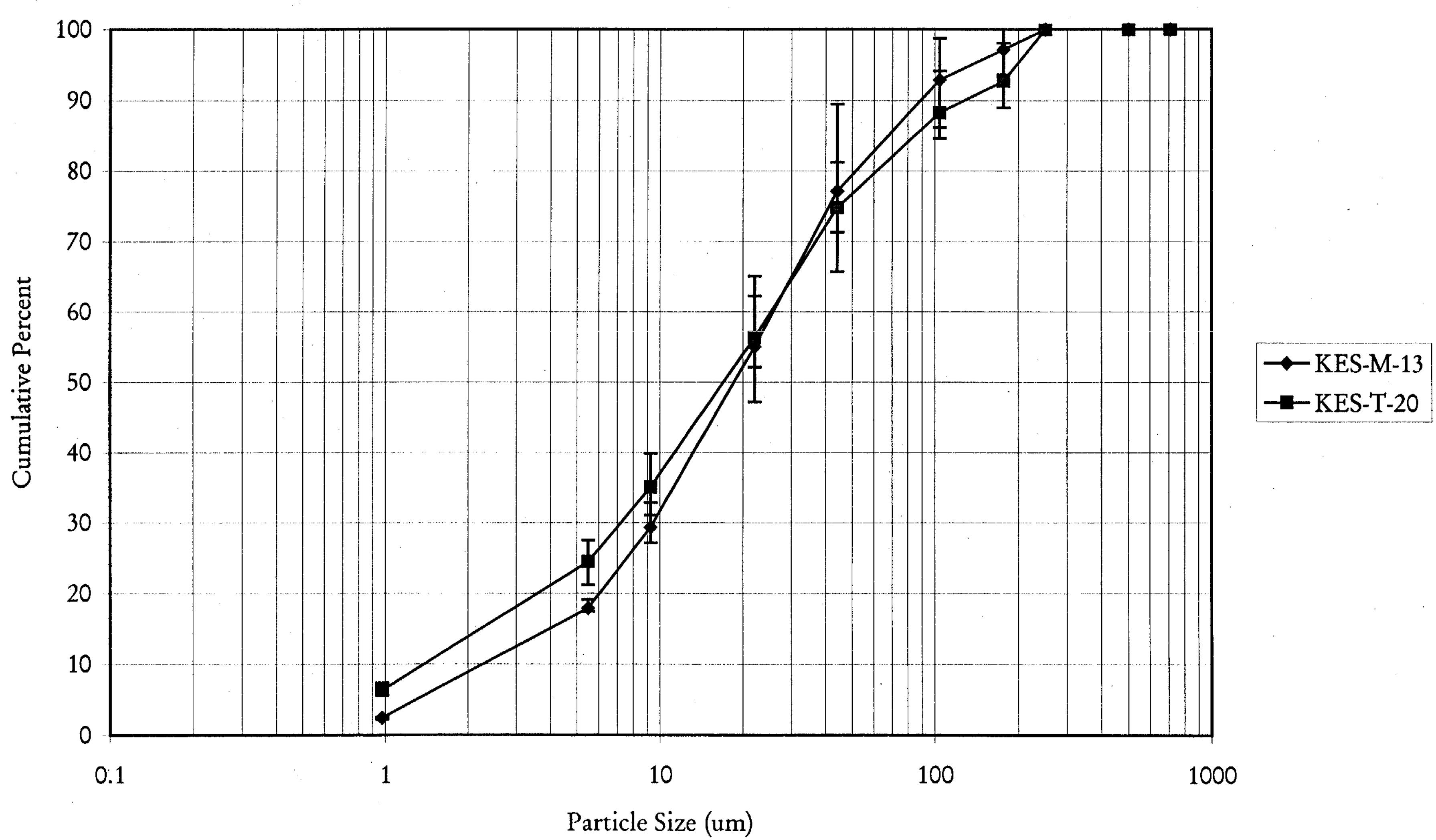


Figure 4.4. Particle Size Distribution Data from Sample 96-04 (KE Canister Sludge) Before Sonication Without Sieving (Microtrac X100, 0.12 to 710 um)

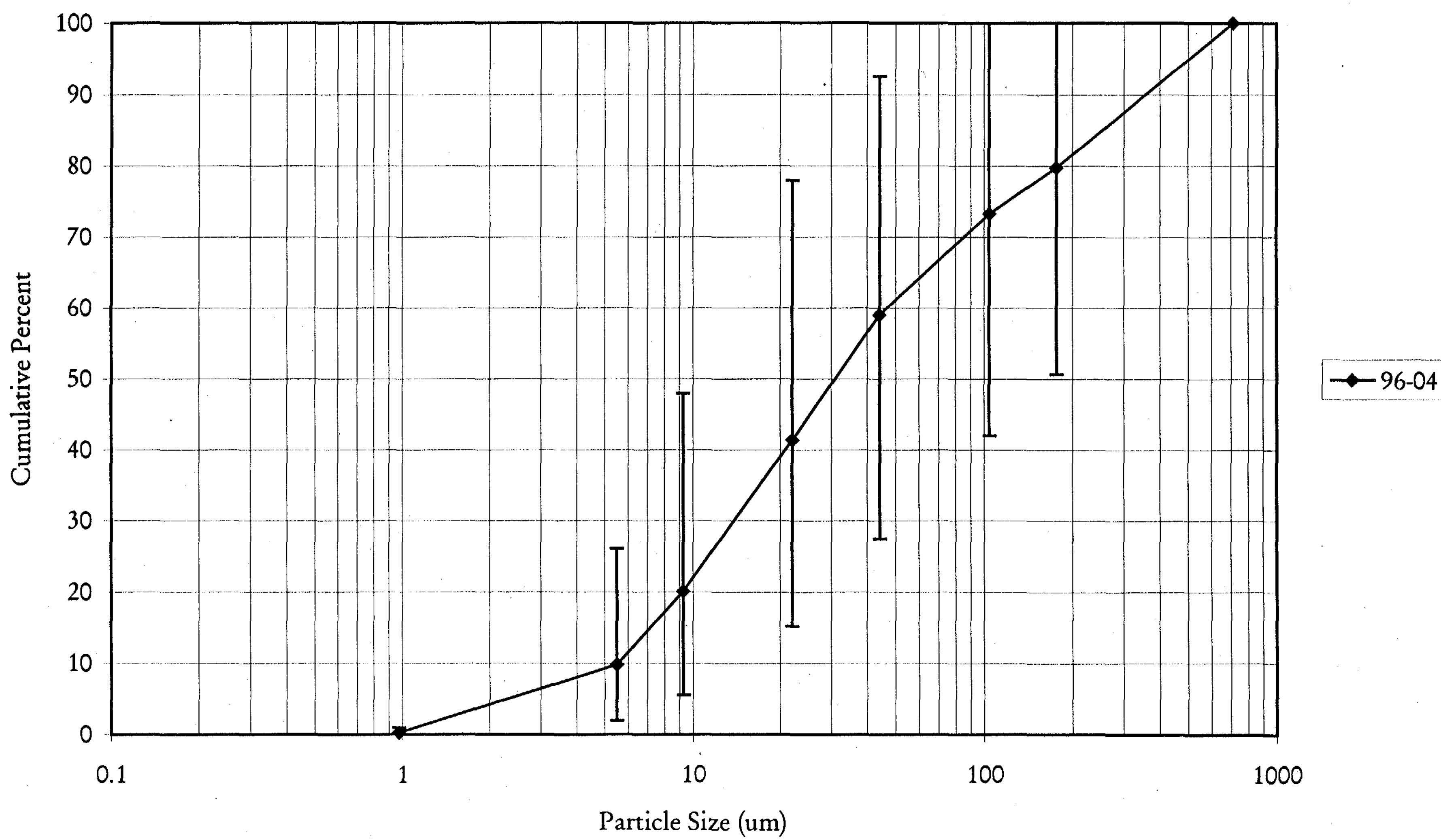


Figure 4.5. Particle Size Distribution Data from Sample 96-06 (KE Canister Sludge) Before Sonication Without Sieving (Microtrac X100, 0.12 to 710 um)

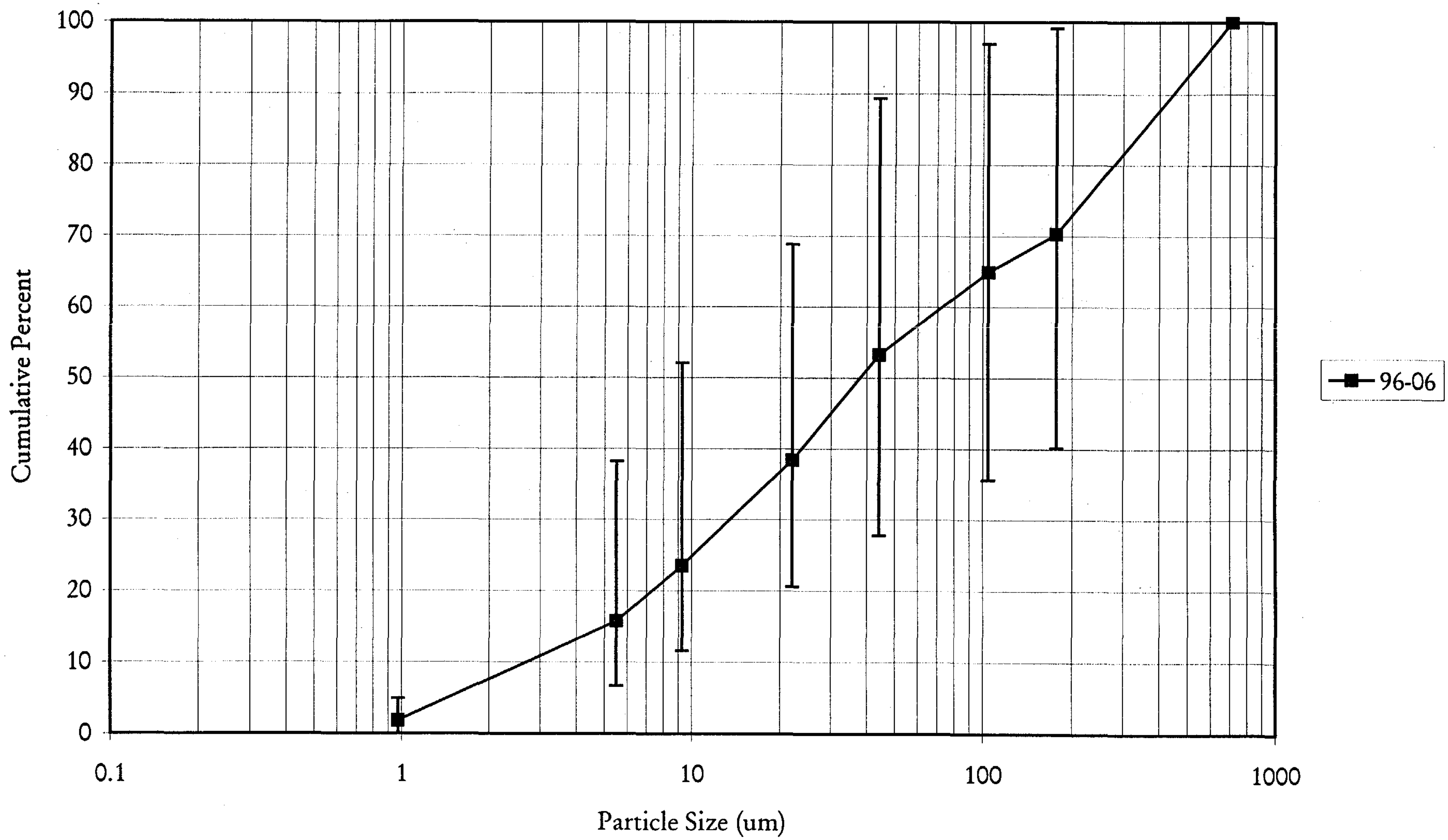


Figure 4.6. Particle Size Distribution Data from Sample 96-11 (KE Canister Sludge) Before Sonication Without Sieving (Microtrac X100, 0.12 to 710 um)

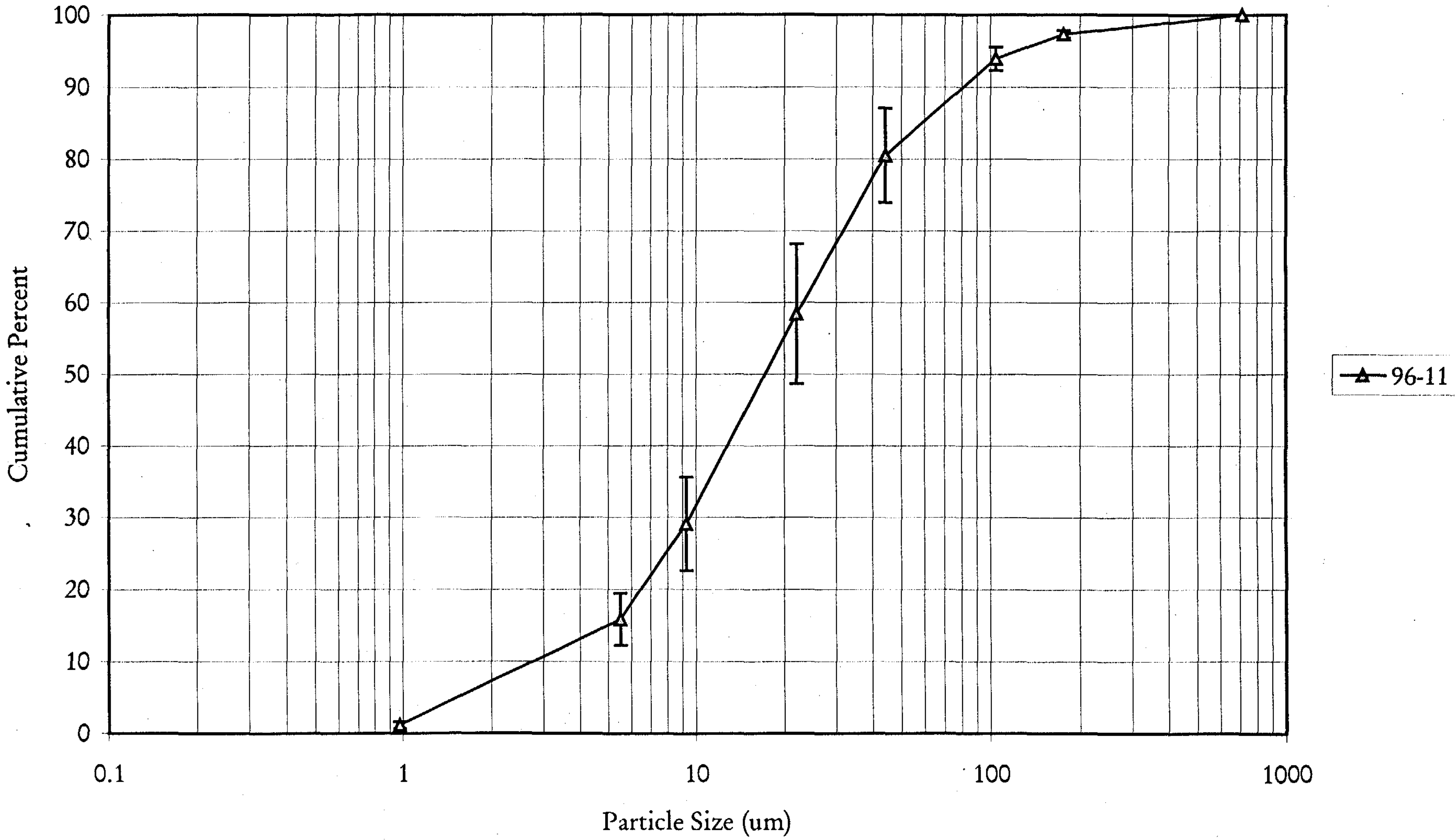


Figure 4.7. Particle Size Distribution Data from KW Canister Sludge Samples Before Sonication Without Sieving (Microtrac X100, 0.12 to 710 um)

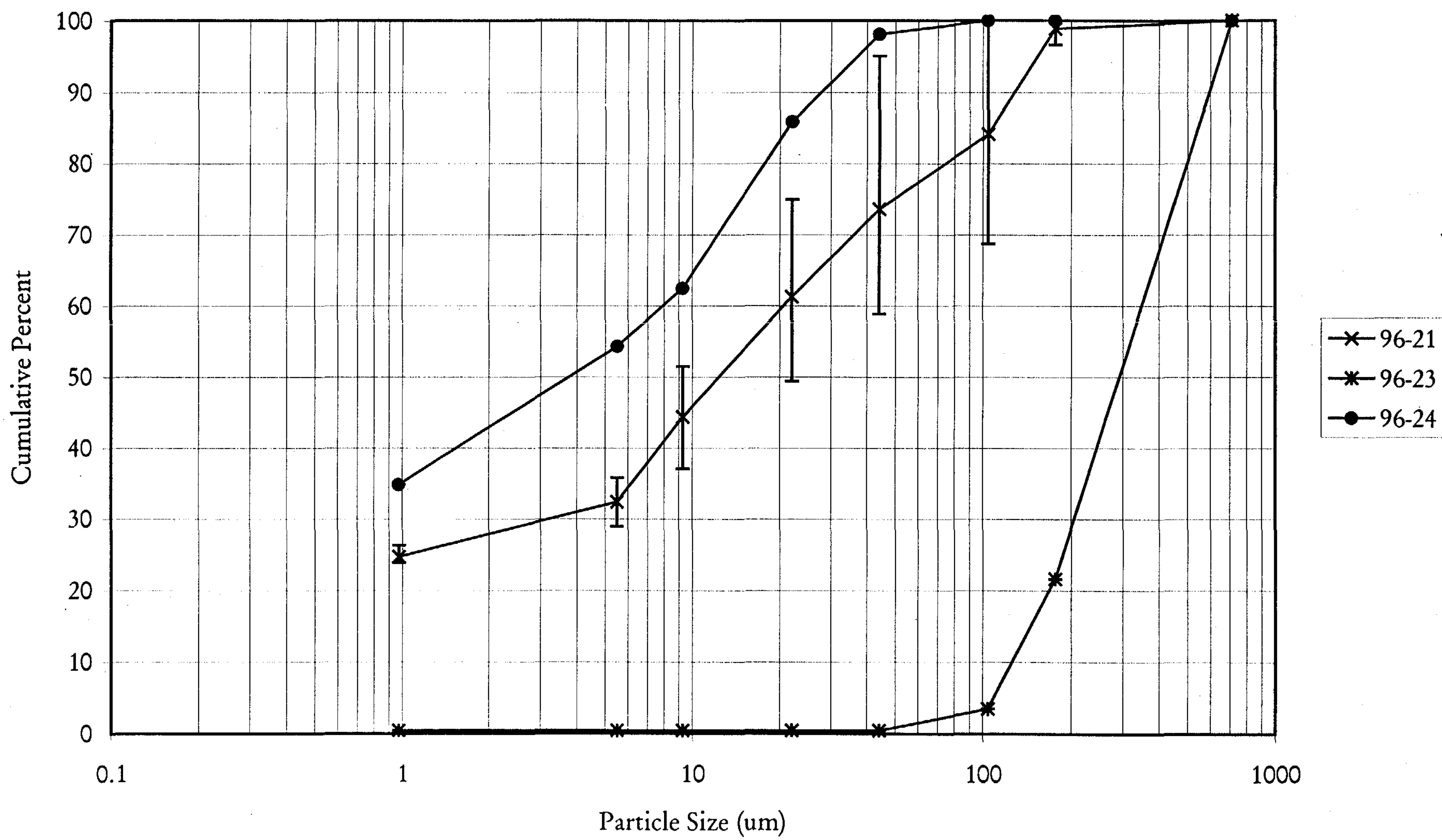


Figure 4.8. Particle Size Distribution Data from Residual Sludge Samples (from Fuel Element Transport) Before Sonication Without Sieving (Microtrac X100, 0.12 to 710 um)

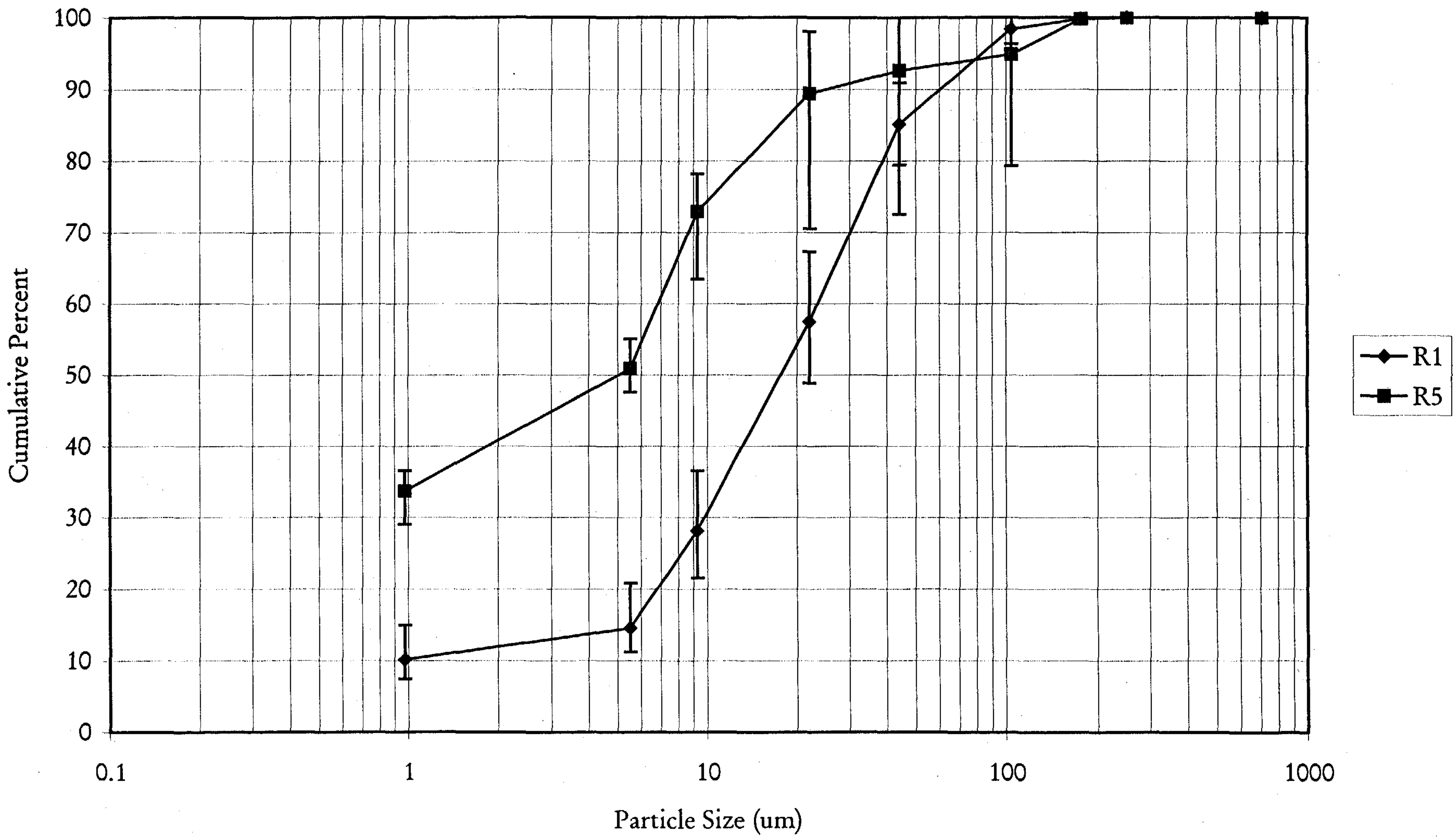


Figure 4.9. Average Particle Size Distribution Results for Samples KES-M-13 (KE Floor Sludge) and KES-T-20 (KE Weasel Pit) After Sonication (Microtrac X100, 0.12 to 710 um)

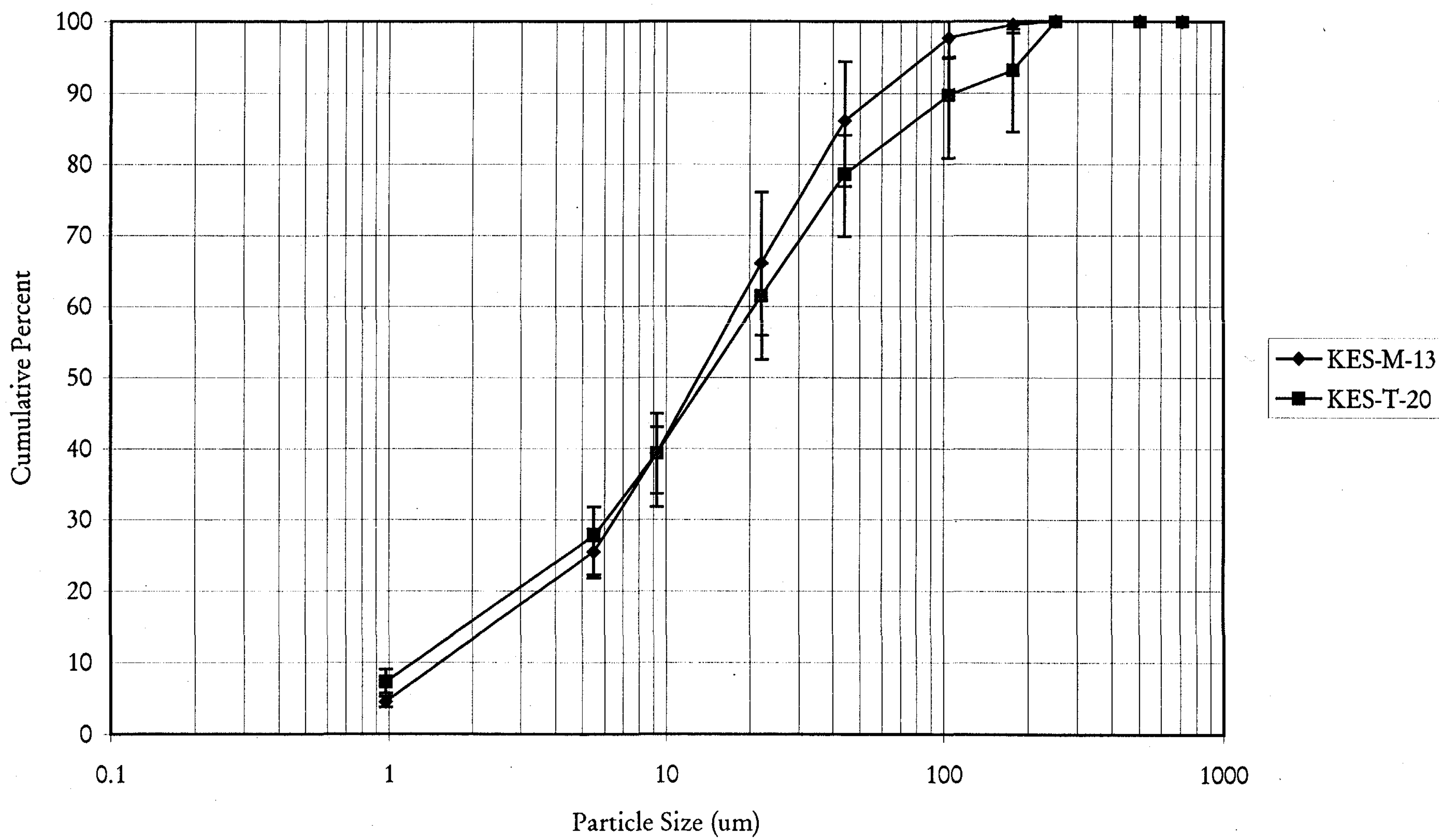


Figure 4.10. Average Particle Size Distribution Results for Sample 96-04 (KE Canister Sludge) After Sonication (Microtrac X100, 0.12 to 710 um)

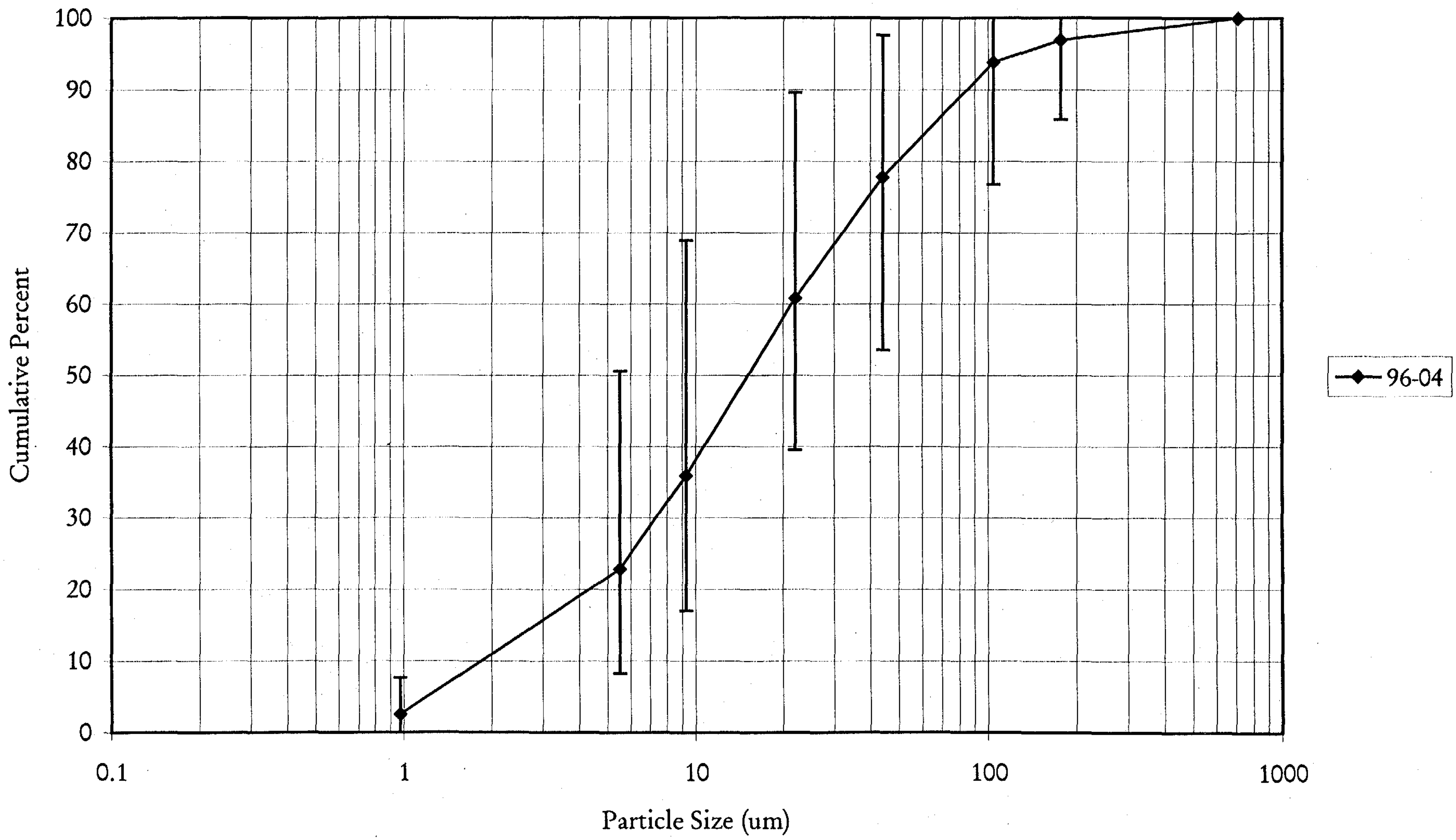


Figure 4.11. Average Particle Size Distribution Results for Sample 96-06 (KE Canister Sludge) After Sonication (Microtrac X100, 0.12 to 710 um)

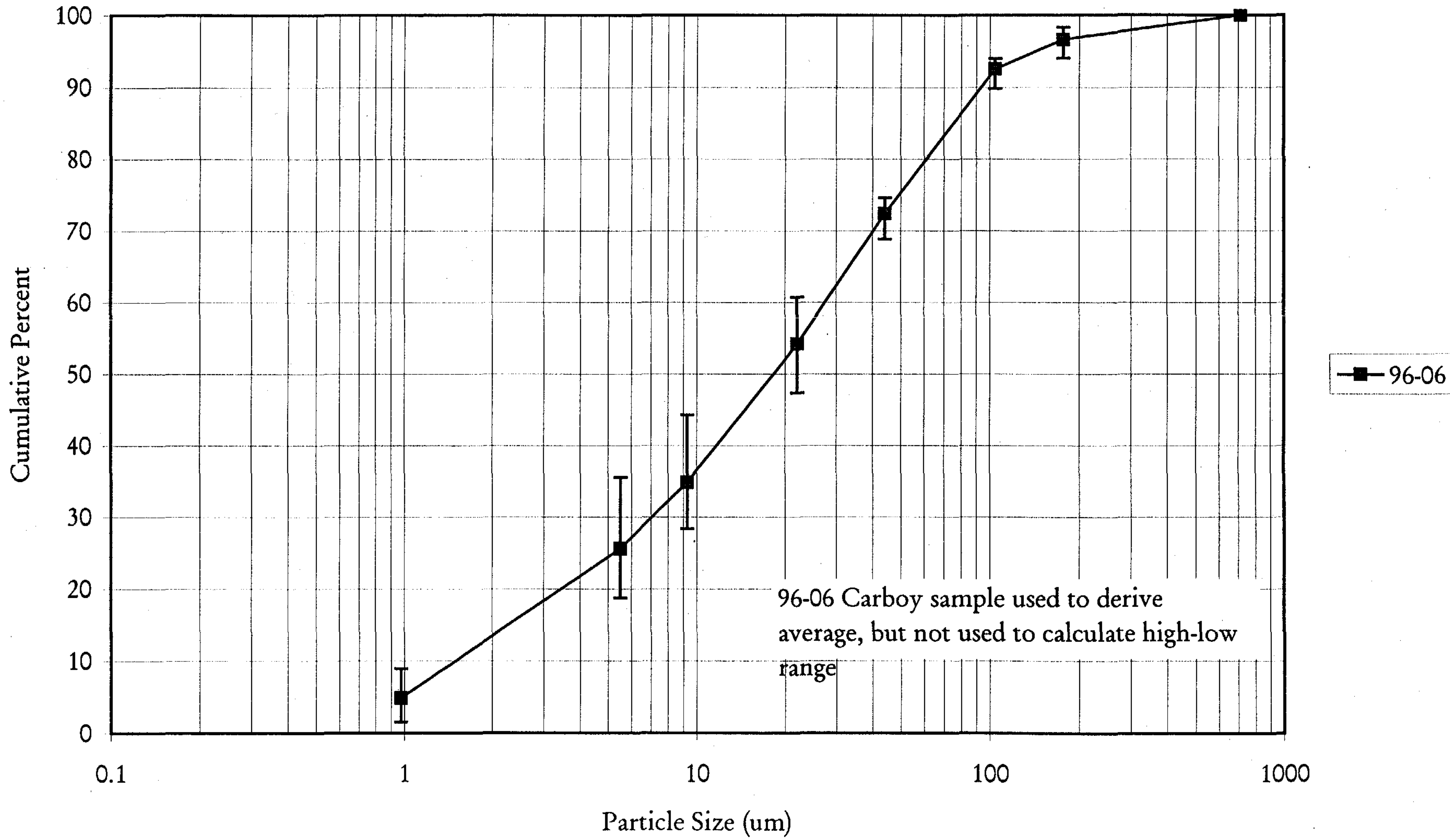


Figure 4.12. Average Particle Size Distribution Results for Sample 96-11 (KE Canister Sludge) After Sonication (Microtrac X100, 0.12 to 710 um)

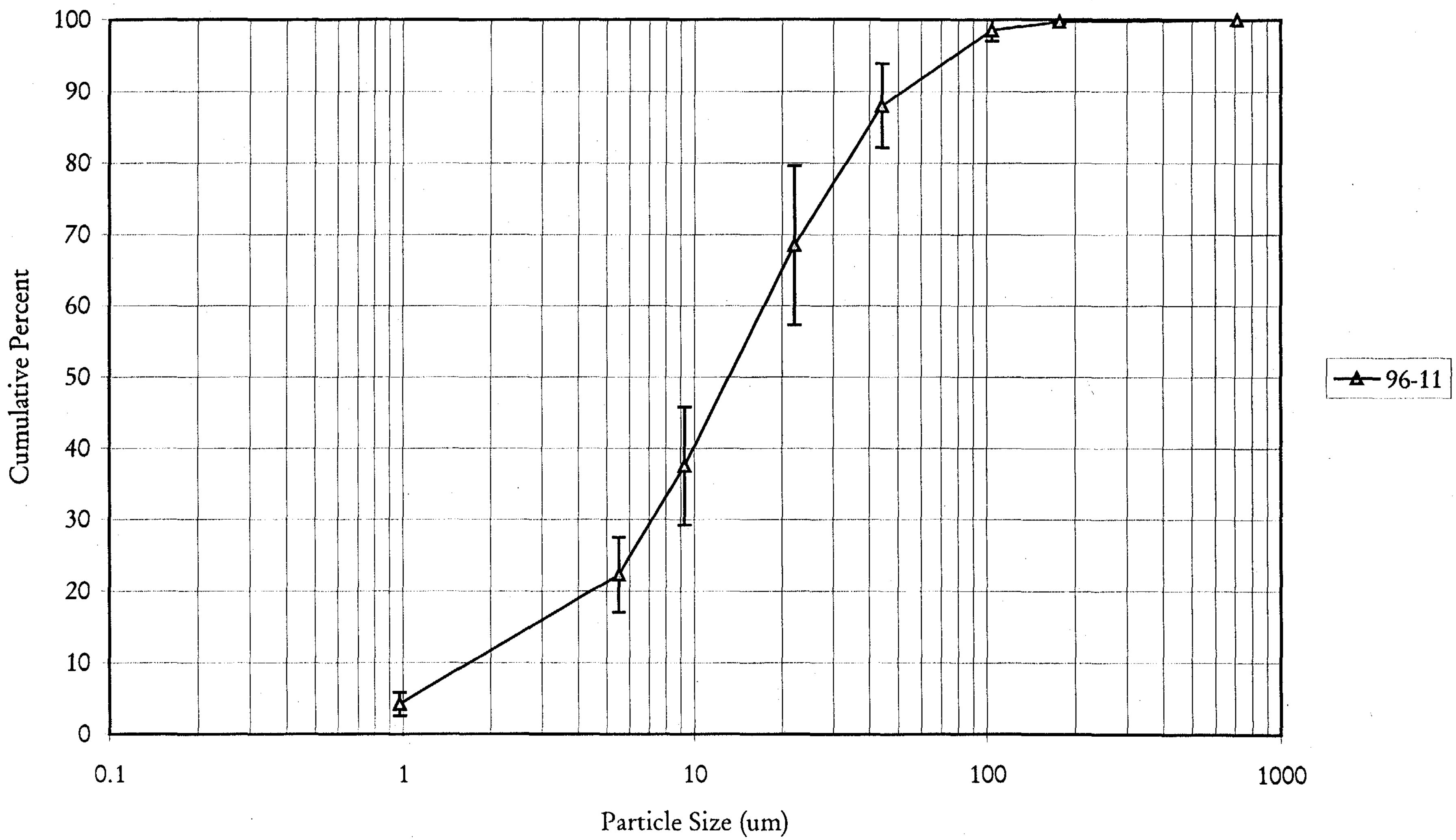


Figure 4.13. Average Particle Size Distribution Results for KW Canister Sludge Samples After Sonication (Microtrac X100, 0.12 to 710 um)

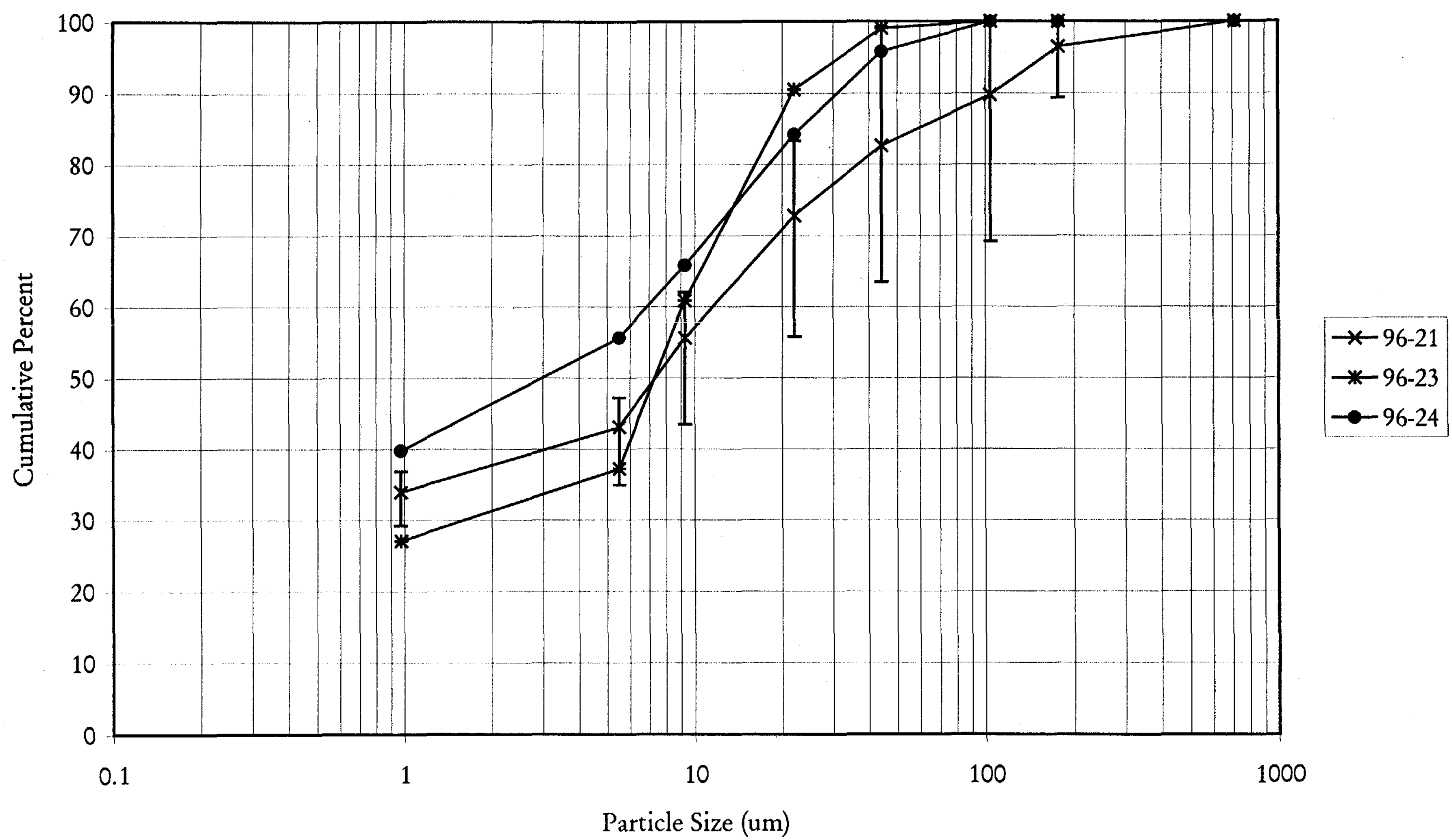


Figure 4.14. Average Particle Size Distribution Results for Residual Sludge Samples (from Fuel Element Transport) After Sonication (Microtrac X100, 0.12 to 710 um)

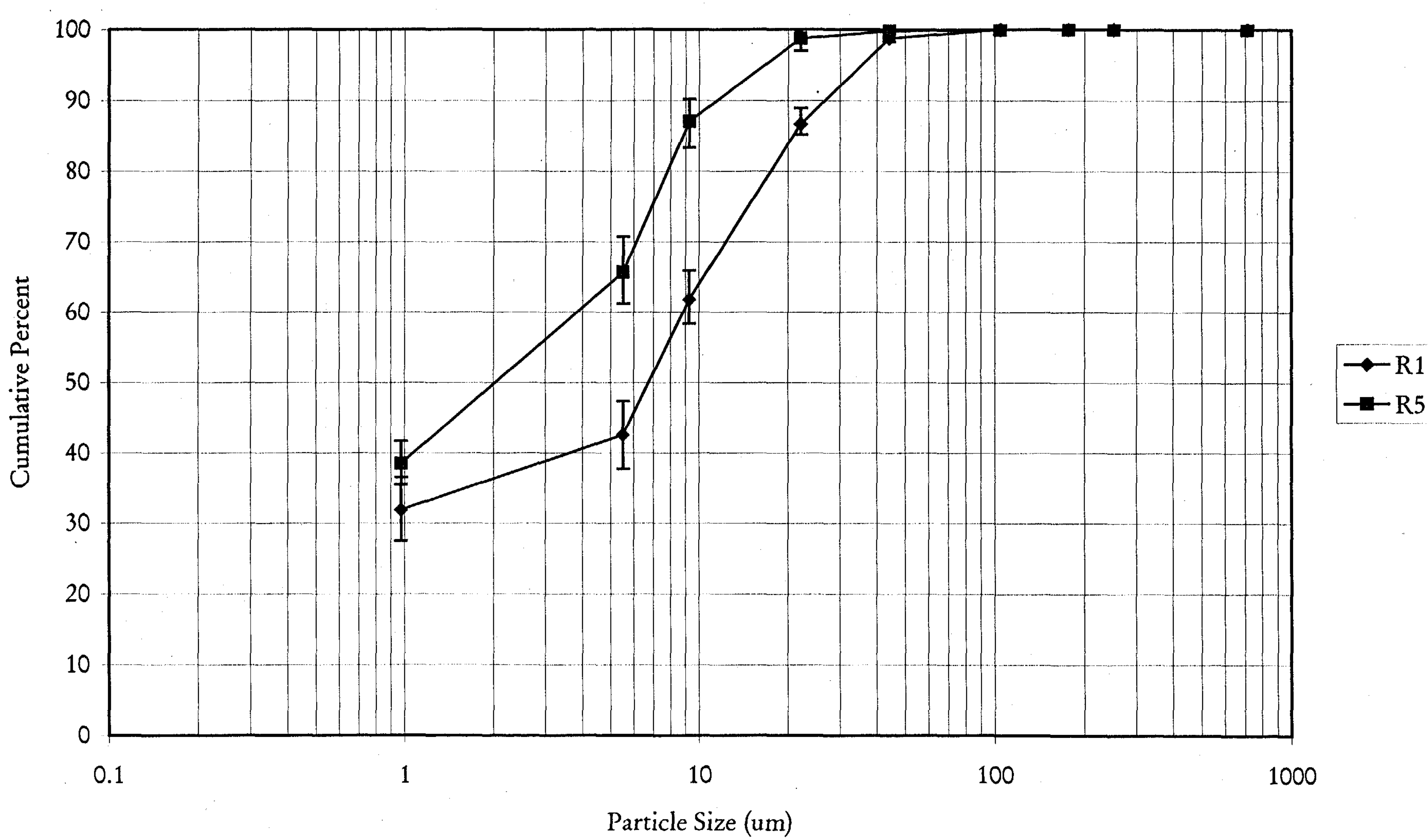


Figure 4.15. Comparison of Average Particle Size Distributions for Sludge Types Before Sonication (Microtrac X100, 0.12 to 710 um)

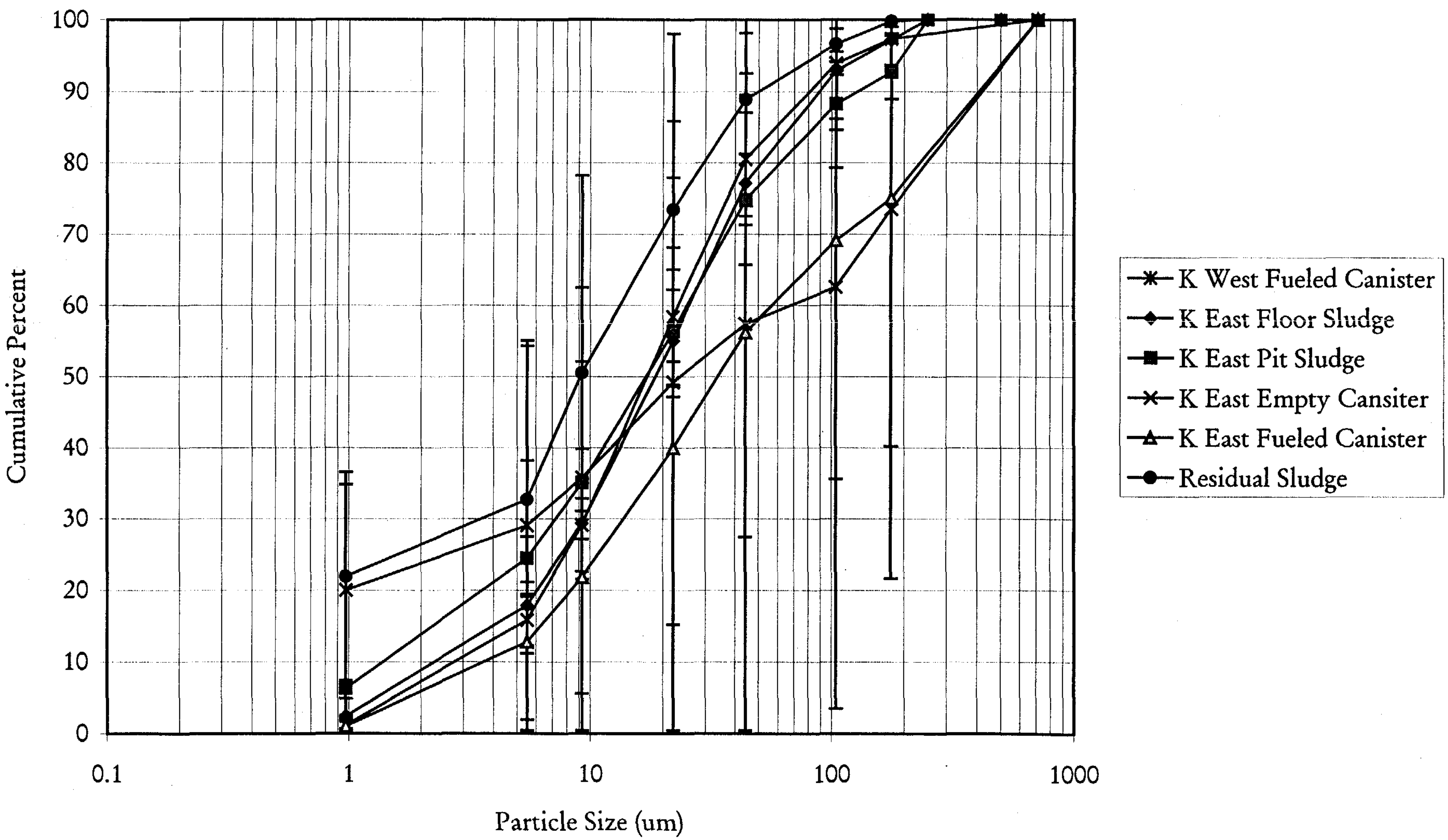


Figure 4.16. Comparison of Average Particle Size Distributions for Sludge Types After Sonication (Microtrac X100, 0.12 to 710 um)

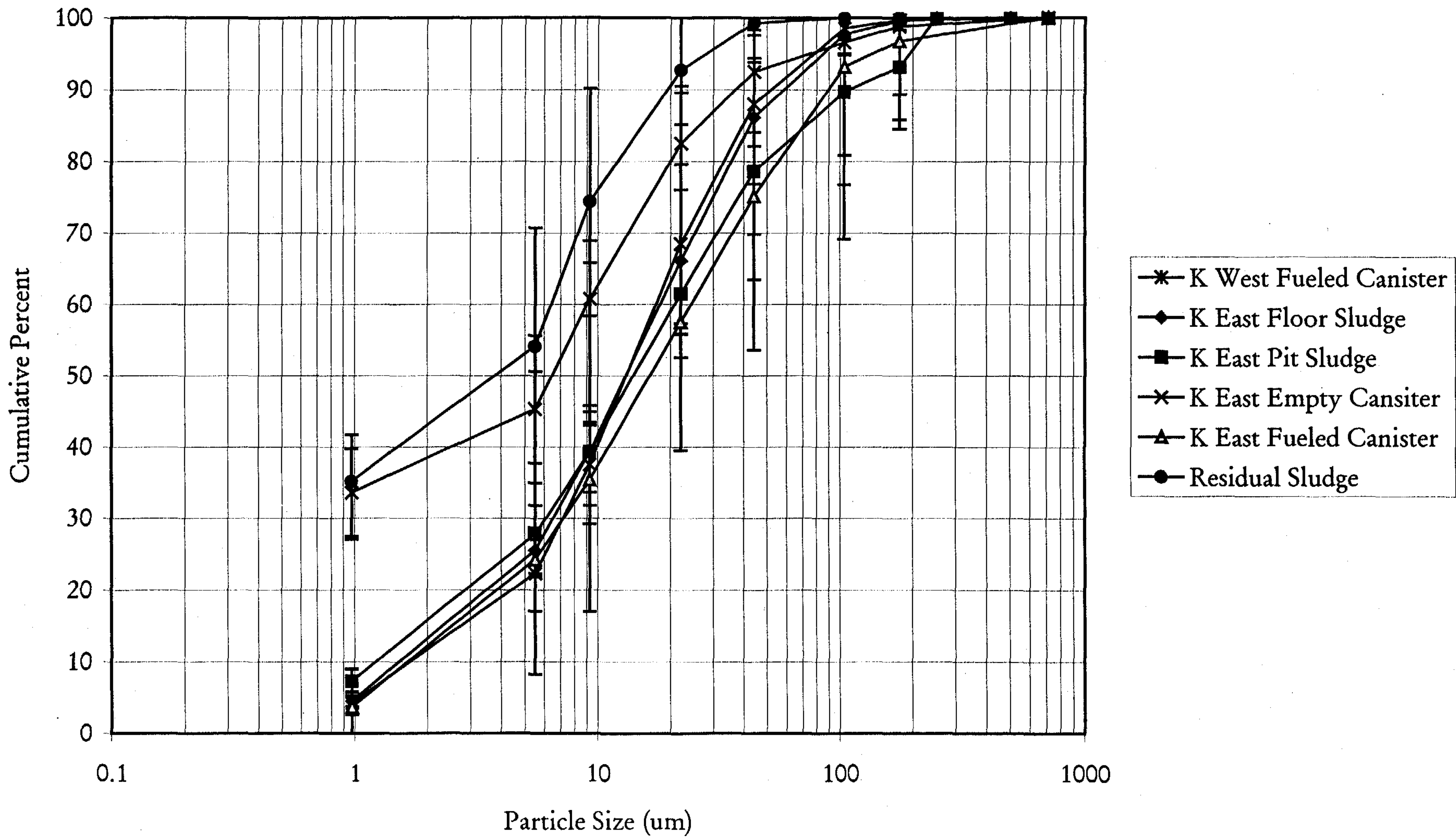


Figure 4.17. Integrated Particle Size Distribution for Samples KES-M-13 (KE Floor Sludge) and KES-T-20 (KE Weasel Pit) After Sonication (Microtrac X100 and Sieving, 0.12 to 6350 um)

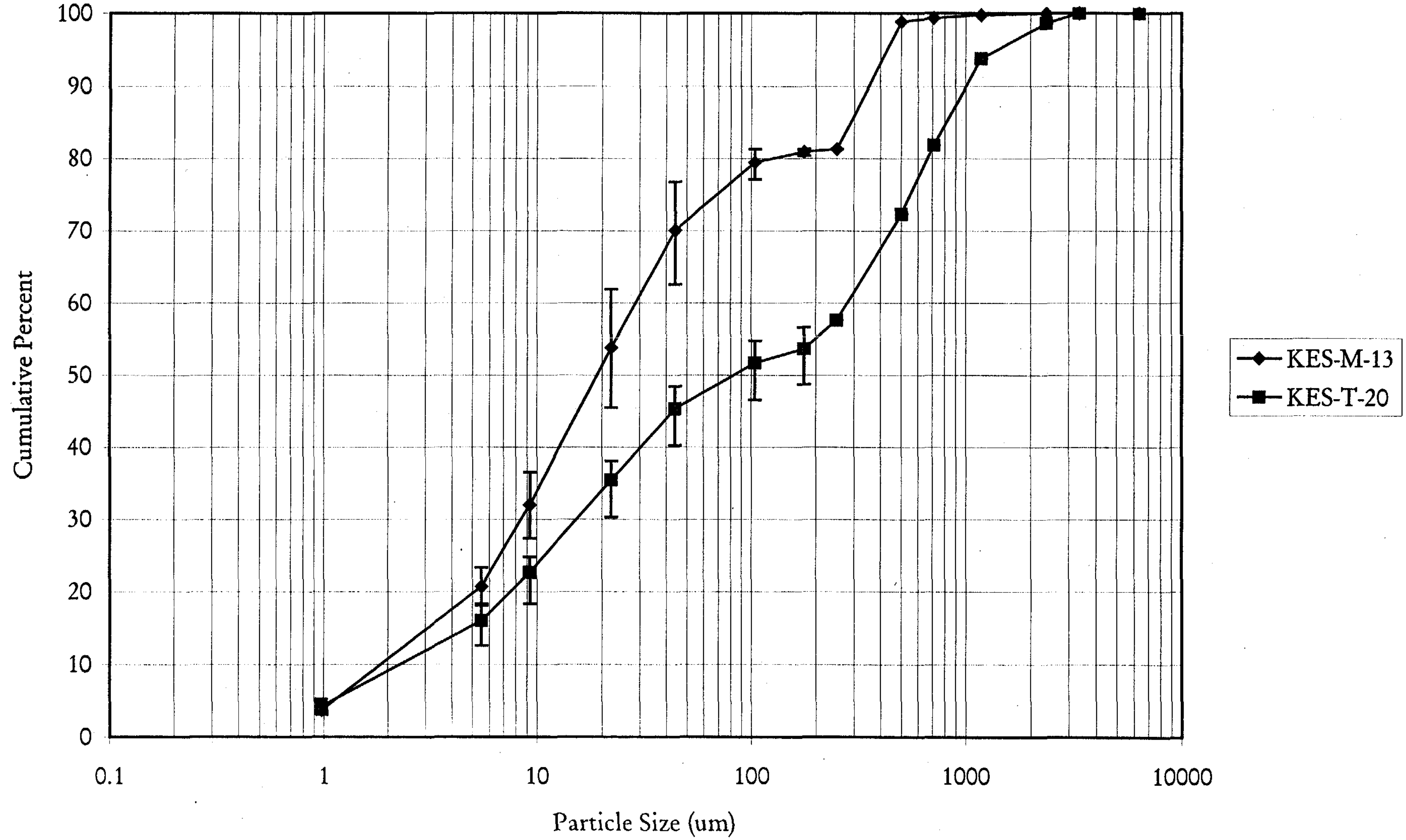


Figure 4.18. Integrated Particle Size Distribution for Sample 96-04 (KE Canister Sludge) After Sonication (Microtrac X100 and Sieving, 0.12 to 6350 um)

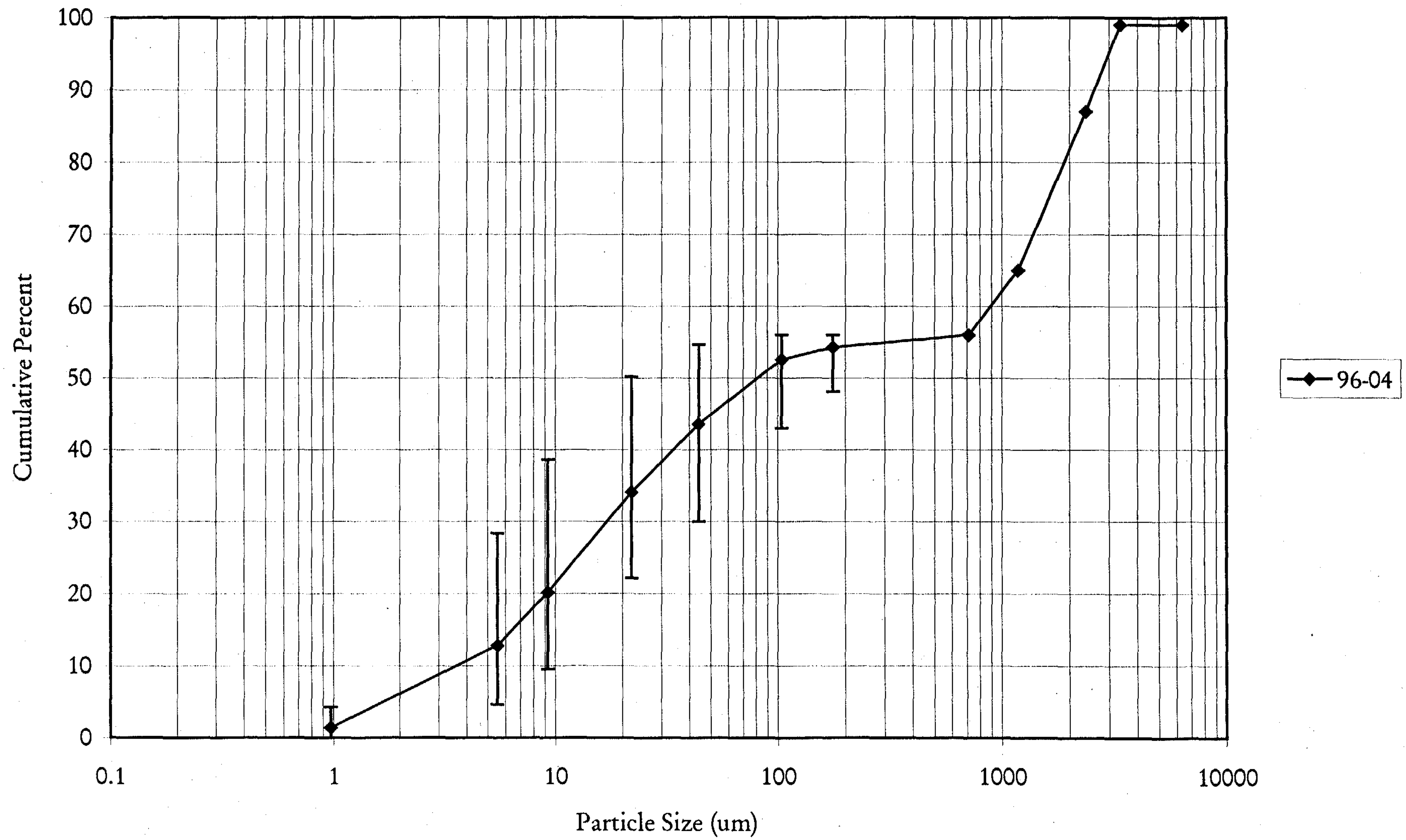


Figure 4.19. Integrated Particle Size Distribution for Sample 96-06 (KE Canister Sludge) After Sonication (Microtrac X100 and Sieving, 0.12 to 6350 um)

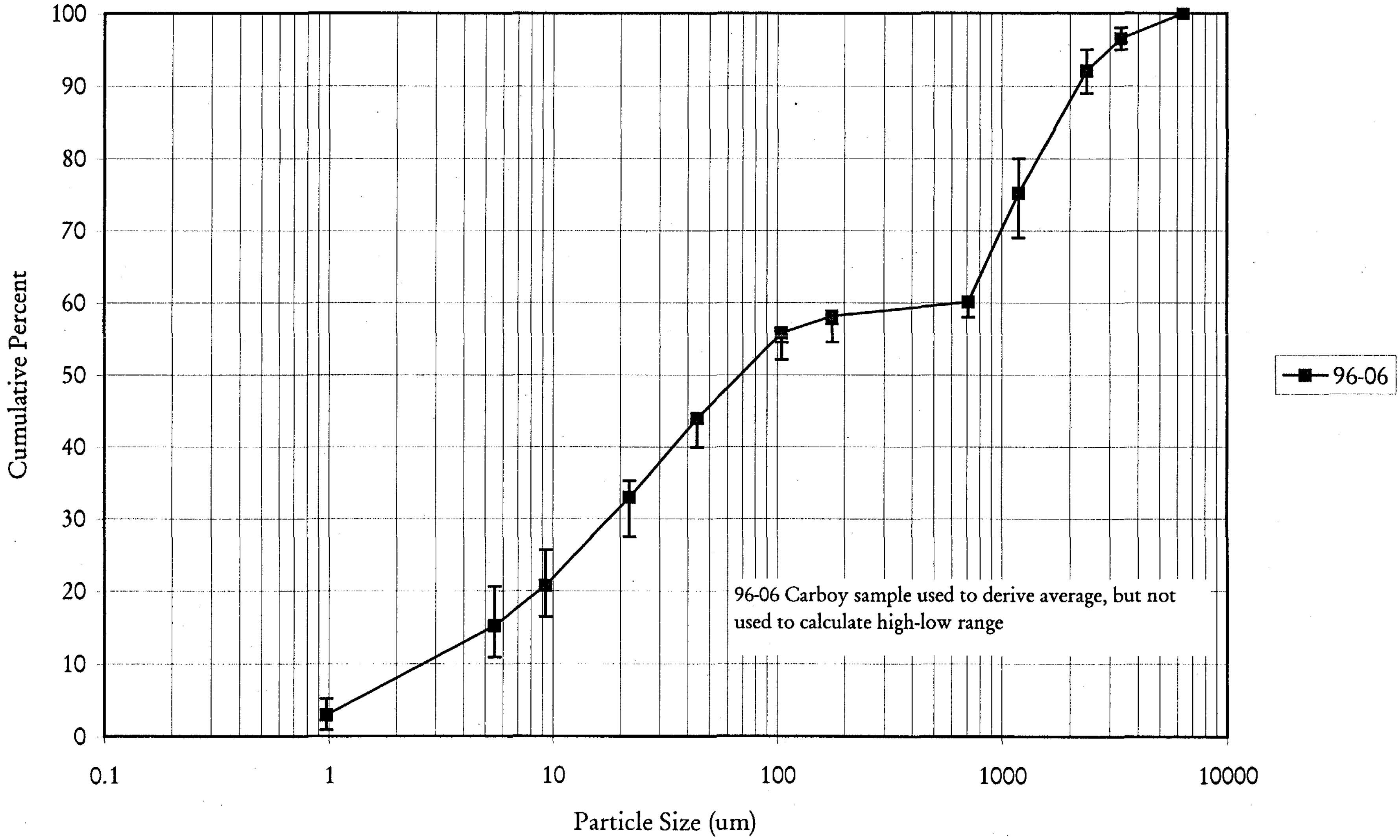


Figure 4.20. Integrated Particle Size Distribution for Sample 96-11 (KE Canister Sludge) After Sonication (Microtrac X100 and Sieving, 0.12 to 6350 um)

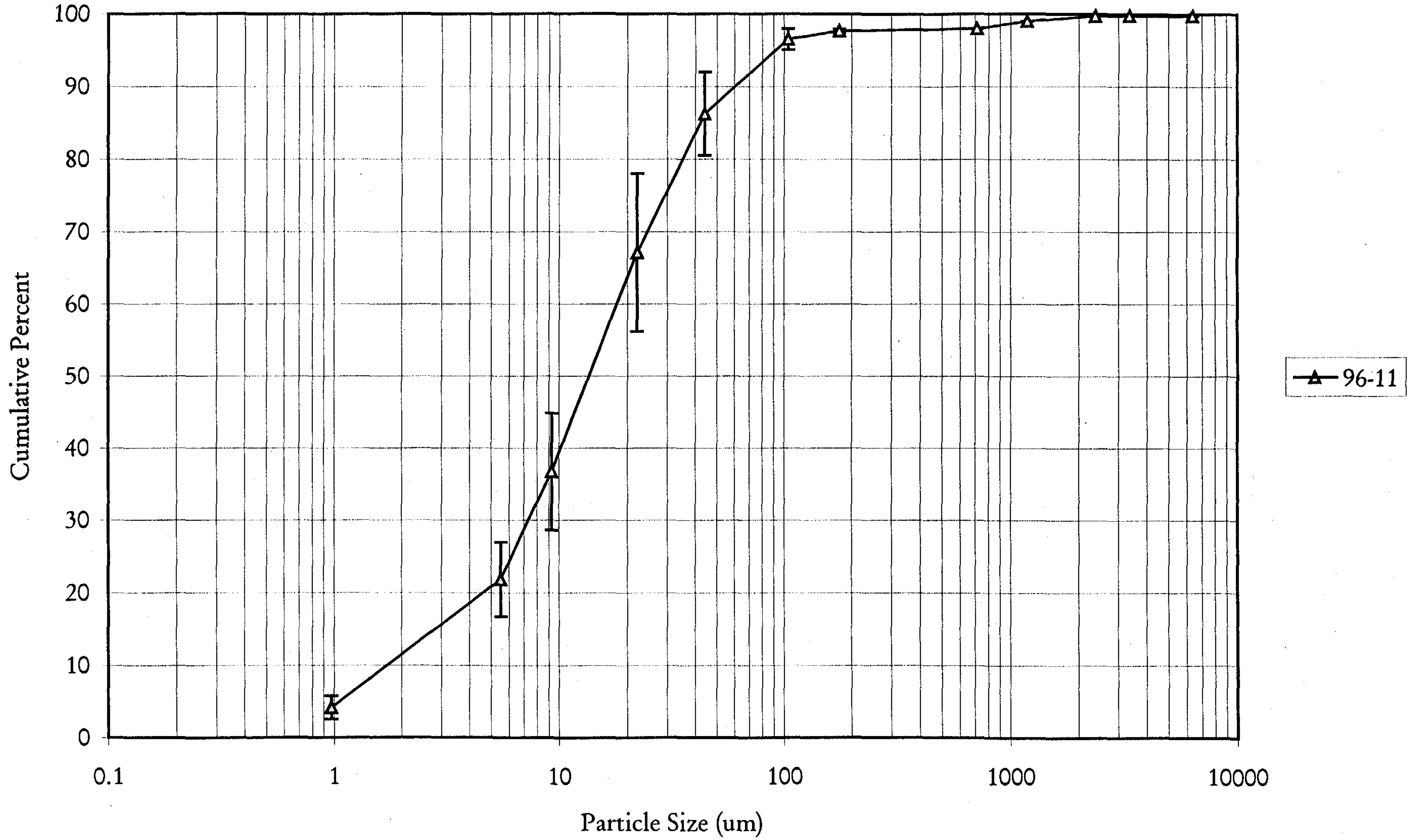


Figure 4.21. Integrated Particle Size Distribution for KW Canister Sludge Samples After Sonication (Microtrac X100 and Sieving, 0.12 to 6350 um)

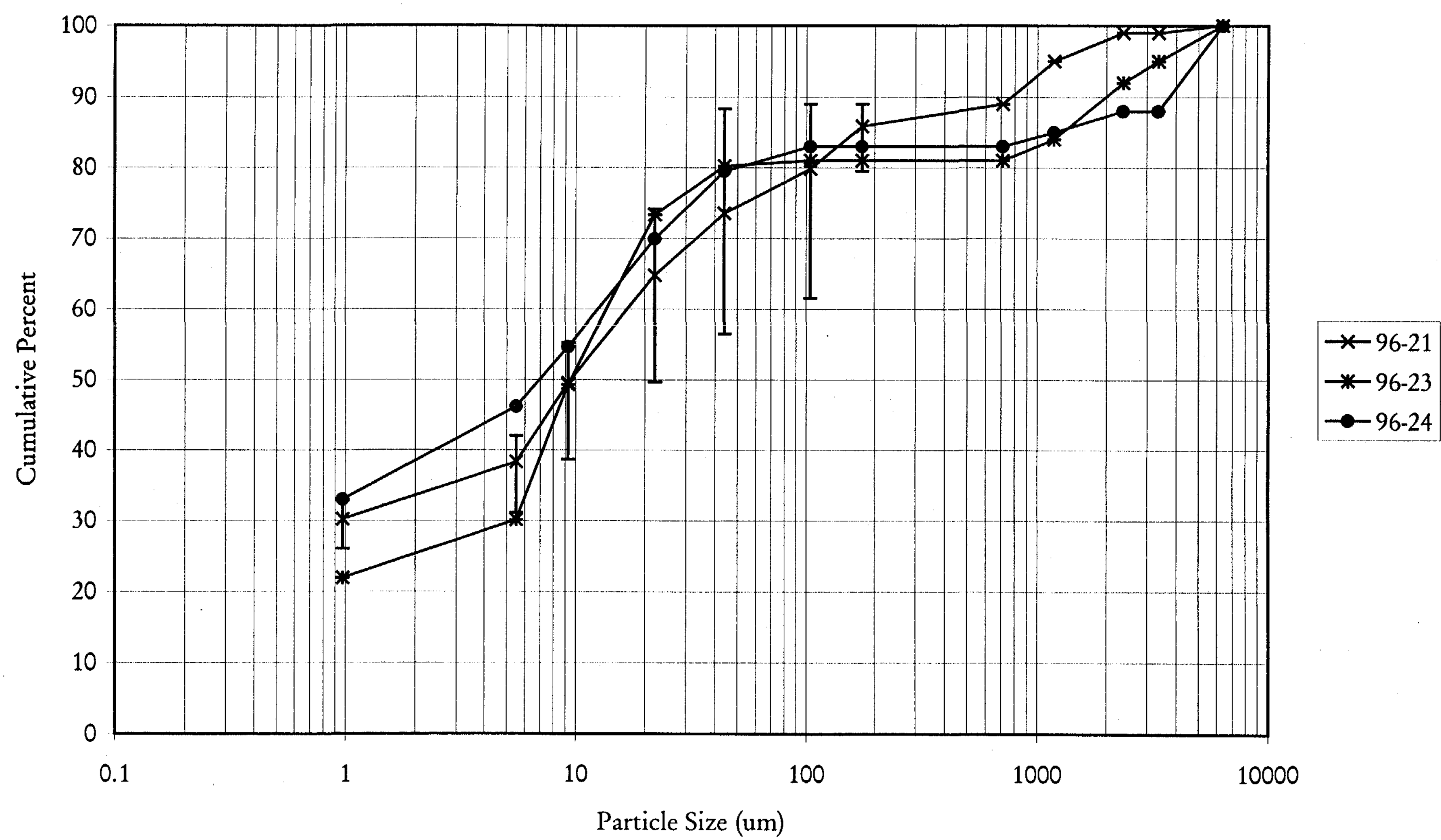


Figure 4.22. Integrated Particle Size Distribution for Residual Sludge Samples (from Fuel Element Transport) After Sonication (Microtrac X100 and Sieving, 0.12 to 6350 um)

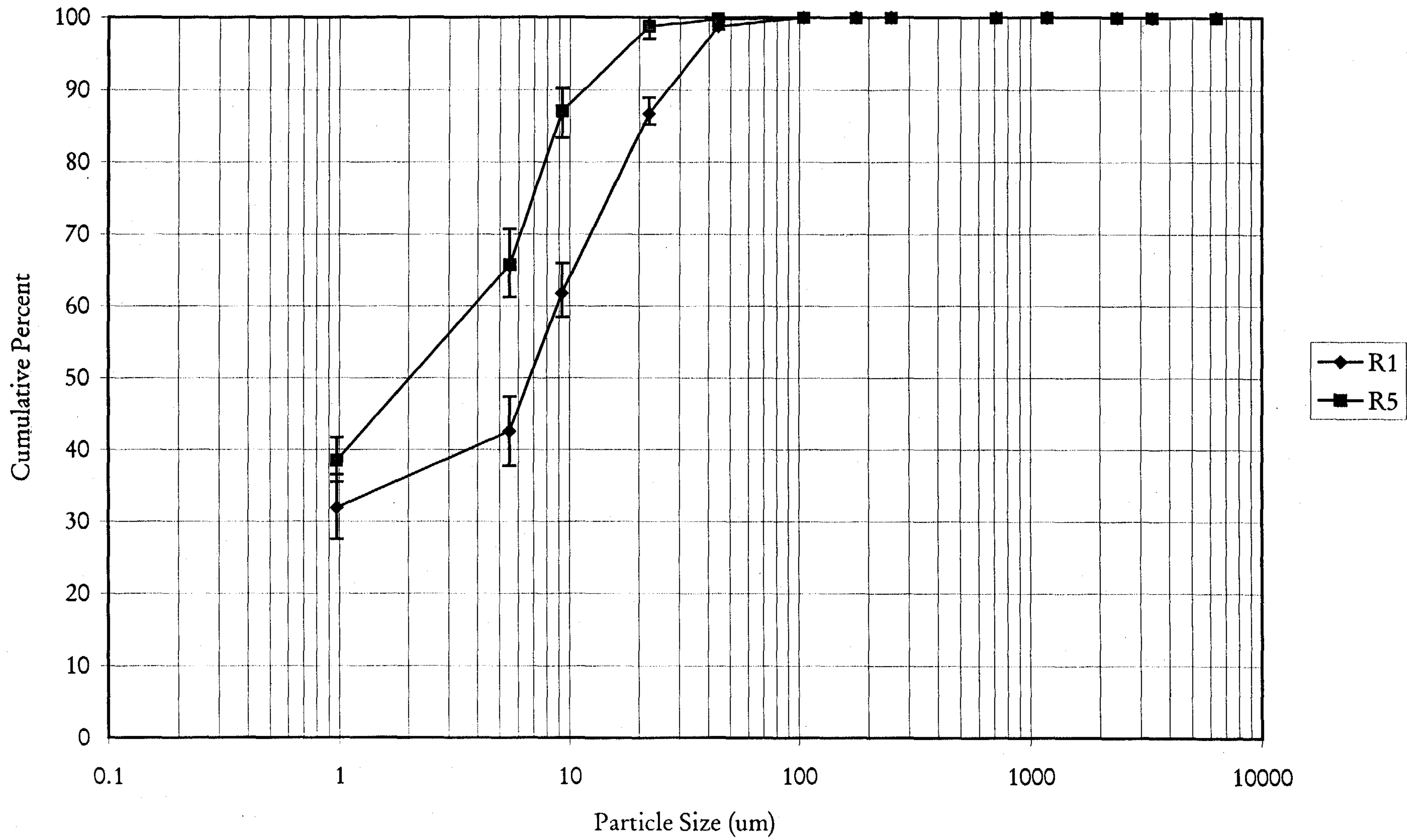


Figure 4.23. Integrated Particle Size Distribution for K East Floor and Weasel Pit Sludge After Sonication (Microtrac X100 and Sieving, 0.12 to 6350 um)

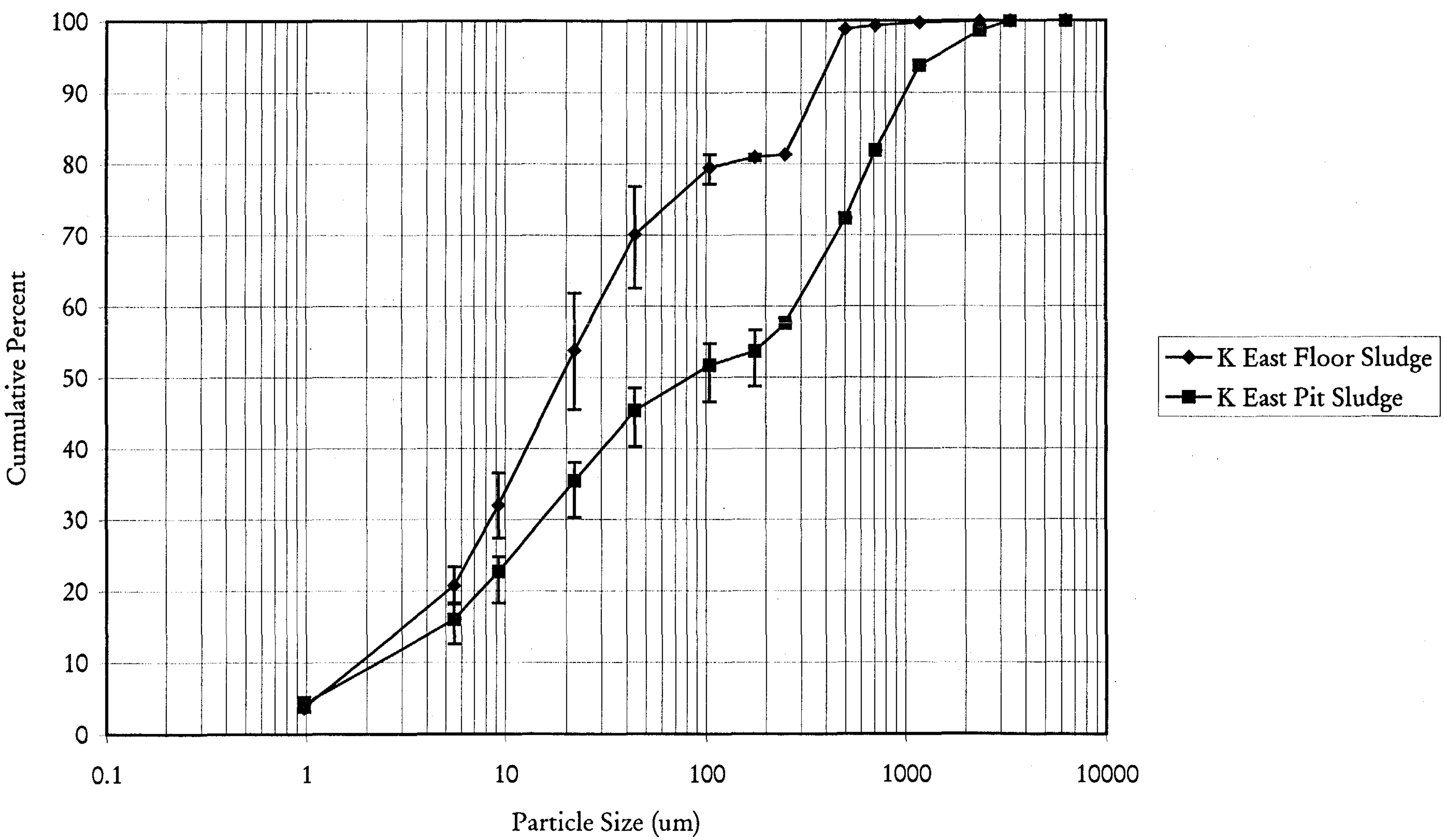


Figure 4.24. Integrated Particle Size Distribution for K East Canister Sludge After Sonication (Microtrac X100 and Sieving, 0.12 to 6350 um)

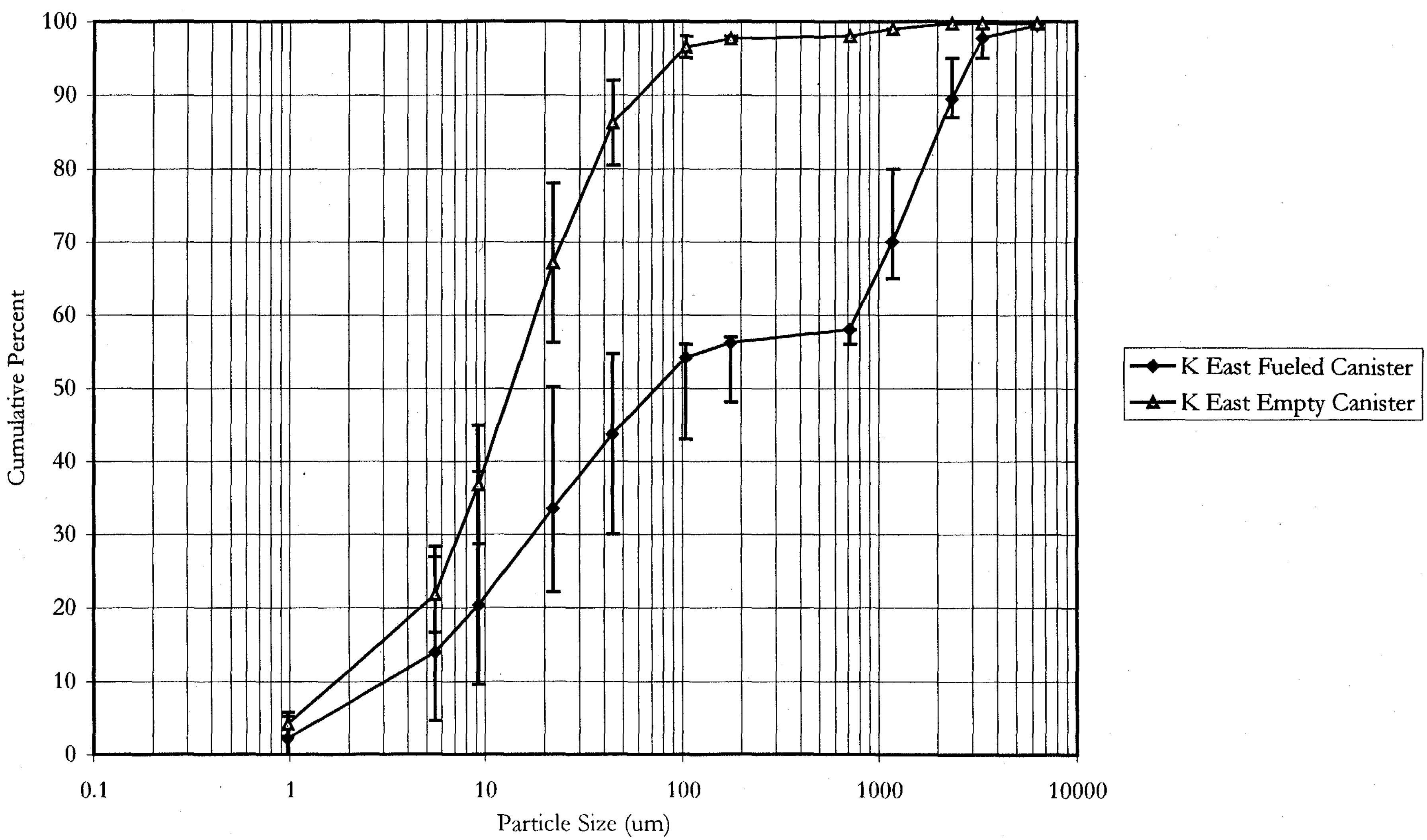


Figure 4.25. Integrated Particle Size Distribution for K West Canister Sludge After Sonication (Microtrac X100 and Sieving, 0.12 to 6350 um)

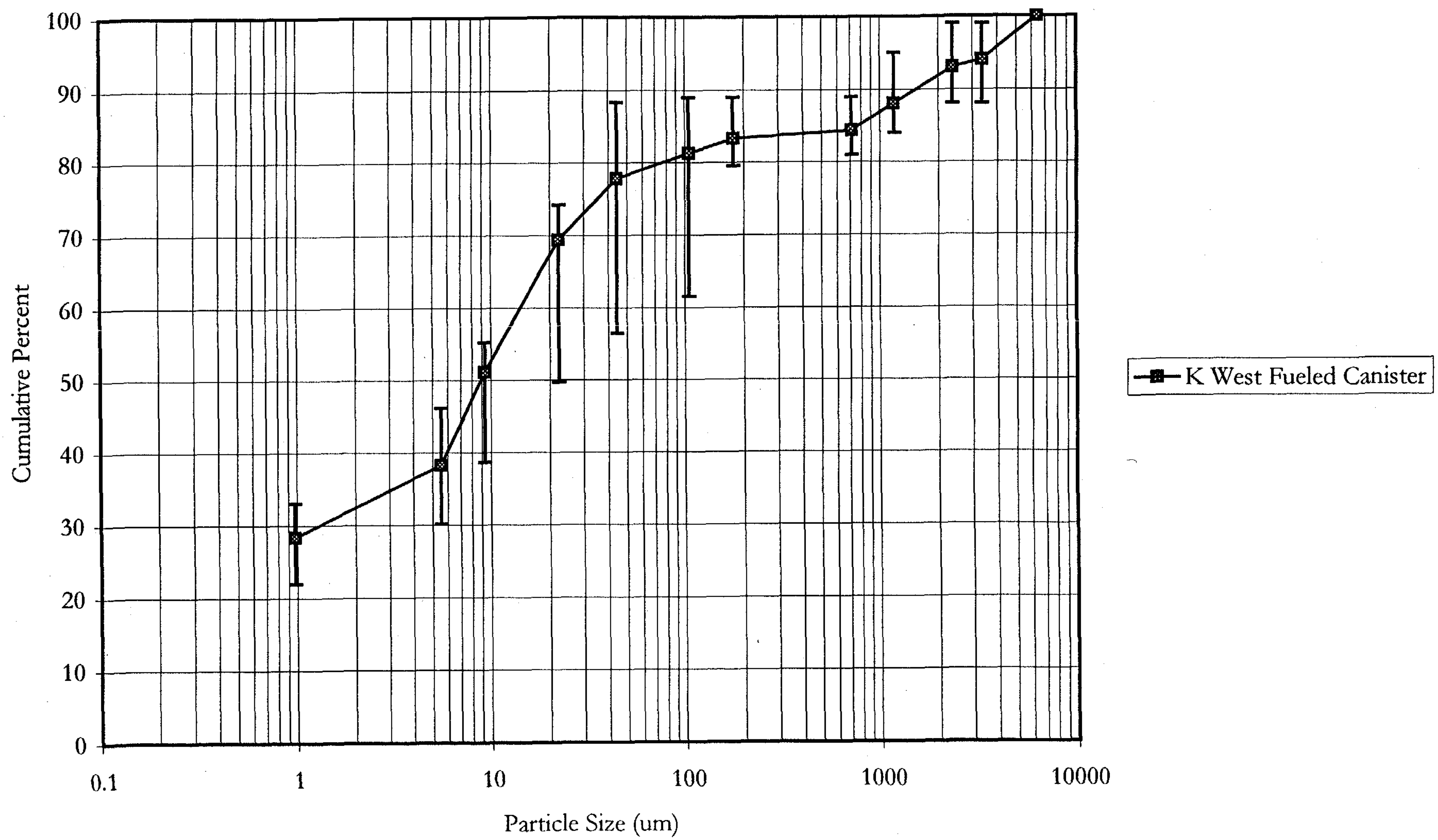


Figure 4.26. Integrated Particle Size Distribution for Residual Sludge (from Fuel Element Transport) After Sonication (Microtrac X100 and Sieving, 0.12 to 6350 um)

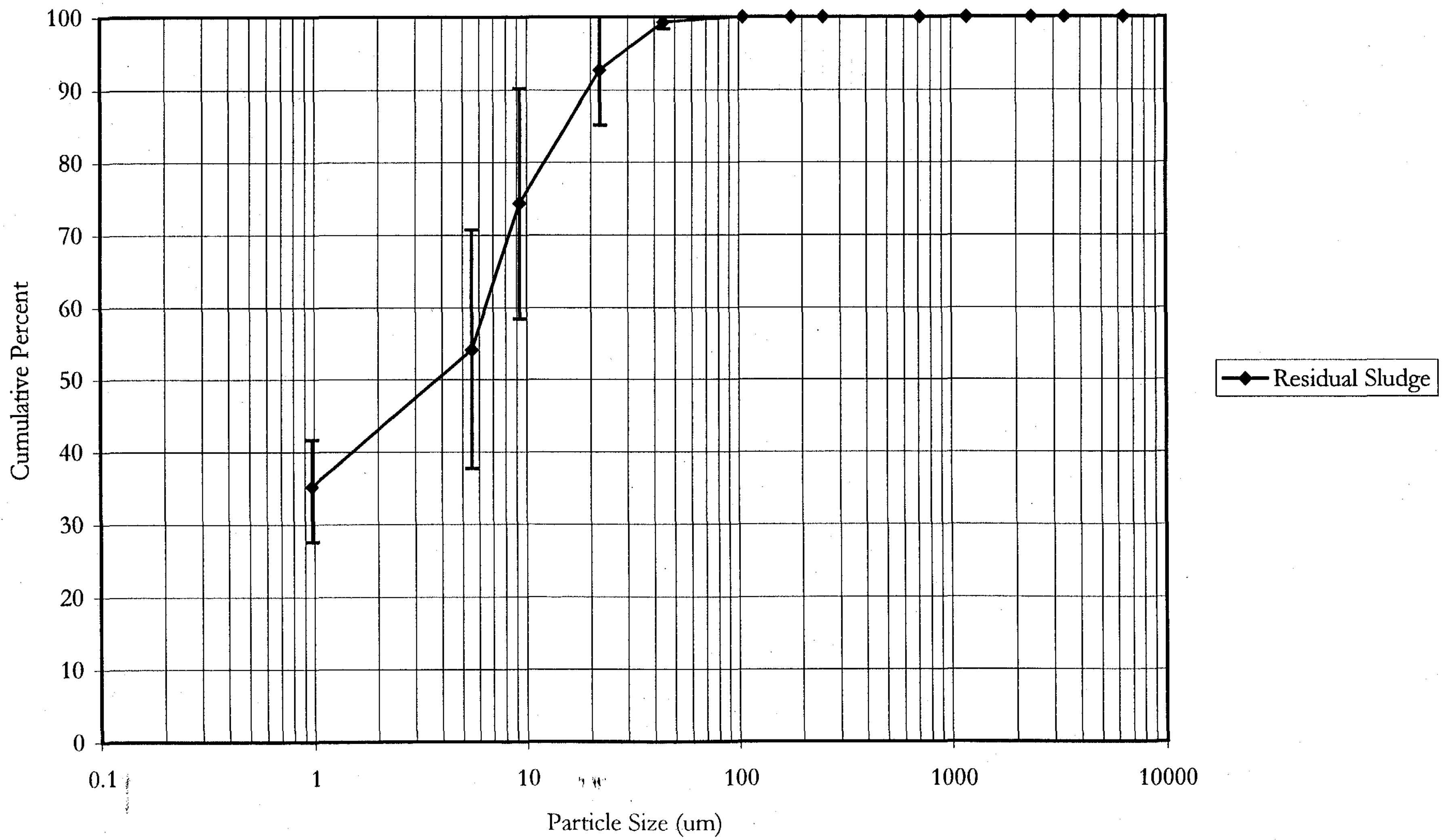
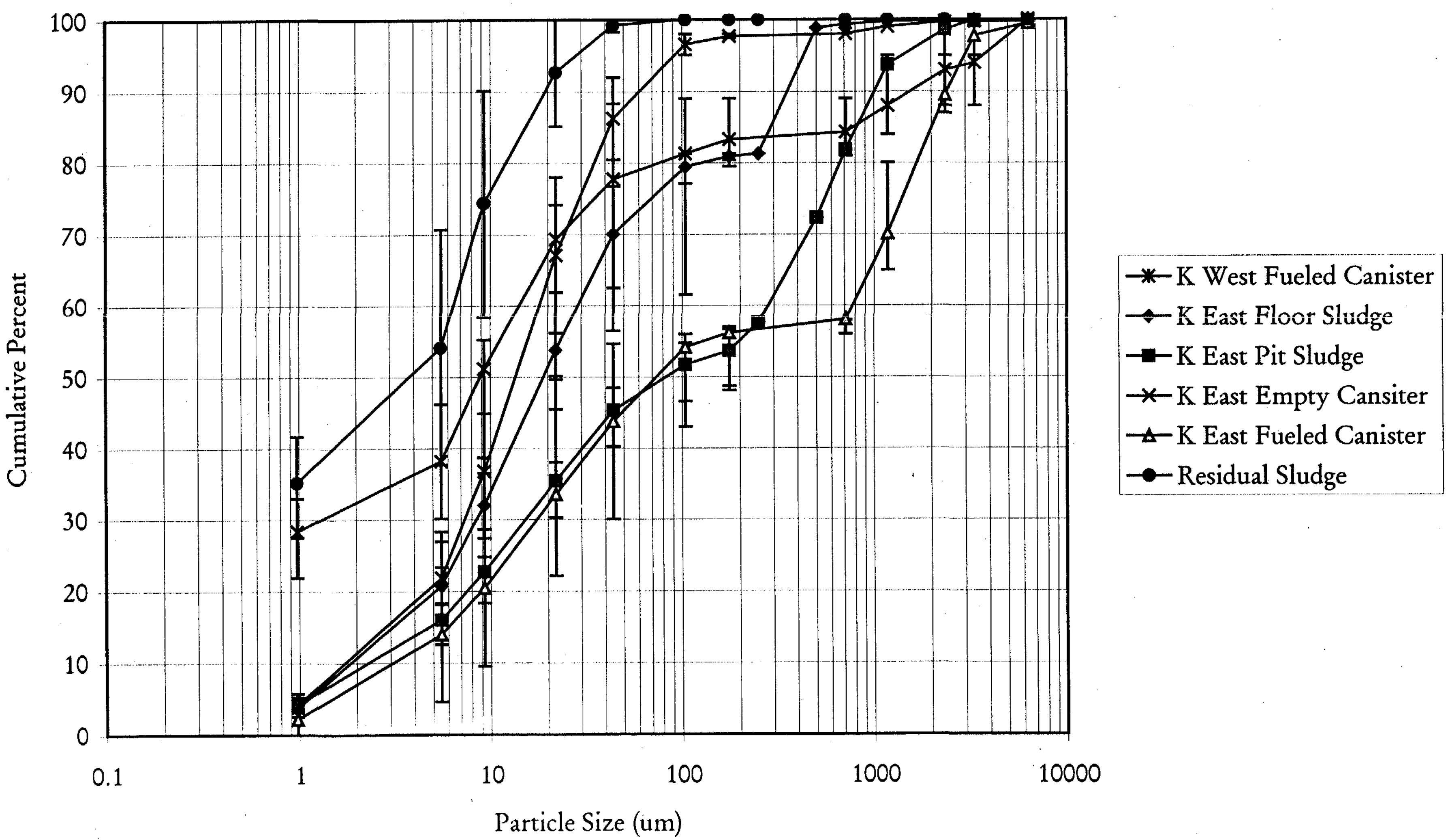


Figure 4.27. Comparison of Integrated Particle Size Distributions for Sludge Types After Sonication (Microtrac X100 and Sieving, 0.12 to 6350 um)



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Appendix

Supporting Particle Size Data from Microtrac X100 Analyses

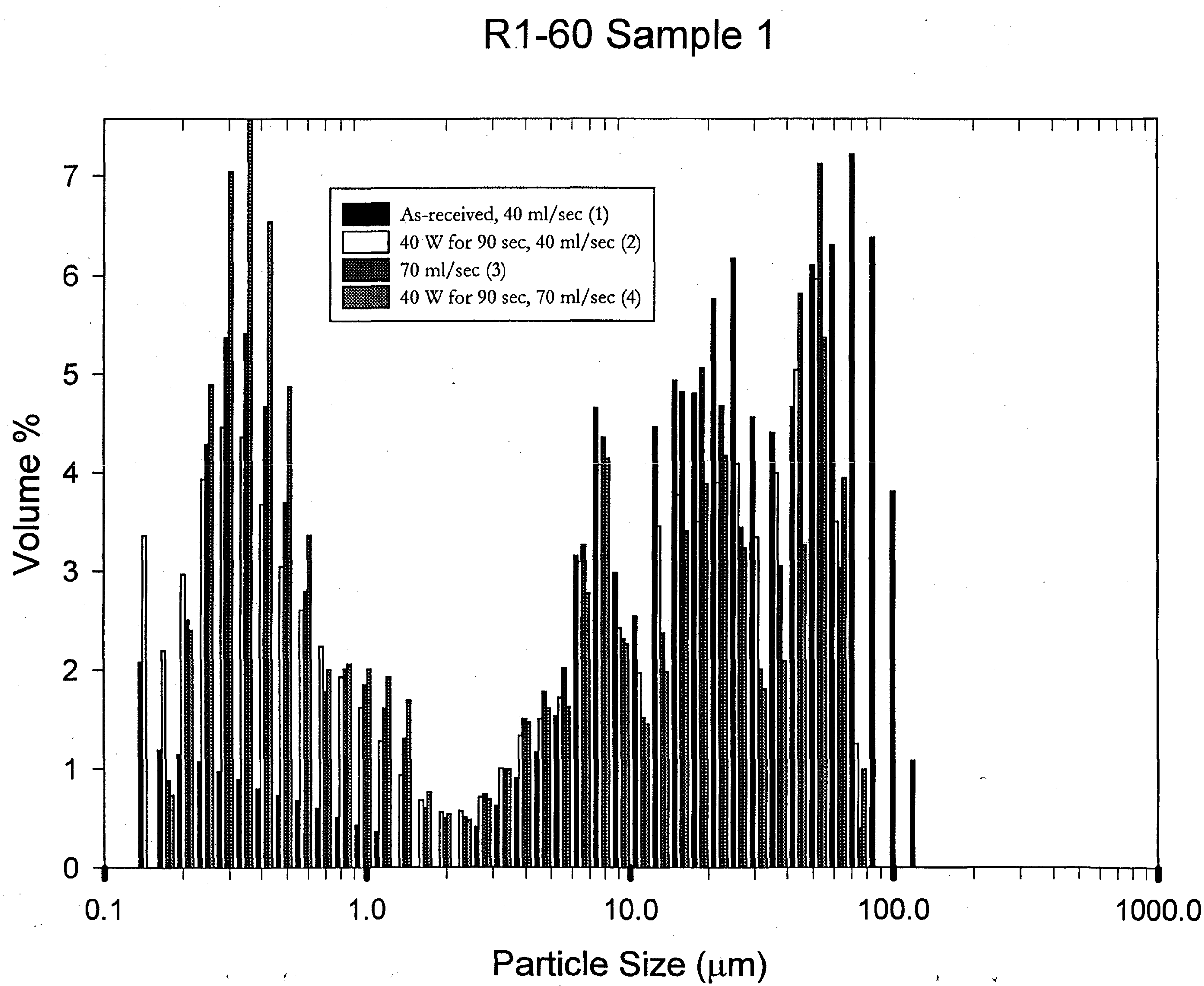
R1 60 Volume Distribution Data

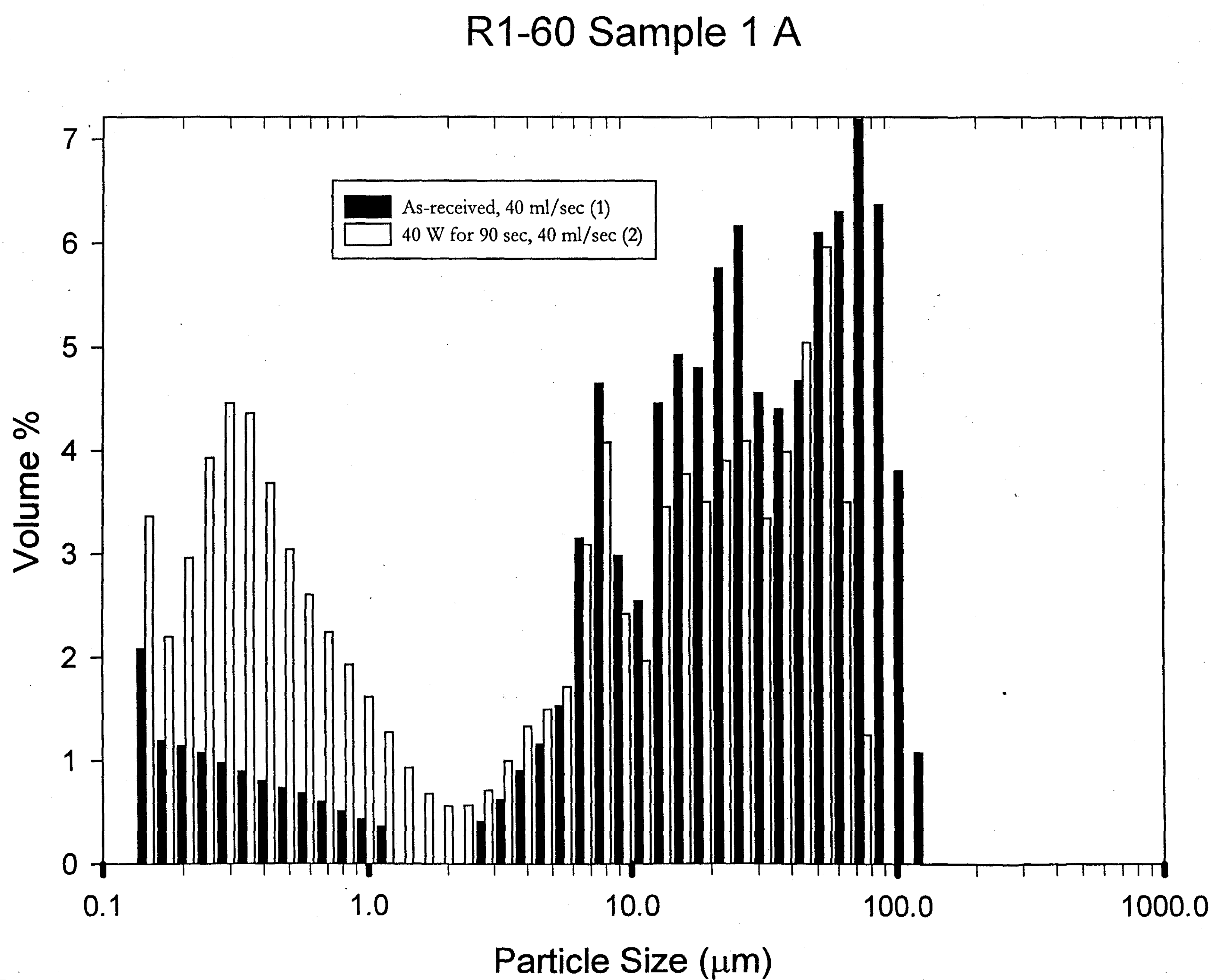
Sample analyzed As-Received 40 ml/sec (1), then sonicated at 40 W and analyzed at 40 ml/sec (2), then flow increased and analyzed at 70 ml/sec (3), then sonicated at 40 W and analyzed at 70 ml/sec (4).

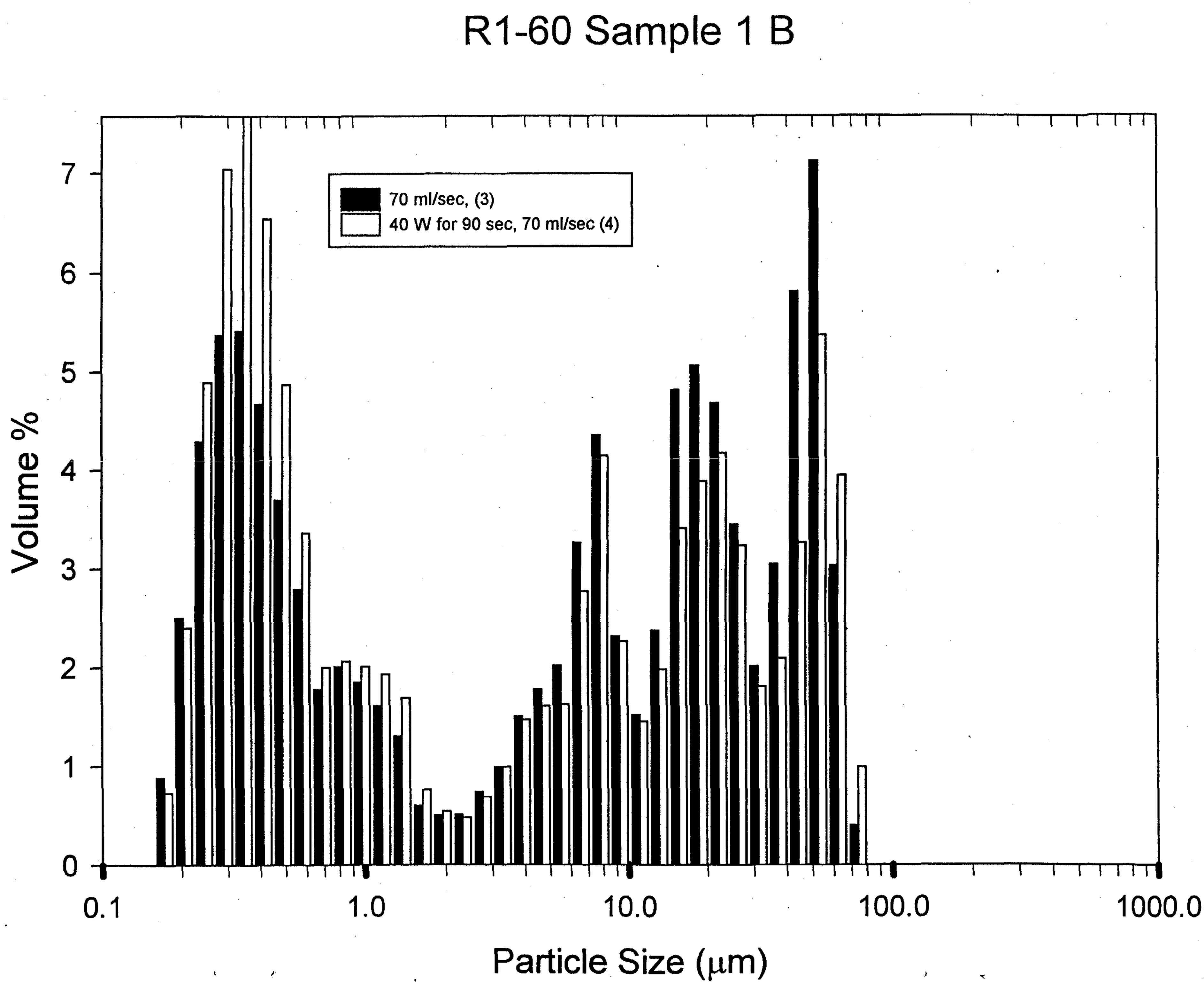
Duplicated several days later, As-Received 40 ml/sec (5), then sonicated at 40 W and analyzed at 40 ml/sec (6), then new sample analyzed at 70 ml/sec (7), then sonicated at 40 W and analyzed at 70 ml/sec (8).

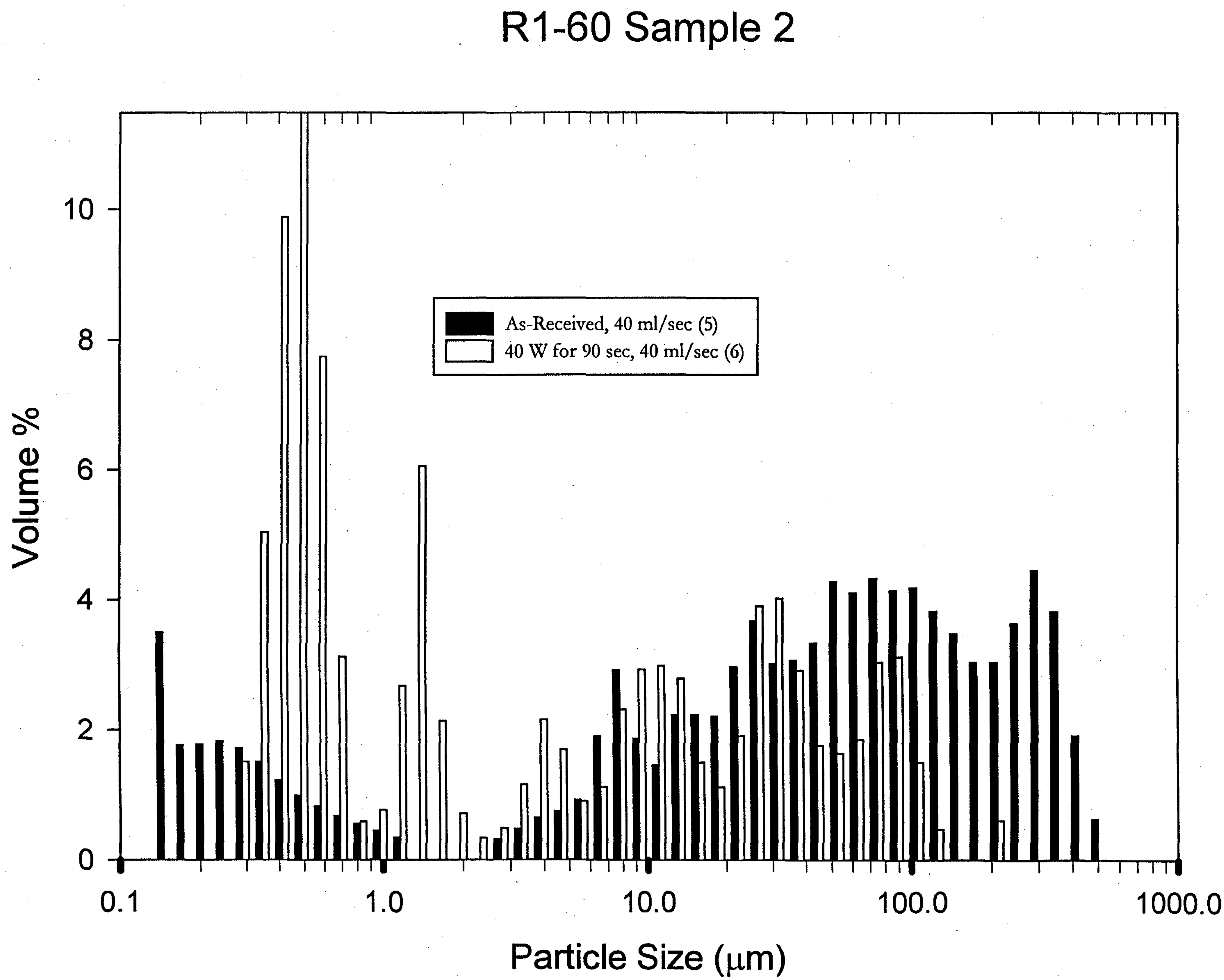
High flow rate required to prevent settling

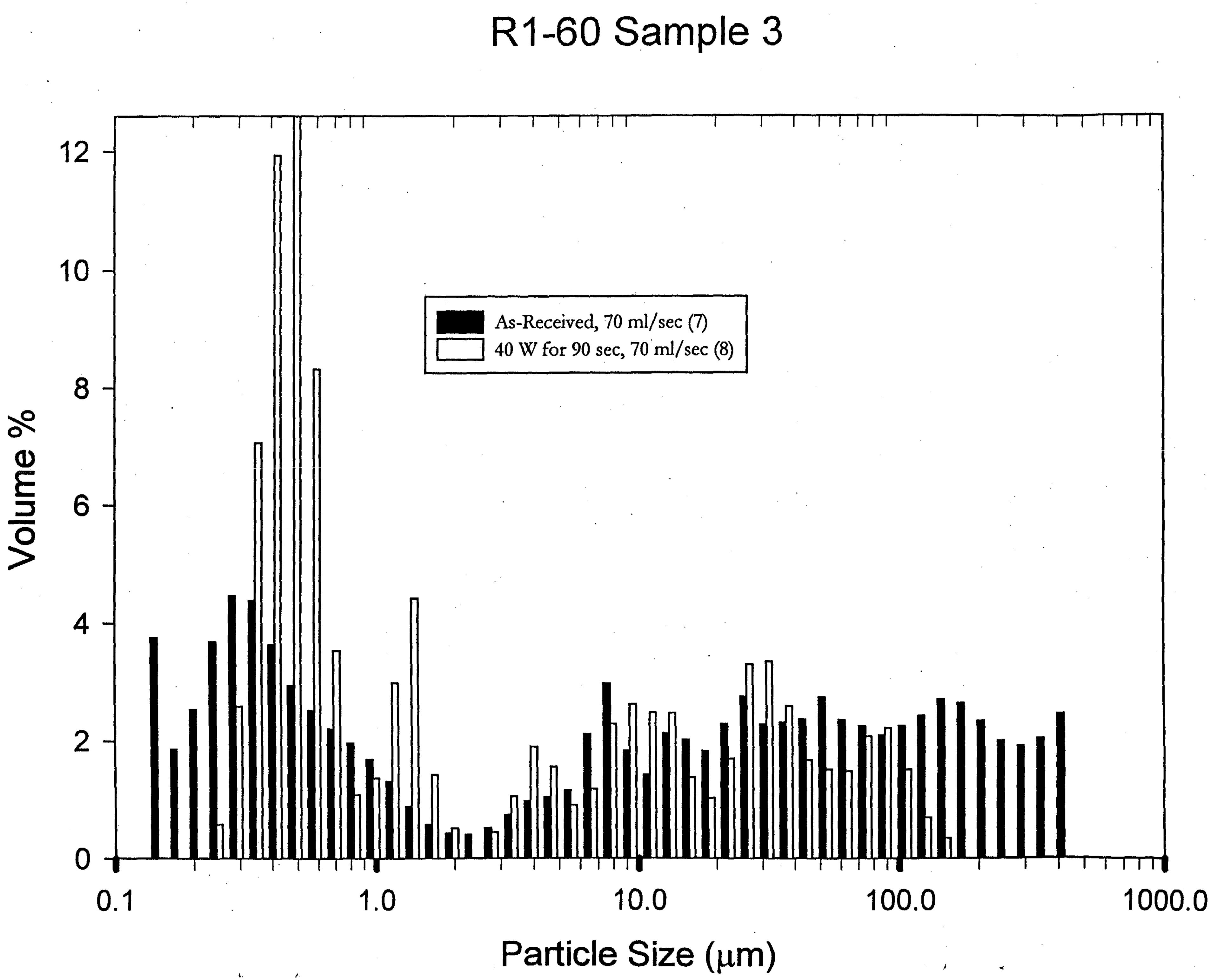
	1	2	3	4	5	6	7	8
	As-Received	40 W		40 W	As-Received	40 W	As-Receiv	40 W
Particle size	40 ml/sec	40 ml/sec	70 ml/sec	70 ml/sec	40 ml/sec	40 ml/sec	70 ml/sec	70 ml/sec
Particle size	1	2	3	4	5	6	7	8
704.000	0	0	0	0	0	0	0	0
592.000	0	0	0	0	0	0	0	0
497.800	0	0	0	0	0.63	0	0	0
418.600	0	0	0	0	1.91	0	2.46	0
352.000	0	0	0	0	3.81	0	2.04	0
296.000	0	0	0	0	4.45	0	1.91	0
248.900	0	0	0	0	3.63	0	2	0
209.300	0	0	0	0	3.03	0.61	2.33	0
176.000	0	0	0	0	3.03	0	2.64	0
148.000	0	0	0	0	3.47	0	2.7	0.34
124.500	1.08	0	0	0	3.82	0.47	2.42	0.69
104.700	3.8	0	0	0	4.18	1.5	2.25	1.51
88.000	6.37	0	0	0	4.14	3.12	2.09	2.21
74.000	7.21	1.25	0.4	0.99	4.32	3.04	2.25	2.07
62.230	6.3	3.5	3.03	3.94	4.1	1.85	2.35	1.48
52.330	6.1	5.96	7.12	5.37	4.27	1.64	2.74	1.51
44.000	4.67	5.04	5.81	3.26	3.33	1.76	2.36	1.67
37.000	4.4	3.99	3.04	2.09	3.08	2.92	2.3	2.59
31.110	4.56	3.34	2.01	1.81	3.02	4.02	2.27	3.34
26.160	6.17	4.09	3.44	3.23	3.67	3.9	2.75	3.29
22.000	5.76	3.9	4.68	4.17	2.97	1.91	2.28	1.69
18.500	4.8	3.5	5.06	3.88	2.21	1.12	1.82	1.02
15.560	4.93	3.77	4.81	3.4	2.24	1.5	2.01	1.37
13.080	4.46	3.45	2.37	1.98	2.23	2.79	2.12	2.47
11.000	2.54	1.97	1.52	1.45	1.46	2.99	1.42	2.48
9.250	2.98	2.42	2.31	2.26	1.87	2.93	1.83	2.62
7.778	4.65	4.08	4.35	4.14	2.92	2.32	2.97	2.28
6.541	3.15	3.09	3.26	2.77	1.9	1.12	2.11	1.17
5.500	1.53	1.72	2.02	1.63	0.92	0.9	1.15	0.9
4.625	1.16	1.5	1.78	1.61	0.75	1.7	1.04	1.55
3.889	0.9	1.33	1.5	1.47	0.65	2.16	0.97	1.89
3.270	0.62	1	0.99	0.99	0.48	1.16	0.74	1.05
2.750	0.41	0.71	0.74	0.69	0.32	0.49	0.52	0.44
2.312	0	0.57	0.51	0.48	0	0.34	0.41	0
1.945	0	0.56	0.5	0.54	0	0.71	0.43	0.51
1.635	0	0.68	0.6	0.76	0	2.14	0.57	1.42
1.375	0	0.93	1.3	1.69	0	6.06	0.88	4.41
1.156	0.36	1.27	1.61	1.93	0.35	2.69	1.3	2.99
0.972	0.43	1.62	1.85	2.01	0.45	0.77	1.68	1.36
0.818	0.51	1.93	2.01	2.06	0.56	0.59	1.96	1.08
0.688	0.6	2.24	1.78	2	0.68	3.13	2.2	3.53
0.578	0.68	2.6	2.79	3.36	0.83	7.75	2.52	8.32
0.486	0.73	3.04	3.69	4.87	0.99	11.47	2.94	12.59
0.409	0.8	3.68	4.67	6.54	1.22	9.88	3.62	11.93
0.344	0.89	4.36	5.41	7.57	1.51	5.04	4.37	7.06
0.289	0.97	4.46	5.37	7.04	1.72	1.51	4.45	2.59
0.243	1.07	3.93	4.29	4.89	1.83	0	3.68	0.58
0.204	1.14	2.96	2.5	2.4	1.78	0	2.54	0
0.172	1.19	2.2	0.88	0.73	1.77	0	1.86	0
0.145	2.08	3.36	0	0	3.5	0	3.75	0


R1-60 Sample 1
As-received, 40 ml/sec (1)
40 W for 90 sec, 40 ml/sec (2)
70 ml/sec (3)
40 W for 90 sec, 70 ml/sec (4)
Volume %
0
1
2
3
4
5
6
7
0.1
1.0
10.0
100.0
1000.0
Particle Size (µm)


R1-60 Sample 1 A
As-received, 40 ml/sec (1)
40 W for 90 sec, 40 ml/sec (2)
Volume %
7
6
5
4
3
2
1
0
0.1
1.0
10.0
100.0
1000.0
Particle Size (μm)


R1-60 Sample 1 B
70 ml/sec, (3)
40 W for 90 sec, 70 ml/sec (4)
Volume %
0
1
2
3
4
5
6
7
0.1
1.0
10.0
100.0
1000.0
Particle Size (μm)


R1-60 Sample 2
Volume %
0
2
4
6
8
10
0.1
1.0
10.0
100.0
1000.0
Particle Size (μm)
As-Received, 40 ml/sec (5)
40 W for 90 sec, 40 ml/sec (6)

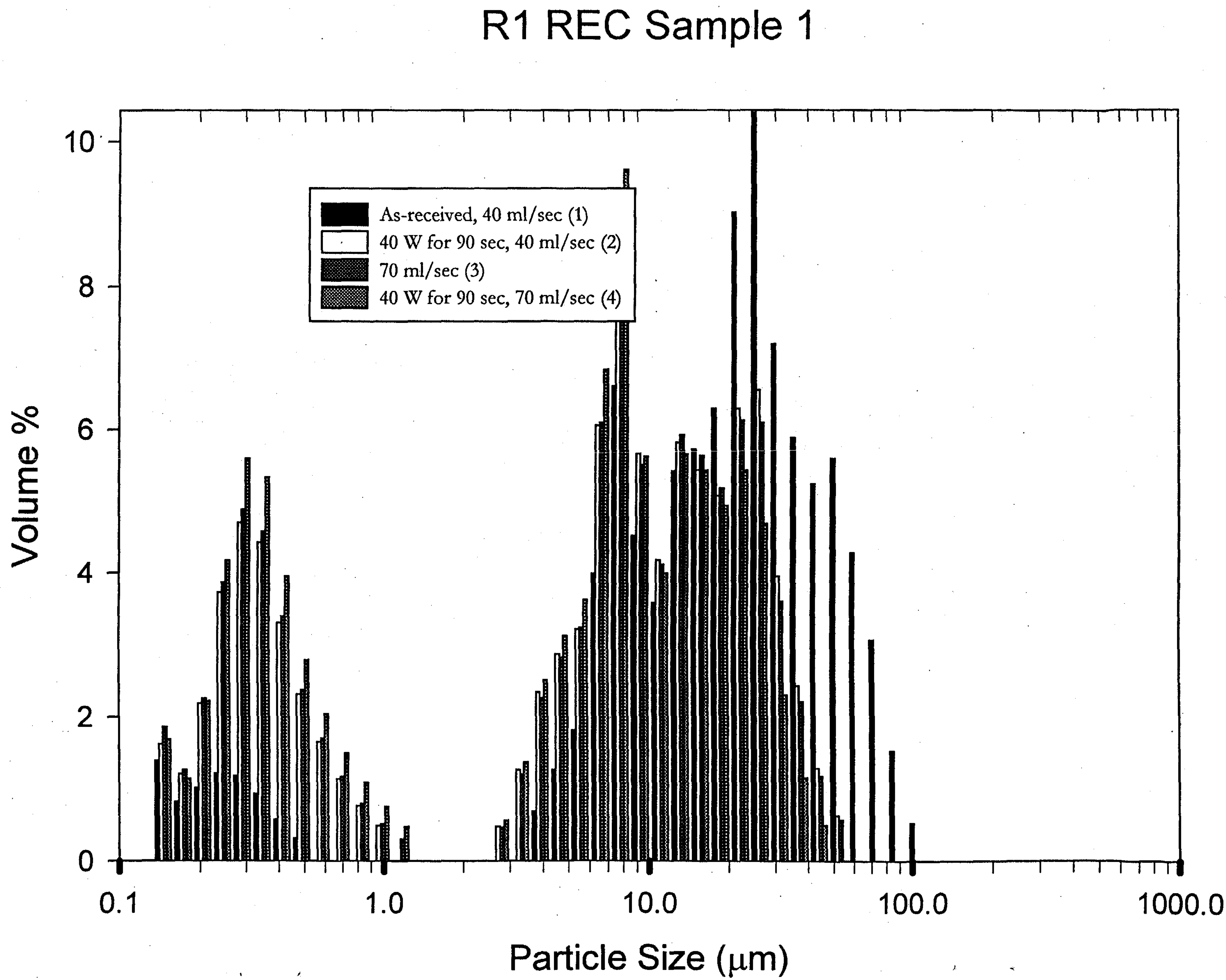

R1-60 Sample 3
Volume %
12
10
8
6
4
2
0
As-Received, 70 ml/sec (7)
40 W for 90 sec, 70 ml/sec (8)
0.1
1.0
10.0
100.0
1000.0
Particle Size (μm)

R1 REC Volume Distribution Data

Sample analyzed As-Received 40 ml/sec (1), then sonicated at 40 W and analyzed at 40 ml/sec (2), then flow increased and analyzed at 70 ml/sec (3), then sonicated at 40 W and analyzed at 70 ml/sec (4).

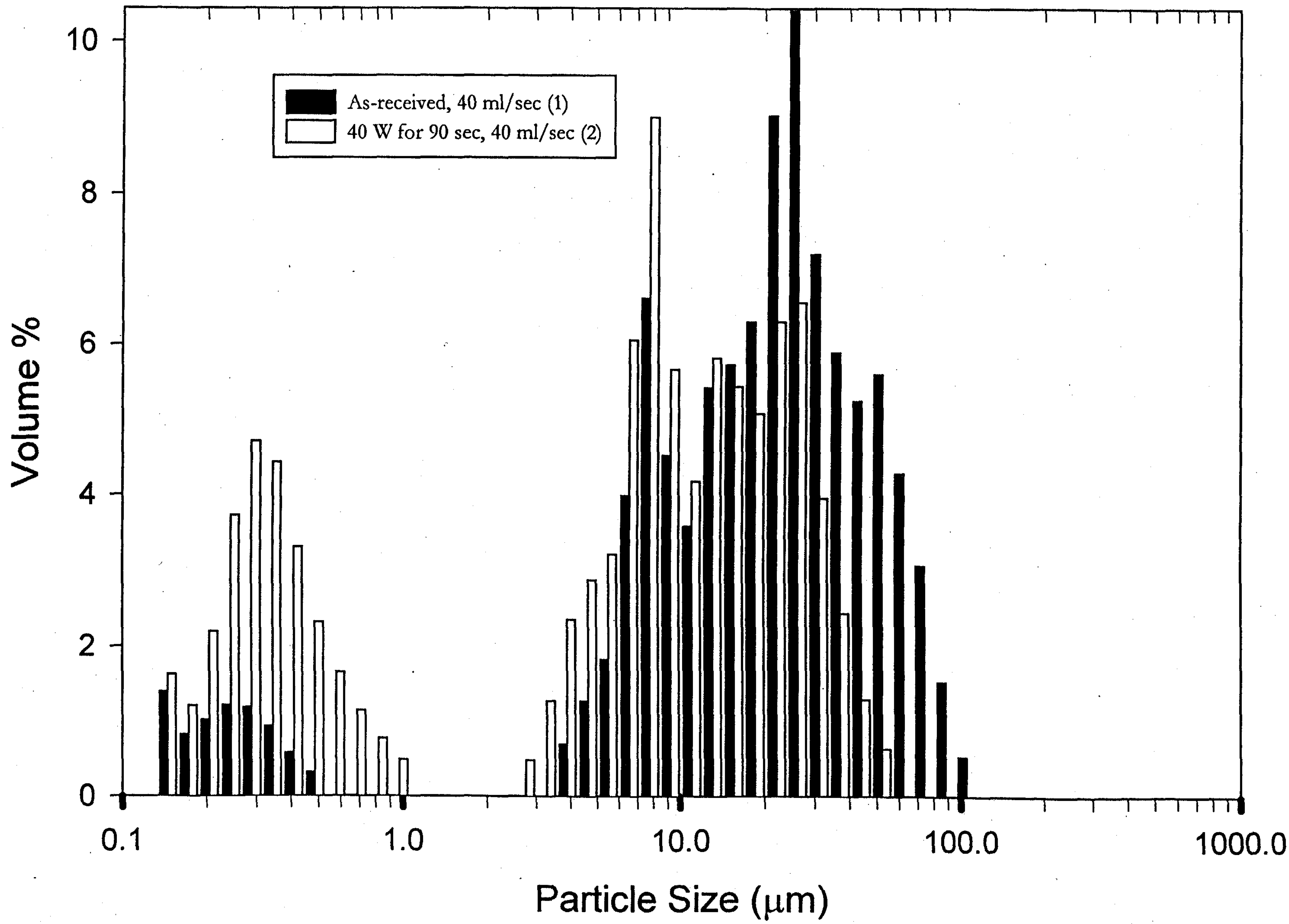
Duplicated several days later, As-Received 40 ml/sec (5), then sonicated at 40 W and analyzed at 40 ml/sec (6), then new sample analyzed at 70 ml/sec (7), then sonicated at 40 W and analyzed at 70 ml/sec (8).

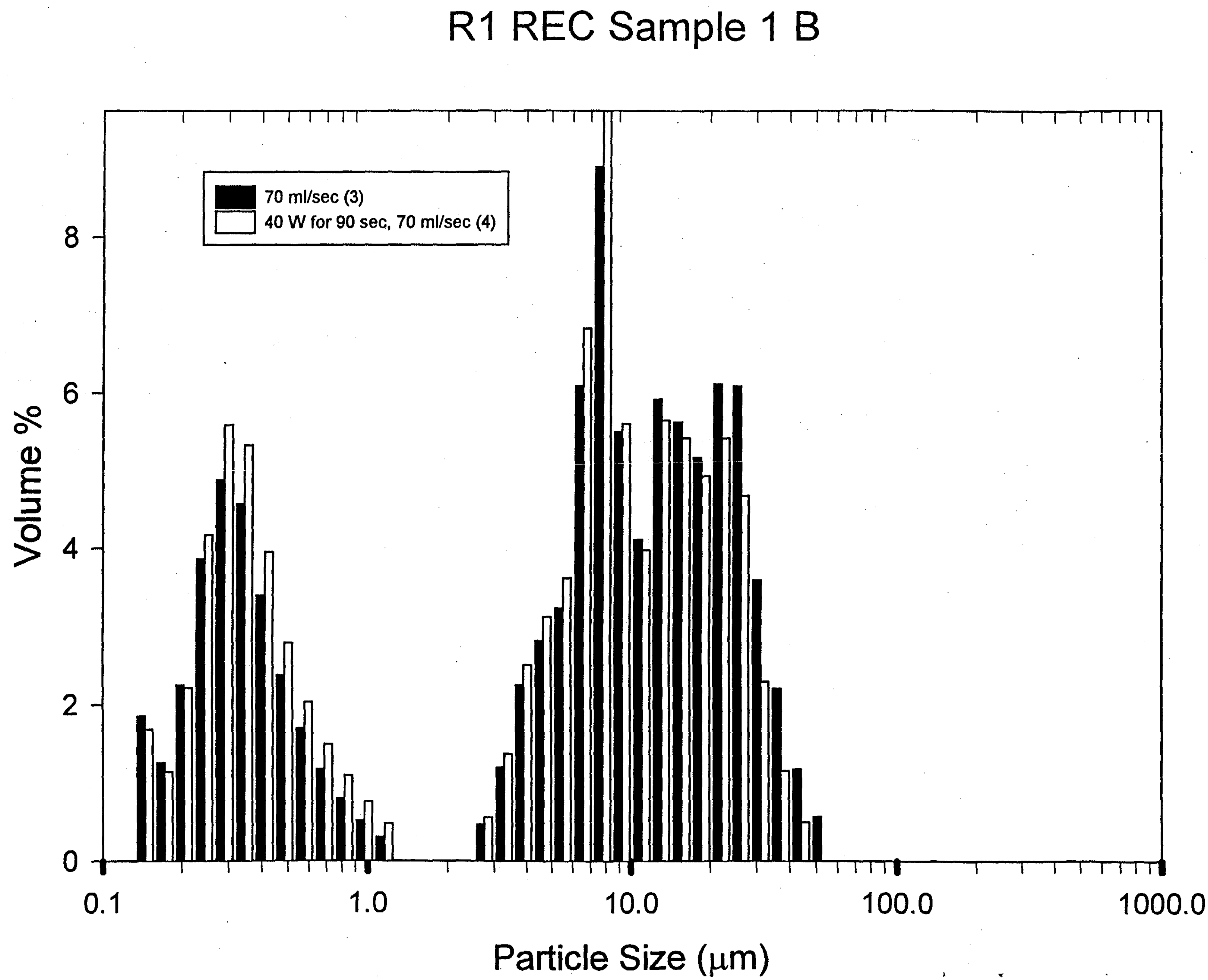
	1	2	3	4	5	6	7	8
	As-Received	40 W		40 W	As-Received	40 W	As-Receiv	40 W
Particle size	40 ml/sec	40 ml/sec	70 ml/sec	70 ml/sec	40 ml/sec	40 ml/sec	70 ml/sec	70 ml/sec
Particle size	1	2	3	4	5	6	7	8
704.000	0	0	0	0	0	0	0	0
592.000	0	0	0	0	0	0	0	0
497.800	0	0	0	0	0	0	0	0
418.600	0	0	0	0	0	0	0	0
352.000	0	0	0	0	0	0	0	0
296.000	0	0	0	0	0	0	0	0
248.900	0	0	0	0	0	0	0	0
209.300	0	0	0	0	0.66	0	0	0
176.000	0	0	0	0	0.72	0	0	0
148.000	0	0	0	0	1.01	0	0.64	0
124.500	0	0	0	0	1.28	0	0.52	0
104.700	0.53	0	0	0	1.58	0	0.56	0
88.000	1.52	0	0	0	2.04	0	0.72	0
74.000	3.06	0	0	0	3.2	0.33	1.21	0.31
62.230	4.27	0	0	0	4.43	0.48	2.03	0.42
52.330	5.59	0.63	0.57	0	5.68	0.88	3.4	0.7
44.000	5.23	1.28	1.17	0.5	5.03	1.28	3.8	0.98
37.000	5.88	2.43	2.21	1.15	5.86	2.1	4.64	1.57
31.110	7.19	3.94	3.59	2.3	8.06	3.32	5.99	2.54
26.160	10.43	6.55	6.09	4.68	11.61	5.69	9.18	4.55
22.000	9.02	6.29	6.12	5.42	7.72	5.79	8.4	4.93
18.500	6.29	5.07	5.17	4.93	4.02	4.91	6.17	4.44
15.560	5.72	5.43	5.63	5.42	4.03	5.27	5.99	4.96
13.080	5.41	5.81	5.92	5.65	6.04	5.37	6.06	5.17
11.000	3.57	4.17	4.11	3.98	5.49	3.6	4.12	3.51
9.250	4.51	5.66	5.5	5.61	4.93	4.79	4.96	4.76
7.778	6.6	9	8.89	9.61	3.5	7.92	6.75	8.04
6.541	3.98	6.05	6.09	6.83	1.5	5.64	4.07	5.78
5.500	1.81	3.21	3.23	3.62	1.18	3.02	2.19	3.1
4.625	1.26	2.87	2.82	3.12	1.68	2.63	1.96	2.68
3.889	0.69	2.35	2.26	2.51	0.72	2.21	1.29	2.18
3.270	0	1.26	1.2	1.37	0	1.36	0.38	1.29
2.750	0	0.48	0.47	0.56	0	0.67	0	0.6
2.312	0	0	0	0	0	0.35	0	0
1.945	0	0	0	0	0	0	0	0
1.635	0	0	0	0	0	0	0	0
1.375	0	0	0	0	0	0.32	0	0.37
1.156	0	0	0.31	0.48	0	0.45	0	0.61
0.972	0	0.49	0.52	0.76	0	0.6	0	0.91
0.818	0	0.77	0.8	1.09	0	0.79	0	1.24
0.688	0	1.14	1.17	1.49	0	1.04	0	1.62
0.578	0	1.65	1.7	2.04	0	1.44	0.43	2.13
0.486	0.32	2.32	2.38	2.79	0	2.04	0.94	2.81
0.409	0.58	3.3	3.39	3.95	0.82	3.02	1.78	3.86
0.344	0.93	4.42	4.57	5.33	2.18	4.27	2.81	5.14
0.289	1.18	4.7	4.88	5.59	2.83	4.94	3.25	5.6
0.243	1.21	3.72	3.86	4.17	1.7	4.51	2.62	4.74
0.204	1.01	2.19	2.26	2.22	0.5	3.24	1.48	3.11
0.172	0.82	1.2	1.26	1.14	0	2.22	0.75	2
0.145	1.39	1.62	1.86	1.69	0	3.51	0.91	3.35



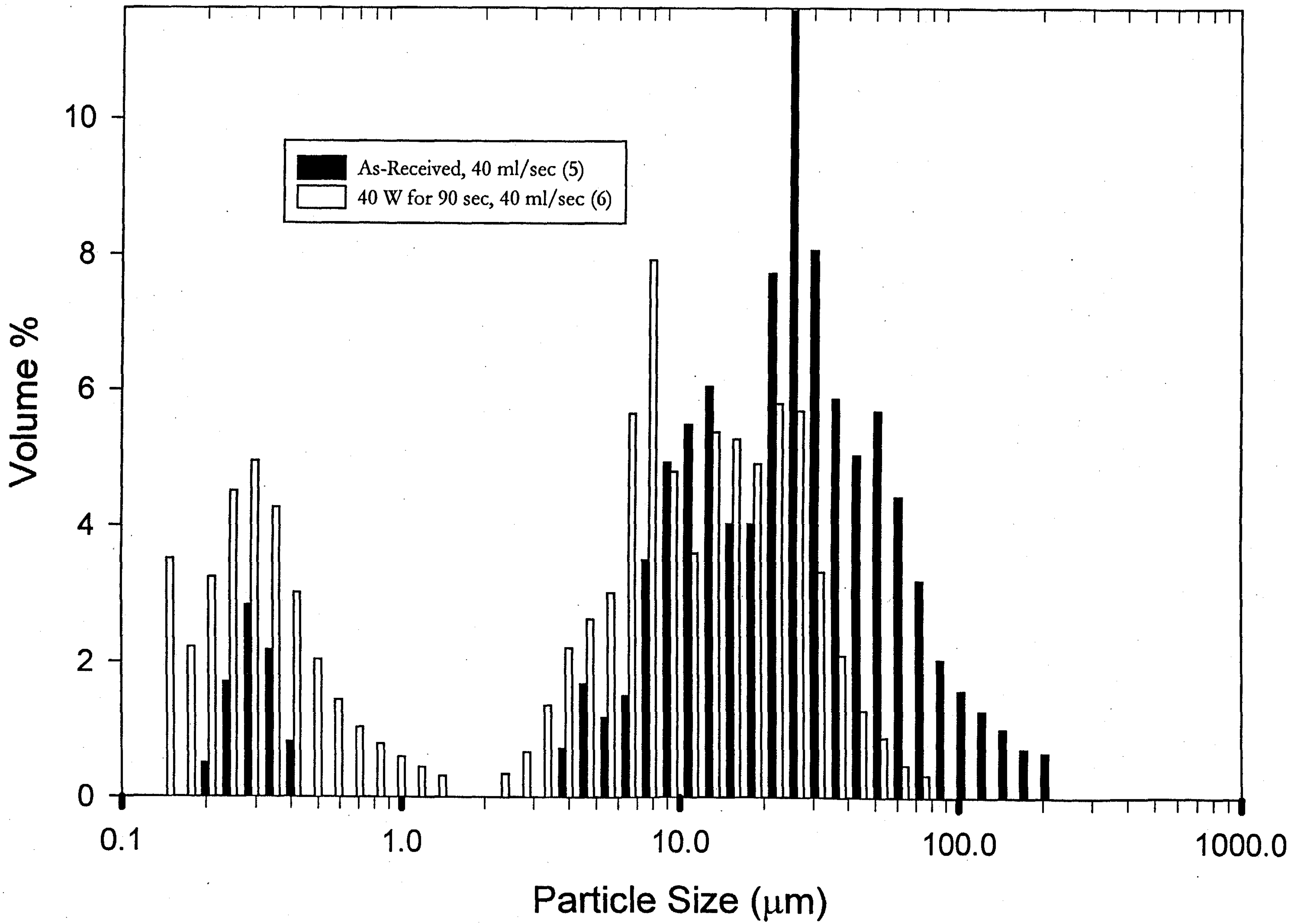
R1 REC Sample 1
As-received, 40 ml/sec (1)
40 W for 90 sec, 40 ml/sec (2)
70 ml/sec (3)
40 W for 90 sec, 70 ml/sec (4)
Volume %
0
2
4
6
8
10
0.1
1.0
10.0
100.0
1000.0
Particle Size (μm)

R1 REC Sample 1 A

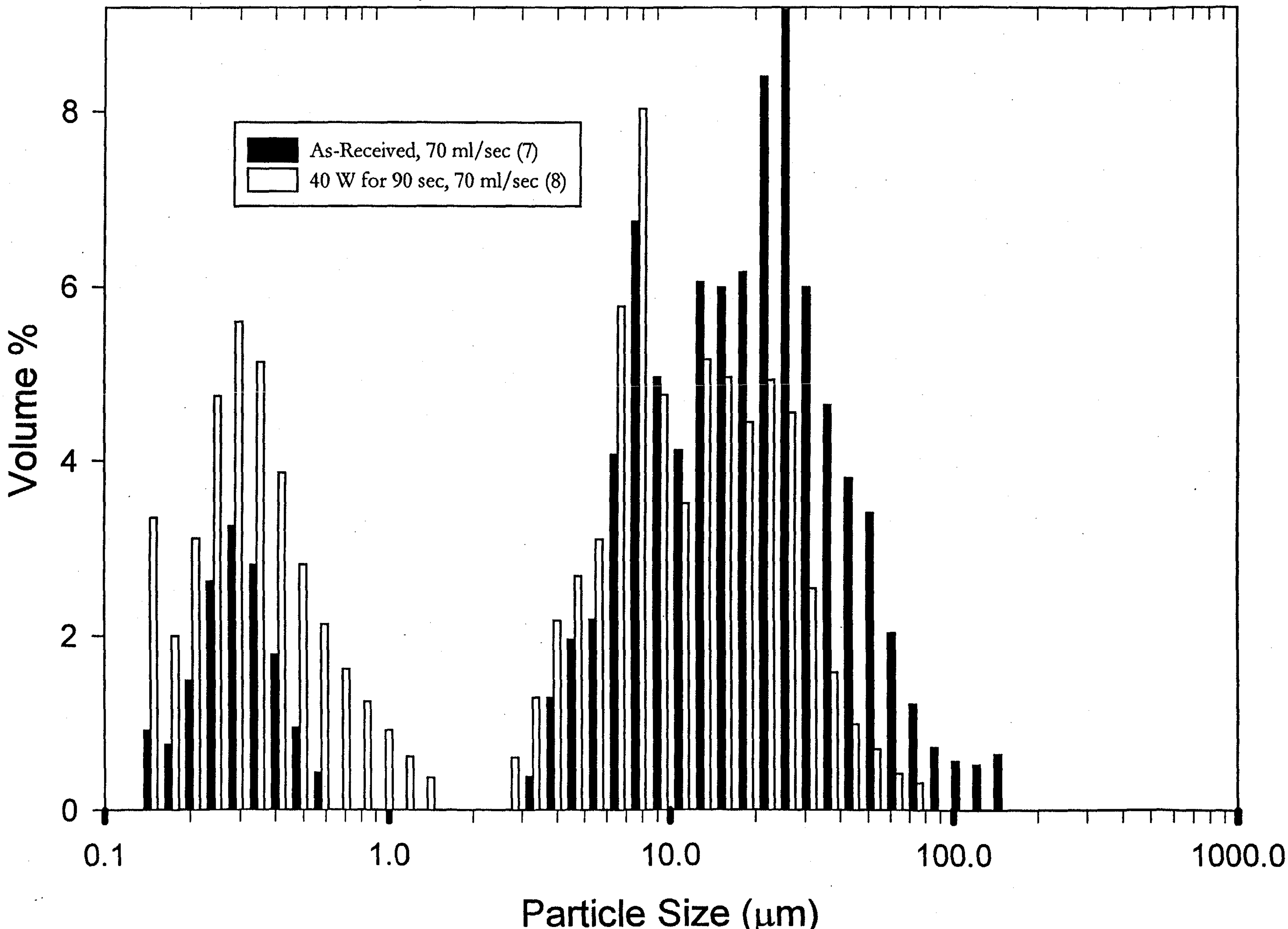



R1 REC Sample 1 B
70 ml/sec (3)
40 W for 90 sec, 70 ml/sec (4)
Volume %
0
2
4
6
8
0.1
1.0
10.0
100.0
1000.0
Particle Size (μm)

R1 REC Sample 2



R1 REC Sample 3



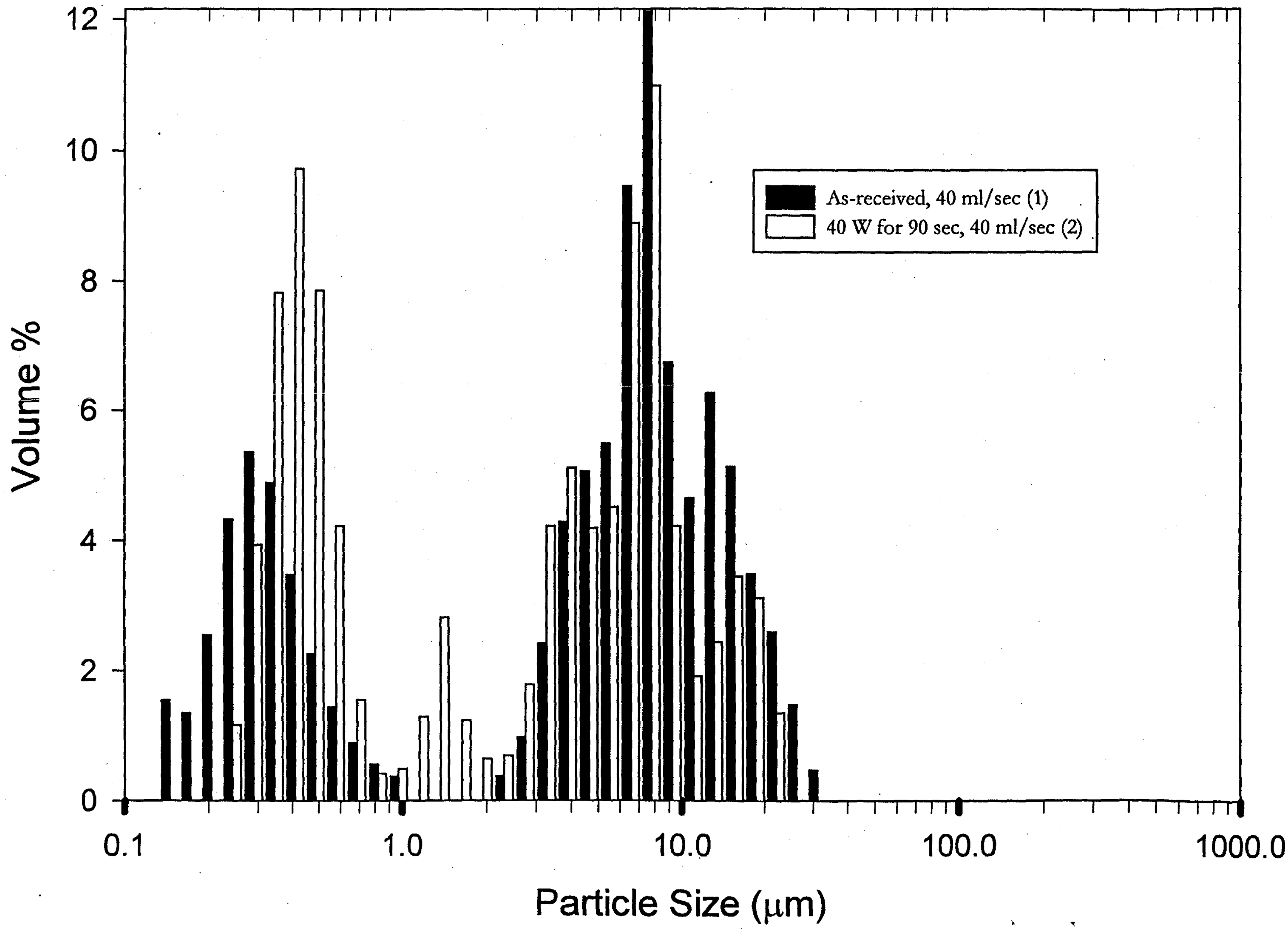
R5 REC Volume Distribution Data

Sample analyzed As-Received 40 ml/sec (1), then sonicated at 40 W and analyzed at 40 ml/sec (2), then flow increased possibly new sample and analyzed at 70 ml/sec (3), then sonicated at 40 W and analyzed at 70 ml/sec (4).

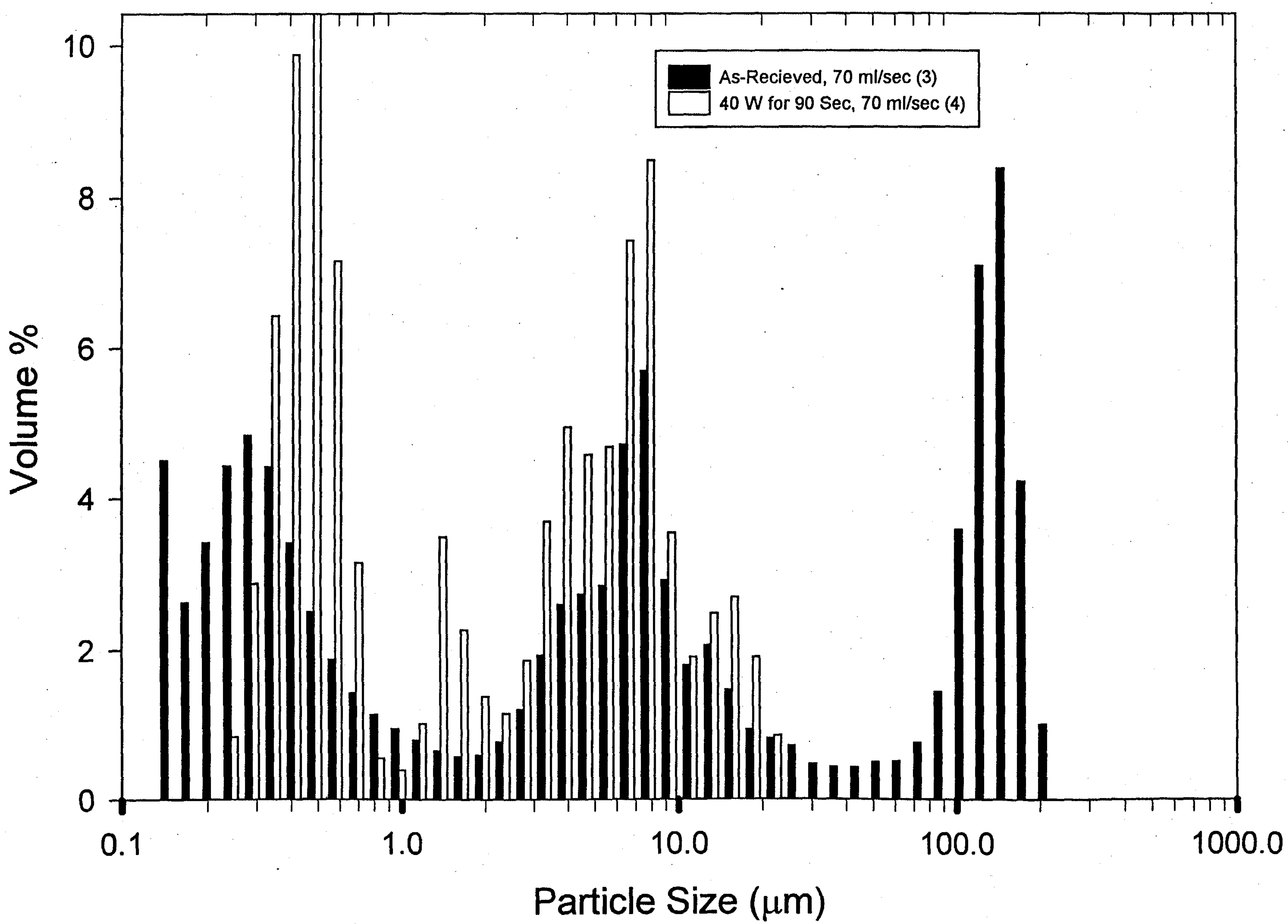
Duplicated several days later, As-Received 40 ml/sec (5), then sonicated at 40 W and analyzed at 40 ml/sec (6), then new sample introduced and analyzed at 70 ml/sec (7), then sonicated at 40 W and analyzed at 70 ml/sec (8).

	1	2	3	4	5	6	7	8
Particle size	As-Received 40 ml/sec	40 W 40 ml/sec	As-Received 70 ml/sec	40 W 70 ml/sec	As-Received 40 ml/sec	40 W 40 ml/sec	As-Receiv 70 ml/sec	40 W 70 ml/sec
704.000	0	0	0	0	0	0	0	0
592.000	0	0	0	0	0	0	0	0
497.800	0	0	0	0	0	0	0	0
418.600	0	0	0	0	0	0	0	0
352.000	0	0	0	0	0	0	0	0
296.000	0	0	0	0	0	0	0	0
248.900	0	0	0	0	0	0	0	0
209.300	0	0	1	0	0	0	0	0
176.000	0	0	4.22	0	0	0	0	0
148.000	0	0	8.38	0	0	0	0	0
124.500	0	0	7.09	0	0	0	0	0
104.700	0	0	3.58	0	0	0	0	0
88.000	0	0	1.44	0	0	0	0	0
74.000	0	0	0.76	0	0.41	0	0	0
62.230	0	0	0.51	0	0.41	0	0.31	0
52.330	0	0	0.5	0	0.53	0.4	0.41	0.53
44.000	0	0	0.42	0	0.59	0	0.45	0
37.000	0	0	0.43	0	0.84	0.36	0.62	0
31.110	0.47	0	0.47	0	1.29	0.64	0.94	0.42
26.160	1.47	0	0.71	0	2.43	1.59	1.76	1.13
22.000	2.59	1.34	0.81	0.85	2.97	2.32	2.23	1.85
18.500	3.48	3.12	0.93	1.9	3.2	2.38	2.59	2.07
15.560	5.13	3.45	1.46	2.69	4.31	2.73	3.8	2.39
13.080	6.26	2.44	2.06	2.48	5.19	3.07	4.89	2.48
11.000	4.65	1.91	1.79	1.9	3.87	3.15	3.78	2.28
9.250	6.74	4.22	2.91	3.54	5.57	5.74	5.5	4.27
7.778	12.15	10.98	5.69	8.49	9.96	10	9.9	8.78
6.541	9.46	8.89	4.71	7.43	7.75	6.43	7.75	6.61
5.500	5.49	4.51	2.84	4.68	4.51	3.93	4.57	3.98
4.625	5.06	4.19	2.73	4.58	4.18	4.91	4.29	4.56
3.889	4.28	5.12	2.6	4.94	3.67	5.56	3.81	5.14
3.270	2.42	4.22	1.93	3.69	2.3	3.36	2.43	3.58
2.750	0.97	1.78	1.2	1.86	1.1	1.37	1.2	1.71
2.312	0.37	0.69	0.77	1.15	0.53	0.73	0.6	1.01
1.945	0	0.64	0.59	1.38	0.32	0.94	0.38	1.24
1.635	0	1.23	0.57	2.27	0	1.65	0.34	2.08
1.375	0	2.82	0.65	3.49	0	2.49	0.39	3.32
1.156	0	1.29	0.79	1.01	0.37	0.77	0.51	1.03
0.972	0.37	0.49	0.94	0.39	0.49	0	0.67	0.4
0.818	0.56	0.42	1.13	0.55	0.66	0.31	0.9	0.49
0.688	0.89	1.55	1.42	3.14	0.95	1.51	1.25	2.56
0.578	1.44	4.22	1.87	7.16	1.42	4.1	1.79	6.18
0.486	2.26	7.85	2.5	10.42	2.11	7.51	2.55	9.61
0.409	3.48	9.72	3.4	9.88	3.19	9.23	3.67	9.78
0.344	4.89	7.82	4.41	6.43	4.52	7.55	5	6.65
0.289	5.36	3.93	4.83	2.87	5.24	3.99	5.65	3.01
0.243	4.33	1.16	4.43	0.83	4.86	1.28	5.13	0.86
0.204	2.55	0	3.41	0	3.57	0	3.7	0
0.172	1.34	0	2.62	0	2.53	0	2.51	0
0.145	1.54	0	4.5	0	4.16	0	3.73	0

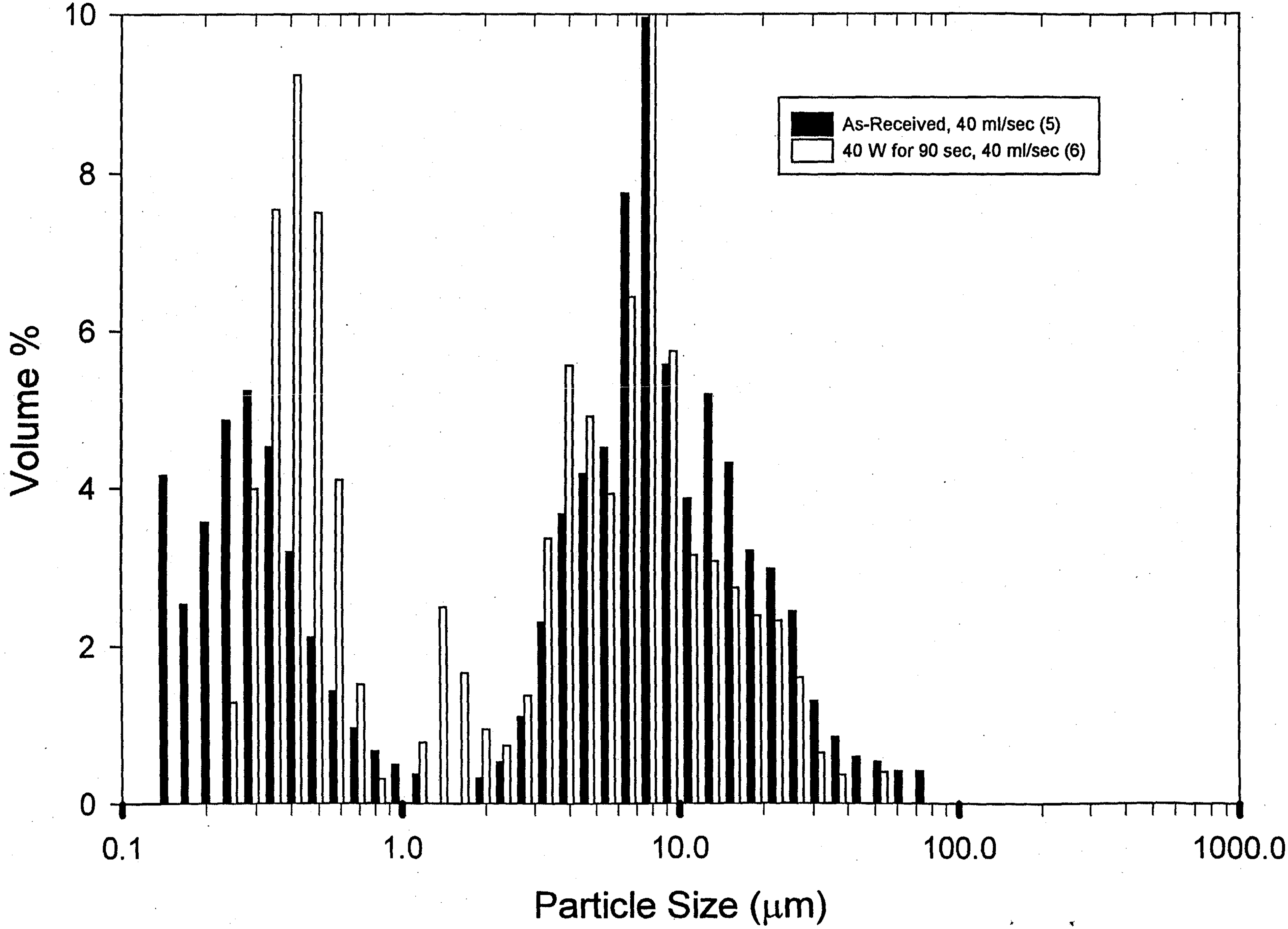
R5 REC Sample 1



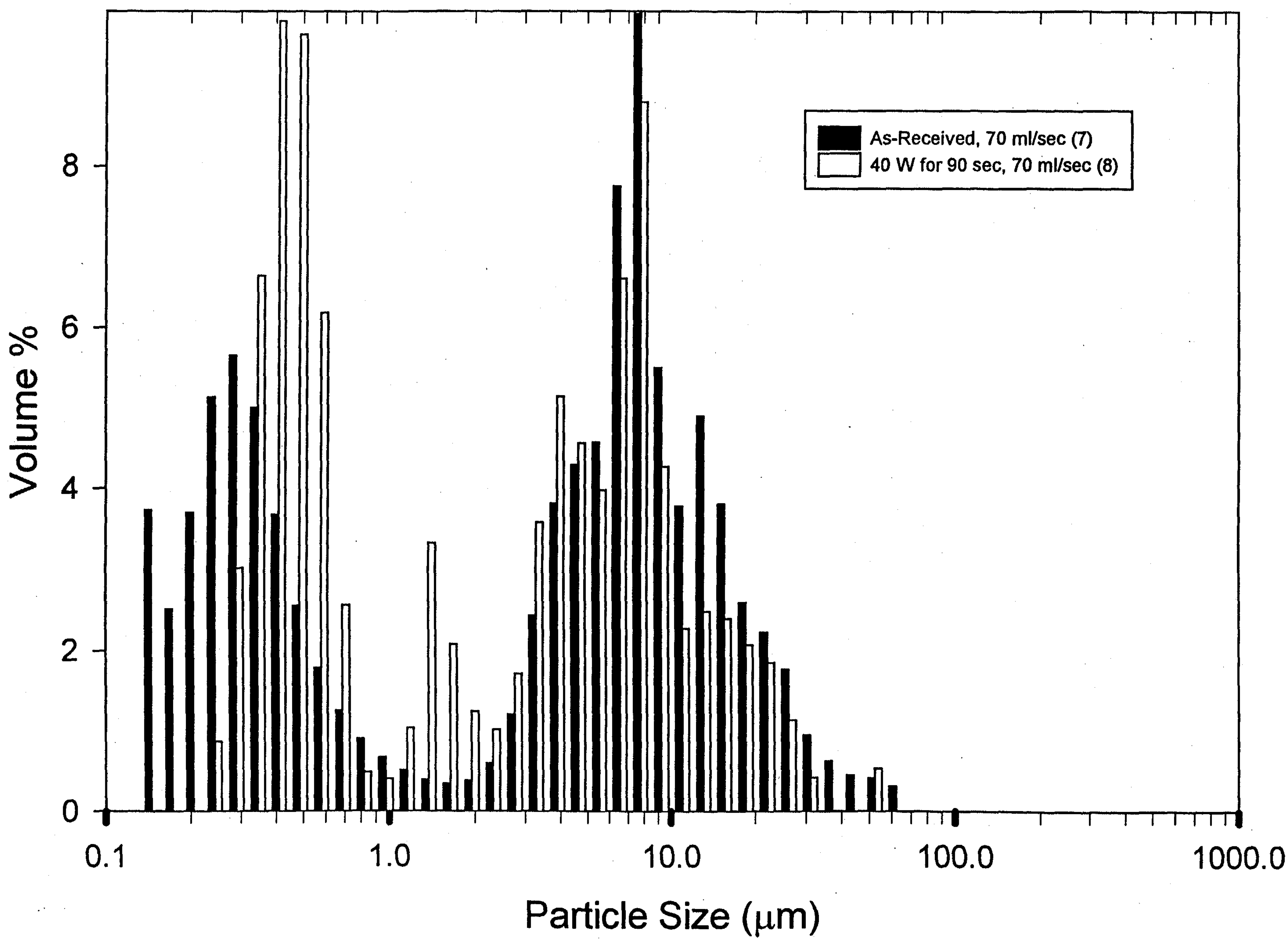
R5 REC Sample 2



R5 REC Sample 3



R5 REC Sample 4

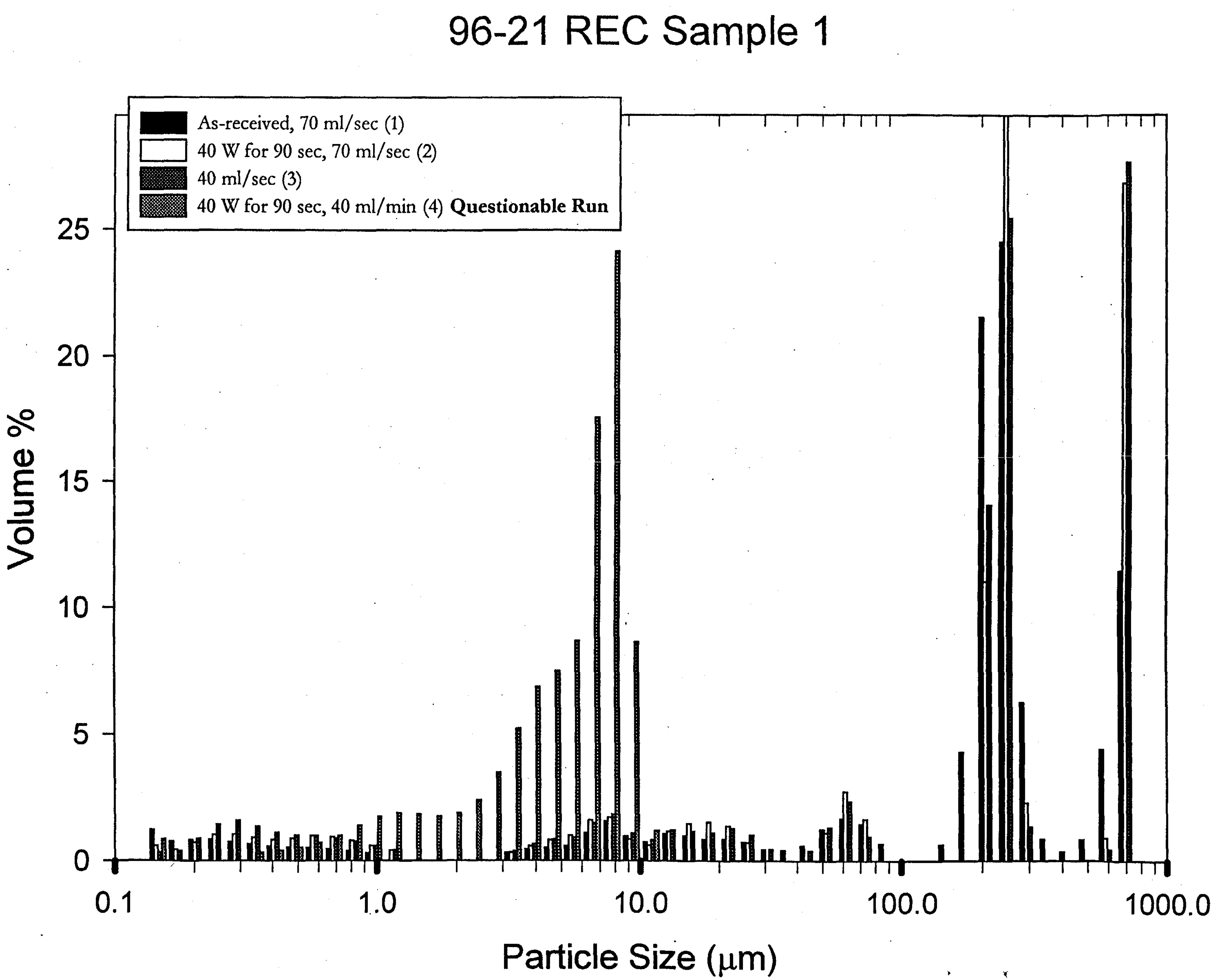


96-21 REC Volume Distribution Data

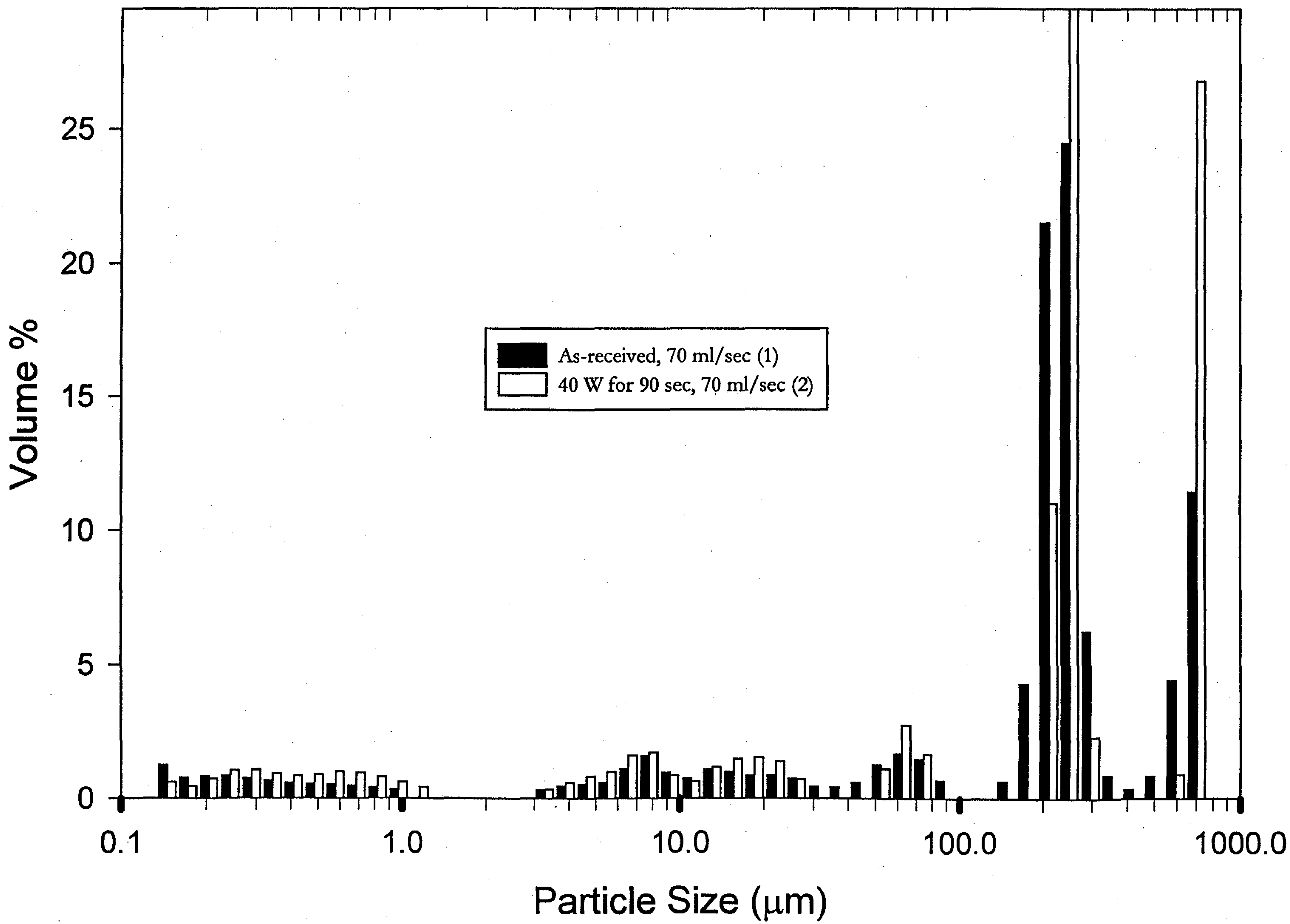
Sample tested at 70 ml/sec (1) then solicated and measured at 70 ml/sec (2) then slowed flow to 40 ml/sec (3) Then sonicated at 40W for 90 seconds and Measured at 40 ml/sec (4).

Duplicate tested at 70 ml/sec (5) then solicated and measured at 70 ml/sec (6) then slowed flow to 40 ml/sec (7) system perturbed possibly sample changed and analyzed at 40 ml/sec (8) Then sonicated at 40W for 90 seconds and Measured at 40 ml/sec (9).

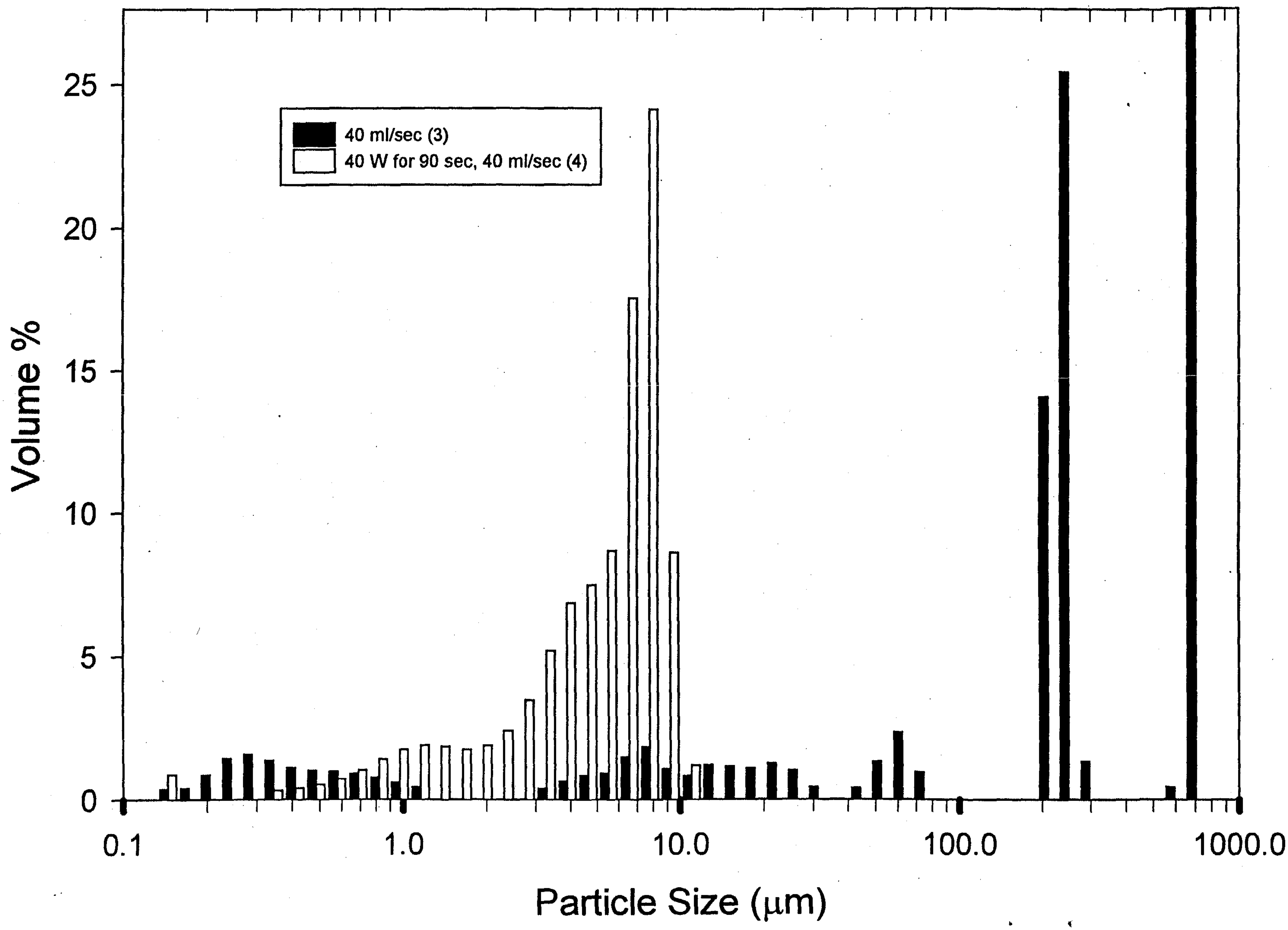
	1	2	3	4	5	6	7	8	9
	First				Duplicate				
	As-Received	40 W		40W	As-Received	40 W			40W
Particle size	70 ml/sec	70 ml/sec	40 ml/sec	40 ml/sec	70 ml/sec	70 ml/sec	40 ml/sec	40 ml/sec	40 ml/sec
704.000	11.43	26.77	27.61	0	0	0	0	0	1.5
592.000	4.39	0.91	0.44	0	0	0	0	0	1.04
497.800	0.83	0	0	0	0	0	0	0	0.43
418.600	0.35	0	0	0	0	0	0	0	0
352.000	0.85	0	0	0	0	0	0	0	0
296.000	6.23	2.29	1.34	0	0	0	0	0	0.64
248.900	24.48	29.46	25.41	0	0	0	0	0.35	2.5
209.300	21.5	11.01	14.04	0	0	0	0	3.03	7.18
176.000	4.27	0	0	0	0	0	0	10.32	10.06
148.000	0.64	0	0	0	0	0	0	11.86	6.76
124.500	0	0	0	0	0	0	0	5.7	2.8
104.700	0	0	0	0	0	0	0	2.36	1.28
88.000	0.68	0	0	0	0	0	0	1.33	0.8
74.000	1.46	1.65	0.97	0	0.36	0	0	1.4	0.84
62.230	1.66	2.73	2.34	0	1.32	0	0	1.92	1.07
52.330	1.25	1.11	1.33	0	3.2	0.75	0.67	2.82	1.52
44.000	0.59	0	0.4	0	4.1	1.85	1.72	2.51	1.49
37.000	0.41	0	0	0	4.74	3.36	3.24	2.2	1.57
31.110	0.44	0	0.44	0	5.01	4.55	4.51	2.05	1.77
26.160	0.74	0.72	1.02	0	6.34	6.17	6.17	2.75	2.62
22.000	0.86	1.37	1.27	0	5.48	5.14	5.14	2.76	2.68
18.500	0.83	1.52	1.1	0	4.47	3.98	3.95	2.51	2.42
15.560	0.98	1.47	1.16	0	4.93	4.31	4.28	2.75	2.66
13.080	1.06	1.16	1.21	0	5.17	4.63	4.61	2.66	2.6
11.000	0.73	0.63	0.83	1.19	3.41	3.2	3.19	1.64	1.62
9.250	0.96	0.85	1.08	8.62	4.29	4.15	4.15	2.09	2.08
7.778	1.57	1.71	1.84	24.13	6.7	6.51	6.52	3.49	3.54
6.541	1.09	1.6	1.48	17.54	4.64	4.45	4.48	2.56	2.63
5.500	0.58	1	0.92	8.67	2.54	2.42	2.45	1.39	1.42
4.625	0.5	0.82	0.84	7.48	2.36	2.26	2.3	1.2	1.21
3.889	0.44	0.58	0.65	6.85	2.18	2.09	2.13	1.02	1.02
3.270	0.32	0.33	0.38	5.2	1.53	1.42	1.44	0.67	0.71
2.750	0	0	0	3.48	0.88	0.76	0.78	0.38	0.43
2.312	0	0	0	2.4	0.53	0.43	0.44	0	0
1.945	0	0	0	1.9	0.4	0.32	0.33	0	0
1.635	0	0	0	1.77	0.4	0.35	0.37	0	0
1.375	0	0	0	1.85	0.5	0.51	0.53	0	0.31
1.156	0	0.42	0.43	1.9	0.64	0.77	0.8	0.38	0.43
0.972	0.32	0.6	0.6	1.75	0.78	1.08	1.12	0.56	0.6
0.818	0.39	0.8	0.77	1.41	0.91	1.39	1.42	0.78	0.82
0.688	0.46	0.96	0.91	1.02	1.05	1.71	1.74	1.04	1.09
0.578	0.5	0.99	0.99	0.72	1.26	2.17	2.19	1.37	1.44
0.486	0.52	0.89	1.01	0.52	1.57	2.84	2.86	1.78	1.87
0.409	0.57	0.84	1.12	0.4	2.09	3.9	3.95	2.37	2.5
0.344	0.65	0.93	1.38	0.33	2.75	5.13	5.21	3.04	3.24
0.289	0.76	1.06	1.6	0	3.09	5.37	5.48	3.29	3.63
0.243	0.85	1.04	1.46	0	2.89	4.3	4.37	2.93	3.48
0.204	0.84	0.74	0.88	0	2.25	2.7	2.72	2.17	2.86
0.172	0.77	0.44	0.4	0	1.81	1.78	1.73	1.64	2.39
0.145	1.25	0.6	0.35	0.87	3.43	3.25	3.01	2.93	4.45


96-21 REC Sample 1
As-received, 70 ml/sec (1)
40 W for 90 sec, 70 ml/sec (2)
40 ml/sec (3)
40 W for 90 sec, 40 ml/min (4) Questionable Run
Volume %
25
20
15
10
5
0
0.1
1.0
10.0
100.0
1000.0
Particle Size (μm)

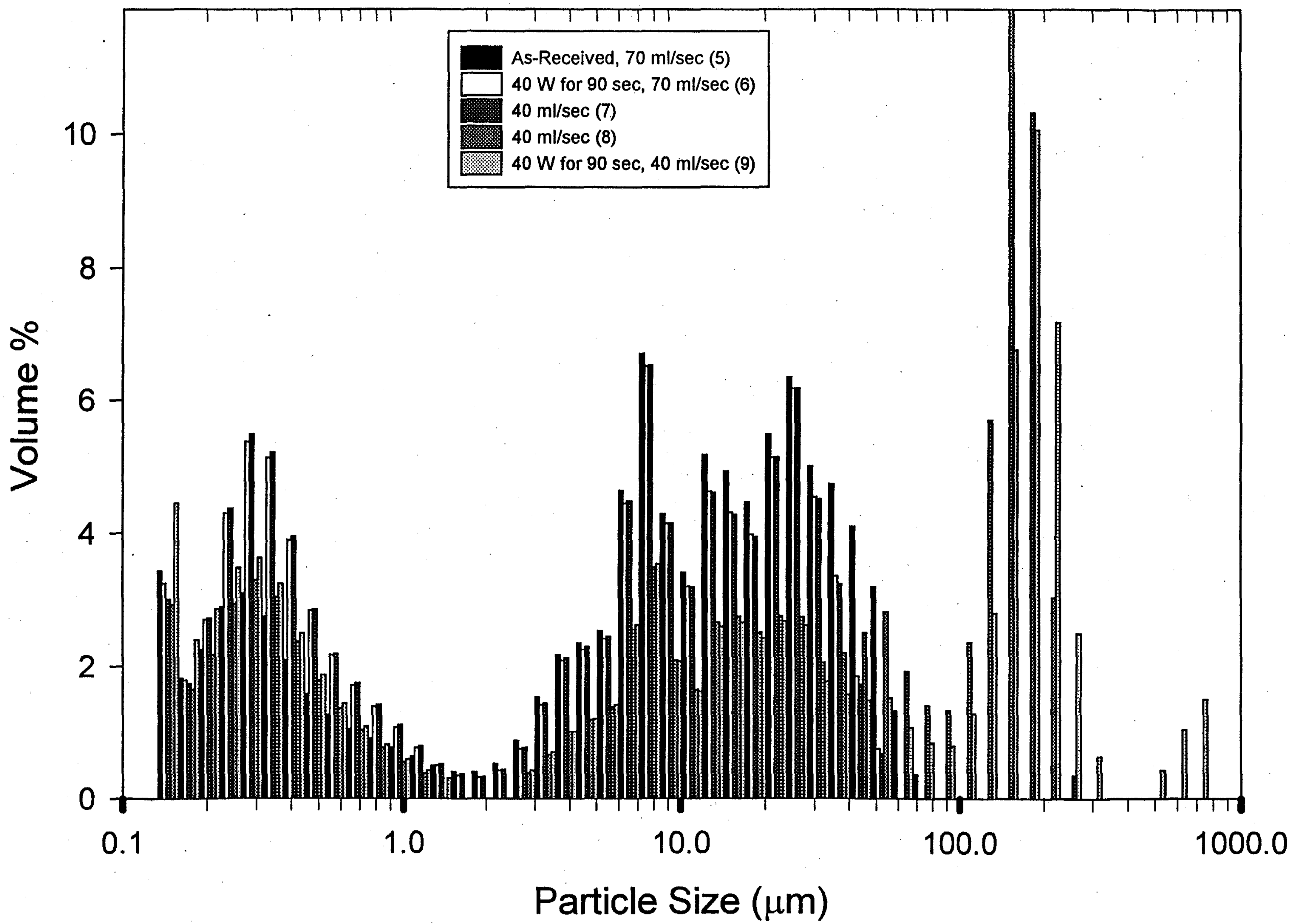
96-21 REC Sample 1 A



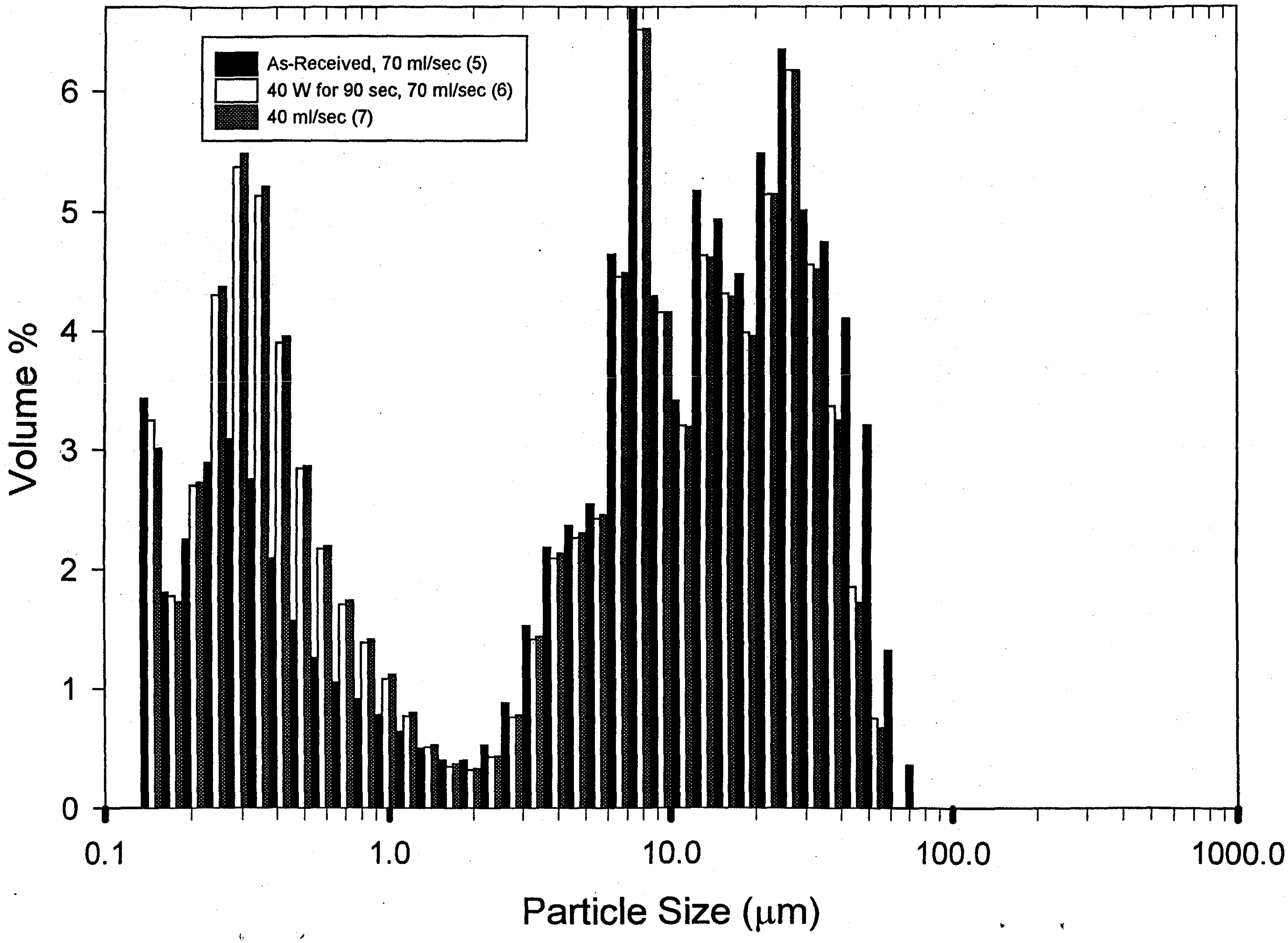
96-21 REC Sample 1 B



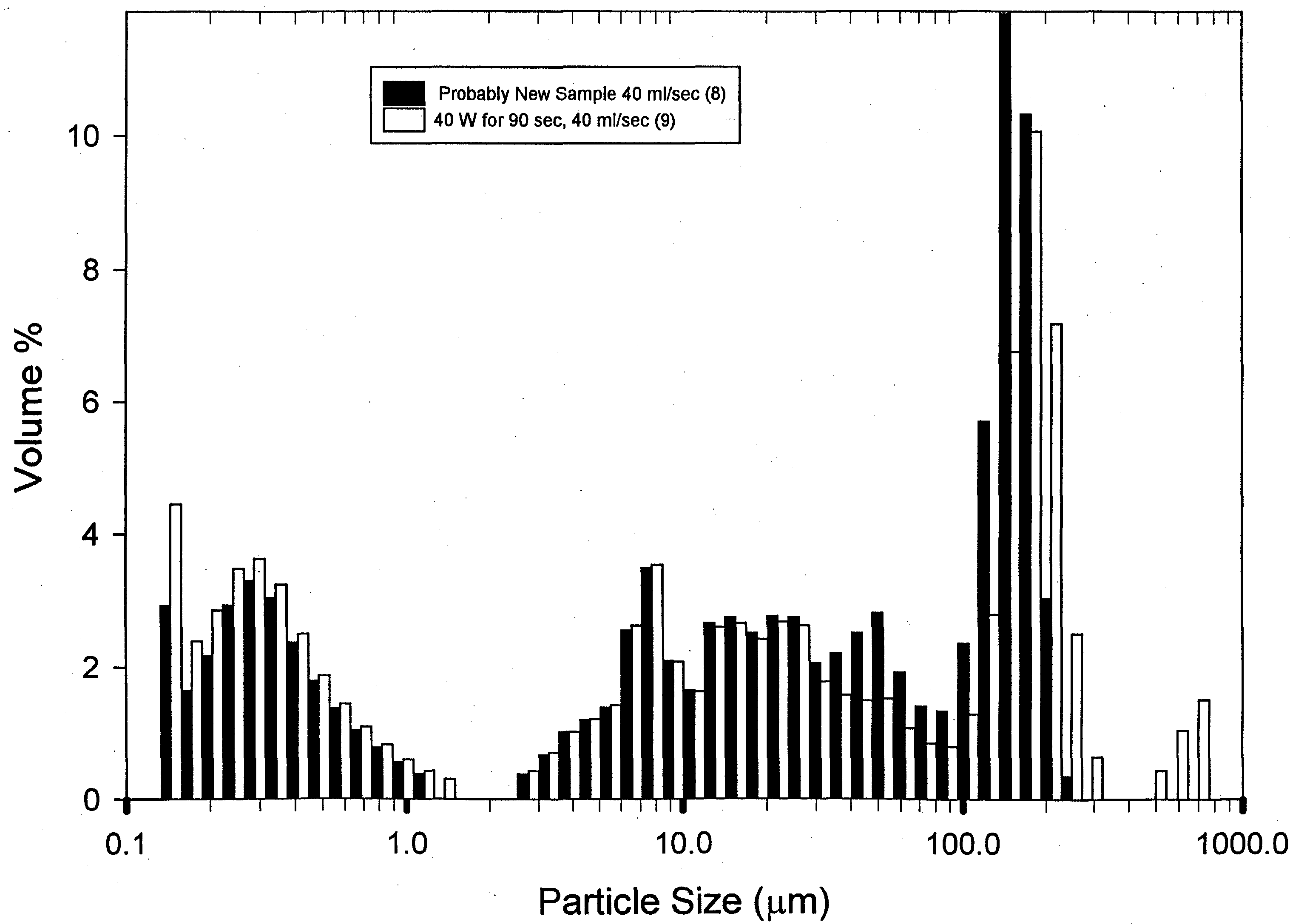
96-21 REC Sample 2



96-21 REC Sample 2 A



96-21 REC Sample 2 B



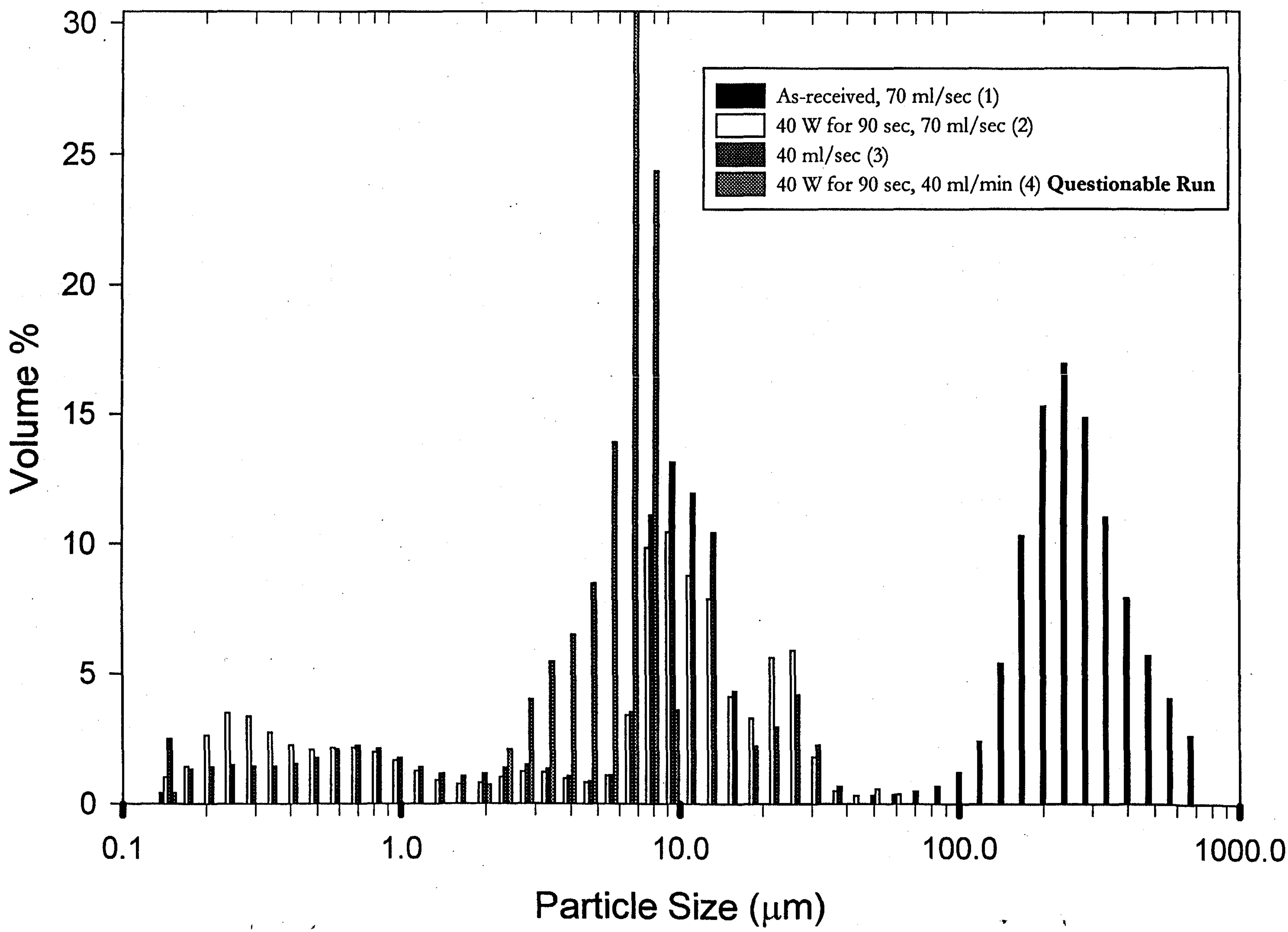
96-23 REC Volume Distribution Data

Sample tested at 70 ml/sec (1) then solicated and measured at 70 ml/sec (2) then slowed flow to 40 ml/sec (3) Then sonicated at 40W for 90 seconds and Measured at 40 ml/sec (4).

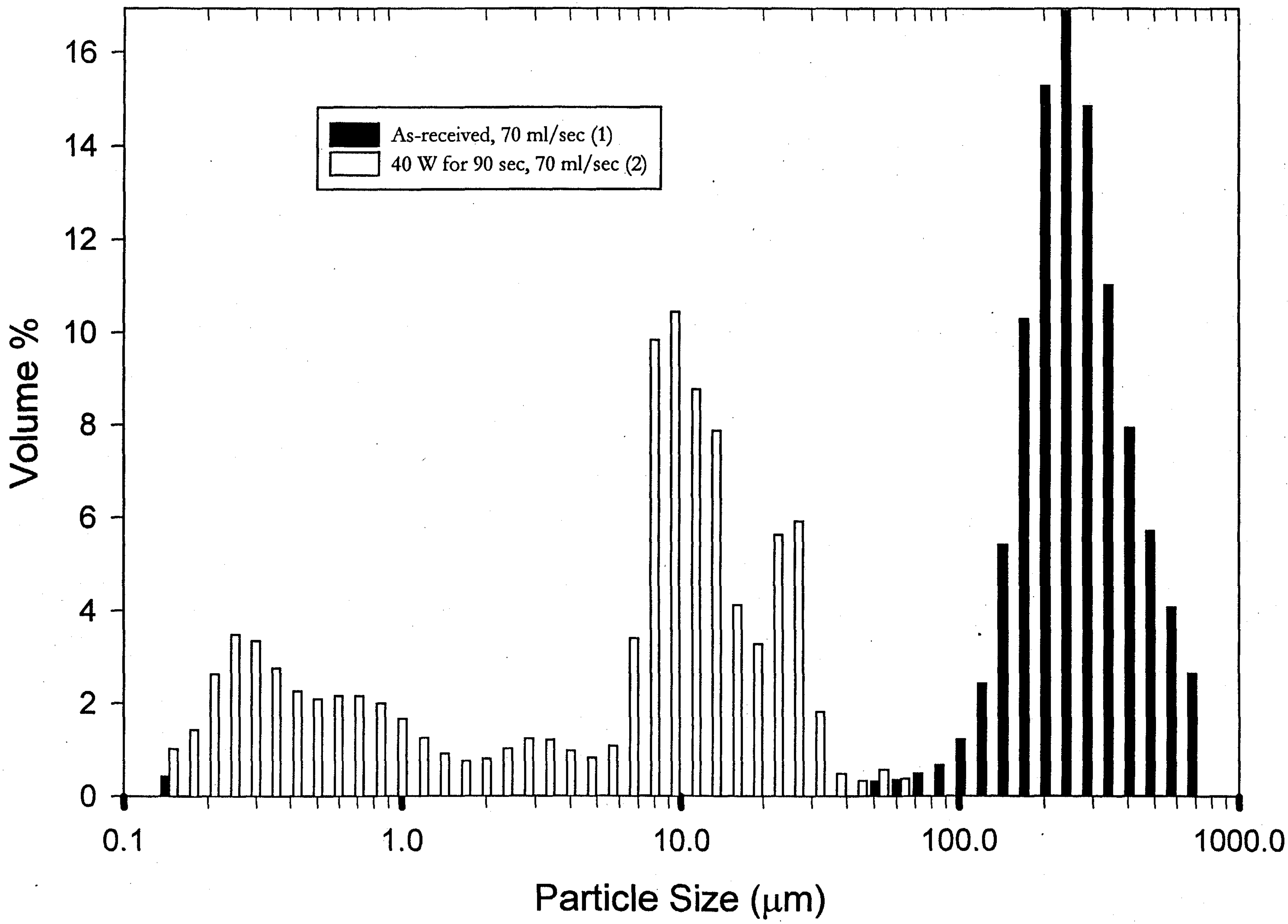
No Duplicate

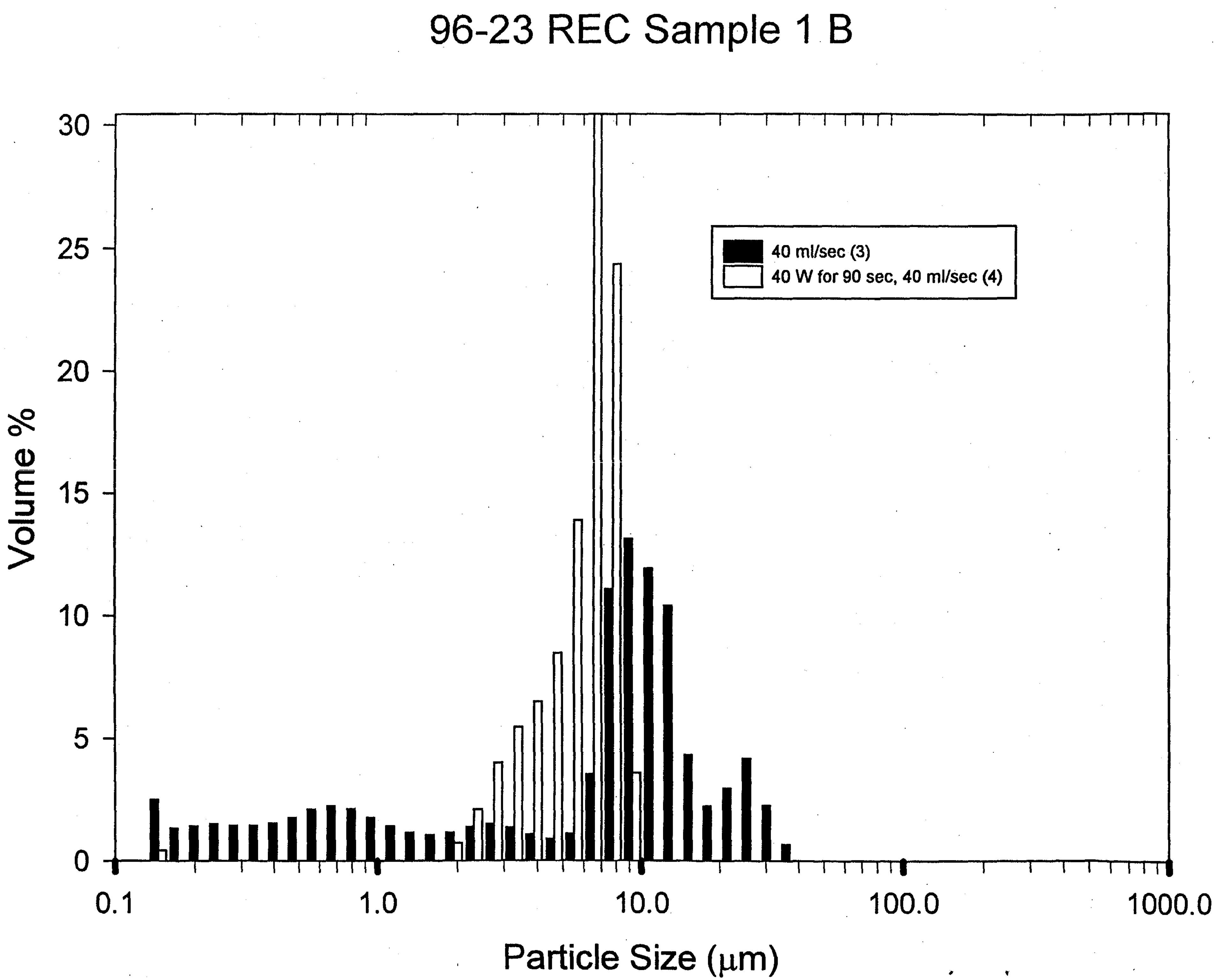
	1	2	3	4
Particle size	First As-Received 70 ml/sec	40 W 70 ml/sec	40 ml/sec	40W 40 ml/sec
704.000	2.63	0	0	0
592.000	4.07	0	0	0
497.800	5.71	0	0	0
418.600	7.92	0	0	0
352.000	11.01	0	0	0
296.000	14.84	0	0	0
248.900	16.94	0	0	0
209.300	15.28	0	0	0
176.000	10.28	0	0	0
148.000	5.4	0	0	0
124.500	2.42	0	0	0
104.700	1.22	0	0	0
88.000	0.68	0	0	0
74.000	0.49	0	0	0
62.230	0.36	0.38	0	0
52.330	0.32	0.57	0	0
44.000	0	0.33	0	0
37.000	0	0.49	0.68	0
31.110	0	1.81	2.26	0
26.160	0	5.91	4.18	0
22.000	0	5.62	2.95	0
18.500	0	3.29	2.23	0
15.560	0	4.12	4.31	0
13.080	0	7.87	10.4	0
11.000	0	8.76	11.92	0
9.250	0	10.44	13.11	3.6
7.778	0	9.83	11.08	24.37
6.541	0	3.41	3.53	30.43
5.500	0	1.08	1.1	13.9
4.625	0	0.83	0.87	8.46
3.889	0	0.98	1.07	6.51
3.270	0	1.21	1.36	5.47
2.750	0	1.24	1.5	4.01
2.312	0	1.03	1.38	2.1
1.945	0	0.81	1.17	0.72
1.635	0	0.76	1.07	0
1.375	0	0.92	1.16	0
1.156	0	1.26	1.42	0
0.972	0	1.67	1.78	0
0.818	0	2	2.12	0
0.688	0	2.16	2.25	0
0.578	0	2.16	2.11	0
0.486	0	2.1	1.78	0
0.409	0	2.26	1.54	0
0.344	0	2.76	1.46	0
0.289	0	3.35	1.46	0
0.243	0	3.49	1.5	0
0.204	0	2.63	1.42	0
0.172	0	1.44	1.34	0
0.145	0.43	1.03	2.49	0.43

96-23 REC Sample 1



96-23 REC Sample 1 A




96-23 REC Sample 1 B
40 ml/sec (3)
40 W for 90 sec, 40 ml/sec (4)
Volume %
0
5
10
15
20
25
30
0.1
1.0
10.0
100.0
1000.0
Particle Size (μm)

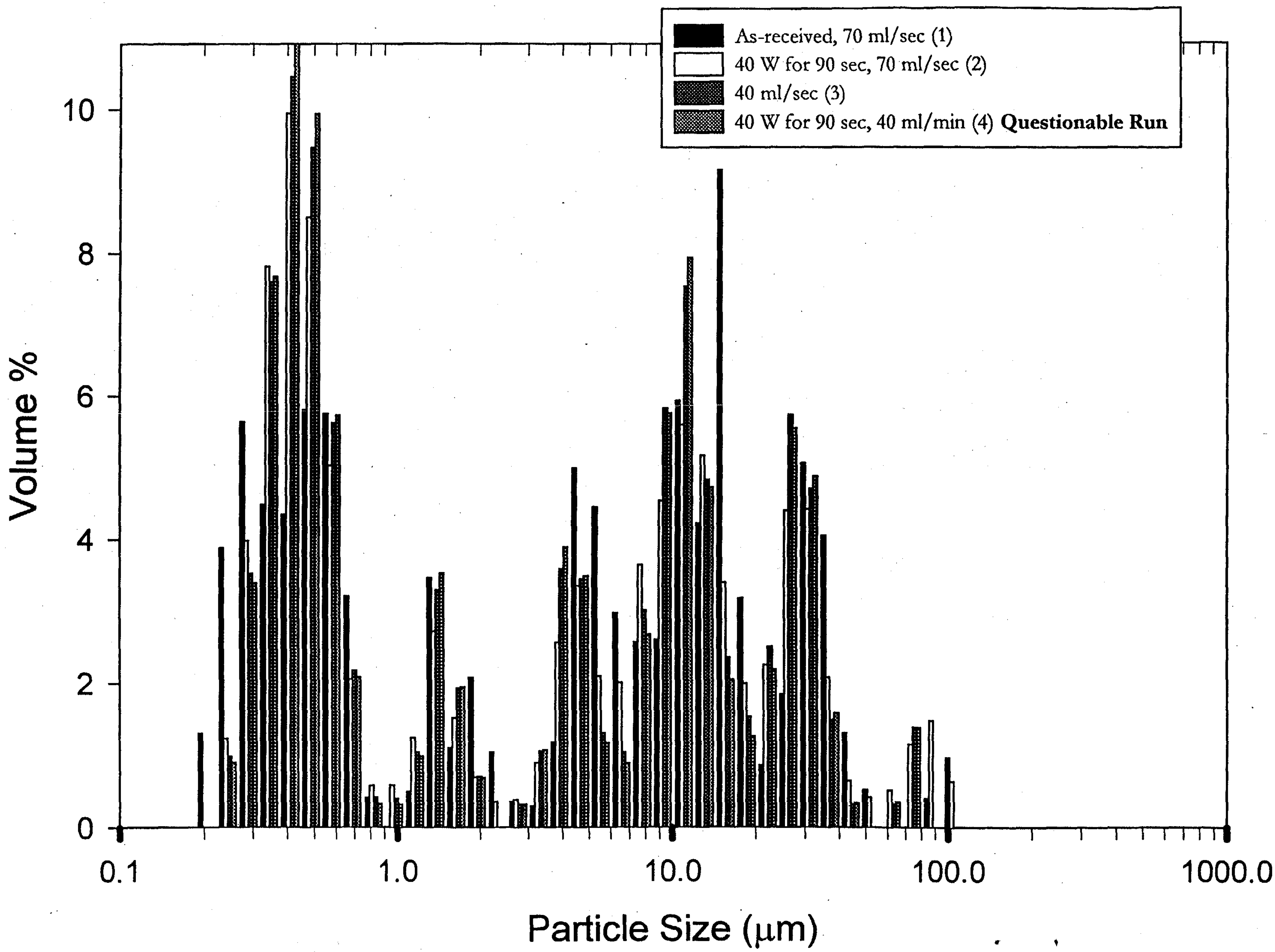
96-24 REC Volume Distribution Data

Sample tested at 70 ml/sec (1) then solicated and measured at 70 ml/sec (2) then slowed flow to 40 ml/sec (3) Then sonicated at 40W for 90 seconds and Measured at 40 ml/sec (4).

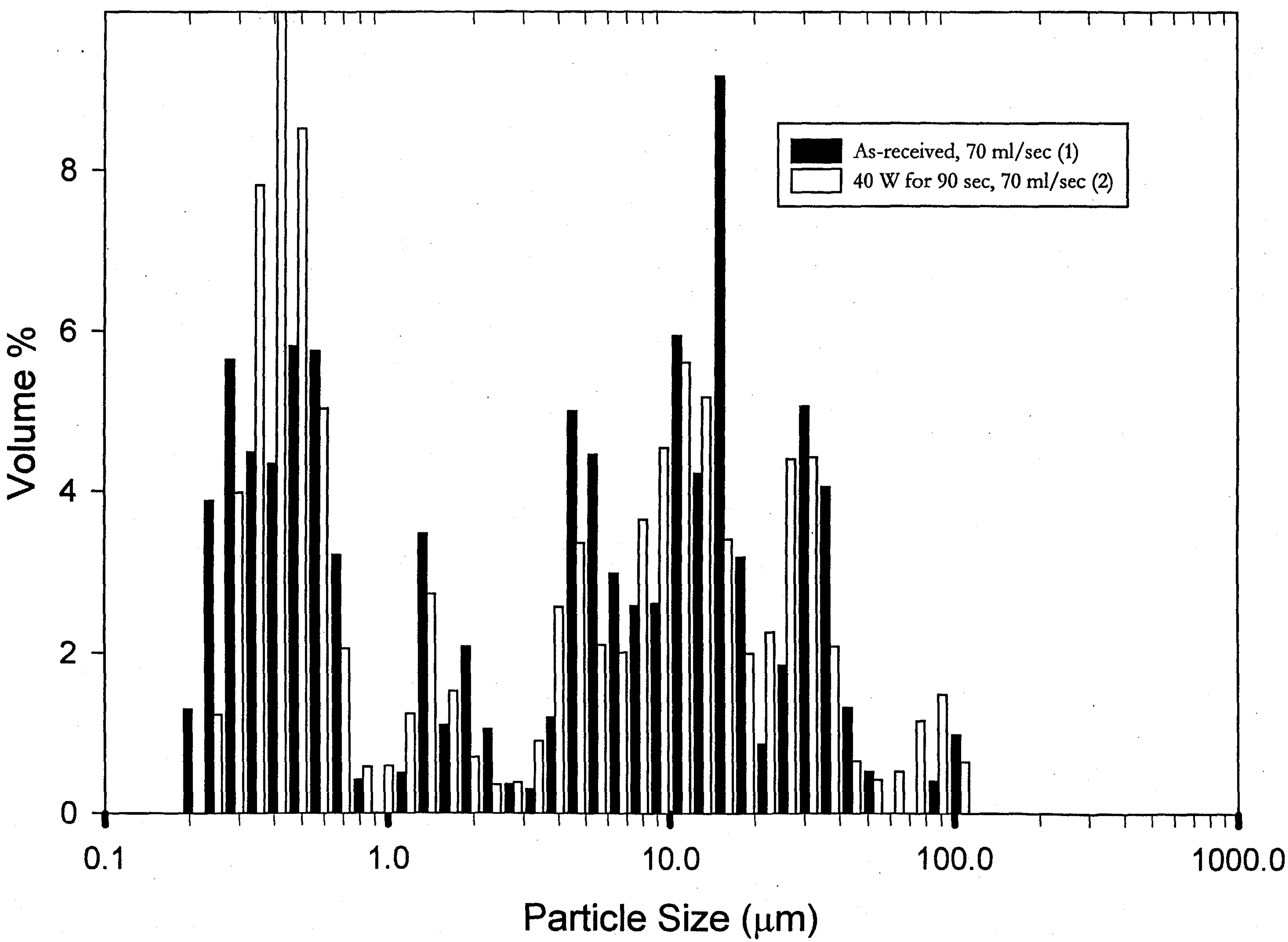
No Duplicate

Particle size	1 First As-Received 70 ml/sec	2 40 W 70 ml/sec	3 40 ml/sec	4 40W 40 ml/sec
704.000	0	0	0	0
592.000	0	0	0	0
497.800	0	0	0	0
418.600	0	0	0	0
352.000	0	0	0	0
296.000	0	0	0	0
248.900	0	0	0	0
209.300	0	0	0	0
176.000	0	0	0	0
148.000	0	0	0	0
124.500	0	0	0	0
104.700	0.97	0.64	0	0
88.000	0.4	1.48	0	0
74.000	0	1.15	1.39	1.39
62.230	0	0.52	0.32	0.35
52.330	0.52	0.42	0	0
44.000	1.31	0.65	0.32	0.34
37.000	4.05	2.08	1.49	1.58
31.110	5.06	4.42	4.7	4.88
26.160	1.84	4.4	5.74	5.55
22.000	0.86	2.26	2.51	2.19
18.500	3.18	1.99	1.53	1.26
15.560	9.16	3.4	2.36	2.05
13.080	4.22	5.17	4.83	4.72
11.000	5.94	5.6	7.54	7.94
9.250	2.61	4.54	5.83	5.76
7.778	2.58	3.65	3.02	2.68
6.541	2.98	2.01	1.04	0.89
5.500	4.45	2.1	1.31	1.17
4.625	4.99	3.36	3.45	3.49
3.889	1.19	2.57	3.59	3.89
3.270	0.3	0.9	1.06	1.08
2.750	0.36	0.39	0.33	0.32
2.312	1.05	0.36	0	0
1.945	2.08	0.7	0.71	0.7
1.635	1.1	1.52	1.94	1.95
1.375	3.47	2.73	3.3	3.53
1.156	0.5	1.24	1.04	0.99
0.972	0	0.59	0.4	0.32
0.818	0.42	0.58	0.42	0.33
0.688	3.21	2.06	2.18	2.09
0.578	5.75	5.03	5.62	5.73
0.486	5.81	8.51	9.47	9.94
0.409	4.34	9.95	10.46	10.92
0.344	4.48	7.82	7.6	7.68
0.289	5.64	3.98	3.52	3.39
0.243	3.88	1.23	0.98	0.9
0.204	1.3	0	0	0
0.172	0	0	0	0
0.145	0	0	0	0

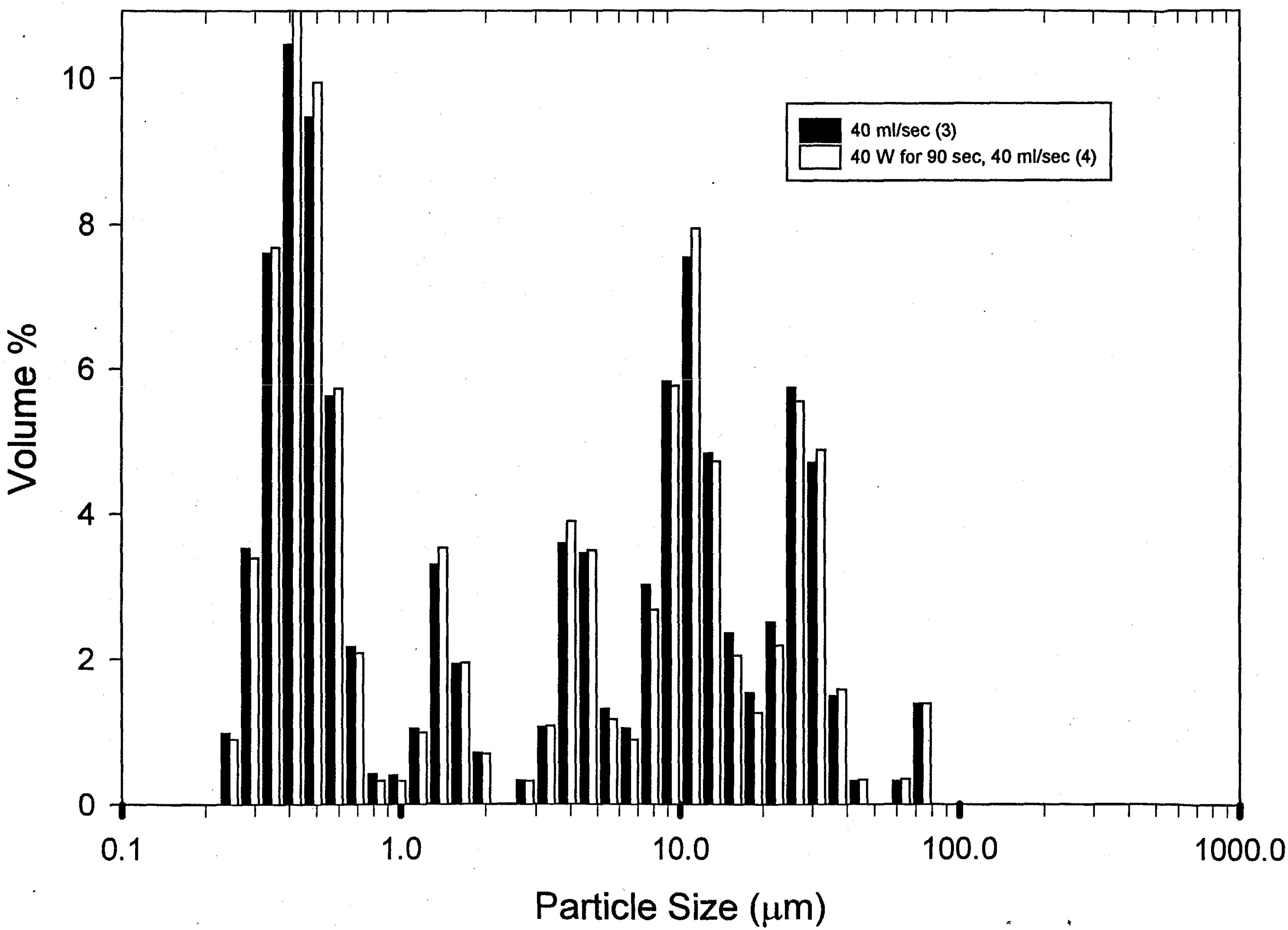
96-24 REC Sample 1



96-24 REC Sample 1 A



96-24 REC Sample 1 B



96-04 U/L Volume Distribution Data

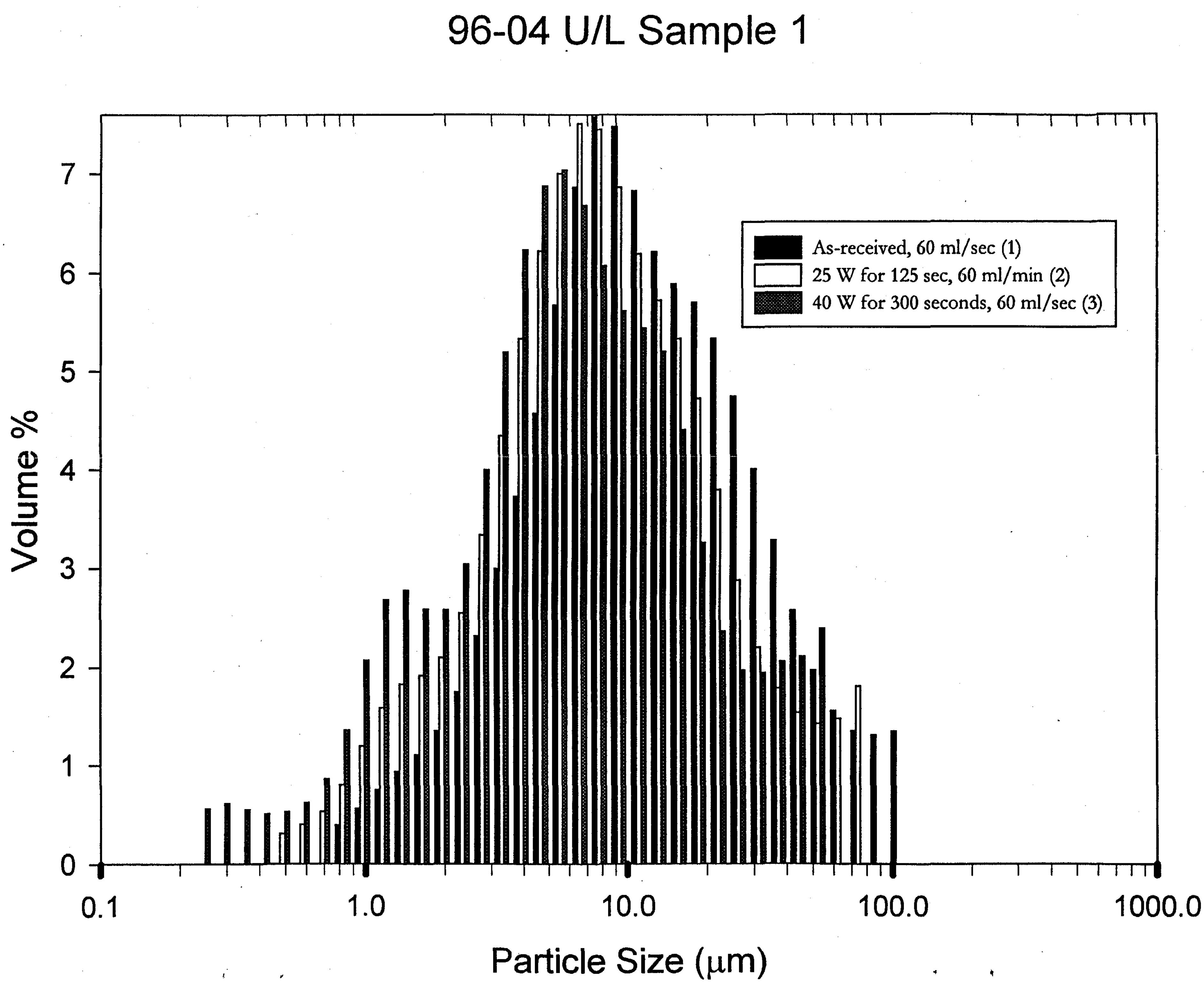
This sample was used to study the effects of circulation time, flow rate and circulation time on PSD results

Sample analyzed at 60 ml/sec (1), then sonicated at 25 W for 125 seconds and analyzed at 60 ml/sec (2), then sonicated at 40 W for 300 seconds and analyzed at 60 ml/sec (3).

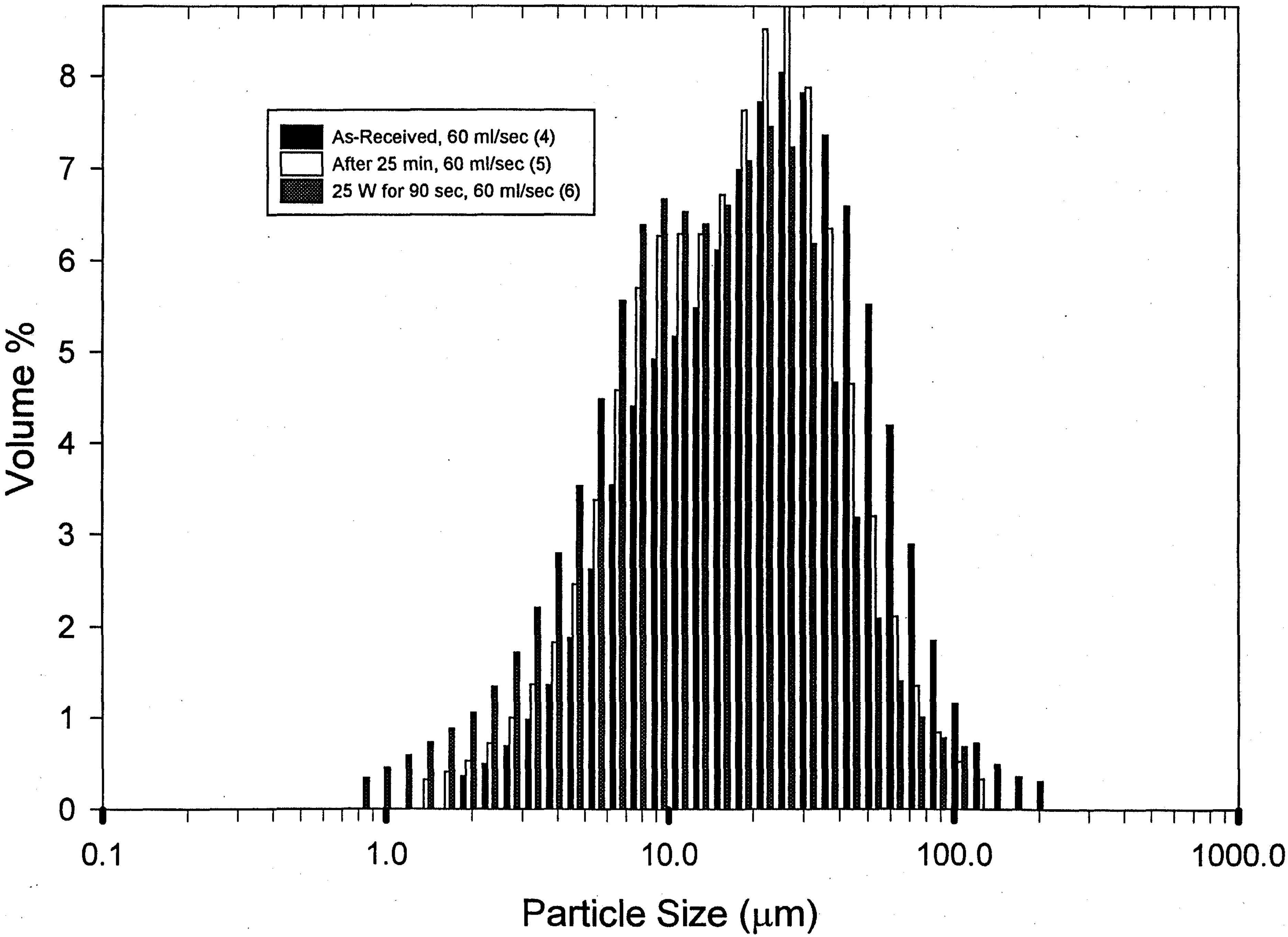
New sample was installed and analyzed at 60 ml/sec after 2 minutes (4) and then after 25 minutes (5), then sonicated at 25 W for 90 sec and analyzed at 60ml/sec (6).

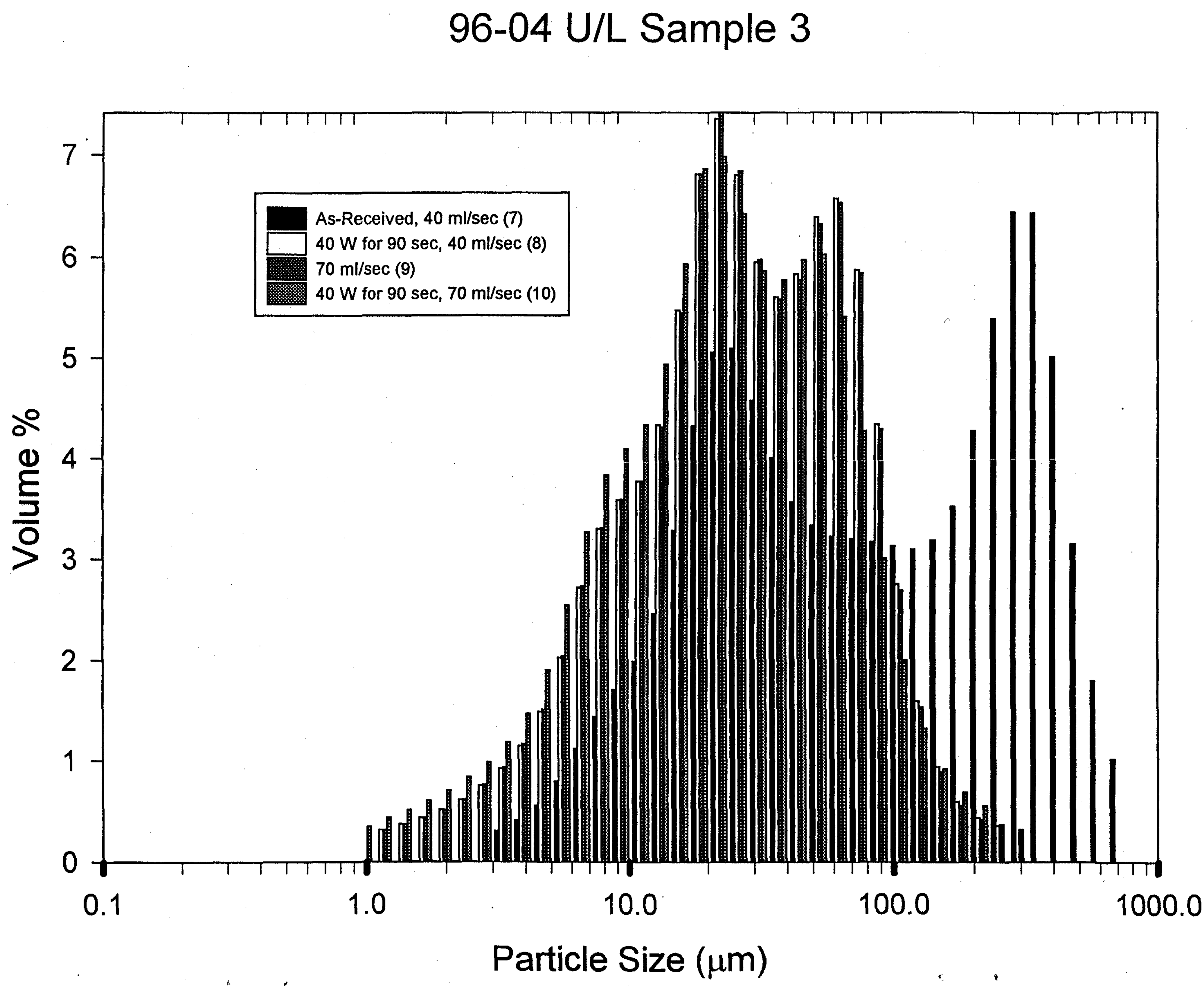
New sample was installed and analyzed at 40 ml/sec after 2 minutes (7), then sonicated at 40 W for 90 sec and analyzed at 40ml/sec (8), then flow increased and analyzed at 70 ml/sec (9), then sonicated at 40W for 90 sec and anlayzed at 70 ml/sec(10).

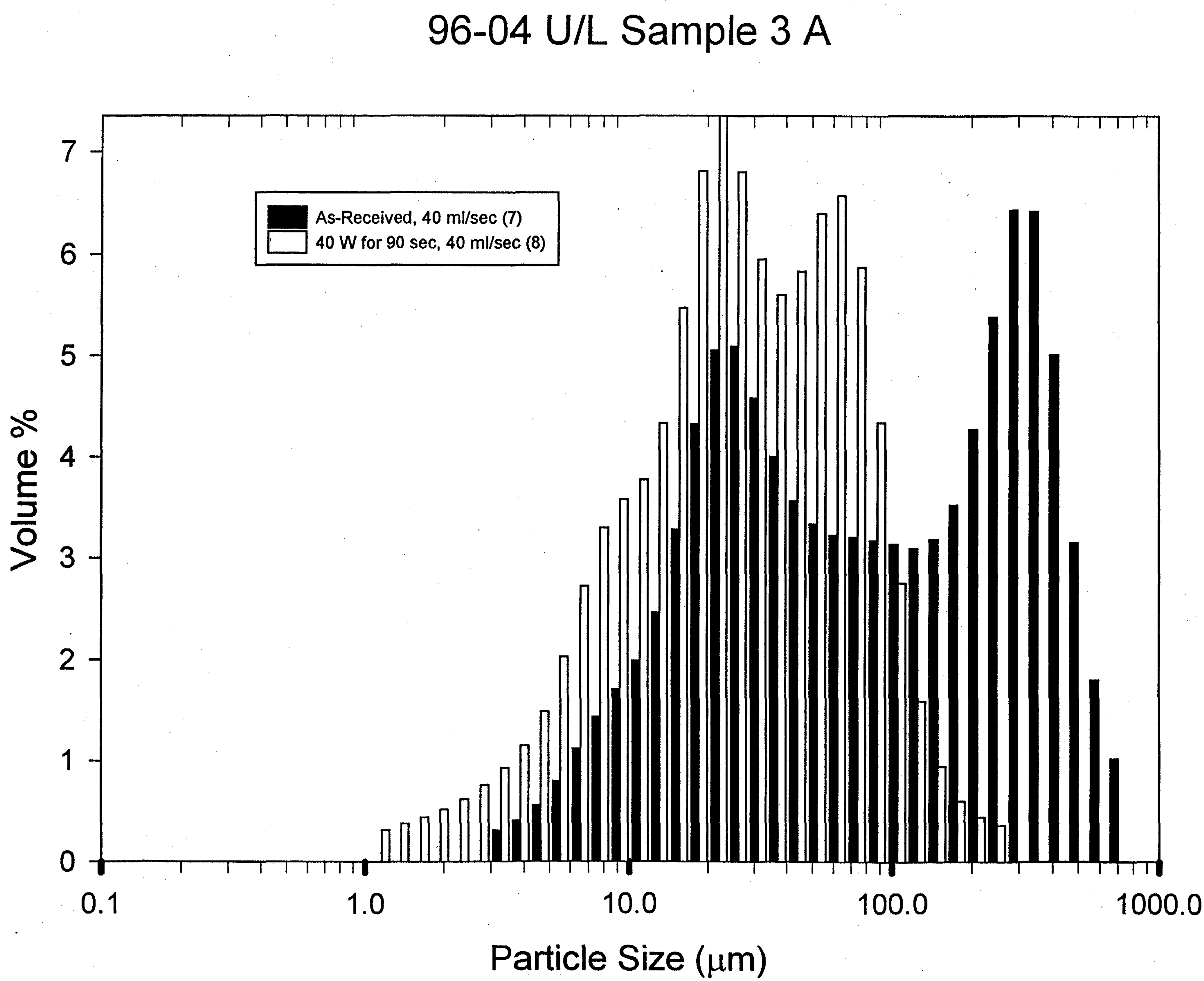
	1	2	3	4	5	6	7	8	9	10
	As-received	25 W	40 W	As-received		25 W	As-receive	40 W		40W
Particle size	60 ml/sec	60 ml/sec	60 ml/sec	60 ml/sec	60 ml/sec	60 ml/sec	40 ml/sec	40 ml/sec	70 ml/sec	70 ml/sec
704.000	0	0	0	0	0	0	1.02	0	0	0
592.000	0	0	0	0	0	0	1.8	0	0	0
497.800	0	0	0	0	0	0	3.15	0	0	0
418.600	0	0	0	0	0	0	5.01	0	0	0
352.000	0	0	0	0	0	0	6.42	0	0	0
296.000	0	0	0	0	0	0	6.43	0	0.32	0
248.900	0	0	0	0	0	0	5.38	0.36	0.37	0
209.300	0	0	0	0.31	0	0	4.27	0.44	0.42	0.56
176.000	0	0	0	0.36	0	0	3.52	0.6	0.56	0.69
148.000	0	0	0	0.49	0	0	3.18	0.94	0.89	0.92
124.500	0	0	0	0.72	0.33	0	3.09	1.59	1.53	1.32
104.700	1.34	0	0	1.15	0.52	0.68	3.13	2.75	2.69	2
88.000	1.3	0	0	1.85	0.84	0.78	3.17	4.34	4.29	3.01
74.000	1.34	1.8	0	2.89	1.35	1	3.2	5.87	5.84	4.27
62.230	1.55	1.47	0	4.19	2.11	1.4	3.22	6.57	6.53	5.41
52.330	1.96	1.42	2.38	5.52	3.2	2.09	3.33	6.39	6.32	6.02
44.000	2.56	1.53	2.1	6.59	4.65	3.18	3.56	5.83	5.77	5.97
37.000	3.27	1.78	2.05	7.36	6.35	4.66	4	5.6	5.58	5.77
31.110	4	2.19	1.93	7.82	7.88	6.18	4.58	5.95	5.97	5.86
26.160	4.74	2.87	1.96	8.04	8.76	7.23	5.09	6.8	6.84	6.42
22.000	5.33	3.79	2.35	7.72	8.51	7.45	5.05	7.35	7.41	6.98
18.500	5.7	4.72	3.25	6.99	7.63	7.08	4.32	6.81	6.81	6.86
15.560	5.89	5.33	4.4	6.11	6.72	6.6	3.28	5.47	5.44	5.93
13.080	6.21	5.72	5.2	5.48	6.29	6.4	2.46	4.33	4.31	4.93
11.000	6.82	6.19	5.44	5.17	6.29	6.53	1.99	3.77	3.77	4.33
9.250	7.47	6.86	5.61	4.92	6.27	6.67	1.71	3.58	3.59	4.09
7.778	7.59	7.44	6.07	4.4	5.7	6.39	1.44	3.3	3.31	3.83
6.541	6.86	7.5	6.68	3.54	4.58	5.56	1.12	2.72	2.73	3.27
5.500	5.67	7	7.03	2.62	3.38	4.48	0.8	2.03	2.04	2.54
4.625	4.57	6.22	6.87	1.88	2.46	3.53	0.56	1.49	1.51	1.9
3.889	3.72	5.33	6.23	1.36	1.83	2.79	0.41	1.15	1.17	1.47
3.270	2.99	4.34	5.19	0.98	1.37	2.21	0.31	0.93	0.94	1.19
2.750	2.31	3.33	3.99	0.69	1	1.72	0	0.76	0.77	0.99
2.312	1.74	2.54	3.03	0.49	0.72	1.34	0	0.62	0.62	0.84
1.945	1.34	2.09	2.57	0.36	0.53	1.06	0	0.52	0.52	0.71
1.635	1.1	1.91	2.58	0	0.41	0.88	0	0.44	0.44	0.61
1.375	0.93	1.82	2.77	0	0.32	0.73	0	0.38	0.38	0.52
1.156	0.75	1.58	2.67	0	0	0.59	0	0.32	0.32	0.44
0.972	0.56	1.19	2.06	0	0	0.45	0	0	0	0.35
0.818	0.39	0.8	1.35	0	0	0.34	0	0	0	0
0.688	0	0.53	0.86	0	0	0	0	0	0	0
0.578	0	0.4	0.62	0	0	0	0	0	0	0
0.486	0	0.31	0.53	0	0	0	0	0	0	0
0.409	0	0	0.51	0	0	0	0	0	0	0
0.344	0	0	0.55	0	0	0	0	0	0	0
0.289	0	0	0.61	0	0	0	0	0	0	0
0.243	0	0	0.56	0	0	0	0	0	0	0
0.204	0	0	0	0	0	0	0	0	0	0
0.172	0	0	0	0	0	0	0	0	0	0
0.145	0	0	0	0	0	0	0	0	0	0

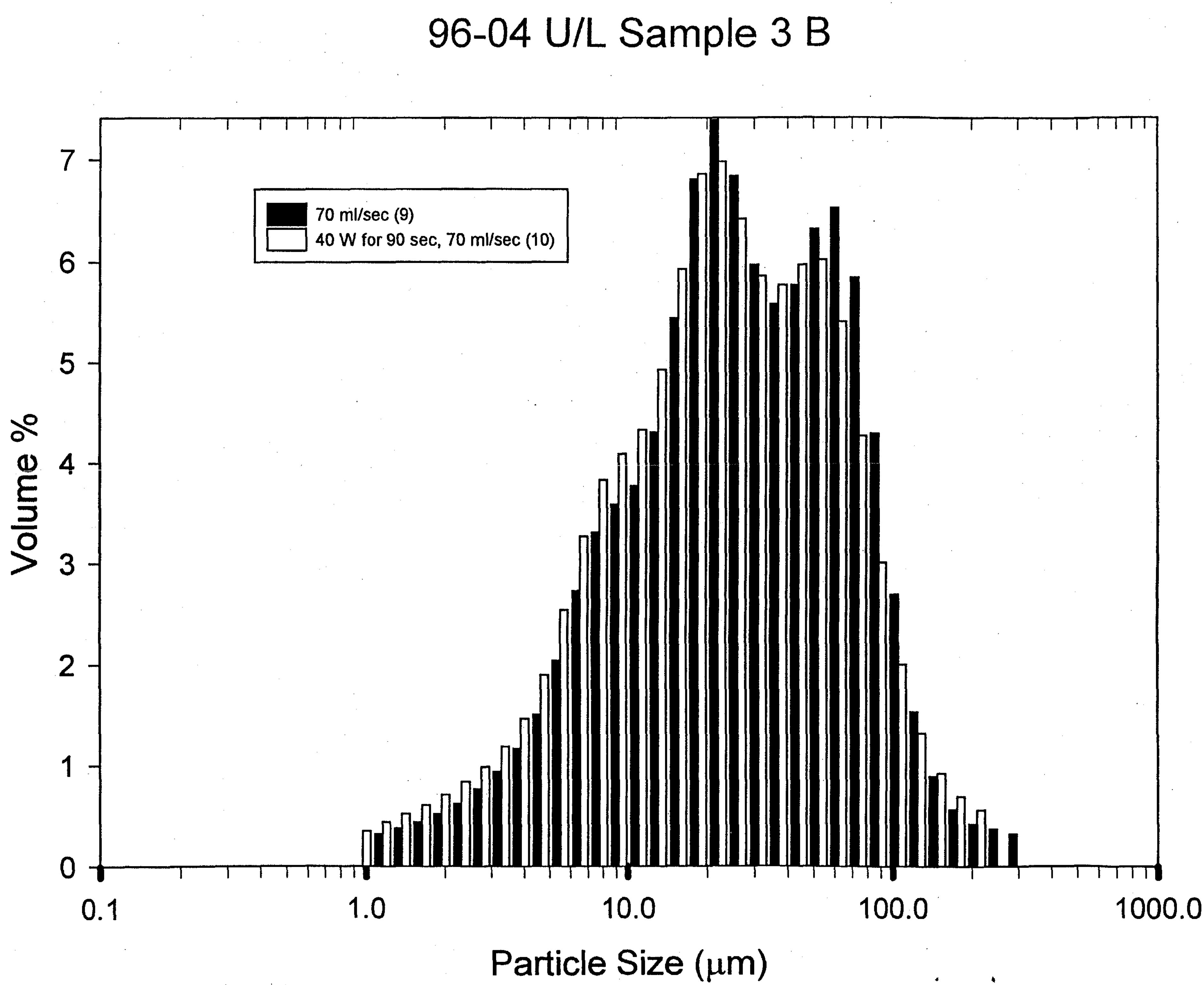

96-04 U/L Sample 1
Volume %
7
6
5
4
3
2
1
0
Particle Size (µm)
0.1
1.0
10.0
100.0
1000.0
As-received, 60 ml/sec (1)
25 W for 125 sec, 60 ml/min (2)
40 W for 300 seconds, 60 ml/sec (3)

96-04 U/L Sample 2




96-04 U/L Sample 3
As-Received, 40 ml/sec (7)
40 W for 90 sec, 40 ml/sec (8)
70 ml/sec (9)
40 W for 90 sec, 70 ml/sec (10)
Volume %
0
1
2
3
4
5
6
7
0.1
1.0
10.0
100.0
1000.0
Particle Size (μm)


96-04 U/L Sample 3 A
As-Received, 40 ml/sec (7)
40 W for 90 sec, 40 ml/sec (8)
Volume %
0
1
2
3
4
5
6
7
0.1
1.0
10.0
100.0
1000.0
Particle Size (μm)

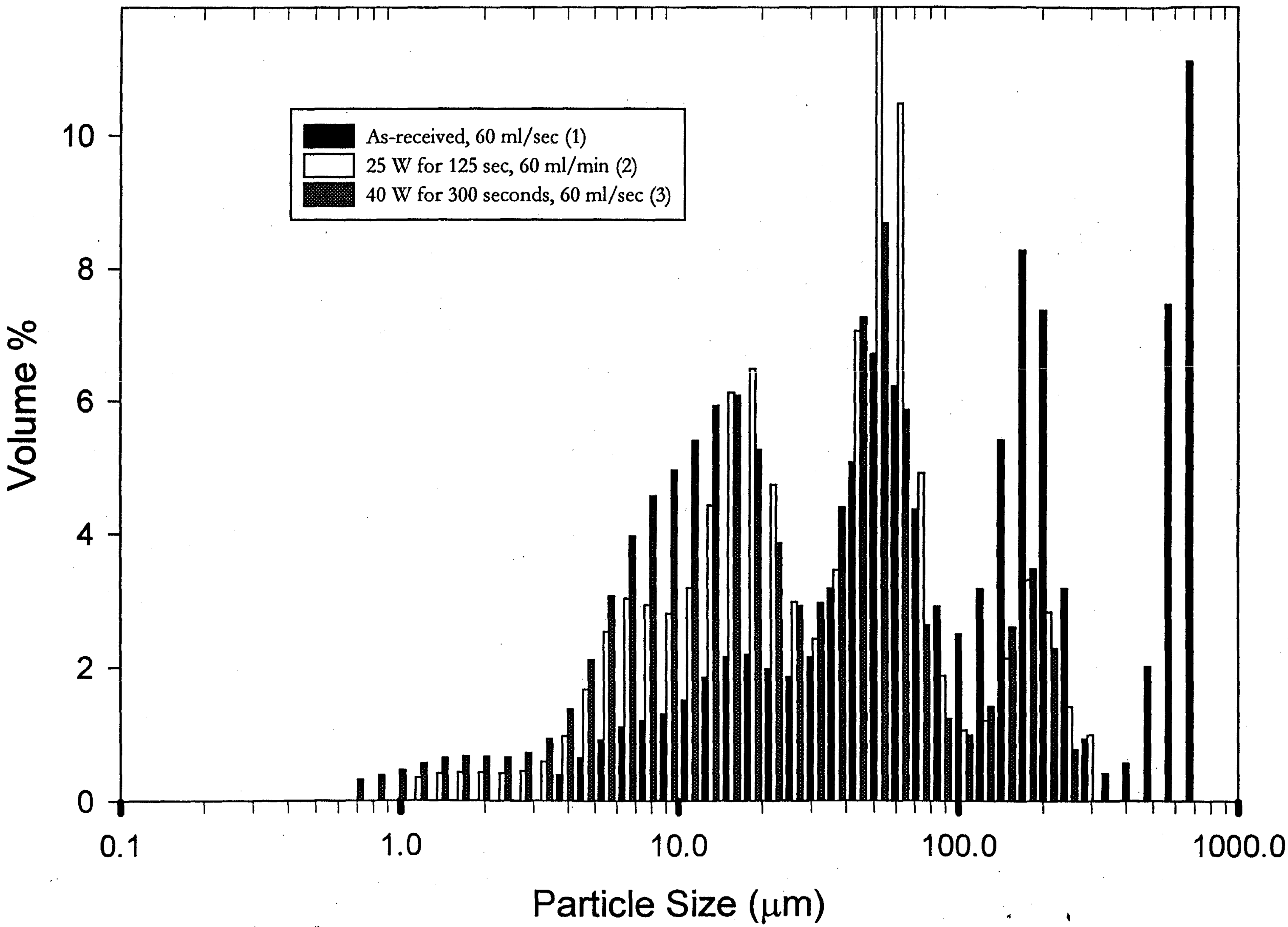

96-04 U/L Sample 3 B
70 ml/sec (9)
40 W for 90 sec, 70 ml/sec (10)
Volume %
0
1
2
3
4
5
6
7
0.1
1.0
10.0
100.0
1000.0
Particle Size (μm)

96-04 L Volume Distribution Data

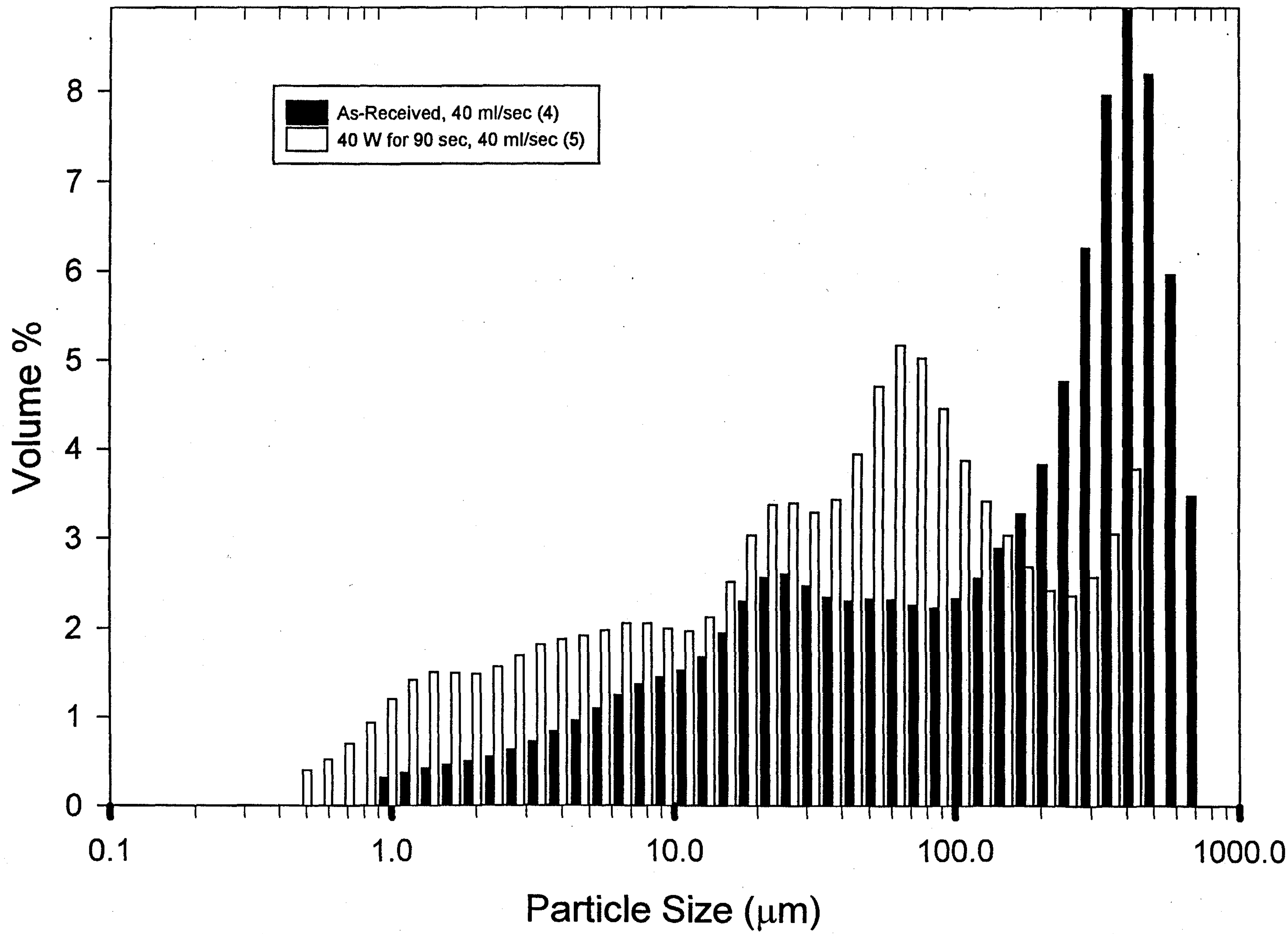
The sample was analyzed at 60 ml/sec (1), then sonicated at 25W for 120 seconds and analyzed at 60 ml/sec (2), Then sonicated at 40 W for 300 seconds and analyzed at 60 ml/sec (3).
New sample was installed and analyzed at 40 ml/sec (4), then sonicated at 40 W for 90 seconds and analyzed at 40 ml/sec (5), then flow increased to 70 ml/second and analyzed (6), then sonicated at 40 W for 90 seconds and analyzed at 70 ml/sec (7).

	1	2	3	4	5	6	7
	As-received	25 W	40 W	As-received	40 W		40 W
Particle size	60 ml/sec	60 ml/sec	60 ml/sec	40 ml/sec	40 ml/sec	70 ml/sec	70 ml/sec
704.000	11.1	0	0	3.47	0	0	18.49
592.000	7.46	0	0	5.96	0	0	11.15
497.800	2.01	0	0	8.19	0	0	2.24
418.600	0.56	0	0	8.93	3.77	3.39	0.4
352.000	0.41	0	0	7.95	3.04	1.41	0
296.000	0.93	0.99	0	6.25	2.56	1.07	0.37
248.900	3.18	1.41	0.77	4.75	2.35	1.28	1.22
209.300	7.36	2.83	2.28	3.82	2.41	1.84	3.27
176.000	8.27	3.31	3.47	3.26	2.67	2.44	4.53
148.000	5.4	2.13	2.6	2.88	3.03	2.79	3.45
124.500	3.17	1.2	1.41	2.55	3.41	2.87	2.05
104.700	2.49	1.06	0.98	2.32	3.87	2.97	1.4
88.000	2.91	1.87	1.23	2.22	4.45	3.27	1.33
74.000	4.36	4.91	2.63	2.25	5.02	3.8	1.67
62.230	6.22	10.47	5.86	2.31	5.16	4.25	2.23
52.330	6.71	11.9	8.68	2.32	4.7	4.34	2.58
44.000	5.07	7.06	7.26	2.3	3.94	4.02	2.42
37.000	3.18	3.46	4.39	2.34	3.43	3.77	2.07
31.110	2.14	2.43	2.96	2.47	3.28	3.78	1.81
26.160	1.85	2.98	2.92	2.6	3.39	3.98	1.72
22.000	1.97	4.74	3.86	2.56	3.37	3.95	1.67
18.500	2.19	6.48	5.26	2.29	3.03	3.51	1.59
15.560	2.15	6.13	6.08	1.94	2.51	2.89	1.48
13.080	1.85	4.43	5.93	1.67	2.12	2.48	1.45
11.000	1.51	3.19	5.4	1.52	1.96	2.39	1.54
9.250	1.3	2.81	4.96	1.44	1.99	2.51	1.7
7.778	1.2	2.94	4.57	1.36	2.05	2.63	1.83
6.541	1.11	3.03	3.96	1.24	2.05	2.58	1.85
5.500	0.91	2.54	3.07	1.09	1.97	2.44	1.8
4.625	0.64	1.67	2.11	0.95	1.91	2.33	1.76
3.889	0.39	0.97	1.37	0.83	1.87	2.28	1.74
3.270	0	0.59	0.93	0.72	1.81	2.21	1.72
2.750	0	0.45	0.72	0.63	1.69	2.08	1.66
2.312	0	0.41	0.65	0.55	1.56	1.94	1.61
1.945	0	0.42	0.66	0.5	1.48	1.88	1.64
1.635	0	0.43	0.67	0.46	1.49	1.93	1.74
1.375	0	0.41	0.64	0.42	1.5	1.97	1.82
1.156	0	0.35	0.56	0.37	1.41	1.85	1.73
0.972	0	0	0.46	0.32	1.2	1.53	1.43
0.818	0	0	0.38	0	0.93	1.15	1.08
0.688	0	0	0.32	0	0.7	0.83	0.8
0.578	0	0	0	0	0.52	0.6	0.61
0.486	0	0	0	0	0.4	0.44	0.5
0.409	0	0	0	0	0	0.33	0.45
0.344	0	0	0	0	0	0	0.4
0.289	0	0	0	0	0	0	0
0.243	0	0	0	0	0	0	0
0.204	0	0	0	0	0	0	0
0.172	0	0	0	0	0	0	0
0.145	0	0	0	0	0	0	0

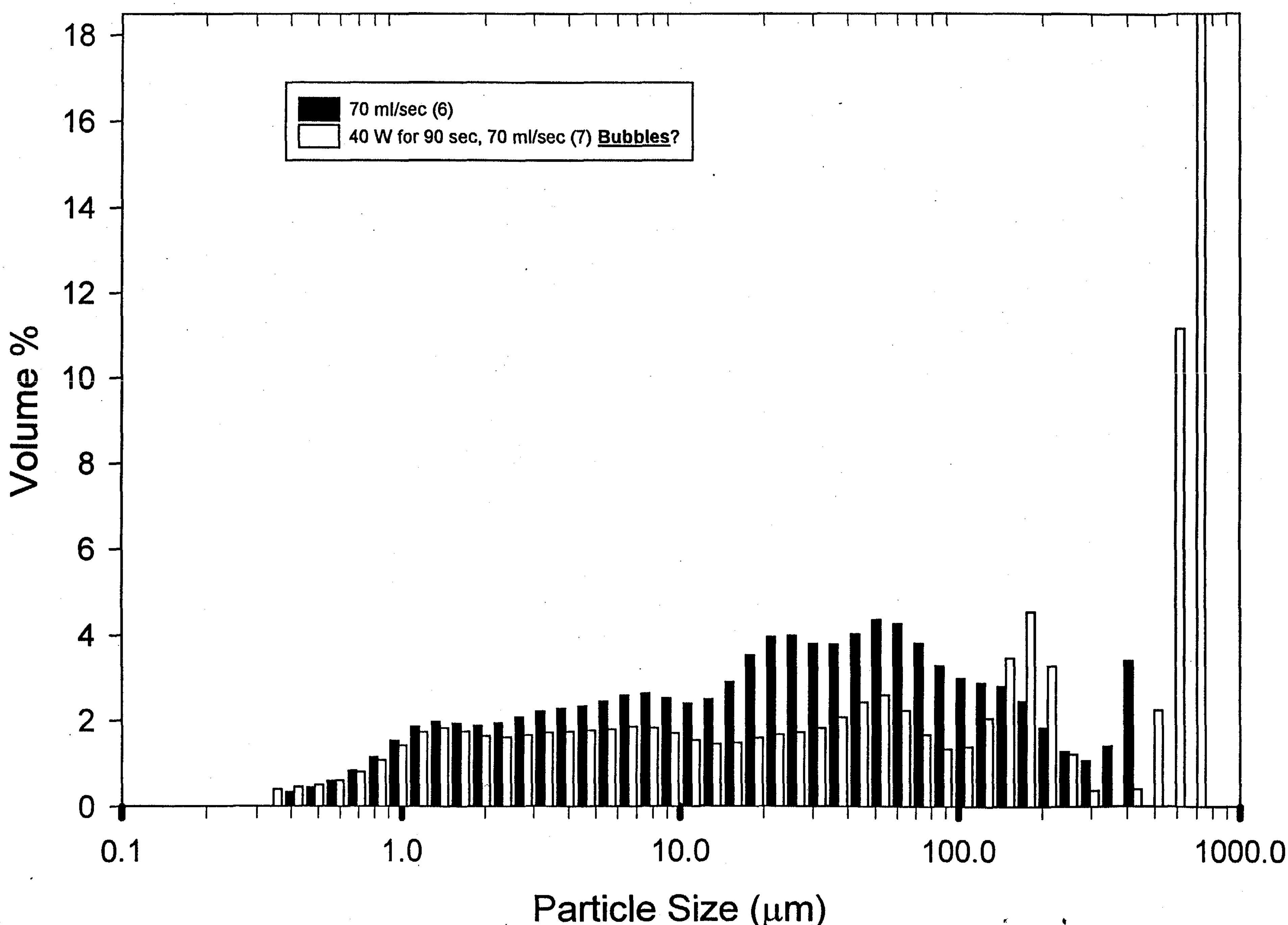
96-04 L Sample 1



96-04 L Sample 2 A



96-04 L Sample 2 B



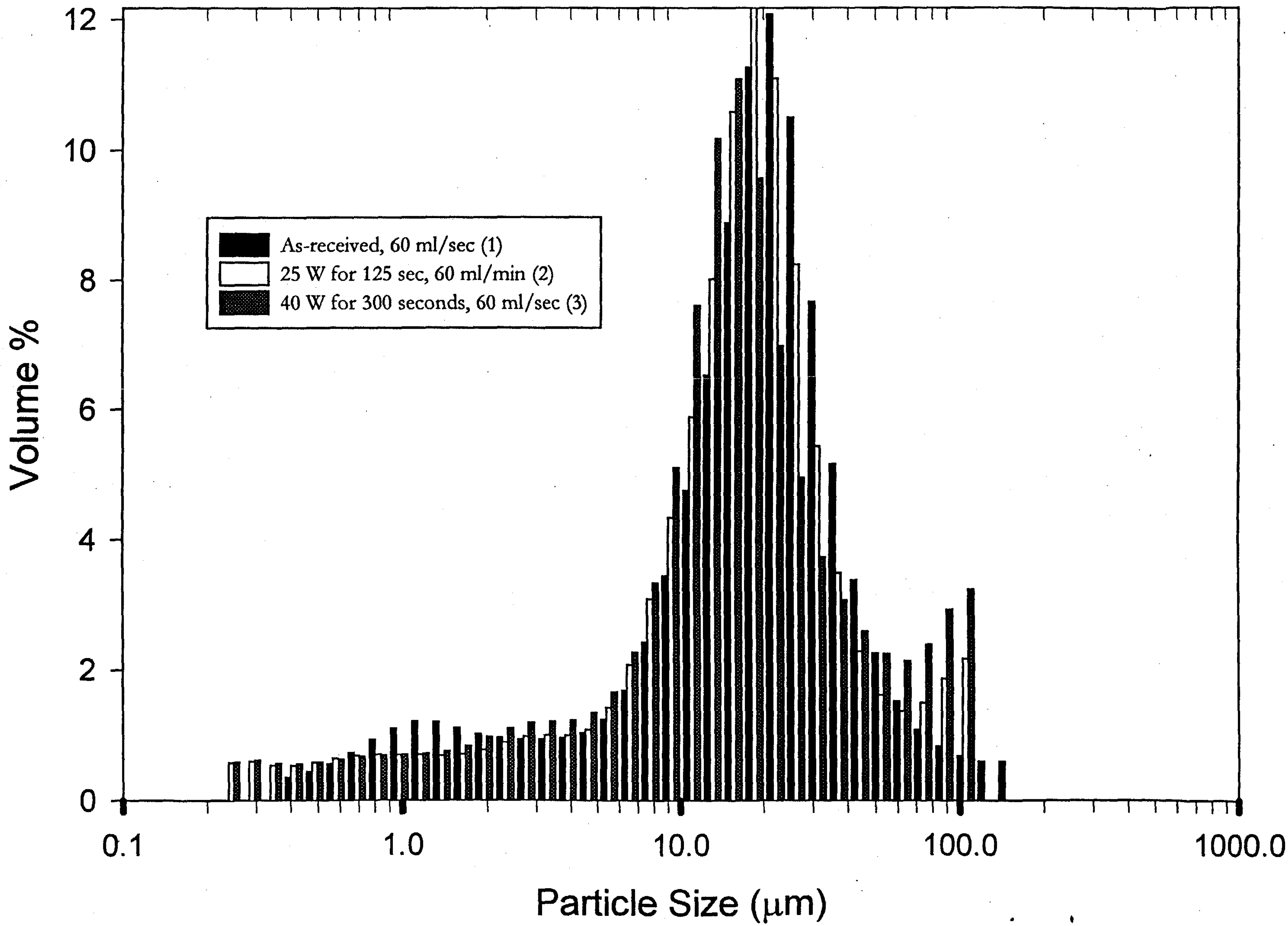
96-06 Carboy Volume Distribution Data

sample was analyzed at 60 ml/min (1), then sonicated at 25 W for 120 seconds and analyzed at 60 ml/sec (2), then sonicated at 40 W for 300 sec and analyzed at 60 ml/sec (3).

No duplicate

	1	2	3
	As-Received	25 W	40 W
Particle Size	60 ml/sec	60 ml/sec	60 ml/sec
704.000	0	0	0
592.000	0	0	0
497.800	0	0	0
418.600	0	0	0
352.000	0	0	0
296.000	0	0	0
248.900	0	0	0
209.300	0	0	0
176.000	0	0	0
148.000	0.6	0	0
124.500	0.6	0	0
104.700	0.68	2.17	3.24
88.000	0.83	1.87	2.93
74.000	1.08	1.5	2.39
62.230	1.52	1.37	2.13
52.330	2.25	1.61	2.24
44.000	3.38	2.28	2.59
37.000	5.15	3.49	3.07
31.110	7.66	5.43	3.73
26.160	10.5	8.24	4.94
22.000	12.08	11.09	6.98
18.500	11.26	12.17	9.57
15.560	8.88	10.59	11.08
13.080	6.52	8.01	10.17
11.000	4.75	5.87	7.6
9.250	3.44	4.33	5.09
7.778	2.42	3.09	3.33
6.541	1.69	2.08	2.27
5.500	1.24	1.42	1.66
4.625	1.03	1.09	1.34
3.889	0.96	1	1.23
3.270	0.94	1	1.21
2.750	0.94	0.98	1.19
2.312	0.96	0.89	1.11
1.945	1.01	0.78	0.97
1.635	1.11	0.71	0.84
1.375	1.2	0.69	0.76
1.156	1.21	0.7	0.72
0.972	1.1	0.7	0.7
0.818	0.93	0.7	0.69
0.688	0.73	0.68	0.67
0.578	0.56	0.64	0.63
0.486	0.44	0.58	0.59
0.409	0.35	0.53	0.56
0.344	0	0.54	0.57
0.289	0	0.6	0.62
0.243	0	0.58	0.59
0.204	0	0	0
0.172	0	0	0
0.145	0	0	0

96-06 Carboy



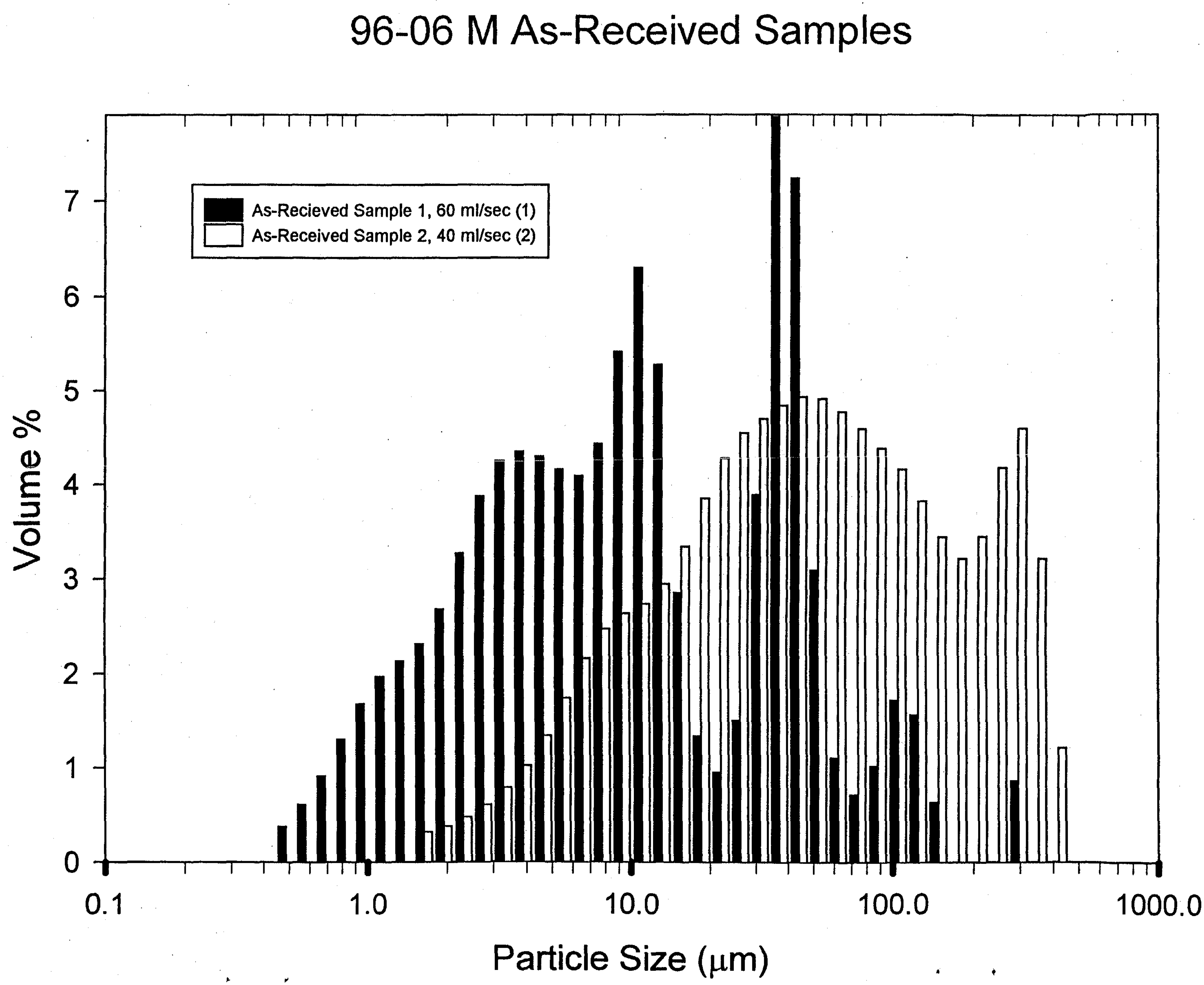
Volume %
12
10
8
6
4
2
0
0.1
1.0
10.0
100.0
1000.0
Particle Size (μm)
As-received, 60 ml/sec (1)
25 W for 125 sec, 60 ml/min (2)
40 W for 300 seconds, 60 ml/sec (3)

96-06 M Volume Distribution Data

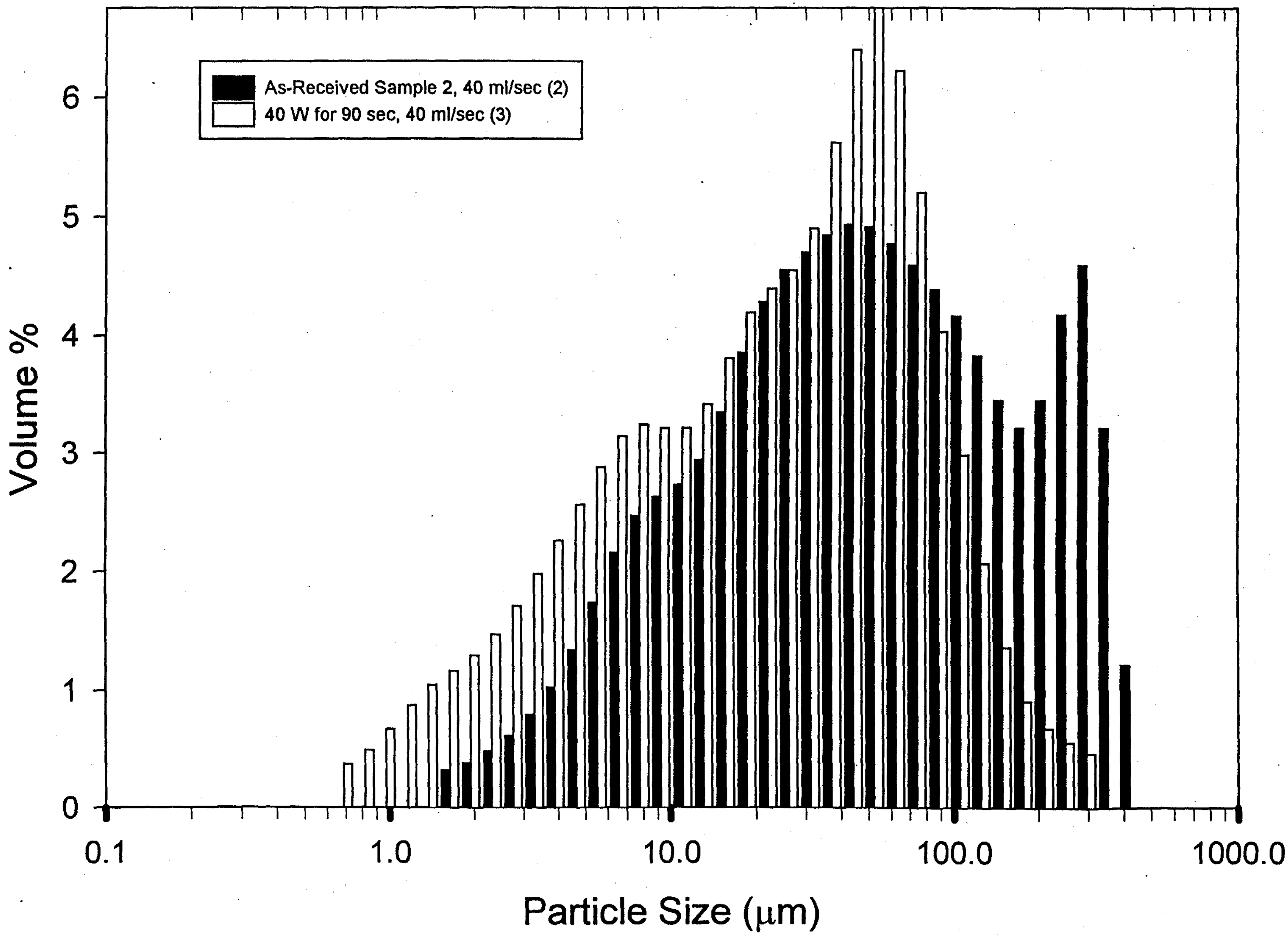
Sample was installed and analyzed at 60 ml/sec (1).

New sample was installed and analyzed at 40 ml/sec (2), then sonicated at 40 W for 90 sec and analyzed at 40 ml/sec (3). Flow rate was increased and sample analyzed at 70 ml/sec (4), then sample sonicated at 40 W for 90 sec and analyzed at 70 ml/sec(5).

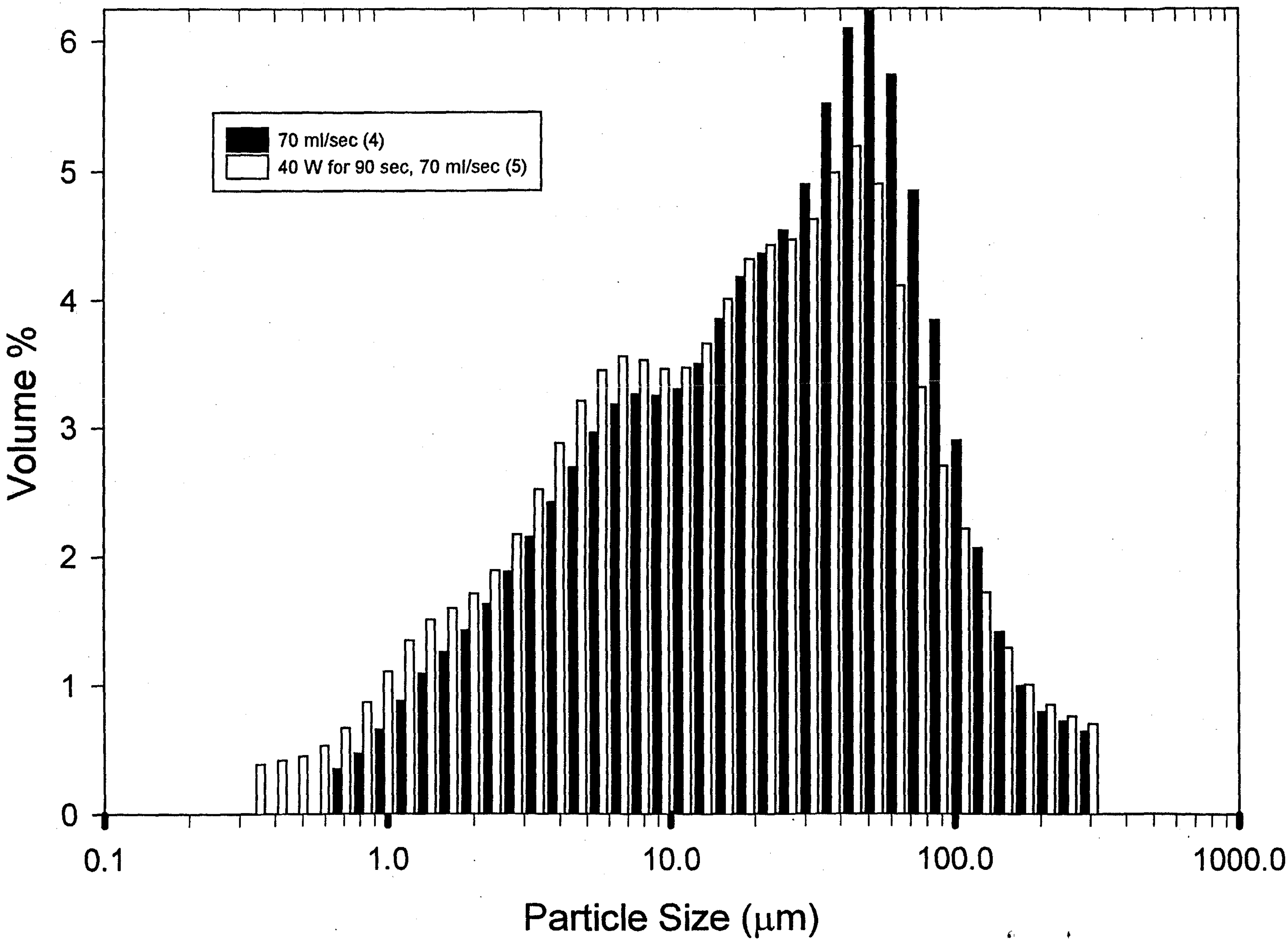
	1	2	3	4	5
	As-receive	As-received	40 W		40 W
Particle size	60 ml/sec	40 ml/sec	40 ml/sec	70 ml/sec	70 ml/sec
704.000	0	0	0	0	0
592.000	0	0	0	0	0
497.800	0	0	0	0	0
418.600	0	1.21	0	0	0
352.000	0	3.21	0	0	0
296.000	0.86	4.59	0.46	0.64	0.7
248.900	0	4.17	0.55	0.72	0.76
209.300	0	3.44	0.67	0.79	0.85
176.000	0	3.21	0.9	0.99	1
148.000	0.63	3.44	1.36	1.41	1.29
124.500	1.56	3.82	2.07	2.06	1.72
104.700	1.71	4.16	2.98	2.9	2.21
88.000	1.01	4.38	4.03	3.84	2.7
74.000	0.71	4.59	5.2	4.85	3.31
62.230	1.1	4.77	6.22	5.74	4.11
52.330	3.09	4.91	6.75	6.25	4.9
44.000	7.23	4.93	6.4	6.1	5.19
37.000	7.9	4.84	5.62	5.52	4.99
31.110	3.89	4.7	4.9	4.9	4.63
26.160	1.5	4.55	4.55	4.54	4.47
22.000	0.95	4.28	4.39	4.36	4.43
18.500	1.33	3.85	4.19	4.18	4.32
15.560	2.85	3.34	3.8	3.85	4.01
13.080	5.27	2.94	3.41	3.5	3.66
11.000	6.3	2.73	3.21	3.3	3.47
9.250	5.41	2.63	3.21	3.25	3.46
7.778	4.43	2.47	3.24	3.26	3.53
6.541	4.09	2.16	3.14	3.18	3.56
5.500	4.16	1.74	2.88	2.96	3.45
4.625	4.3	1.34	2.56	2.69	3.21
3.889	4.35	1.02	2.26	2.42	2.88
3.270	4.25	0.79	1.98	2.15	2.52
2.750	3.88	0.61	1.71	1.88	2.17
2.312	3.27	0.48	1.47	1.63	1.89
1.945	2.68	0.38	1.29	1.43	1.71
1.635	2.31	0.32	1.16	1.26	1.6
1.375	2.13	0	1.04	1.09	1.51
1.156	1.97	0	0.87	0.88	1.35
0.972	1.68	0	0.67	0.66	1.11
0.818	1.3	0	0.49	0.47	0.87
0.688	0.91	0	0.37	0.35	0.67
0.578	0.61	0	0	0	0.53
0.486	0.38	0	0	0	0.45
0.409	0	0	0	0	0.42
0.344	0	0	0	0	0.39
0.289	0	0	0	0	0
0.243	0	0	0	0	0
0.204	0	0	0	0	0
0.172	0	0	0	0	0
0.145	0	0	0	0	0


96-06 M As-Received Samples
As-Recieved Sample 1, 60 ml/sec (1)
As-Received Sample 2, 40 ml/sec (2)
Volume %
7
6
5
4
3
2
1
0
0.1
1.0
10.0
100.0
1000.0
Particle Size (μm)

96-06 M Sample 2 A



96-06 M Sample 2 B

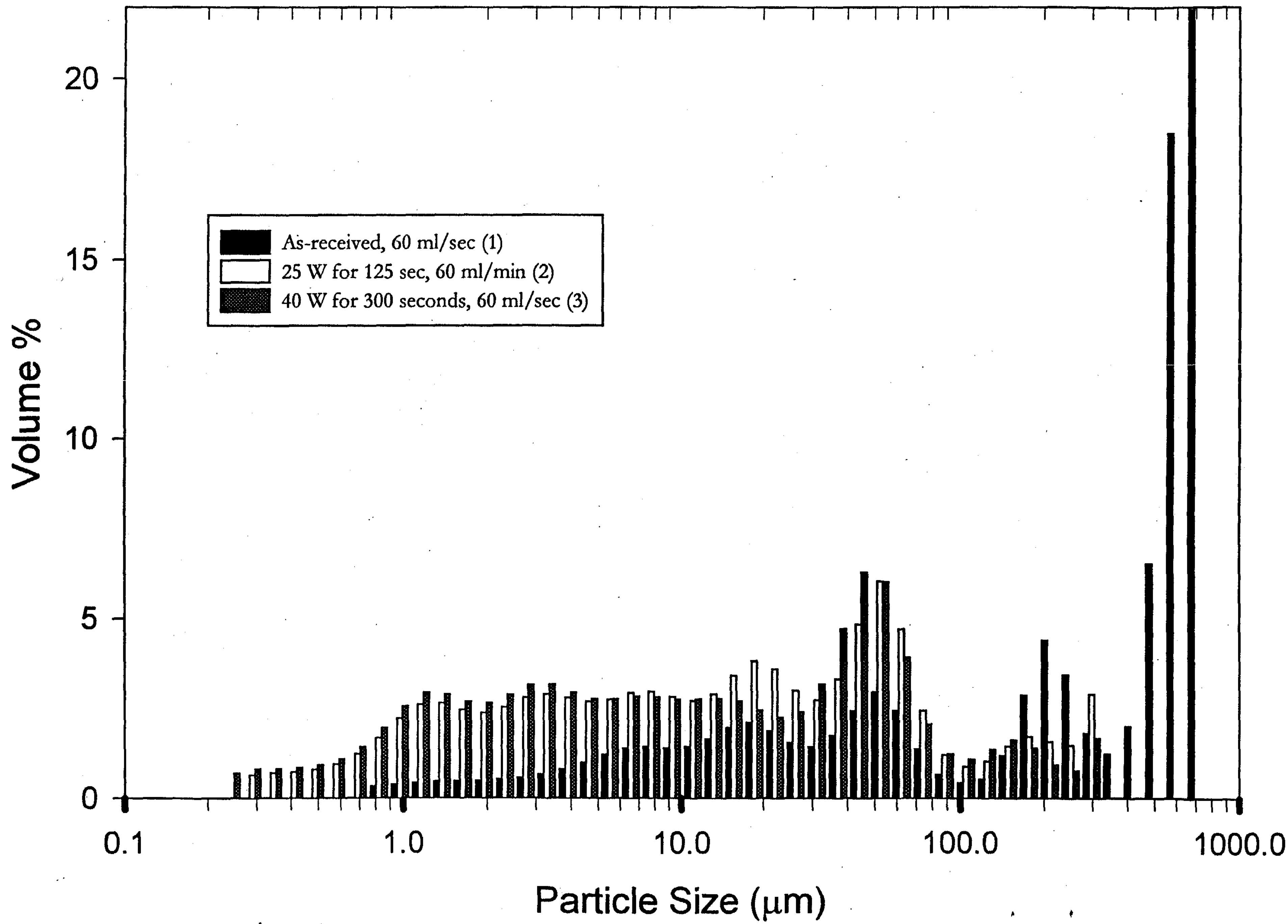


96-06 L *Volume Distribution Data*

sample was analyzed at 60 ml/min (1), then sonicated at 25 W for 120 seconds and analyzed at 60 ml/sec (2), then sonicated at 40 W for 300 sec and analyzed at 60 ml/sec (3).
No duplicate

	1	2	3
	As-Received	25 W	40 W
Particle Size	60 ml/sec	60 ml/sec	60 ml/sec
704.000	21.97	0	0
592.000	18.48	0	0
497.800	6.51	0	0
418.600	2	0	0
352.000	1.23	0	0
296.000	1.8	2.9	1.67
248.900	3.43	1.48	0.77
209.300	4.4	1.57	0.93
176.000	2.86	1.72	1.4
148.000	1.18	1.45	1.62
124.500	0.53	1.03	1.35
104.700	0.43	0.88	1.09
88.000	0.66	1.21	1.23
74.000	1.36	2.44	2.06
62.230	2.44	4.7	3.91
52.330	2.95	6.03	6.02
44.000	2.42	4.84	6.28
37.000	1.74	3.31	4.71
31.110	1.44	2.73	3.17
26.160	1.55	3	2.4
22.000	1.88	3.6	2.25
18.500	2.11	3.83	2.45
15.560	1.96	3.41	2.7
13.080	1.64	2.9	2.78
11.000	1.44	2.71	2.75
9.250	1.4	2.82	2.76
7.778	1.44	2.97	2.83
6.541	1.39	2.93	2.84
5.500	1.22	2.76	2.77
4.625	0.99	2.69	2.77
3.889	0.8	2.8	2.95
3.270	0.67	2.9	3.17
2.750	0.58	2.8	3.16
2.312	0.52	2.54	2.89
1.945	0.49	2.38	2.66
1.635	0.48	2.46	2.68
1.375	0.47	2.64	2.89
1.156	0.43	2.61	2.93
0.972	0.38	2.22	2.56
0.818	0.33	1.67	1.95
0.688	0	1.23	1.43
0.578	0	0.95	1.1
0.486	0	0.8	0.93
0.409	0	0.73	0.85
0.344	0	0.71	0.82
0.289	0	0.65	0.82
0.243	0	0	0.7
0.204	0	0	0
0.172	0	0	0
0.145	0	0	0

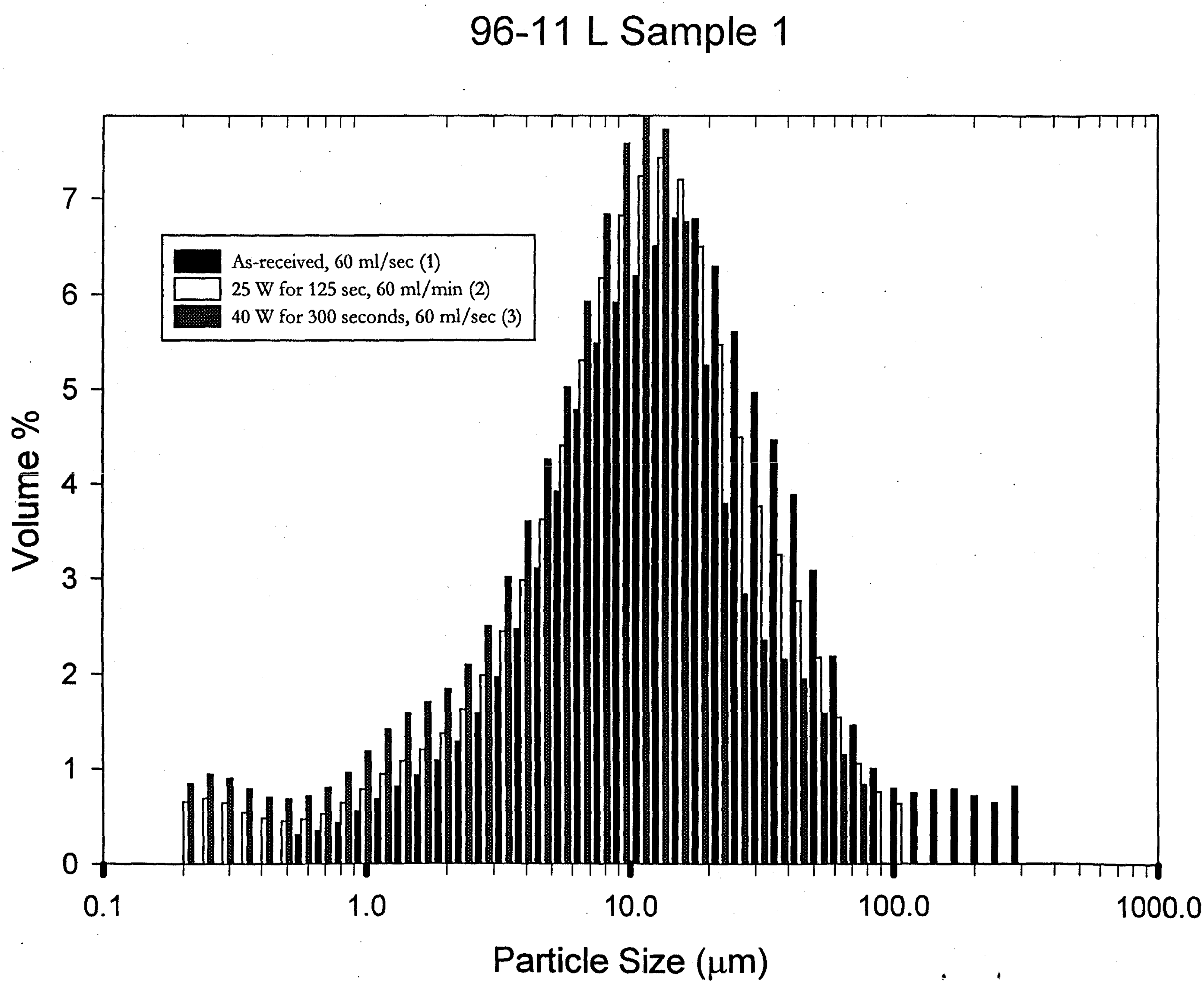
96-06 L Sample 1



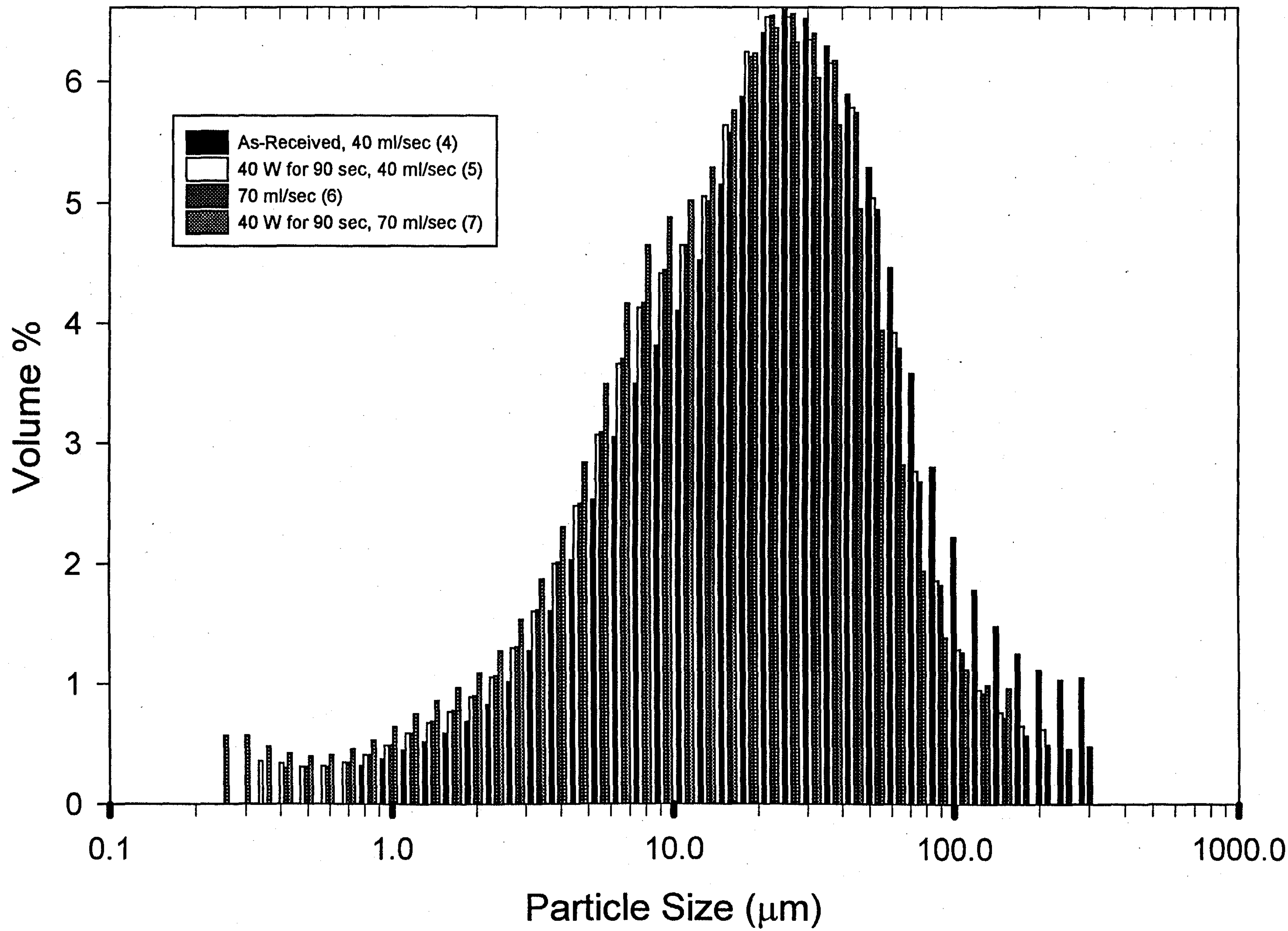
96-11 L Volume Distribution Data

Sample was analyzed at 60 ml/sec (1), then sonicated at 25W for 120 seconds and analyzed at 60 ml/sec (2), then sonicated at 40 W for 300 sec and analyzed at 60 ml/sec (3).
New sample was installed and analyzed at 40 ml/sec (4), then sonicated at 40W for 90 sec and analyzed at 40 ml/sec (5), then flow rate was increased and analyzed at 70 ml/sec (6), then sonicated at 40 W for 90 sec and analyzed at 70 ml/sec (7).

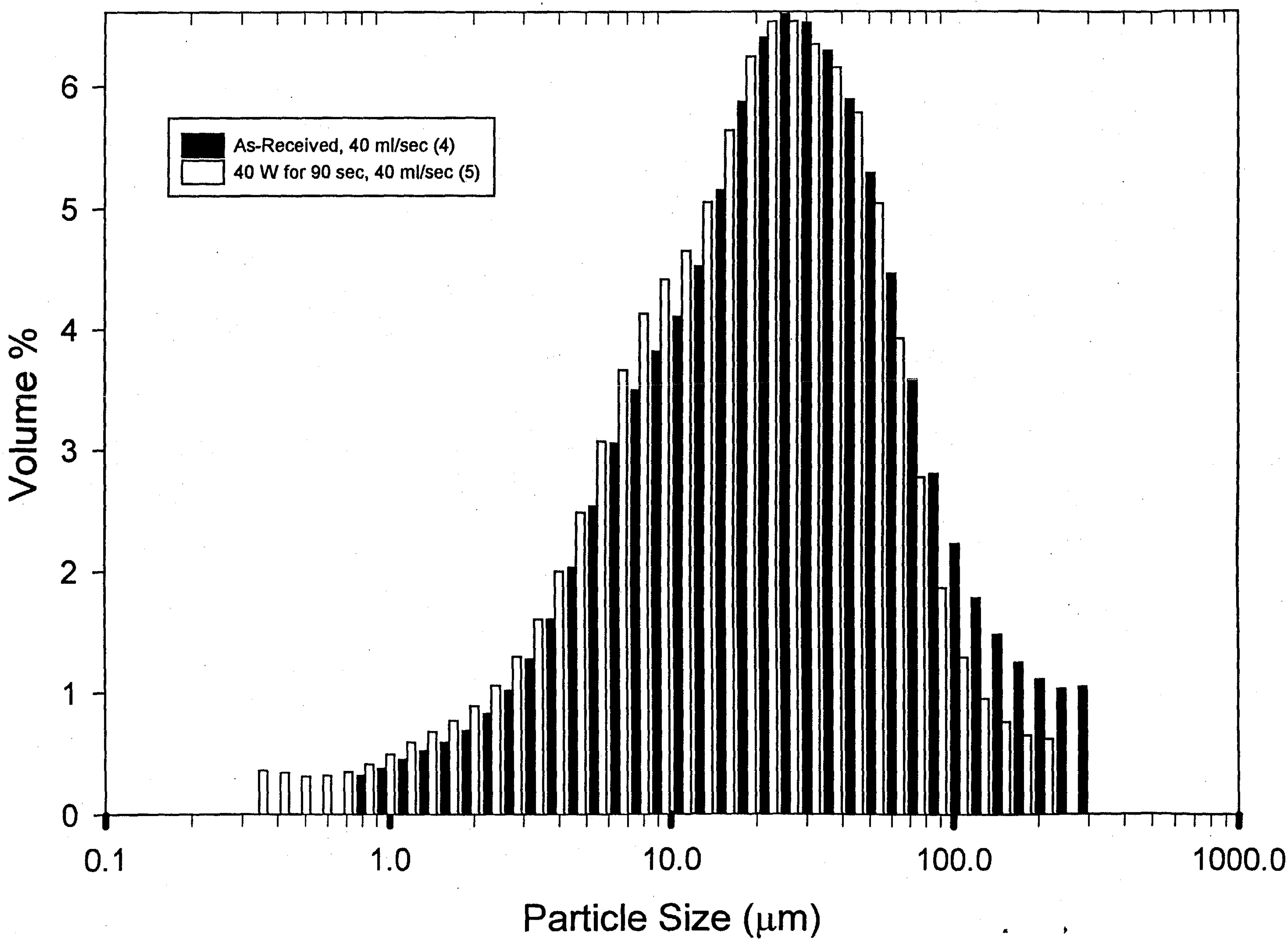
	1	2	3	4	5	6	7
	As-received	25 W	40 W	As-received	40 W		40W
Particle size	60 ml/sec	60 ml/sec	60 ml/sec	40 ml/sec	40 ml/sec	70 ml/sec	70 ml/sec
704.000	0	0	0	0	0	0	0
592.000	0	0	0	0	0	0	0
497.800	0	0	0	0	0	0	0
418.600	0	0	0	0	0	0	0
352.000	0	0	0	0	0	0	0
296.000	0.81	0	0	1.05	0	0.48	0
248.900	0.64	0	0	1.03	0	0.46	0
209.300	0.71	0	0	1.11	0.62	0.49	0
176.000	0.78	0	0	1.25	0.65	0.57	0
148.000	0.77	0	0	1.48	0.76	0.71	0.96
124.500	0.74	0	0	1.78	0.95	0.92	0.99
104.700	0.79	0.63	0	2.22	1.29	1.26	1.12
88.000	1	0.75	0	2.8	1.86	1.82	1.39
74.000	1.45	1.05	0.83	3.58	2.77	2.68	1.94
62.230	2.18	1.54	1.14	4.46	3.92	3.79	2.82
52.330	3.08	2.17	1.58	5.29	5.04	4.94	3.94
44.000	3.88	2.76	1.94	5.89	5.78	5.74	4.95
37.000	4.46	3.25	2.15	6.29	6.15	6.17	5.64
31.110	4.96	3.76	2.35	6.52	6.34	6.39	6.03
26.160	5.6	4.49	2.83	6.61	6.53	6.56	6.32
22.000	6.29	5.47	3.79	6.4	6.53	6.54	6.44
18.500	6.78	6.5	5.25	5.87	6.24	6.2	6.23
15.560	6.79	7.19	6.75	5.15	5.64	5.58	5.76
13.080	6.5	7.42	7.72	4.52	5.05	5.01	5.29
11.000	6.19	7.23	7.86	4.1	4.65	4.65	5.02
9.250	5.91	6.82	7.57	3.81	4.41	4.44	4.88
7.778	5.48	6.17	6.83	3.49	4.13	4.17	4.65
6.541	4.78	5.3	5.92	3.05	3.66	3.7	4.16
5.500	3.91	4.4	5.02	2.53	3.07	3.09	3.49
4.625	3.1	3.62	4.25	2.03	2.48	2.5	2.84
3.889	2.46	2.98	3.6	1.61	2	2.01	2.3
3.270	1.96	2.44	3.01	1.28	1.61	1.62	1.87
2.750	1.58	1.98	2.49	1.02	1.3	1.31	1.54
2.312	1.28	1.62	2.09	0.83	1.06	1.07	1.28
1.945	1.08	1.37	1.84	0.69	0.89	0.9	1.09
1.635	0.93	1.2	1.7	0.59	0.77	0.78	0.97
1.375	0.81	1.08	1.58	0.52	0.68	0.69	0.86
1.156	0.68	0.94	1.41	0.45	0.59	0.59	0.75
0.972	0.55	0.78	1.18	0.38	0.49	0.49	0.64
0.818	0.43	0.64	0.96	0.32	0.41	0.41	0.53
0.688	0.35	0.53	0.8	0	0.35	0.35	0.46
0.578	0.31	0.47	0.71	0	0.32	0.31	0.41
0.486	0	0.45	0.68	0	0.31	0.31	0.4
0.409	0	0.48	0.7	0	0.34	0.3	0.42
0.344	0	0.54	0.79	0	0.36	0	0.48
0.289	0	0.64	0.9	0	0	0	0.57
0.243	0	0.69	0.94	0	0	0	0.57
0.204	0	0.65	0.84	0	0	0	0
0.172	0	0	0	0	0	0	0
0.145	0	0	0	0	0	0	0

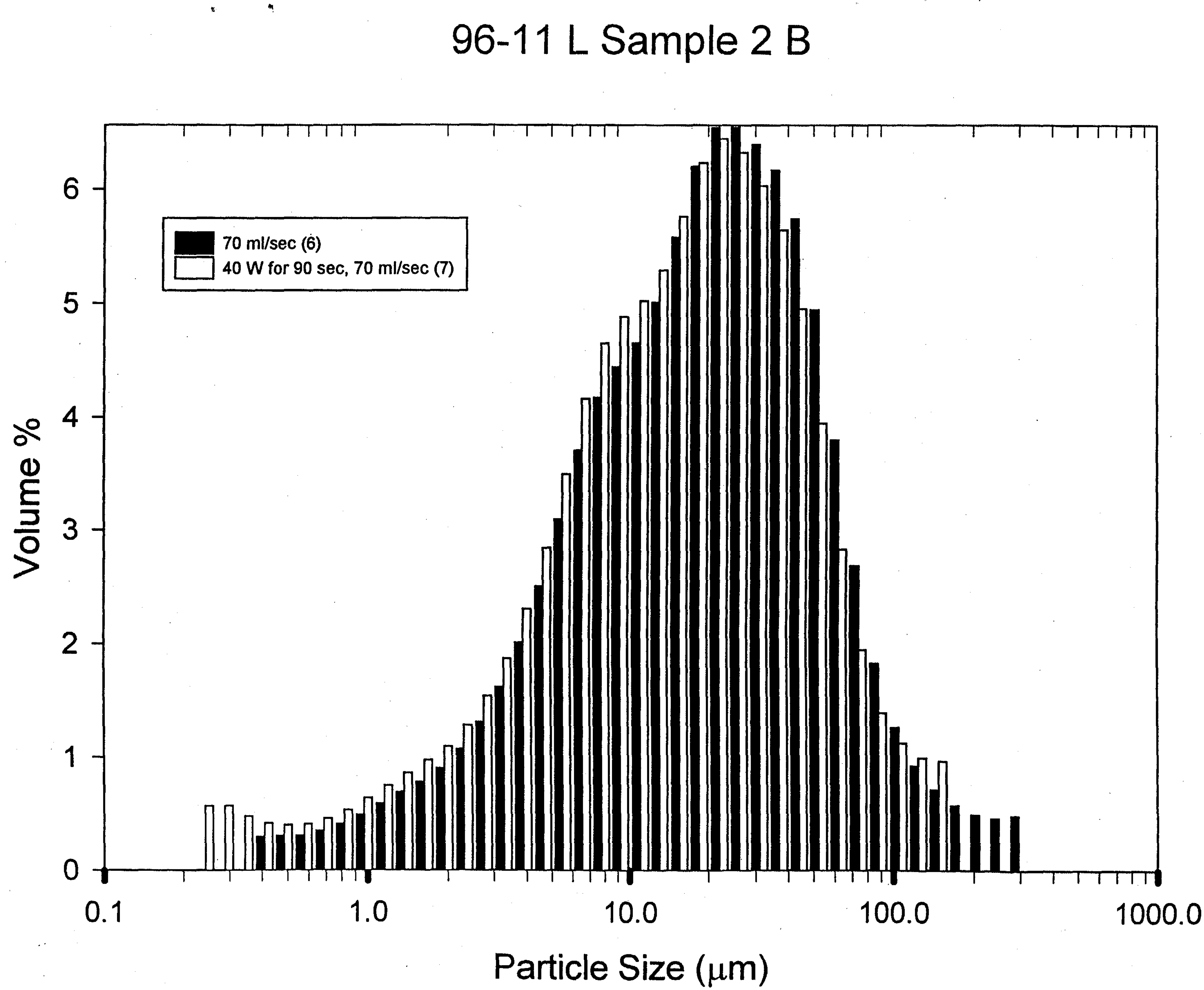

96-11 L Sample 1
As-received, 60 ml/sec (1)
25 W for 125 sec, 60 ml/min (2)
40 W for 300 seconds, 60 ml/sec (3)
Volume %
0
1
2
3
4
5
6
7
0.1
1.0
10.0
100.0
1000.0
Particle Size (μm)

96-11 L Sample 2



96-11 L Sample 2 A




96-11 L Sample 2 B
70 ml/sec (6)
40 W for 90 sec, 70 ml/sec (7)
Volume %
0
1
2
3
4
5
6
0.1
1.0
10.0
100.0
1000.0
Particle Size (µm)

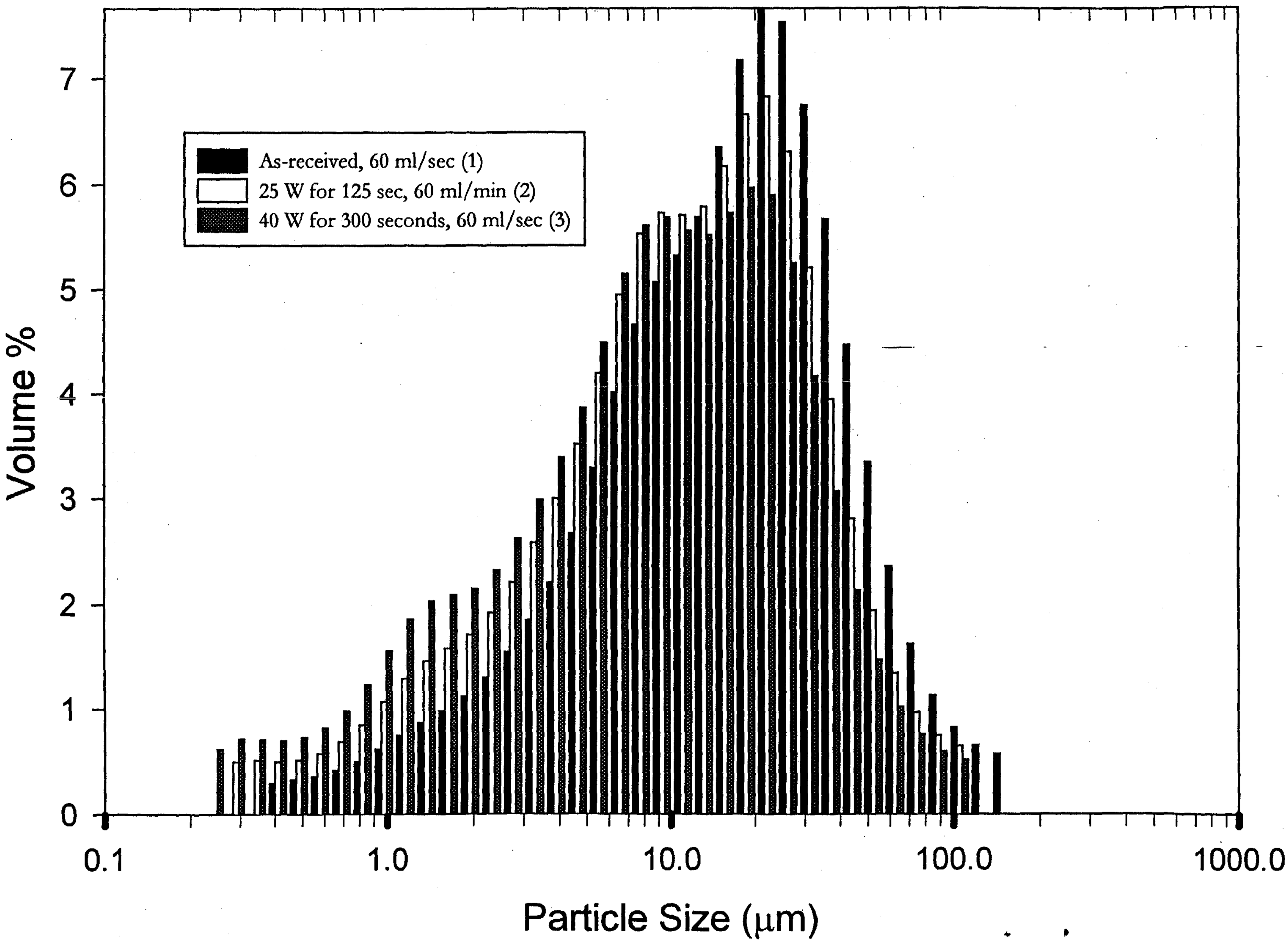
KES-M-13 top   Volume Distribution Data

Sample was analyzed at 60 ml/sec (1), then sonicated at 25W for 120 seconds and analyzed at 60 ml/sec (2), then sonicated at 40 W for 300 sec and analyzed at 60 ml/sec (3).

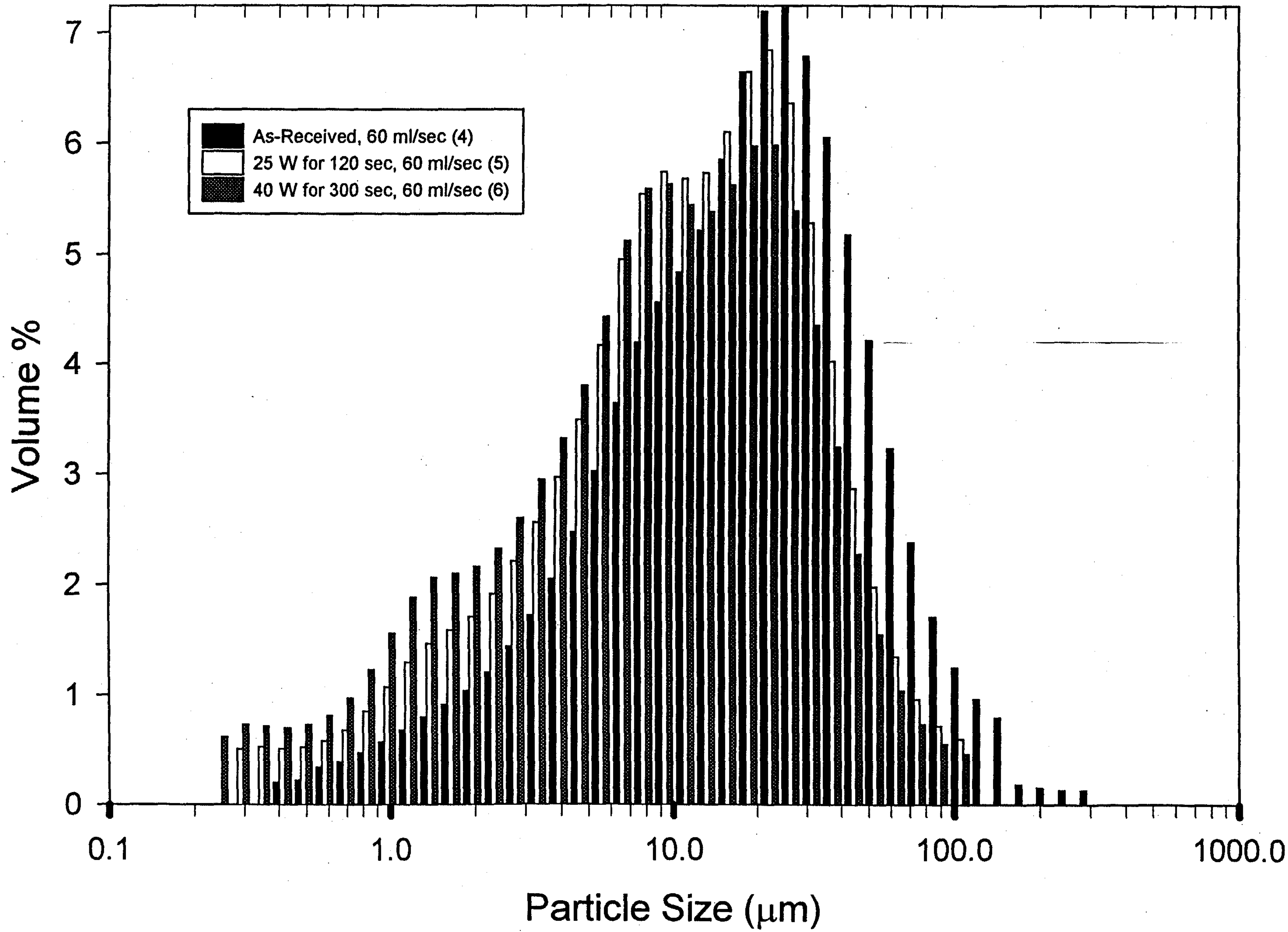
New sample was installed and analyzed at 60 ml/sec (4), then sonicated at 25W for 120 seconds and analyzed at 60 ml/sec (5), then sonicated at 40 W for 300 sec and analyzed at 60 ml/sec (6).

	1	2	3	4	5	6
	As-received	25 W	40 W	As-received	25 W	40 W
Particle size	60 ml/sec	60 ml/sec	60 ml/sec	60 ml/sec	60 ml/sec	60 ml/sec
704.000	0	0	0	0	0	0
592.000	0	0	0	0	0	0
497.800	0	0	0	0	0	0
418.600	0	0	0	0	0	0
352.000	0	0	0	0	0	0
296.000	0	0	0	0.13	0	0
248.900	0	0	0	0.13	0	0
209.300	0	0	0	0.15	0	0
176.000	0	0	0	0.18	0	0
148.000	0.58	0	0	0.79	0	0
124.500	0.66	0	0	0.96	0	0
104.700	0.83	0.65	0.52	1.25	0.6	0.46
88.000	1.13	0.75	0.6	1.71	0.72	0.55
74.000	1.62	0.97	0.76	2.38	0.96	0.73
62.230	2.36	1.34	1.02	3.23	1.35	1.04
52.330	3.35	1.94	1.47	4.21	1.98	1.55
44.000	4.47	2.81	2.13	5.17	2.87	2.27
37.000	5.67	3.95	3.07	6.05	4.02	3.24
31.110	6.75	5.21	4.17	6.78	5.28	4.35
26.160	7.53	6.31	5.25	7.24	6.36	5.39
22.000	7.66	6.83	5.9	7.19	6.84	5.98
18.500	7.17	6.66	5.97	6.64	6.64	5.97
15.560	6.35	6.17	5.73	5.85	6.1	5.62
13.080	5.69	5.79	5.52	5.21	5.73	5.38
11.000	5.32	5.71	5.56	4.83	5.68	5.44
9.250	5.07	5.73	5.69	4.56	5.74	5.63
7.778	4.66	5.53	5.61	4.19	5.54	5.59
6.541	4.01	4.95	5.15	3.64	4.95	5.12
5.500	3.29	4.2	4.49	3.02	4.17	4.43
4.625	2.67	3.52	3.87	2.47	3.49	3.8
3.889	2.21	3	3.39	2.05	2.97	3.32
3.270	1.85	2.58	2.99	1.72	2.56	2.95
2.750	1.55	2.21	2.62	1.44	2.21	2.6
2.312	1.3	1.92	2.32	1.21	1.92	2.32
1.945	1.12	1.71	2.15	1.04	1.71	2.16
1.635	0.98	1.58	2.09	0.91	1.59	2.1
1.375	0.87	1.46	2.03	0.8	1.47	2.06
1.156	0.75	1.29	1.86	0.68	1.3	1.88
0.972	0.62	1.07	1.56	0.57	1.07	1.56
0.818	0.5	0.85	1.23	0.47	0.85	1.23
0.688	0.42	0.69	0.98	0.39	0.68	0.97
0.578	0.36	0.58	0.82	0.34	0.58	0.81
0.486	0.33	0.52	0.73	0.22	0.52	0.73
0.409	0.3	0.5	0.7	0.2	0.51	0.7
0.344	0	0.52	0.71	0	0.53	0.72
0.289	0	0.5	0.72	0	0.51	0.73
0.243	0	0	0.62	0	0	0.62
0.204	0	0	0	0	0	0
0.172	0	0	0	0	0	0
0.145	0	0	0	0	0	0

KES-M-13 Top Sample 1



KES-M-13 Top Sample 2



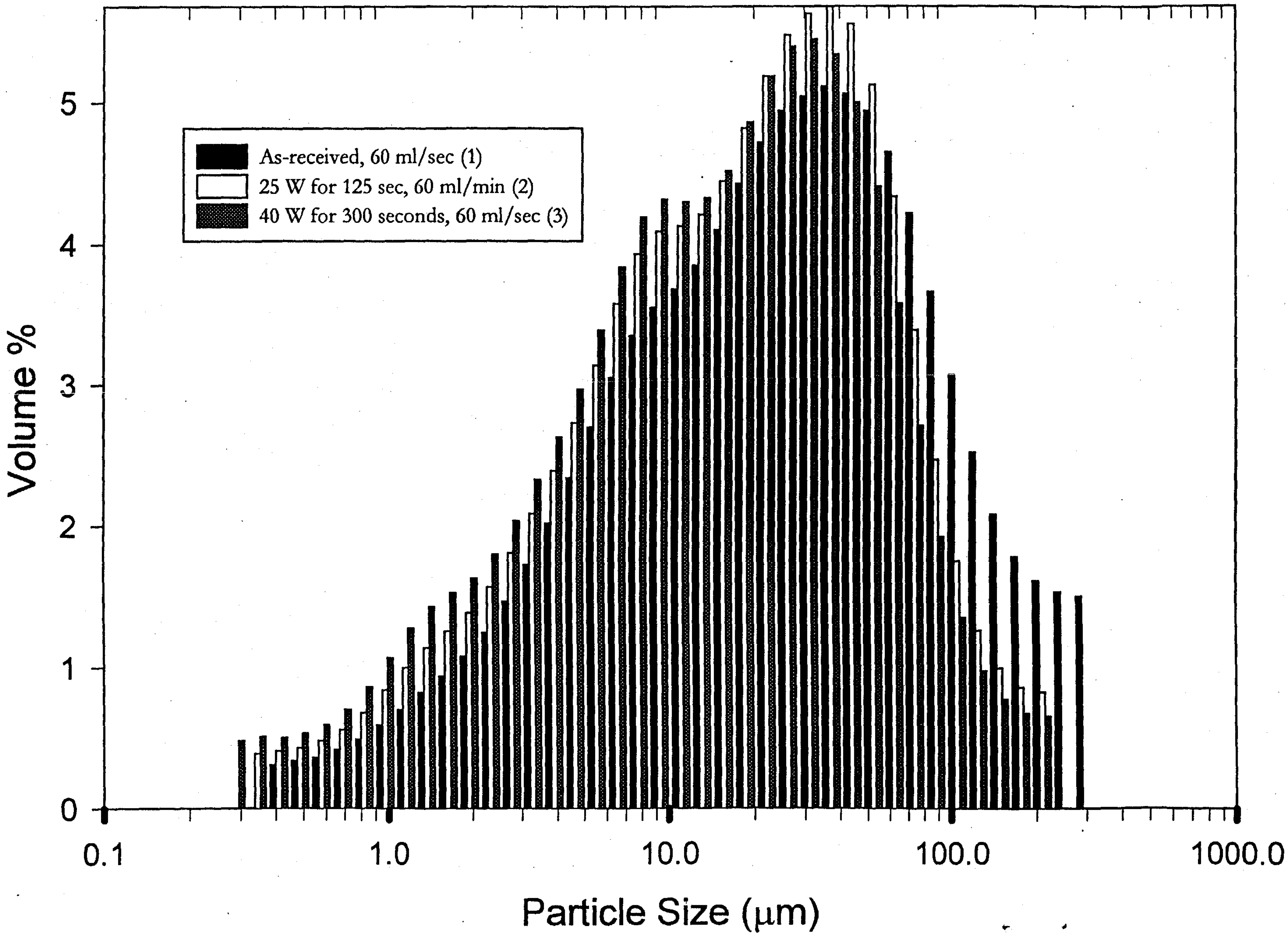
KES-M-13 Bottom Volume Distribution Data

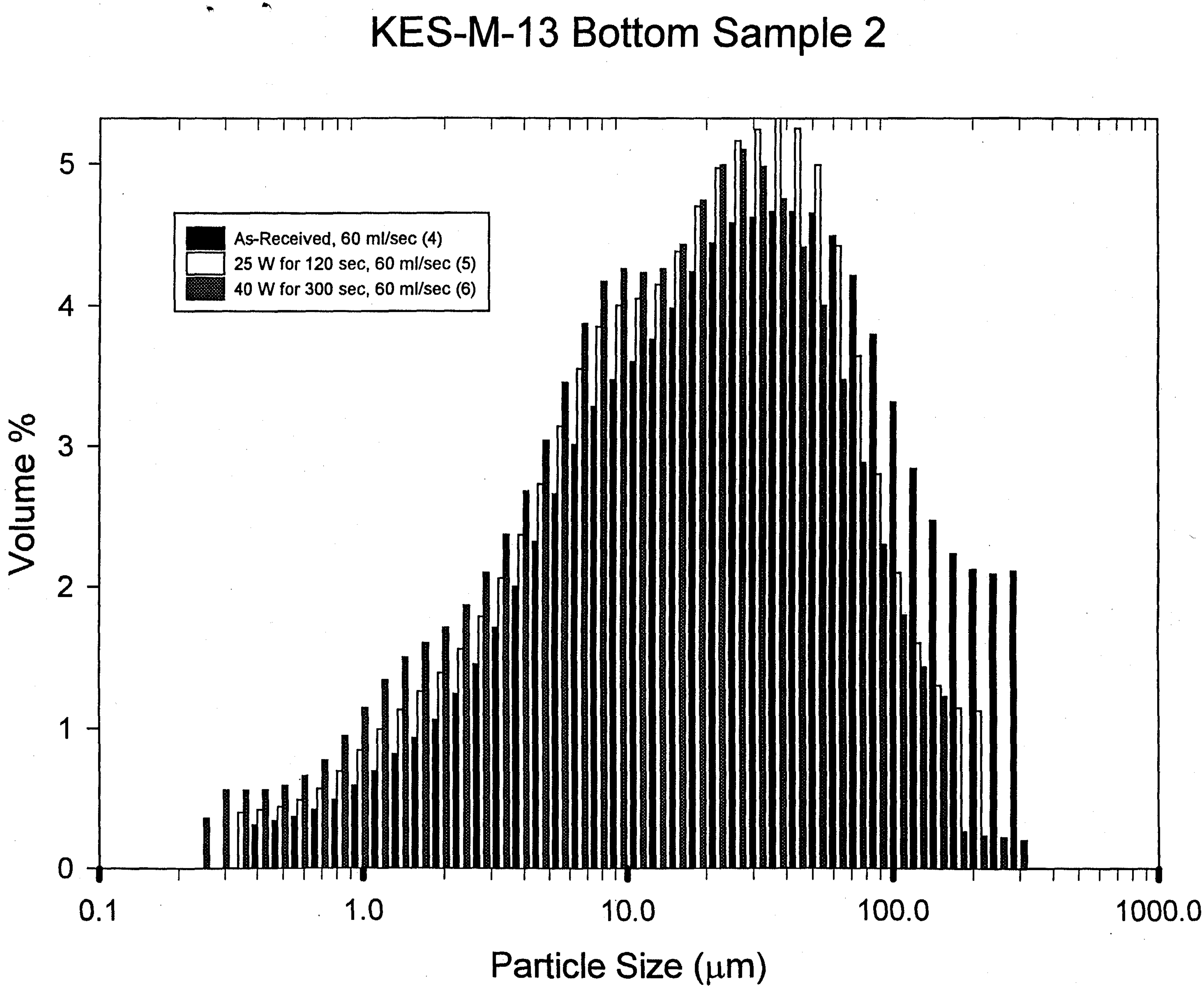
Sample was analyzed at 60 ml/sec (1), then sonicated at 25W for 120 seconds and analyzed at 60 ml/sec (2), then sonicated at 40 W for 300 sec and analyzed at 60 ml/sec (3).

New sample was installed and analyzed at 60 ml/sec (4), then sonicated at 25W for 120 seconds and analyzed at 60 ml/sec (5), then sonicated at 40 W for 300 sec and analyzed at 60 ml/sec (6).

	1	2	3	4	5	6
	As-received	25 W	40 W	As-receive	25 W	40 W
Particle size	60 ml/sec	60 ml/sec	60 ml/sec	60 ml/sec	60 ml/sec	60 ml/sec
704.000	0	0	0	0	0	0
592.000	0	0	0	0	0	0
497.800	0	0	0	0	0	0
418.600	0	0	0	0	0	0
352.000	0	0	0	0	0	0
296.000	1.5	0	0	2.11	0	0.2
248.900	1.53	0	0	2.09	0	0.22
209.300	1.61	0.82	0.65	2.12	1.12	0.23
176.000	1.78	0.85	0.67	2.23	1.14	0.26
148.000	2.08	0.99	0.77	2.47	1.3	1.22
124.500	2.52	1.26	0.97	2.84	1.6	1.43
104.700	3.07	1.75	1.35	3.31	2.1	1.8
88.000	3.66	2.47	1.92	3.79	2.8	2.3
74.000	4.22	3.39	2.71	4.21	3.64	2.88
62.230	4.65	4.34	3.58	4.49	4.42	3.47
52.330	4.94	5.12	4.41	4.65	4.99	4
44.000	5.06	5.55	5	4.66	5.25	4.41
37.000	5.11	5.67	5.33	4.66	5.32	4.75
31.110	5.04	5.62	5.44	4.62	5.24	4.98
26.160	4.94	5.47	5.39	4.58	5.16	5.1
22.000	4.72	5.18	5.18	4.44	4.97	4.99
18.500	4.43	4.82	4.86	4.24	4.7	4.74
15.560	4.1	4.45	4.52	3.98	4.38	4.43
13.080	3.85	4.21	4.33	3.76	4.15	4.26
11.000	3.68	4.13	4.3	3.6	4.05	4.23
9.250	3.55	4.09	4.32	3.47	4	4.26
7.778	3.35	3.93	4.19	3.28	3.85	4.17
6.541	3.05	3.58	3.84	3.01	3.55	3.87
5.500	2.7	3.14	3.39	2.66	3.14	3.45
4.625	2.34	2.73	2.97	2.32	2.73	3.04
3.889	2.02	2.39	2.63	2	2.37	2.68
3.270	1.73	2.09	2.33	1.71	2.06	2.37
2.750	1.47	1.81	2.04	1.45	1.79	2.1
2.312	1.25	1.57	1.8	1.24	1.56	1.87
1.945	1.08	1.39	1.63	1.06	1.39	1.71
1.635	0.94	1.26	1.53	0.93	1.26	1.6
1.375	0.82	1.14	1.43	0.81	1.13	1.5
1.156	0.7	1	1.28	0.69	0.99	1.34
0.972	0.59	0.84	1.07	0.59	0.84	1.14
0.818	0.49	0.68	0.86	0.49	0.69	0.94
0.688	0.42	0.56	0.7	0.42	0.57	0.77
0.578	0.36	0.48	0.59	0.37	0.49	0.66
0.486	0.34	0.43	0.53	0.34	0.44	0.59
0.409	0.31	0.41	0.5	0.31	0.42	0.56
0.344	0	0.39	0.51	0	0.4	0.56
0.289	0	0	0.48	0	0	0.56
0.243	0	0	0	0	0	0.36
0.204	0	0	0	0	0	0
0.172	0	0	0	0	0	0
0.145	0	0	0	0	0	0

KES-M-13 Bottom Sample 1



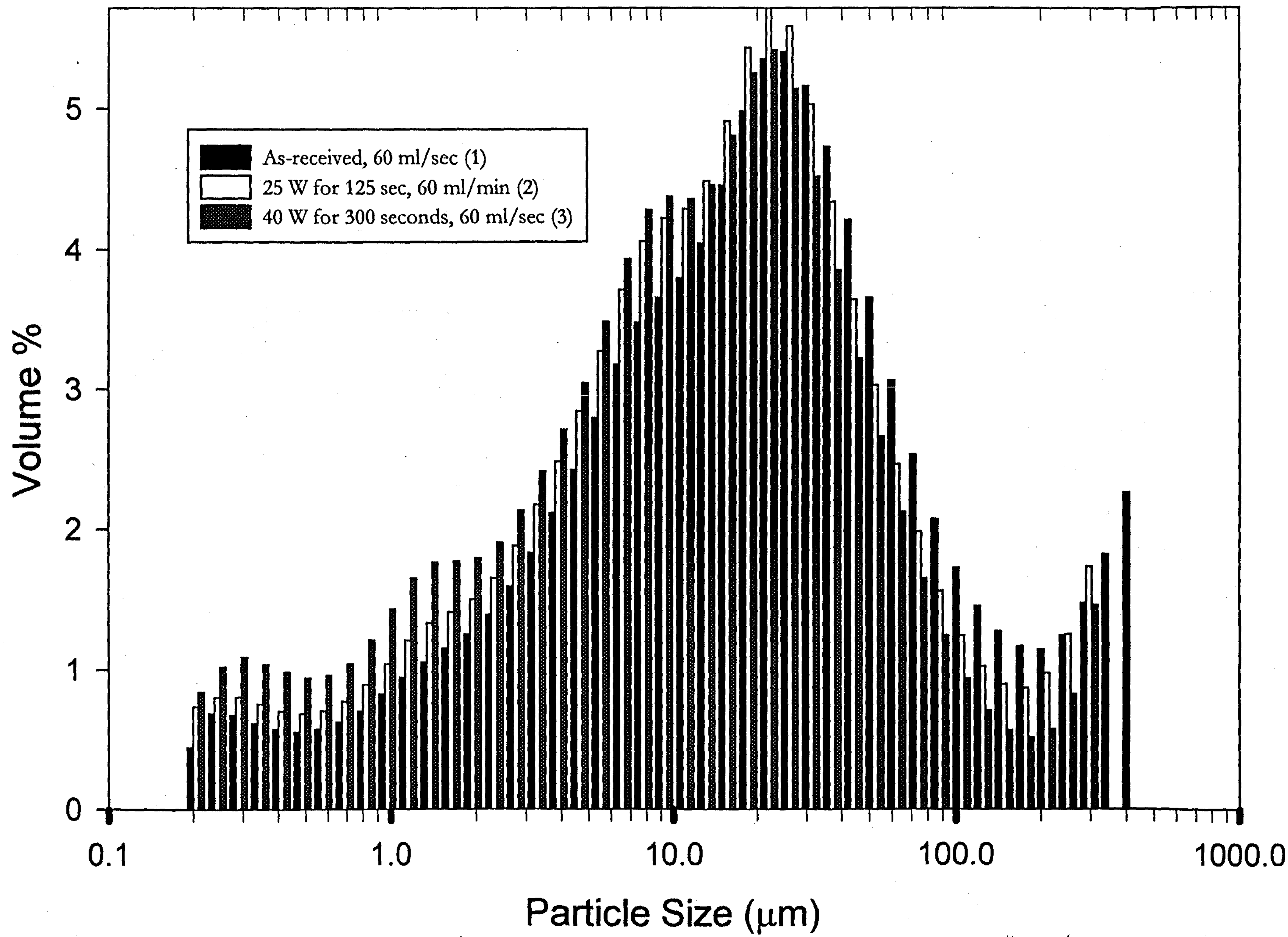

KES-M-13 Bottom Sample 2
As-Received, 60 ml/sec (4)
25 W for 120 sec, 60 ml/sec (5)
40 W for 300 sec, 60 ml/sec (6)
Volume %
0
1
2
3
4
5
0.1
1.0
10.0
100.0
1000.0
Particle Size (μm)

KES-T-20 top Volume Distribution Data

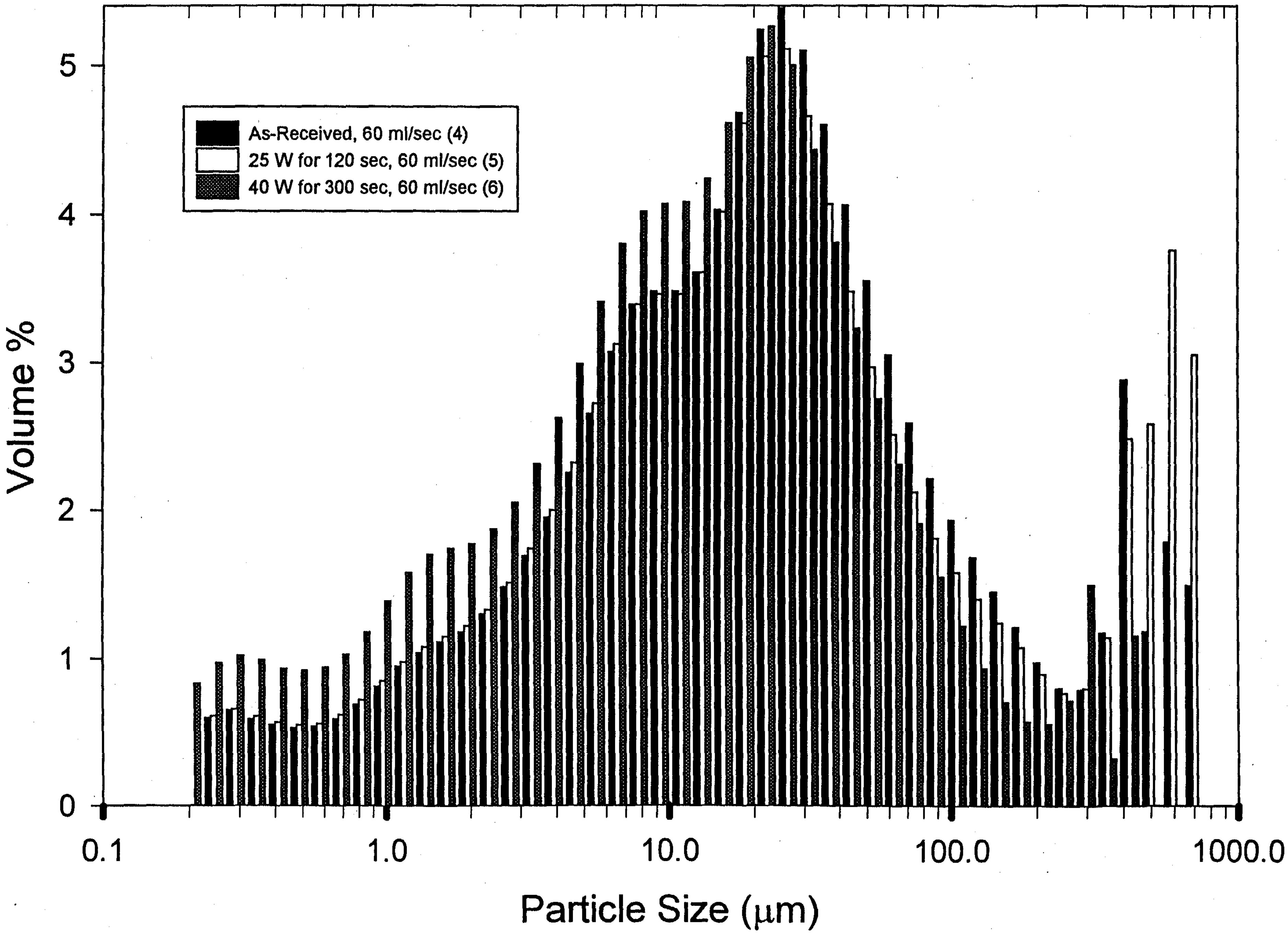
Sample was analyzed at 60 ml/sec (1), then sonicated at 25W for 120 seconds and analyzed at 60 ml/sec (2), then sonicated at 40 W for 300 sec and analyzed at 60 ml/sec (3).
New sample was installed and analyzed at 60 ml/sec (4), then sonicated at 25W for 120 seconds and analyzed at 60 ml/sec (5), then sonicated at 40 W for 300 sec and analyzed at 60 ml/sec (6).

	1	2	3	4	5	6
	As-received	25 W	40 W	As-received	25 W	40 W
Particle size	60 ml/sec	60 ml/sec	60 ml/sec	60 ml/sec	60 ml/sec	60 ml/sec
704.000	0	0	0	1.49	3.05	0
592.000	0	0	0	1.78	3.76	0
497.800	0	0	0	1.18	2.58	0
418.600	2.26	0	0	2.88	2.48	1.15
352.000	1.82	0	0	1.17	1.14	0.32
296.000	1.47	1.73	1.46	0.78	0.79	1.49
248.900	1.24	1.25	0.82	0.79	0.76	0.71
209.300	1.14	0.97	0.57	0.97	0.89	0.55
176.000	1.16	0.86	0.51	1.21	1.07	0.57
148.000	1.27	0.89	0.56	1.45	1.24	0.7
124.500	1.45	1.02	0.7	1.68	1.4	0.93
104.700	1.72	1.24	0.93	1.93	1.58	1.22
88.000	2.07	1.56	1.24	2.21	1.81	1.55
74.000	2.53	1.98	1.65	2.59	2.12	1.91
62.230	3.06	2.46	2.12	3.05	2.51	2.31
52.330	3.65	3.03	2.66	3.55	2.97	2.75
44.000	4.21	3.64	3.22	4.06	3.48	3.23
37.000	4.73	4.34	3.85	4.6	4.07	3.81
31.110	5.16	5.03	4.52	5.1	4.66	4.43
26.160	5.4	5.58	5.14	5.4	5.11	5
22.000	5.35	5.71	5.41	5.24	5.06	5.26
18.500	4.98	5.43	5.25	4.68	4.61	5.05
15.560	4.46	4.91	4.81	4.03	4.02	4.61
13.080	4.04	4.49	4.46	3.61	3.61	4.24
11.000	3.79	4.29	4.36	3.48	3.46	4.08
9.250	3.65	4.22	4.38	3.48	3.46	4.07
7.778	3.47	4.06	4.28	3.39	3.39	4.02
6.541	3.17	3.71	3.93	3.07	3.12	3.8
5.500	2.79	3.27	3.48	2.65	2.72	3.41
4.625	2.42	2.84	3.04	2.25	2.32	2.99
3.889	2.11	2.48	2.71	1.95	2	2.62
3.270	1.83	2.17	2.41	1.69	1.74	2.31
2.750	1.59	1.88	2.13	1.48	1.51	2.05
2.312	1.39	1.65	1.9	1.3	1.33	1.87
1.945	1.25	1.5	1.79	1.18	1.22	1.77
1.635	1.15	1.41	1.77	1.11	1.15	1.74
1.375	1.05	1.33	1.76	1.04	1.08	1.7
1.156	0.94	1.21	1.65	0.95	0.98	1.58
0.972	0.82	1.04	1.43	0.81	0.85	1.39
0.818	0.7	0.89	1.21	0.69	0.72	1.18
0.688	0.62	0.77	1.04	0.59	0.62	1.03
0.578	0.57	0.7	0.96	0.54	0.56	0.94
0.486	0.55	0.68	0.94	0.53	0.55	0.92
0.409	0.57	0.7	0.98	0.55	0.57	0.93
0.344	0.61	0.75	1.04	0.59	0.61	0.99
0.289	0.67	0.8	1.09	0.65	0.66	1.02
0.243	0.68	0.8	1.02	0.6	0.61	0.97
0.204	0.44	0.73	0.84	0	0	0.83
0.172	0	0	0	0	0	0
0.145	0	0	0	0	0	0

KES-T-20 Top Sample 1



KES-T-20 Top Sample 2



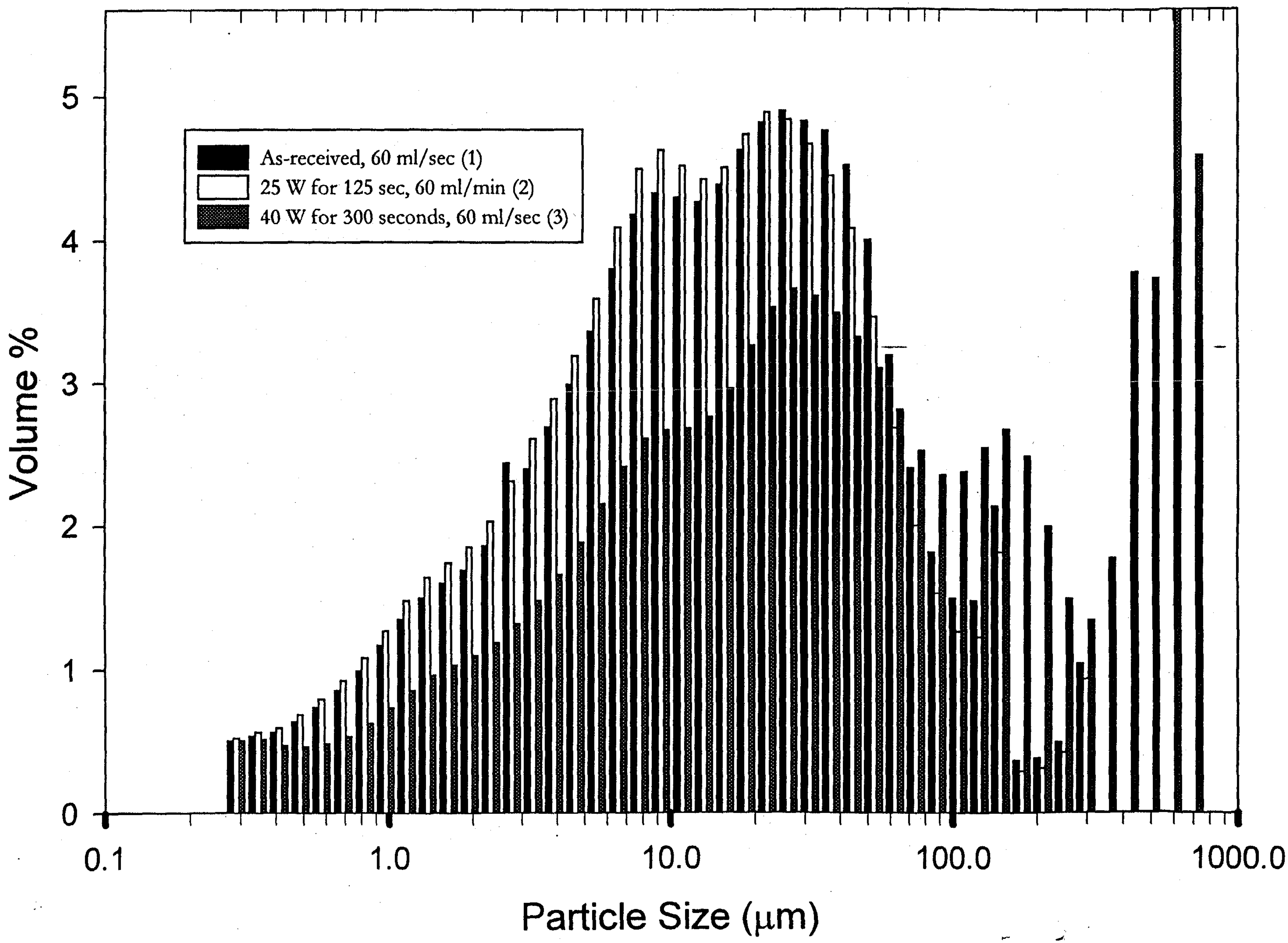
KES-T-20 Bottom   Volume Distribution Data

Sample was analyzed at 60 ml/sec (1), then sonicated at 25W for 120 seconds and analyzed at 60 ml/sec (2).

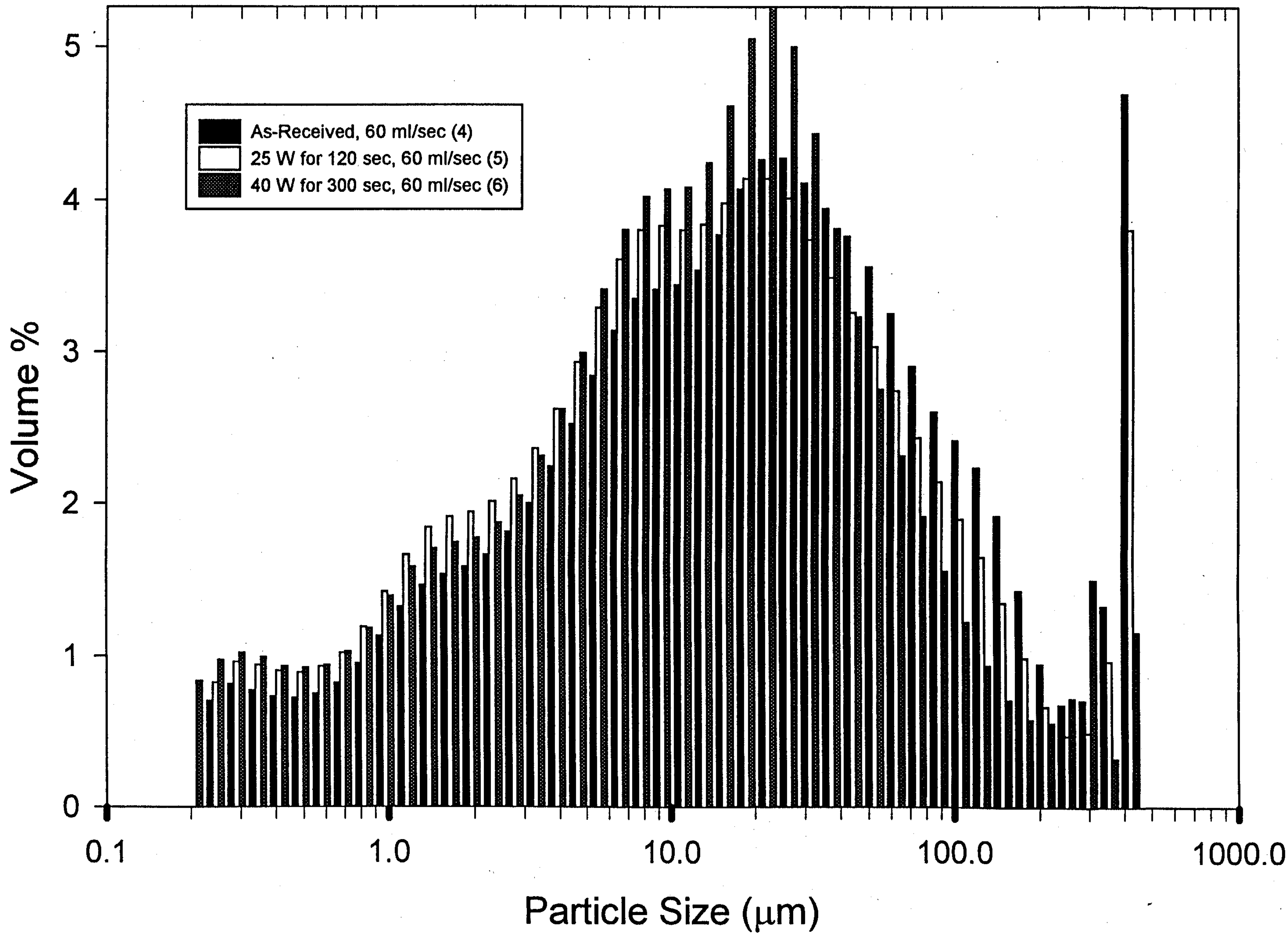
New sample was installed and analyzed at 60 ml/sec (3), then sonicated at 25W for 120 seconds and analyzed at 60 ml/sec (4).

Particle size	1 As-received 60 ml/sec	2 25 W 60 ml/sec	3 As-received 60 ml/sec	4 25 W 60 ml/sec	6 40 W 60 ml/sec
704.000	0	0	4.59	0	0
592.000	0	0	5.6	0	0
497.800	0	0	3.73	0	0
418.600	0	0	3.77	4.69	3.8
352.000	0	0	1.77	1.32	0.96
296.000	1.04	0.93	1.34	0.7	0.49
248.900	0.49	0.42	1.49	0.67	0.47
209.300	0.38	0.31	1.99	0.94	0.66
176.000	0.36	0.29	2.48	1.42	0.98
148.000	2.13	1.81	2.67	1.91	1.34
124.500	1.47	1.22	2.54	2.23	1.64
104.700	1.49	1.26	2.37	2.41	1.89
88.000	1.81	1.53	2.35	2.6	2.14
74.000	2.4	2	2.52	2.9	2.43
62.230	3.19	2.68	2.81	3.25	2.74
52.330	4	3.46	3.1	3.56	3.03
44.000	4.52	4.08	3.32	3.76	3.26
37.000	4.76	4.45	3.49	3.94	3.49
31.110	4.83	4.67	3.61	4.11	3.74
26.160	4.9	4.84	3.66	4.27	4.01
22.000	4.82	4.89	3.53	4.26	4.14
18.500	4.63	4.74	3.26	4.07	4.14
15.560	4.39	4.51	2.96	3.77	3.98
13.080	4.27	4.43	2.76	3.54	3.84
11.000	4.3	4.52	2.68	3.44	3.8
9.250	4.33	4.63	2.67	3.41	3.83
7.778	4.18	4.5	2.61	3.35	3.8
6.541	3.8	4.09	2.41	3.14	3.61
5.500	3.36	3.59	2.15	2.84	3.29
4.625	2.99	3.19	1.88	2.52	2.93
3.889	2.69	2.89	1.66	2.24	2.62
3.270	2.4	2.61	1.48	2	2.36
2.750	2.44	2.31	1.32	1.81	2.16
2.312	1.86	2.03	1.19	1.66	2.01
1.945	1.69	1.85	1.1	1.58	1.94
1.635	1.6	1.74	1.03	1.53	1.91
1.375	1.5	1.64	0.96	1.46	1.84
1.156	1.35	1.48	0.85	1.32	1.66
0.972	1.17	1.27	0.73	1.13	1.42
0.818	0.99	1.08	0.62	0.95	1.19
0.688	0.85	0.92	0.53	0.82	1.02
0.578	0.73	0.79	0.48	0.75	0.93
0.486	0.63	0.68	0.46	0.72	0.89
0.409	0.56	0.59	0.47	0.73	0.9
0.344	0.53	0.56	0.51	0.77	0.94
0.289	0.5	0.52	0.5	0.81	0.96
0.243	0	0	0	0.7	0.82
0.204	0	0	0	0	0
0.172	0	0	0	0	0
0.145	0	0	0	0	0

KES-T-20 Bottom Sample 1



KES-T-20 Bottom Sample 2



NRW 100
Volume Distribution Data

	1
	As-received
Particle size	60 ml/sec
704.000	94.58
592.000	5.42
497.800	0
418.600	0
352.000	0
296.000	0
248.900	0
209.300	0
176.000	0
148.000	0
124.500	0
104.700	0
88.000	0
74.000	0
62.230	0
52.330	0
44.000	0
37.000	0
31.110	0
26.160	0
22.000	0
18.500	0
15.560	0
13.080	0
11.000	0
9.250	0
7.778	0
6.541	0
5.500	0
4.625	0
3.889	0
3.270	0
2.750	0
2.312	0
1.945	0
1.635	0
1.375	0
1.156	0
0.972	0
0.818	0
0.688	0
0.578	0
0.486	0
0.409	0
0.344	0
0.289	0
0.243	0
0.204	0
0.172	0
0.145	0

NRW 400

Volume Distribution Data

Particle size	1 As-received 60 ml/sec
704.000	93.58
592.000	6.42
497.800	0
418.600	0
352.000	0
296.000	0
248.900	0
209.300	0
176.000	0
148.000	0
124.500	0
104.700	0
88.000	0
74.000	0
62.230	0
52.330	0
44.000	0
37.000	0
31.110	0
26.160	0
22.000	0
18.500	0
15.560	0
13.080	0
11.000	0
9.250	0
7.778	0
6.541	0
5.500	0
4.625	0
3.889	0
3.270	0
2.750	0
2.312	0
1.945	0
1.635	0
1.375	0
1.156	0
0.972	0
0.818	0
0.688	0
0.578	0
0.486	0
0.409	0
0.344	0
0.289	0
0.243	0
0.204	0
0.172	0
0.145	0