Pacific Northwest National Laboratory
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January 27, 2000

Mr. Thomas H. May
ASTD Project Manager
CH2M Hill
MSIN: R2-11, 2750E/A215/200E
Richland, WA 99352

RE: REISSUAL OF THE REPORT "QUALIFIACTION OF THE LASENTEC M600P PARTICLE SIZE ANALYZER AND THE RED VALVE MODEL 1151 PRESSURE SENSOR"

Dear Mr. May:

Attached is the PNNL report entitled "Qualification of the Lasentec M600P Particle Size Analyzer and the Red Valve Model 1151 Pressure Sensor". The report is being reissued after the earlier version has been recalled due to numerous printing errors, which were realized after mailing.

If you have any questions, please do not hesitate to contact me.

Sincerely

B. Jagannadha Rao

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Project Manager

encl:

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PNNL-13064

Pacific Northwest
National Laboratory
Operated by Battelle for the
U.S. Department of Energy

Qualification of the Lasentec M600P Particle Size Analyzer and the Red Valve Model 1151 Pressure Sensor

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CH2M Hill, Richland, Washington
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Prepared for the U.S. Department of Energy
under Contract DE-AC06-76RLO 1830.

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Abstract

The Lasentec M600 in-line particle size analyzer was installed at Oak Ridge National Laboratory (ORNL) in August 1998 to support retrieval of the Gunite and Associated Tanks (GAAT). Before installation at ORNL, the sensor underwent validation testing at the Pacific Northwest National Laboratory (PNNL) Instrument Validation facility. Mechanically, the instrument worked well during validation testing and met all expectations. Operationally, much was learned about optimum ways to display and interpret the data. Slurry samples taken during the in-line tests at PNNL were shipped to the vendor for analysis with a benchtop Lasentec sensor. These experiments were performed to determine if off-line analyses yield particle size distributions similar to those generated by the in-line sensor. It was determined that the Lasentec sensor measures repeatable chord lengths as long as particles are "presented" to the sensor window the same way. After the initial non-radioactive simulant testing at PNNL, the instrument was shipped for radioactive validation and acceptance testing in the Slurry Monitoring Test System (SMTS) connected to the Tank W-9 of the GAATs at ORNL. For all acceptance tests conducted at ORNL, the variation in the chord length distribution and the total particle count corresponded very well with the slurry density data as determined using an in-line Promass 63M Coriolis meter. Based on the performance results obtained, the Lasentec M600P FBRM is expected to meet the requirements for measuring the particle size distribution during the slurry transfer operations at Hanford and the Oak Ridge GAAT remediation project.

The Red Valve pressure sensor was endorsed at the Hanford Site following instrument validation tests at PNNL and is currently in operation in the Tank 241-C-106 pump pit. While this instrument measures pressure within a transfer line, this type of pressure sensor could be configured to measure pressure drop over time. In turn, the status of a slurry transfer could be inferred from the pressure-drop measurement. In 1998, four Red Valve pressure sensors (with Sensotech Model AE-213 pressure transducers) were installed before and after the booster pumps of the 4-in. slurry (SL-200) and supernatant (SN-200) transfer lines between Tank 241-C-106 and Tank 241-AY-102. These pressure sensors have been in operation for over 1 year, and to date, the sensors have been trouble-free according to the operators involved with slurry and supernatant transfer operations. Based on these observations, it is apparent that the Red Valve pressure sensors could be installed at the end of the slurry transfer lines and used to measure the pressure drop in the system.

Nomenclature

ASTD	Accelerated Site Technology Deployment project
CERCLA	Comprehensive Environmental Response, Compensation, and Liability Act
DOE	U.S. Department of Energy
TDI	Technology Deployment Initiative
HTI	Hanford Tanks Initiative
FBRM	Focused Beam Reflectance Measurement
GAAT	Gunite and Associated Tanks
IVF	Instrumentation Validation Facility
MVST	Melton Valley Storage Tanks
ORNL	Oak Ridge National Laboratory
PNNL	Pacific Northwest National Laboratory
SMTS	Slurry Monitoring Test System

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1.0 Introduction

Remediation plans for most of the tank wastes stored at both the Hanford and Oak Ridge National Laboratory (ORNL) sites include retrieval operations to remove the wastes from storage tanks and transport operations to transfer the wastes to treatment facilities. Retrieval operations will involve mixing solid and liquid wastes to create slurries that can be transported via pipelines to specified locations. Sedimentation of solids and precipitation or gelation reactions during slurry transport could result in blocked pipelines. The economic penalties for pipeline blockages are steep; for example, the U.S. Department of Energy (DOE) may be required to pay as much as $2 million for each day that tank waste cannot be delivered to the privately owned vitrification facility at the Hanford Site. Additional information regarding waste issues is provided in Appendix A.

To reduce the likelihood of pipeline blockage during waste-transfer operations, the Accelerated Site Technology Deployment (ASTD) project,[a] with funding from Project W-320 (Waste Retrieval Sluicing System) and Hanford Tanks Initiative (HTI), is evaluating three online slurry monitoring devices for use at the Hanford and ORNL sites. These include: (1) the Lasentec M600P Particle Size Analyzer developed by Laser Sensor Technology, Inc., Redmond, WA,[b] (2) the Red Valve Pressure sensor manufactured by Red Valve Company, Inc., Pittsburgh, PA, and (3) a densimeter developed at the Pacific Northwest National Laboratory (PNNL). There are three phases of instrument evaluation: qualification, implementation, and deployment. Instruments are procured and/or fabricated, calibrated, and installed in their target system during the qualification phase. Additionally, at the end of this phase, the instruments undergo acceptance testing and are made available to operations. Instrument performance is documented and shared with all complex-wide slurry-transfer projects during the deployment phase. In the final implementation phase, the slurry monitors are intended to monitor waste slurries during tank waste retrieval operations.

This report documents the qualification phase for the Lasentec Particle Size Analyzer and the Red Valve Pressure Sensor while the scope of the ASTD Slurry Monitoring project includes qualifying the densimeter. Because testing is in progress, qualification of the densimeter will be documented in a separate report during FY 2000. A technical summary of the Lasentec particle size analyzer and Red Valve pressure-sensor qualification tests, along with recommendations, is presented in Section 2. Technical details are presented for the Lasentec in Section 3 and for the Red Valve Pressure Sensor in Section 4.

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[a] Further information about the Slurry Monitoring ASTD project can be found in the *Slurry Monitoring TDI Deployment Plan* (Fluor Daniel Hanford 1997) and the report entitled *Slurry Monitoring ASTD Project Supplemental Information* (Fluor Daniel Hanford 1998).

[b] The Lasentec M600P is an in-line analyzer for measuring chord-length distribution of suspended solid particles. Chord length and particle size are not exactly equivalent terms, but there is a direct correlation between the two. For the purposes of the testing performed, the Lasentec M600P was used to evaluate the particle size distribution of the suspended solid particles in the slurries. As such, the instrument will be referred to as a particle size analyzer elsewhere in this report.

2.0 Technical Summary and Recommendations

This section summarizes the technical achievements of the Lasentec particle size analyzer and the Red Valve pressure sensor based on validation and acceptance test results and offers recommendations for using these instruments.

2.1 Lasentec Particle-Size Analyzer

The Lasentec M600 in-line particle size analyzer was installed at ORNL in August 1998 to support sludge retrieval from the Gunite and Associated Tanks (GAAT). Before installation at ORNL, the sensor underwent validation testing with waste slurry simulants (non-radioactive, non-hazardous analogs of nuclear tank waste). These tests were performed at the PNNL Instrument Validation Facility (IVF). Eight simulants were chosen to test the Lasentec: four different silica/kaolin weight ratios at two total solids concentrations, 5-wt% and 10-wt% solids. Kaolin particles were around 1 μm in size, whereas the silica particles were around 100 to 1000 μm in size. The full range of the Lasentec sensor (0.8 to 1000 μm) was validated by using silica and kaolin.

Mechanically, the instrument worked well during validation testing and met all expectations. Operationally, much was learned about optimum ways to display and interpret the data. Scan time, the amount of time that particles are measured and counted by the Lasentec sensor, was found to be important. If the scan time is less than one minute, the data, particularly for larger particles, were too noisy and inconsistent to be of much use. At one minute or greater scan times, noise in the data was dramatically reduced. The Lasentec calculated several statistical particle size averages by manipulating collected data: unweighted or number average, length-weighted average, length-squared weighted average, and length-cubed weighted average particle size. Only the length-cubed weighted particle size average showed any significant variation during the eight test cases. The other average values calculated by the Lasentec software remained fairly constant despite changes in the silica/kaolin weight ratio. This result was unexpected because an increase in the amount of silica, i.e., large particles, should increase the average particle size. Even though only one statistic appears to be particularly relevant when tracking process changes, the histograms can be very valuable. For example, a sieve analysis on the silica added to the pipe loop was almost perfectly matched by the Lasentec sensor.

Slurry samples taken during the in-line tests at PNNL were shipped to the vendor for analysis with a benchtop Lasentec sensor. These experiments were performed to determine if off-line analyses yield particle size distributions similar to those generated by the in-line sensor. Although the in-line Lasentec data did not match those produced by the benchtop model, several different benchtop units and in-line units (operating in a static mode) measured the same chord-length weighted histograms for the same sample. These results suggest that the Lasentec sensor measures repeatable chord lengths as long as particles are "presented" to the sensor window the same way. The laboratory and in-line measured length-weighted mean particle sizes did not match. This may have been caused by potential deficiencies in mixing the samples during the benchtop tests (e.g., the fast-settling silica particles are difficult to keep homogeneous in the sample bottle) and sampling slurries from the pipe loop, or perhaps by stratifying solids within

the test-loop pipe near the in-line sensor. This finding should be used as a caveat when comparing Lasentec in-line data to benchtop data in the future. If comparisons between in-line and laboratory sensors are desired, it is important to compare Lasentec in-line data to a Lasentec bench-top sensor. Note that the solids must be homogeneous, both in the pipe and the benchtop beaker, for results from both sensors to match.

After the initial non-radioactive simulant testing at PNNL, the instrument was shipped for radioactive validation and acceptance testing in the Slurry Monitoring Test System (SMTS) connected to the Tank W-9 of the GAATs at ORNL. The Lasentec equipment validation runs at ORNL were conducted at three different dwell times for the Pulsair system and two recirculation pump positions. For all acceptance tests conducted at ORNL, the variation in the chord length distribution and the total particle count corresponded very well with the slurry density data as determined using an in-line Promass 63M Coriolis meter. Similarly, the results also show that > 99.9% of the particles have chord lengths < 105 μm and that the instrument was extremely sensitive to small variations in the particle size distribution. Based on the performance results obtained, the Lasentec Focused Beam Reflectance Measurement (FBRM) is expected to meet the requirements of the GAAT Remediation Project for measuring the particle size distribution during the slurry-transfer operations at ORNL.

2.2 Red Valve Pressure Sensor

The Red Valve pressure sensor was endorsed at the Hanford Site following instrument validation tests at PNNL and is currently in operation in the Tank 241-C-106 pump pit. While this instrument measures pressure within a transfer line, this type of pressure sensor could be configured to measure pressure drop over time. In turn, the status of a slurry transfer could be inferred from the pressure-drop measurement. The Red Valve pressure sensor is certified to ± 1% of full scale or 1.0 psig on a 1-to-100 psig scale. Pressure measurement data validated the sensor in the 40 psig to 100 psig range. The pressure measured by the Red Valve pressure sensor in validation tests is within 1% of the actual direct pressure tap readings obtained by the Rosemount Model 3051CG sensor.

The principle behind the Red Valve pressure sensor operation suggests that the transducer plugging and fouling issues can be eliminated. In 1998, four Red Valve pressure sensors (with Sensotech Model AE-213 pressure transducers) were installed before and after the booster pumps of the 4-in. slurry (SL-200) and supernatant (SN-200) transfer lines between Tank 241-C-106 and Tank 241-AY-102. The sensor responds rapidly to changes in the booster pump discharge pressure and appears to be extremely sensitive to variations in the discharge pressure. The pressure sensor components in the SL-200 and SN-200 transfer lines are exposed to a total radiation dosage on the order of 300 R/yr. These pressure sensors have been in operation for over 1 year, and to date, the sensors have been trouble-free according to the operators involved with slurry and supernatant transfer operations. Based on these observations, it is apparent that the Red Valve pressure sensors could be installed at the end of the slurry transfer lines and used to measure the pressure drop in the system.

2.3 Recommendations

Based on the performance results obtained, the Lasentec M600P FBRM and the Red Valve pressure sensors are expected to meet the slurry monitoring requirements for measuring the particle size distribution and pressure drop during the feed delivery, storage, and disposal missions at Hanford and the Oak Ridge GAAT remediation project.

3.0 Lasentec M600P Particle Size Analyzer

This section documents the qualification phase for the Lasentec Particle Size Analyzer. The qualification phase included specifying and procuring the instrument, installing and validating the instrument using simulated waste streams at PNNL, and installing and acceptance testing the instrument in the GAAT retrieval system at ORNL.

3.1 Instrument Description and Specifications

The Lasentec M600P is an in-line analyzer that was developed by Laser Sensor Technology, Inc., Redmond, Washington, for measuring chord-length distribution of suspended solid particles. Although the chord length and particle size are not exactly equivalent terms, but there is a direct correlation between the two. For the purposes of the testing performed, the Lasentec M600P was used to evaluate the particle size distribution of the suspended solid particles in the slurries. As such, the instrument will be referred to as a particle size analyzer elsewhere in this report. This instrument uses a technique known as FBRM to provide continuous in-process and real-time measurement of the rate and degree of change of the particle dimension and particle count. A schematic of the FBRM probe tip consisting of a laser beam source, rotating optics, and a sapphire glass window is shown in Figure 3.1.

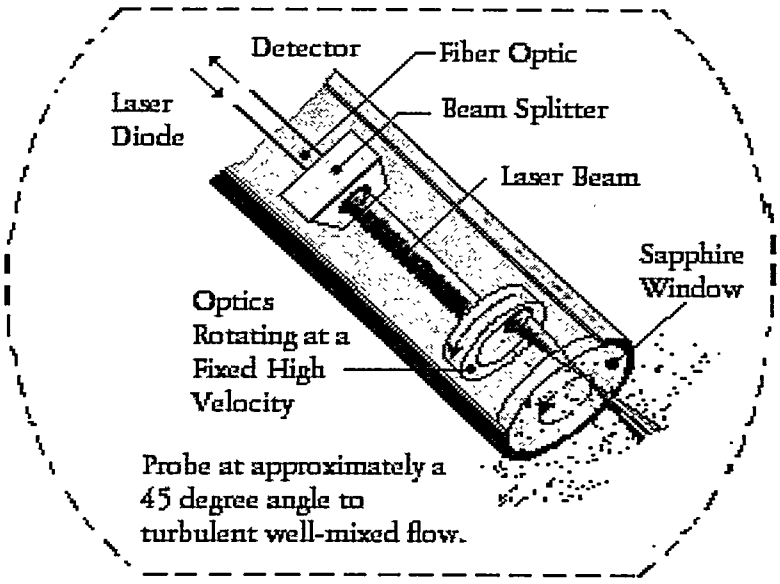

Figure 3.1. Schematic of the FBRM Probe Tip©; Copyright 1999, Laser Sensor Technology, Inc., Reprinted with Permission

The most intense part of the focused beam (or beam waist) is approximately 2 μm in dimension and 10 μm in depth. The light intensity is distributed across the cross section of the beam spot in Gaussian fashion with the center being more intense than the edges. The focal point, which is just outside the probe window, is rotated around the window at a linear velocity of 2 m/s. When the focal point intersects the edge of a particle, the particle begins to backscatter light as shown in Figure 3.2. The particle continues to backscatter light until the focused beam

has reached the edge of the particle. This backscatter is collected by the FBRM optics and converted into an electronic signal. A unique discrimination circuit is then used to isolate the time period of backscatter from one edge of an individual particle to its opposite edge. This time period (t) is multiplied by the scan speed (v), to yield a distance or chord length (c), according to the following equation:

$$c = v \times t \tag{3.1}$$

The chord length c in Equation 3.1 is the straight-line distance between any two edges of a particle and is a function of the particle shape. Typically, thousands of chord lengths are measured per second and counted by the FBRM electronics. The resulting chord length by number distribution is a robust thumbprint of the particle size distribution in the slurry. Any change in the size distribution will have a corresponding change in the chord-length distribution.

The electronics associated with the Lasentec monitor "sort" the measured chord lengths into 38 "bins." The "bins" are on a log scale from 1.9 μm to 1000 μm with an extended bottom "bin" from 0.8 μm to 1.9 μm and an extended top "bin" for counts greater than 1000 μm. At the end of the user-defined measurement duration (between 2 s and 5 min), the Lasentec software constructs a histogram of the measured chord lengths from the number of particles classified in each "bin." Figure 3.3 is an example of the chord-length distribution obtained with the Lasentec monitor during instrument validation tests at PNNL (Daymo et al. 1996).[1]

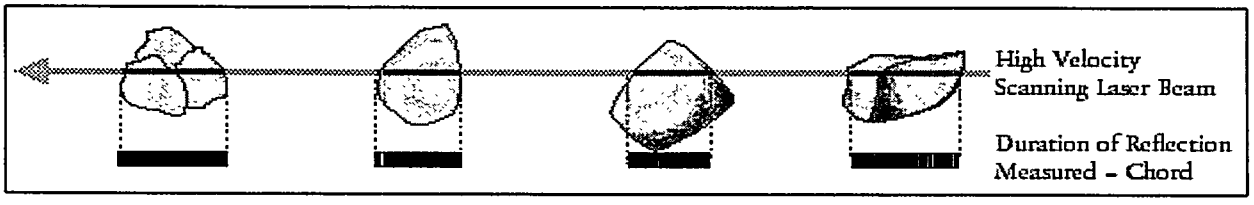

Figure 3.2. FBRM Approach for Measuring the Chord Length Using Lasentec Chord Length Analyzer©; Copyright 1999, Laser Sensor Technology, Inc., Reprinted with Permission

[1] E. A., Daymo, G. R. Golcar, and L. K. Jagoda. *Alternate On-Line Slurry Measurement Techniques.* Letter Report, Pacific Northwest National Laboratory, Richland, Washington (1996).

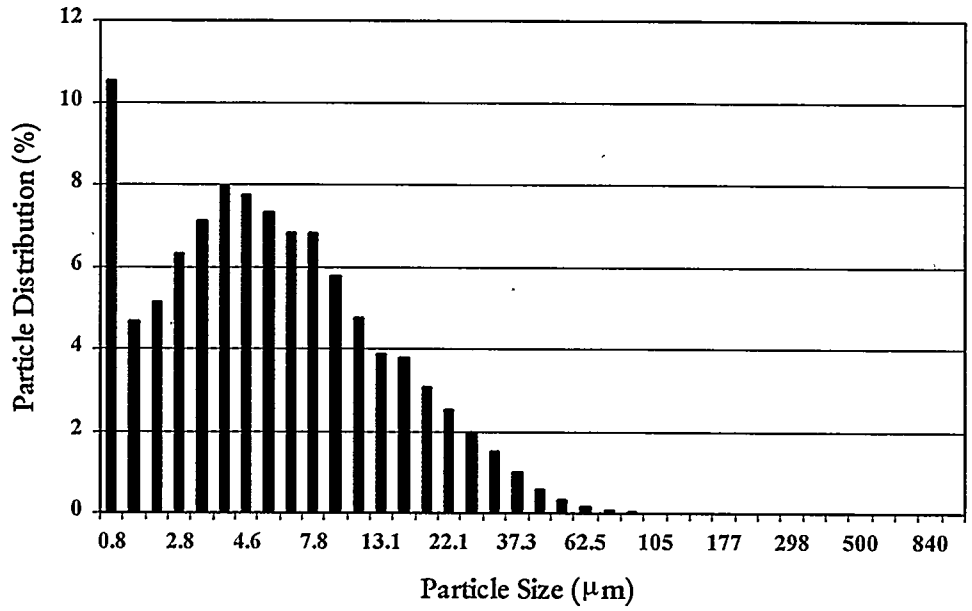

Figure 3.3. Typical Chord Length Distribution from an in-line Lasentec FBRM Monitor Obtained at PNNL Using a 30 vol% Gibbsite/Graphite Slurry at a Slurry Velocity of 1.8 m/s (Daymo et al. 1996 and 1998)

The software that accompanies the Lasentec monitor not only calculates the mean particle size, but also the length, length-squared, and length-cubed weighted mean values. Each weighted mean particle size value is, successively, more heavily influenced by the presence of large particles than the unweighted mean particle size value. The mean particle size data presented in this report are either the "unweighted mean particle size" or the "length-cubed weighted mean particle size". The mean size is the most familiar particle size statistic for operators, and the length-cubed weighted mean is the most sensitive to changes on the course end of the distribution, our primary area of investigation. The unweighted mean particle size is defined as

$$\overline{C}_u = \frac{\sum_{i=1}^{k} Y_{i,u} M_i}{\sum_{i=1}^{k} Y_{i,u}} = \frac{\sum_{i=1}^{k} \left[\left(\frac{n_i}{\sum_{i=1}^{k} n_i} \right) M_i \right]}{\sum_{i=1}^{k} \left(\frac{n_i}{\sum_{i=1}^{k} n_i} \right)} = \frac{\sum_{i=1}^{k} n_i M_i^1}{\sum_{i=1}^{k} n_i M_i^0} \qquad (3.2)$$

where

n_i = Counts in an individual measurement channel
(there are 38 channels over the 0.8 to 1000 μm range of the Lasentec monitor)

M_i = Midpoint size of an individual channel
Y_i = Percentage (%) of counts per channel
$\overline{C}_u$ = Unweighted mean particle size
k = Upper channel # ($2 \le k \le 38$)
u = Unweighted value.

Similarly, the length-cubed weighted mean is defined as

$$\overline{C}_c = \frac{\sum_{i=1}^{k} Y_{i,c} M_i}{\sum_{i=1}^{k} Y_{i,c}} = \frac{\sum_{i=1}^{k} \left[\left(\frac{n_i M_i^3}{\sum_{i=1}^{k} n_i M_i^3} \right) M_i \right]}{\sum_{i=1}^{k} \left(\frac{n_i M_i^3}{\sum_{i=1}^{k} n_i M_i^3} \right)} = \frac{\sum_{i=1}^{k} n_i M_i^4}{\sum_{i=1}^{k} n_i M_i^3} \qquad (3.3)$$

where

$\overline{C}_c$ = length-cubed weighted mean particle size
c = length-cubed value.

The Lasentec monitor does not directly account for the velocity of particles as they pass the monitor. To offset this effect, the focal point is scanned at 2 m/s. In addition, the manufacturer recommends that the probe be installed in a vertical up-flow section of pipe with the probe window positioned at a 45° angle to the flow. The 2 m/s scan compensates for fluctuations in the slurry velocity (at average slurry velocities of 1.8 m/s or slower), whereas the angle of the probe slows the particles in the measurement zone. The slurry flow should also be turbulent because turbulence mixes the particles in the pipe and ensures that "uniformly random" material is presented to the probe window.

According to the manufacturer, in a process with a slurry velocity greater than 1.8 m/s, the flow speed should be held constant so there is a linear offset to the measured data. That is, if the slurry velocity is greater than 1.8 m/s, there is less time for the Lasentec monitor to reflect light off a given particle than if the slurry were traveling at a velocity less than 1.8 m/s. To the Lasentec monitor, if light is reflected off the surface for a shorter period of time, the particle appears smaller. Likewise, the measured particle size would be greater if the velocity is decreased to a new velocity that is still greater than 1.8 m/s. If the flow speed is greater than 1.8 m/s and fluctuates with time, an external flow speed measurement should be provided to the Lasentec FBRM electronics for a real-time correction to the shift in measured particle size.

3.2 Installation and Validation Testing at PNNL

In 1996 and 1998, the Lasentec particle size and M300 and M600P population monitors were cold tested at PNNL's Instrument Validation Facility (IVF) that houses a 3 in. Schedule 40 inner-diameter pipe loop, a 250-gal feed tank, and a 225-gpm centrifugal pump. A schematic of the test loop is provided in Figure 3.4. For validation tests, the slurry was passed through the W-211 loop. More detailed drawings of the IVF are presented in Reynolds et al. 1996.

According to the Lasentec manual and marketing literature, the monitor should not be installed in the down-flow configuration because fluid may not completely fill the pipe in this configuration. Lasentec also argues that the solids in the pipe will distribute differently in the up-flow configuration as opposed to the down-flow configuration. However, installation of the Lasentec analyzer in the down-flow leg of the pipe loop did not significantly affect test results for the following two reasons:

1. There is no evidence that the fluid does not fill the pipe in the down-flow leg of the test loop. If the flow were discontinuous as Lasentec argues, then the instrument readings would fluctuate with time. No such fluctuations in the total particle counts were observed.

2. There is no evidence that solids stratified differently in the up-flow and the down-flow configurations. This is evidenced by similar density cup measurements of the samples taken from the up- and down-leg sample ports, which indicates there is probably little difference in the solids concentration in the two legs. Refer to Figure 3.5.

Validation tests in 1998 were performed using the simulant test matrix shown in Table 3.1. In addition to varying the particle size distribution, other parameters that were investigated during the Lasentec acceptance testing at PNNL include the effect of (1) air bubbles in the system, (2) solids that could coat the probe window, (3) simulant color, (4) flow rate, and (5) scan time. Additionally, grab samples of the slurry also were collected and analyzed off-line to determine the correlation between the in-line and off-line measurements. The results from these investigations are described in the following paragraphs.

3.2.1 Effect of Air Bubbles

Because retrieval of tank waste may entrain bubbles into the slurry line, every selected instrument must be able to yield relatively stable, useful readings in the presence of air bubbles. To study the effect of air bubbles in the system, the average particle size of a 2-vol% graphite slurry was compared before and after air was injected at 1 cfm into the flow loop. The results from this test are illustrated in Figure 3.6. This figure shows that although bubbles increased the noise in the Lasentec monitor's measurement, the measured mean particle size changed by less than 1% from the value before bubbles were introduced.

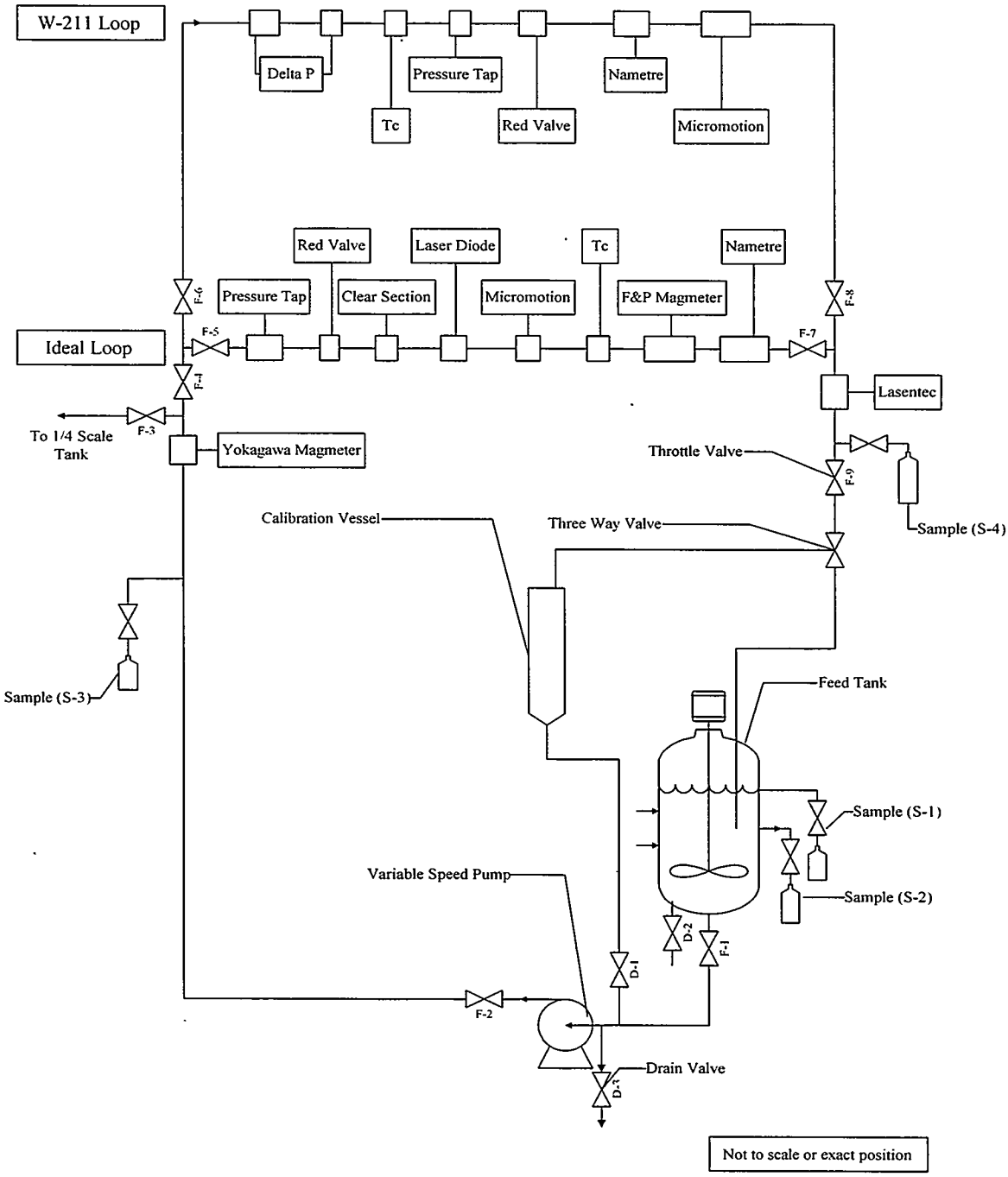

Figure 3.4. Schematic of the Test Loop at the Instrument Validation Facility at PNNL (Reynolds et al. 1996)

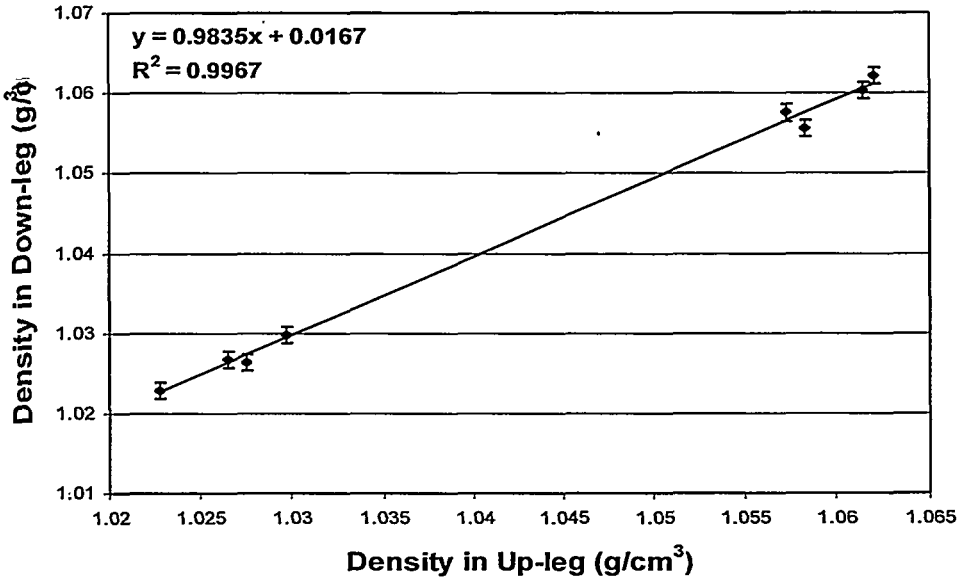

Figure 3.5. Density in Down-Leg vs. Density in the Up-Leg Section of the Pipe Loop as Determined by the Density Cup Measurements (Daymo et al.1996)

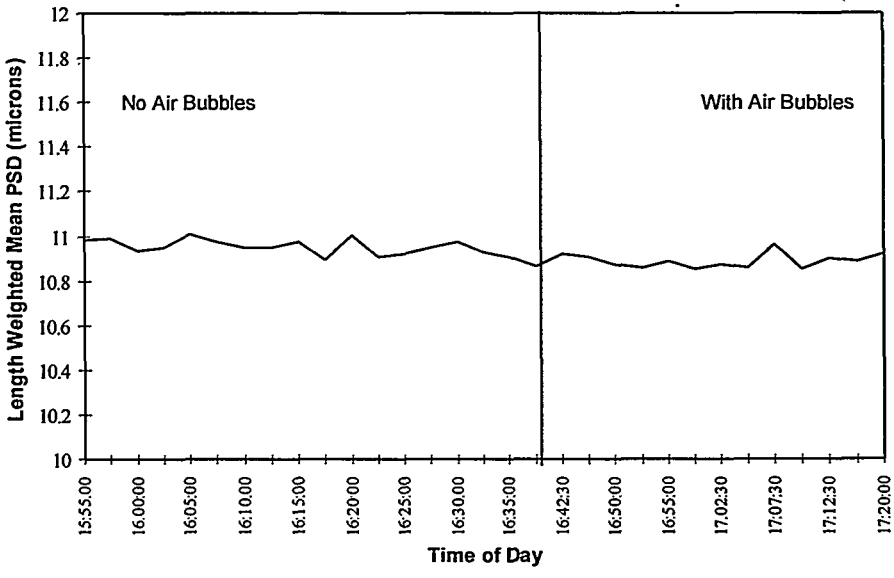

Figure 3.6. Lasentec Mean Particle Size Distribution Obtained Using Graphite Slurry with and Without Air Bubbles Introduced into the Pipeline (Daymo et al. 1996)

Table 3.1. Matrix of Simulant Slurries Used to Validate the Instruments at the Hanford Site

Solids Material(s)	Solids Concentration (wt%)	Lasentec Monitor Tested
Graphite (mean size: 4 μm)	6 11 15	M300
Gibbsite (mean size: 7.5 μm)	11 45 53	M300
Graphite and Gibbsite	9% Graphite, 13% Gibbsite 11% Graphite, 16% Gibbsite 6% Graphite, 25% Gibbsite	M300
Bentonite (mean size: 0.8 μm)	3 6 11	M300
Bentonite and Mica flakes (Mica mean size: 6 μm)	10% Bentonite, 4% Mica	M300
Silica (mean size: 3.5 μm)	12 34 54	M300
Plastic Beads (mean size: 18 μm)	5 20 35	M300
Kaolin (Kaolin mean size: 0.8 μm)	5 10	M600P
Kaolin and Silica (Silica mean size: 410 μm)	4.2% Kaolin, 0.8% Silica 3.5% Kaolin, 1.5% Silica 2.8% Kaolin, 2.2% Silica 8.5% Kaolin, 1.5% Silica 7.0% Kaolin, 3.0% Silica 5.5% Kaolin, 4.5% Silica	M600P

3.2.2 Effect of Solids Coating

The Lasentec monitor optics are protected by a sapphire window that is chemically compatible with the caustic and highly radioactive tank waste. One concern was whether solids could coat the window and hinder accurate particle size measurements. To address this concern, the M300 monitor was tested with graphite slurries that coated the entire pipeline. As a result of the coating nature of the graphite slurries, two magnetic flow meters installed in the pipe loop had to be removed and cleaned on two separate occasions. Because the Lasentec probe is

inserted into the flow direction at a 45° angle, the sapphire window is self-cleaning. No residue was found on the probe window when the monitor was removed from the pipe loop after flow tests with graphite were completed.

3.2.3 Effect of Simulant Color

The color of tank waste could vary slightly during waste-retrieval operations, and some concern exists about whether color changes would affect the monitor's capability to adequately measure particle size. During one simulant test (4 vol% bentonite), a total of 0.76 L (0.2 gal) of red and orange food coloring were added to the feed tank containing 795 L (210 gal) of slurry to change the color of the simulant. As a result of the food coloring, the bentonite slurry changed color from olive-green to peach. The results of the mean particle size distribution obtained before and after the addition of the coloring agent to the simulant are shown in Figure 3.7. This figure shows that the Lasentec showed no significant change in the average particle size distribution after the food coloring was added to the slurry.

3.2.4 Effect of Change in the Simulant Flow Rate

For most of the Lasentec monitor validation tests, a nominal flow rate of 9 L/s was selected, corresponding to an average velocity of about 1.8 m/s (6 ft/s) in the 3-in. inner-diameter pipe. This slurry velocity was selected because it is the target velocity for the pipeline in the GAATs at ORNL. There is some concern as to the effect that the flow rate change would have on the sensitivity of the instrument, as flow rates tend to fluctuate during normal operation of the slurry transfer lines. Also, another concern was that the Lasentec monitor does not directly account for the velocity of particles as they pass the monitor. To offset the effect of flow rate fluctuations, the focal point of the monitor is scanned at 2 m/s, and it is recommended that the probe be installed in a vertical up-flow section of pipe with the probe window positioned at a 45° angle to the flow. The 2 m/s scan compensates for fluctuations in the slurry velocity (at average slurry velocities of 1.8 m/s or slower), whereas the angle of the probe slows the particles in the measurement zone.

According to the manufacturer, in a process with a slurry velocity greater than 1.8 m/s, the flow speed should be held constant so there is a linear offset to the measured data. That is, if the slurry velocity is greater than 1.8 m/s, there is less time for the Lasentec monitor to reflect light off a given particle than if the slurry were traveling at a velocity less than 1.8 m/s. To the Lasentec monitor, if light is reflected off the surface for a shorter period of time, the particle appears smaller. Likewise, the measured particle size would be greater if the velocity is decreased to a new velocity that is still greater than 1.8 m/s. If the flow speed is greater than 1.8 m/s and fluctuates with time, an external flow-speed measurement should be provided to the Lasentec FBRM electronics for a real-time correction to the shift in measured particle size.

To investigate the effect of the flow rate on the measured particle size distribution in two tests, the flow rate was changed to 2.7 m/s (13 L/s) and 1.3 m/s (6.3 L/s), respectively. These results are compared to the nominal flow rate of 1.8 m/s (9 L/s) and are shown in Figures 3.8 and

3.9, for the high (2.7 m/s) and low (1.3 m/s) flow rates, respectively. Figure 3.8 shows that a 5% shift in the mean particle size was observed when the volumetric flow rate was increased from 1.8 m/s (13 L/s) to 2.7 m/s (9 L/s). Similarly, Figure 3.9 shows that an 8% increase in the mean particle distribution was observed when the flow rate was decreased from 1.8 m/s (9 L/s) to 1.3 m/s (6.3 L/s). These shifts in the mean particle size with flow rates are to be expected from the Lasentec monitor.

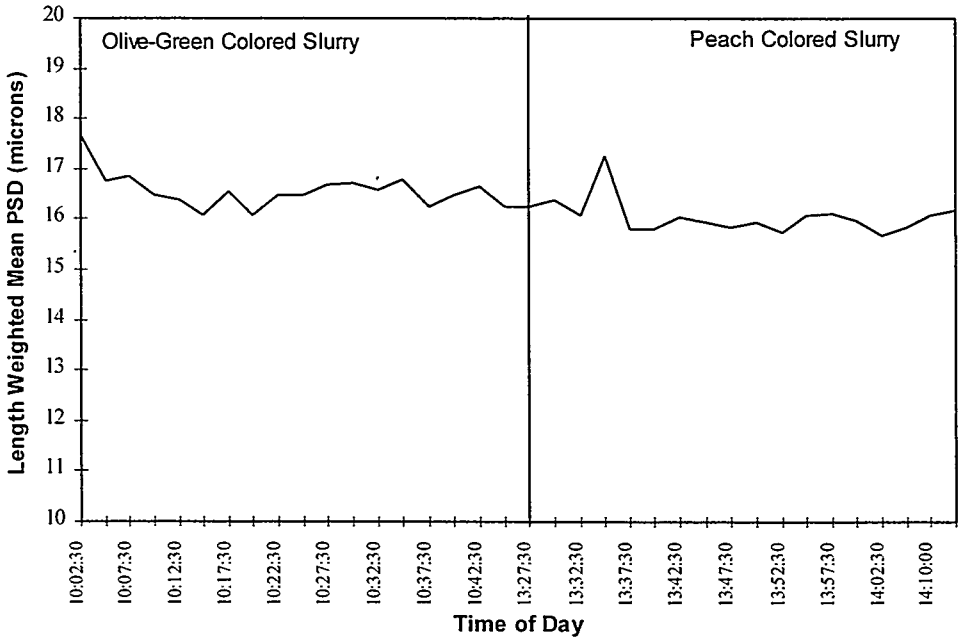

Figure 3.7. Lasentec Mean Particle Size Distribution for a Bentonite Slurry with and Without the Addition of Coloring Agent (Daymo et al. 1996)

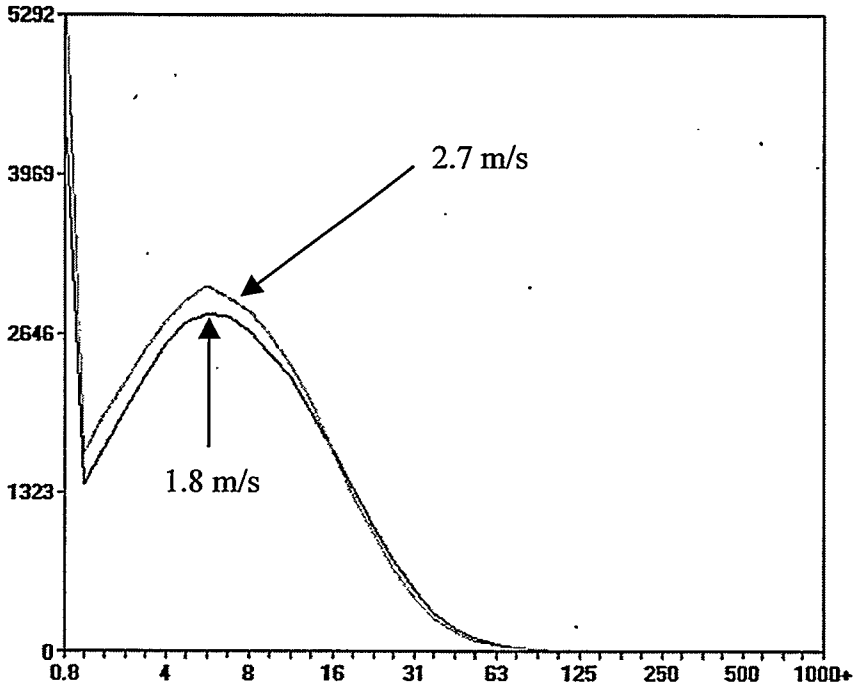

Figure 3.8. Effect of Slurry Velocity on the Lasentec Particle Size Distribution for a 3.5-wt% Kaolin and 1.5-wt% Silica Slurry (Daymo et al. 1996)

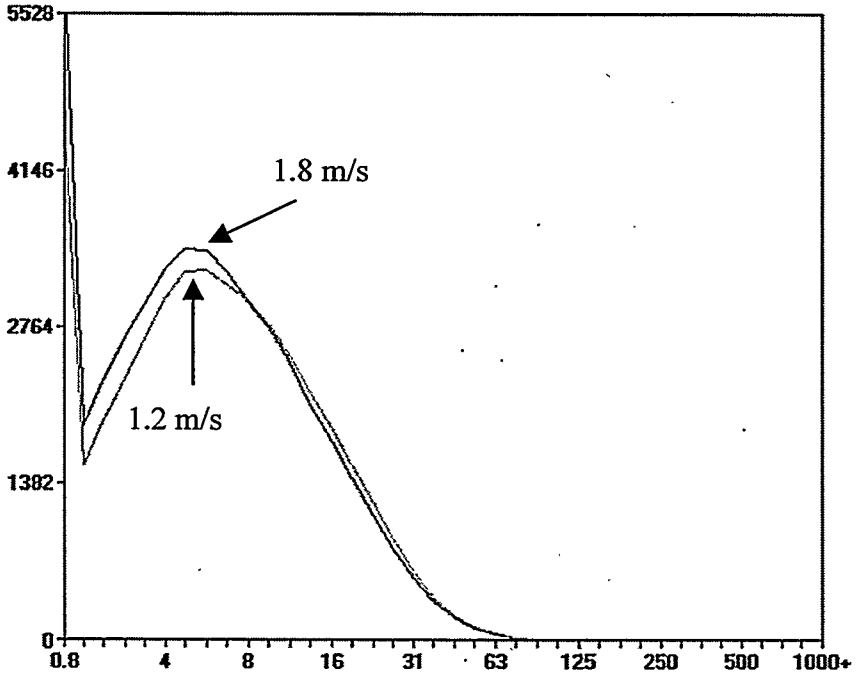

Figure 3.9. Effect of Slurry Velocity on the Lasentec Particle Size Distribution for a 3.5-wt% Kaolin and 1.5-wt% Silica Slurry (Daymo et al 1996)

Light reflected for a shorter time causes the decrease in the mean particle size distribution with an increase in the flow rate. According to Equation 1, the chord lengths would be smaller. Similarly, at lower flow rates, the mean particle size would be larger. Also, the observed 5 to 8% shift in mean particle size with flow rate is acceptable for slurry transport applications at both Hanford and ORNL.

3.2.5 Effect of Changes in Solids Concentration

During the May 1998 acceptance testing of the Lasentec M600P monitor, the solids concentration was varied in two ways. First, the ratio of silica (410 μm mean size, as measured by sieve analysis) to kaolin was increased from 0:1 to 1:5.6, 1:2.33, and 1:1.22. Second, the total solids concentration was increased from 5-wt% solids to 10-wt% solids.

A representative result of the effect of adding silica to 5-wt% kaolin is shown in Figure 3.10. Similarly, the effect of adding kaolin to silica slurry (2.5-wt%) is shown in Figure 3.11. For all the silica/kaolin slurries studied, the average particle size was found to increase as the silica/kaolin ratio was increased because more large particles are present in the slurry. Also, the effect of an increased proportion of silica on average particle size was found to become less significant with each incremental increase of silica as the average measured particle size approached that of the silica. At both 5-wt% and 10-wt% solids concentrations, the total number of counts per second decreased by around 13% when the silica/kaolin ratio was changed from 0:1 to 1:1.22. This decrease in the total number of counts per second was expected, as there were fewer particles in the system, and the larger particles had a smaller surface-to-volume ratio.

At 5-wt% solids, the length-cubed weighted mean particle size increased from around 53 μm to 150 μm when the silica/kaolin ratio increased from 0:1 to 1:5.6. A similar increase in particle size was observed at 10-wt% solids for the same change in silica/kaolin ratios (45 μm at a ratio of 0:1 to 156 μm at a ratio of 1:5.6). As expected, when the proportion of silica was further increased, the length-cubed weighted mean particle size did not increase as significantly since the length-cubed weighted mean of the kaolin/silica system was approaching the length-cubed weighted mean of the silica on its own (e.g., the measured length-cubed weighted mean particle size values at 10-wt% solids were 204 μm at a silica/kaolin ratio of 1:2.33, and 214 μm at a silica/kaolin ratio of 1:1.22). Note that the length-cubed weighted mean (which has a similar effect of a volume weight) heavily weights the change to course particles at the expense of resolution on the fine-particle side of the distribution.

For the cases where the solids concentration increased with the ratio of silica to kaolin being constant, the average length-cubed weighted mean particle sizes at 5-wt% and 10-wt% solids were nearly the same at each case tested. The total number of particles counted increased by around 20% when the solids concentration was increased from 5-wt% to 10-wt% solids.

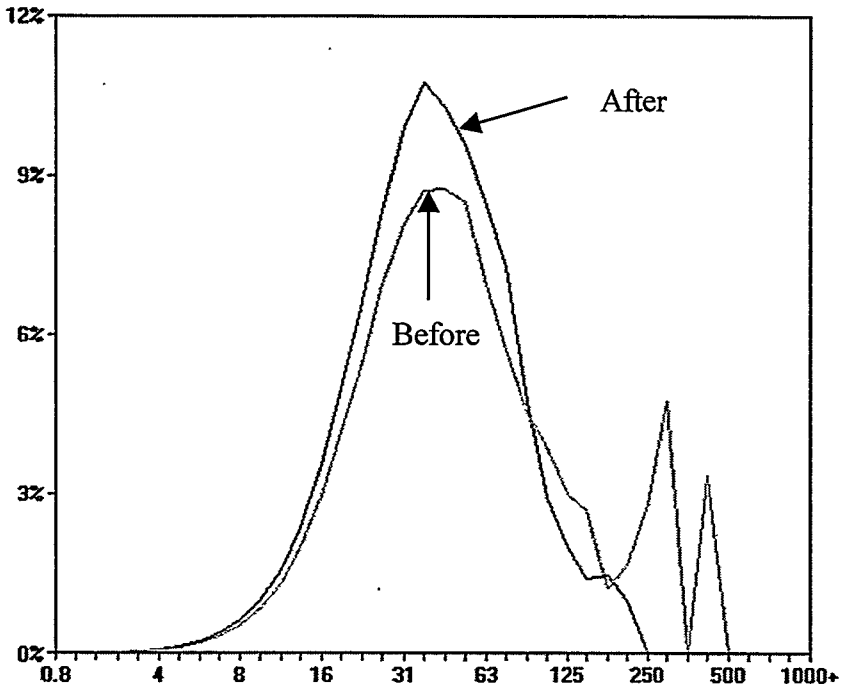

Figure 3.10. Effect of Change in Solids Concentration on the Lasentec (chord length cubed) Particle Size Distribution as Observed Before and After the Addition of Silica to a 5-wt% Kaolin Slurry (Daymo et al. 1996)

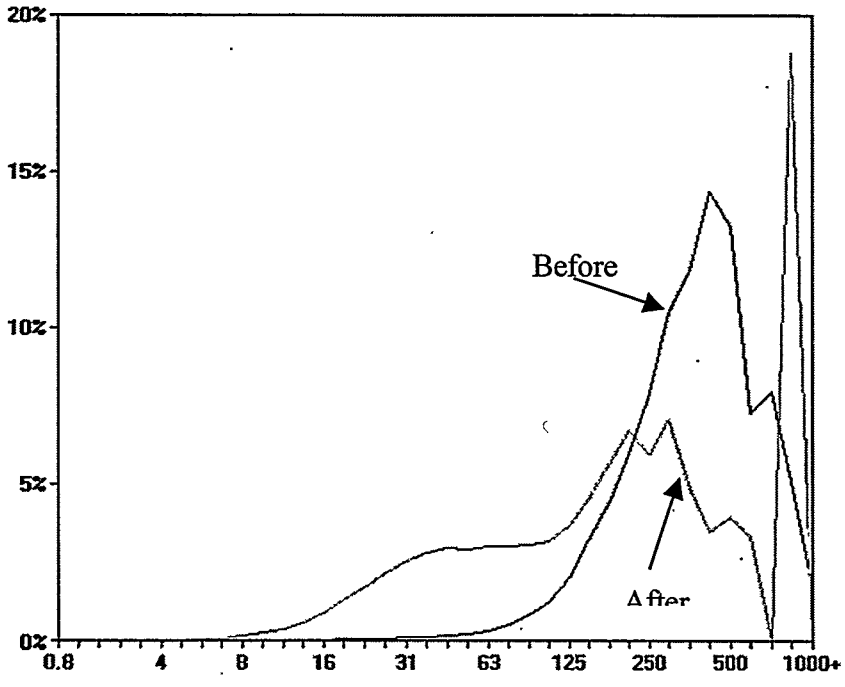

Figure 3.11. Effect of Change in Solids Concentration on the Lasentec (chord length cubed) Particle Size Distribution as Observed Before and after the Addition of Kaolin to a 2.5-wt% Silica Slurry (Dymo et al. 1996)

3.2.6 Effect of Scan Time

High scan time increases the number of counts that contribute to the particle size measurement data. Increasing the number of particles counted results in smoother and more accurate data. However, a large scan time also decreases the frequency at which new data sets can be collected. In a similar manner, low scan rates enable a larger collection of data sample sets at the sacrifice of the quality of the data. The effect of the sampling time on the mean particle size is shown in Figure 3.12. The data in this figure were collected at three sampling intervals of 30 s, 60 s, and 300 s. The 30-s sample time produces the most fluctuation in the data while the 300 s sampling time provides a very uniform mean particle size measurement. Also, at the 60-s scan rate, although some variation in the mean particle size exists, the data are closer to those observed with the 300-s scan time. Therefore, for the Lasentec particle analyzer, the scan time should be 60 s or greater and preferably (if possible) 300 s.

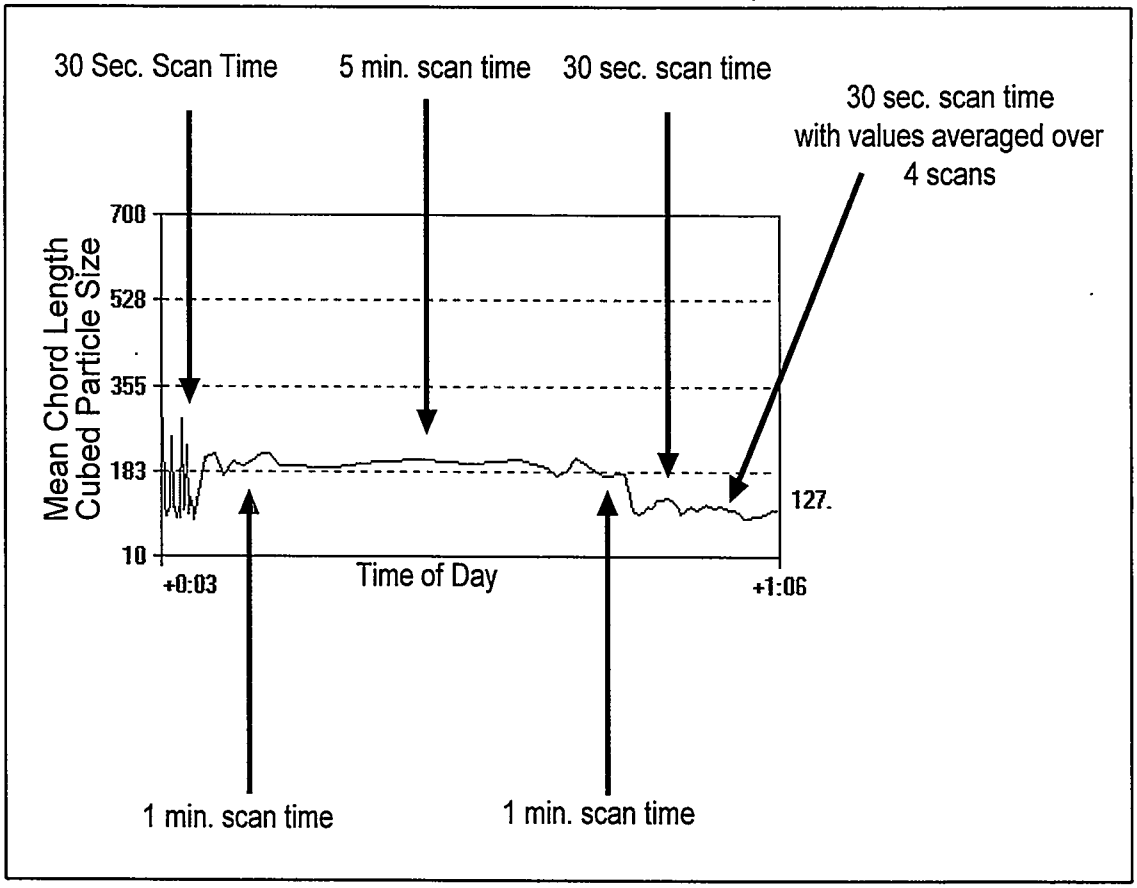

Figure 3.12. Effect of the Scan Time on the Lasentec Mean Chord Length Cubed Particle Size Distribution for a 7-wt% Kaolin and 3-wt% Silica Slurry (Daymo et al. 1996)

3.2.7 Comparison of In Line Lasentec Data to Off Line Sieve Analysis

As described above, the Lasentec FBRM monitor yields particle size histograms by measuring the length of time that laser light backscatters off of particles that pass by the probe window. One important aspect of the Lasentec "acceptance tests"[3] was to show that particle size data from the Lasentec monitor could be compared to another (independent) particle size measurement of the same material.

Figure 3.13 is a comparison between a sieve analysis performed on dry samples of silica and a particle size histogram for the silica slurry measured with the in-line Lasentec monitor (length-cubed weighted particle size data). When the Lasentec collected these data, the flow rate was 9 L/s (an average velocity of 6 ft/s in a 3 in. pipe). The Lasentec measured histogram and the sieve analysis match well, suggesting that the length-cubed weighted particle size data may be roughly correlated with a sieve analysis on materials of this type. Depending on the shape of the particles, though, a sieve analysis may not always compare with the particle size distribution measured by a Lasentec FBRM monitor.

If a certain volume of spherical particles is held constant, but the aspect ratio increased (i.e., the particles become cigar-like), a sieve analysis would indicate that the cigar-like particles are generally smaller than the spheres. If the same system were measured with an FBRM monitor, the unweighted mean particle size would decrease as the spheres become cigar-like because the unweighted mean is strongly dependent on the number of short chords across the width of these cigar-like particles. At the same time, the length-cubed weighted mean particle size would increase because of the large chords measured across the longest dimension of the cigar-like particle. Although such tests were not performed for this report, the Lasentec FBRM sensor can be used to monitor relative changes in the shapes of particles.

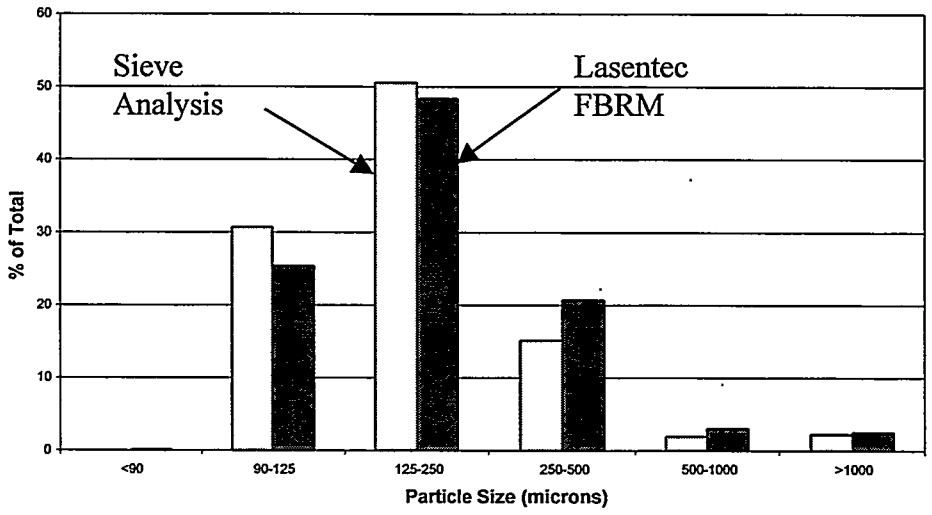

Figure 3.13. Comparison of the Sieve Analysis and the Lasentec (length cubed) Mean Particle Size Distribution Obtained Using a 10-wt% Silica Slurry (Daymo et al. 1996 and 1998)

3.2.8 Comparison of In Line Lasentec Data to Bench-top (off-line) Lasentec Analyzer

The Lasentec monitor may reduce operational costs if in-line particle size measurements could reduce (or eliminate) the number of laboratory particle size analyses that need to be performed on radioactive grab samples taken before and/or after slurry-transfer operations. Ideally, particle size distributions measured by the in-line Lasentec monitor should correlate well with particle size distributions of grab samples measured off-line by laboratory (i.e., bench-top) monitors.

Samples of kaolin and silica/kaolin slurries collected during "acceptance testing" of the Lasentec M600P monitor were sent to the vendor for analysis with a Lasentec M500LF (a laboratory version of the M600P monitor). To make accurate comparisons between in-line and bench-top FBRM monitors, it is crucial that both probes are exposed to identical distributions of particles.

One-liter samples of each slurry type were sent to Lasentec, and aliquots from each 1-L slurry sample container were taken and analyzed with the bench top monitor. Obtaining representative aliquots was difficult for silica/kaolin slurries because the large silica particles settle quickly. In general, the narrower the particle size distribution, the higher the solids concentration, and the smaller the particles, the easier it is to correctly collect slurry samples.

While the presence of fast-settling silica made it impossible to compare results between the bench-top Lasentec M500LF monitor and the in-line M600P instrument for slurries containing silica, the vendor measured the particle size distribution of the silica/kaolin slurry aliquots using several different (independent) M500LF monitors. The company reported that the independent M500LF monitors measured essentially the same particle size histogram when the same silica/kaolin slurry aliquots were presented to each of the monitors. This result suggests that the FBRM method is highly repeatable.

The in-line Lasentec M600P and the laboratory Lasentec M500LF monitors reported similar length-cubed (weighted) mean particle size for the kaolin slurries. A representative comparison result for a 5-wt% kaolin slurry is shown in Figure 3.14. For the 5-wt% kaolin slurry, the in-line length-cubed weighted mean particle size is 53 μm, whereas the laboratory monitor measured 57 μm. Similarly, at 10-wt% kaolin slurry, the in-line monitor measured the length-cubed weighted mean particle size to be 45 μm, while the laboratory monitor measured 56 μm. This difference between the in-line and laboratory monitors is considered to be acceptable for tank waste retrieval applications.

3.3 Acceptance Testing at ORNL

After the initial non-radioactive simulant testing at PNNL, the instrument was shipped for radioactive validation in the SMTS connected to the Tank W-9 of the GAATs at ORNL. The discussion and results presented in this section are taken from the report prepared by Hylton and Bayne (1999) on the testing of in-line slurry monitoring devices at ORNL. A schematic of the

Tank W-9 and the SMTS system is shown in Figure 3.15. A detailed description of the SMTS also is available in Hylton and Bayne (1999).

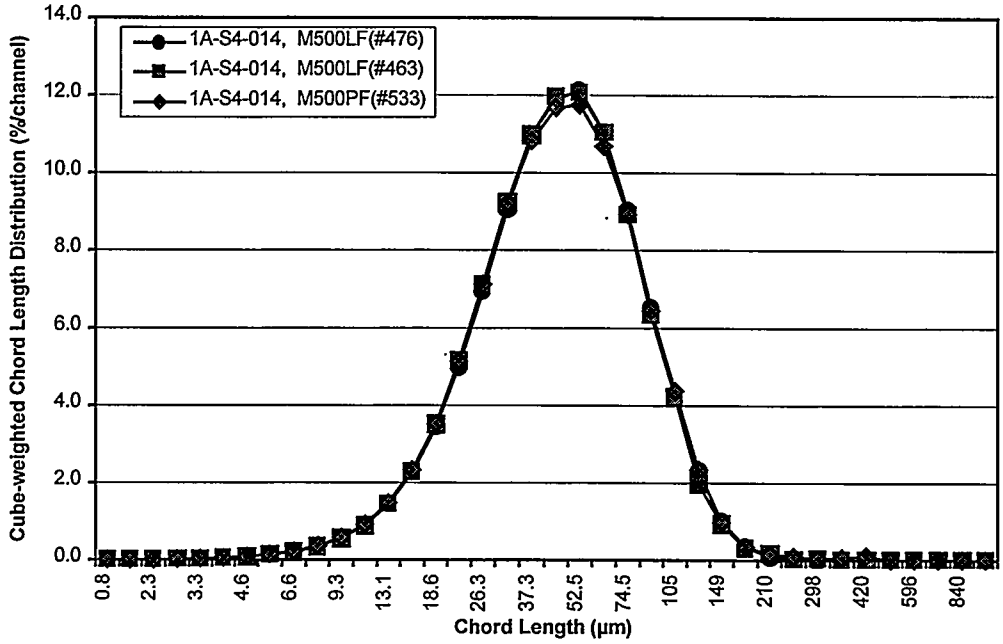

Figure 3.14. Comparison of the Particle Size Distribution (length cubed) as Determined by an in line (M500PF) and Bench Top (M500LF; duplicate) Lasentec Monitor for a 5-wt% Kaolin Slurry (Daymo et al. 1996)

The ORNL slurry transfer system uses 2-in. (~0.051-m) Schedule 40 piping and was designed for a nominal flow rate of 227 L/m. This corresponds to a linear velocity of 1.9 m/s. To meet the Lasentec maximum flow rate requirement of 1.8 m/s, the Lasentec probe was installed in a 2.5-in. (0.064-m) Schedule 40 pipe. The pipe expansion reduced the slurry velocity to the Lasentec probe to about 1.2 m/s. The probe was installed at an angle of 45° in the up-flow configuration as per the manufacturer's recommendation. Such a configuration makes the probe self-cleaning as the impinging slurry keeps the sludge from building up on the probe's sapphire window. A photograph of the actual probe in the SMTS is shown in Figure 3.16. The SMTS was operated and monitored by three computers. The main computer used Intellution Fix 32 software to control, monitor, and record data for everything except a prototype ultrasonic suspended-solids monitor and the Lasentec instrument, which had their own dedicated computers. Data-acquisition hardware was procured from RTP Corporation. To ensure valid comparison between the data collected by the computers, the calendars and clocks for all three computers were synchronized before starting to collect data.

The tank contents of Tank W-9 were mixed using a technology developed by Pulsair™ Systems, Inc. In this technology, compressed air pulsed from the accumulator plates placed at the bottom of the tank creates a shock wave that immediately displaces the liquid and initiates

the mixing process. As the air begins to form a bubble above the accumulator plate, the liquid and sludge particles are swept away from the plate. The bubble begins its rise, and low pressure under the bubble draws liquid and sludge particles back to the accumulator plate. As the bubble

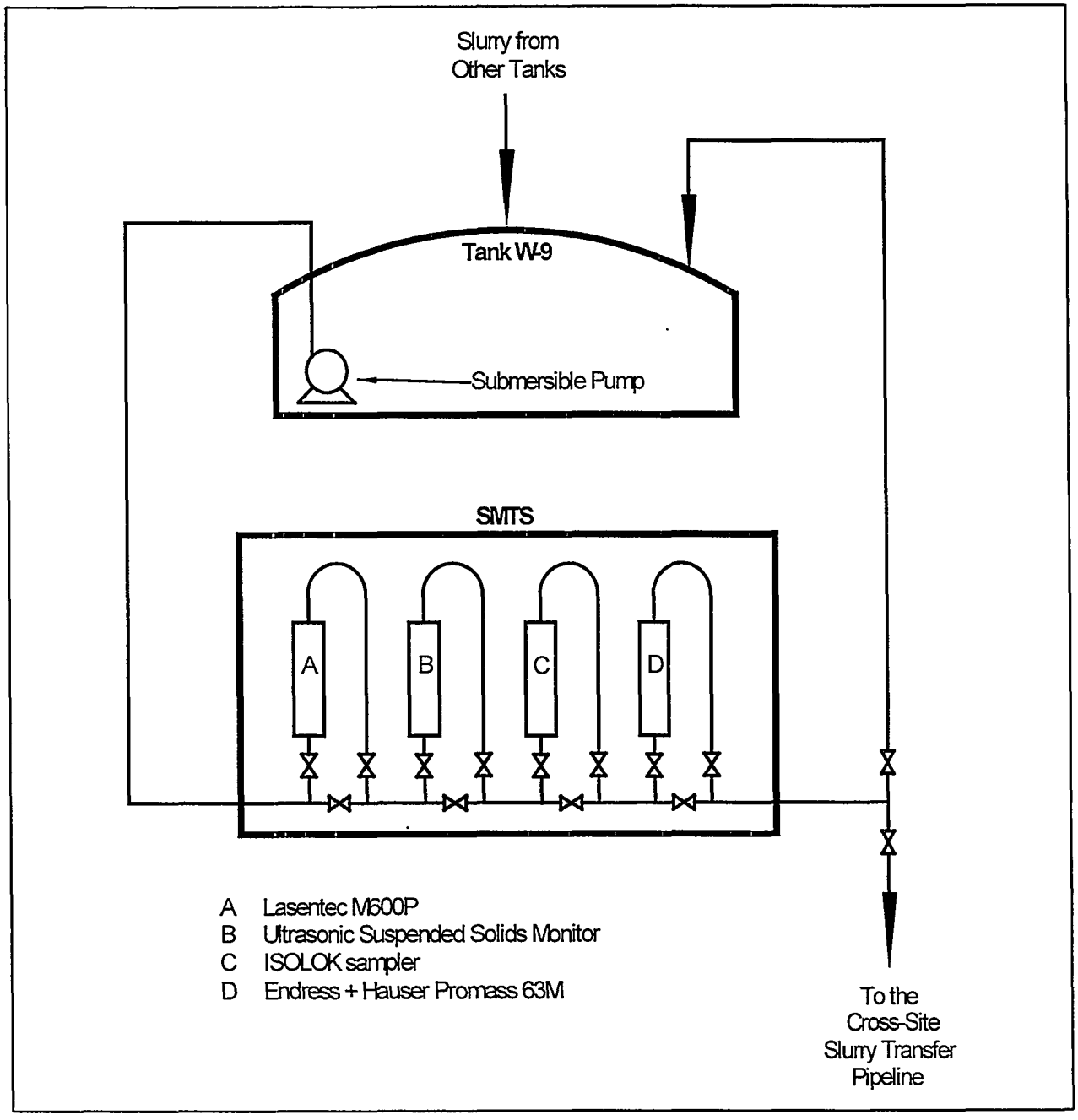

Figure 3.15. Schematic of the Flow Path from Tank W-9 to the Slurry Monitoring Test System (SMTS, Hylton and Bayne 1999)

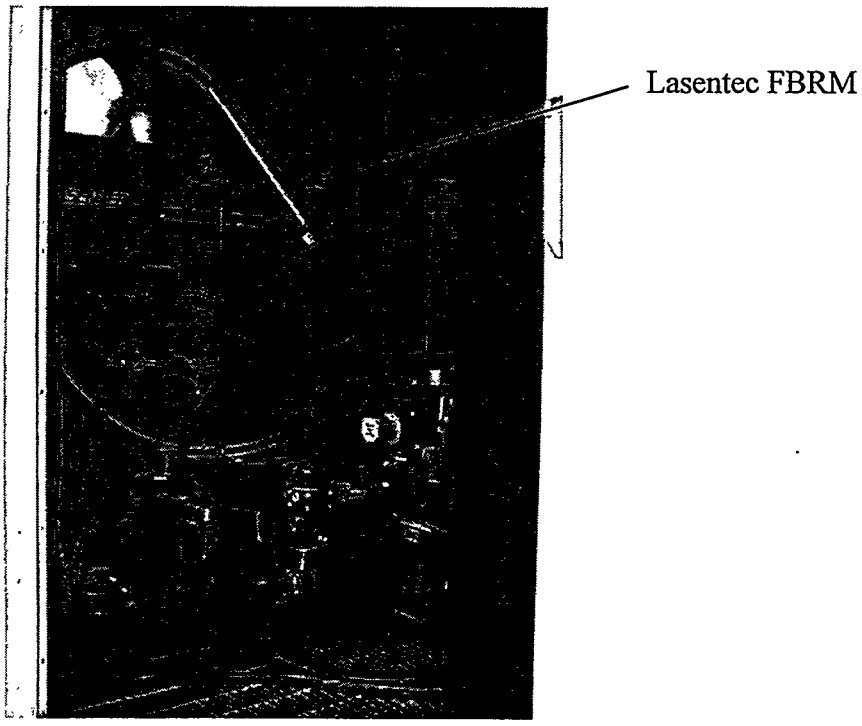

Figure 3.16. The Lasentec M600P Monitor (indicated by the arrow) Installed in the Slurry Monitoring Test System (SMTS) at ORNL (Hylton and Bayne 1999)

rises, the liquid above the bubble is forced up and away and liquid and sludge particles are pulled from the bottom and mixed with the lighter liquid. The bubble breaks on the liquid surface, and the mixing changes from vertical to horizontal. A surface mixing force moves the liquid to the tank wall, where it travels down the wall to the tank bottom to complete the mixing cycle. The operating parameters that were variable for the Pulsair mixing system were (1) dwell time, i.e., time between air injections, (2) injection time, i.e., the amount of time that air was injected, and (3) the air supply pressure. Of these parameters, the dwell time was considered to have the most influence on the mixing performance. Also, since a concentration gradient would exist in the tank, the position of the recirculation pump could also influence the mixing of the tank contents. Therefore, the instrument validation runs were conducted at three different dwell times and two recirculation pump positions as shown in Table 3.2.

The results of the six acceptance tests at ORNL are shown in Figures 3.17 to 3.18. The results in Figure 3.17 (a-e) represent the complete chord-length distributions from the start to the termination of the Pulsair system. The results in Figure 3.18 (a-e) represent the total particle count as a function of time from the start to the termination of the recirculation. The results in Figures 3.19 (a-e) represent the time dependent variation (from the start to the termination of the

recirculation pump) in number of particles greater than 105 μm. Also shown in Figures 3.18 and 3.19 for comparison purposes are the time-dependent variations in the density of the slurry for the six runs from the start to the termination of recirculation pump. The following sections present a detailed description of the results included in Figures 3.17 to 3.19.

Table 3.2. Conditions for Testing the Slurry Monitors at ORNL

Test Number	Date	Tank W-9 Volume (Gal)	Recirculation Pump Position (ft)	Pulsair Mixing Parameters		
				Dwell Time (s)	Injection Time (s)	Air Supply Pressure (psi)
1	02/17/1999	104,000	4	10	1	35
2	02/22/1999	104,000	4	18	1	35
3	02/25/1999	103,000	4	14	1	35
4	03/02/1999	105,000	6	10	1	35
5	03/05/1999	105,000	6	14	1	35
6	03/11/1999	113,000	6	18	1	35

3.3.1 Slurry Test 1

During the Slurry Test 1, the recirculation pump was operated for 1 hr before the Pulsair system was turned on. The tank contents were mixed for approximately 2.5 hrs before the slurry was pumped through the SMTS and data collection was initiated. The Pulsair system was then stopped after 12 minutes of initiating the data collection while the recirculation through the SMTS was continued for another 1.5 hrs.

The total number of particles measured by the Lasentec instrument includes only those particles that come close to the window to be counted. A graph of the time-dependent variation of the chord length distribution and the total number of particles measured by the Lasentec probe is shown in Figure 3.17a and 3.18a, respectively, for the Slurry Test 1. Also shown in Figure 3.18a for comparison purposes are the density results for the same test as measured by the Promass 63M Coriolis meter. As might be expected, the results show that the particle count responds in a similar fashion to the density; when the density decreases, the particle count decreases and vice versa.

The current acceptance criteria for transferring the slurries through the ORNL cross-site pipeline are that the particles be less than 100 μm. The Lasentec software divides the count data by chord lengths into 38 bins as discussed in Chapter 2. The channel closest to the 100 μm bin is 105 μm. Figure 3.19a is a plot of the time variation in total number of particles and the particles with chord length > 105μm. Although the number of particles that were >105 μm was small, Figure 3.19a shows that this number increased slightly after the Pulsair mixing system was started, indicating that the instrument responds very well to small changes in the particle count.

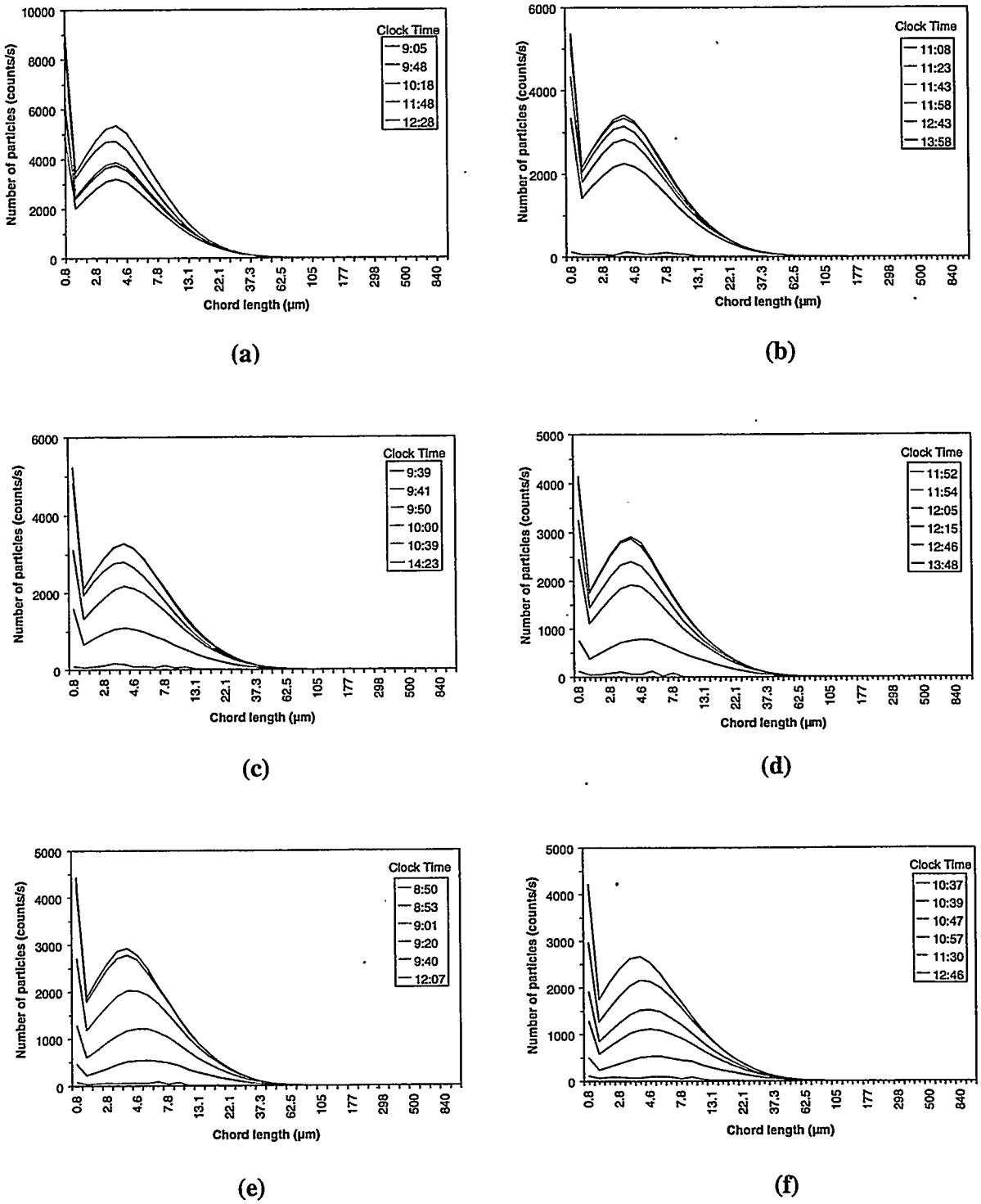

Figure 3.17. Particle Chord Length Distribution as a Function of Time for the Radioactive Acceptance Testing of the Lasentec Monitor at ORNL. (a) Test 1, (b) Test 2, (c) Test 3, (d) Test 4, (e) Test 5, and (f) Test 6 (Hylton and Bayne 1999).

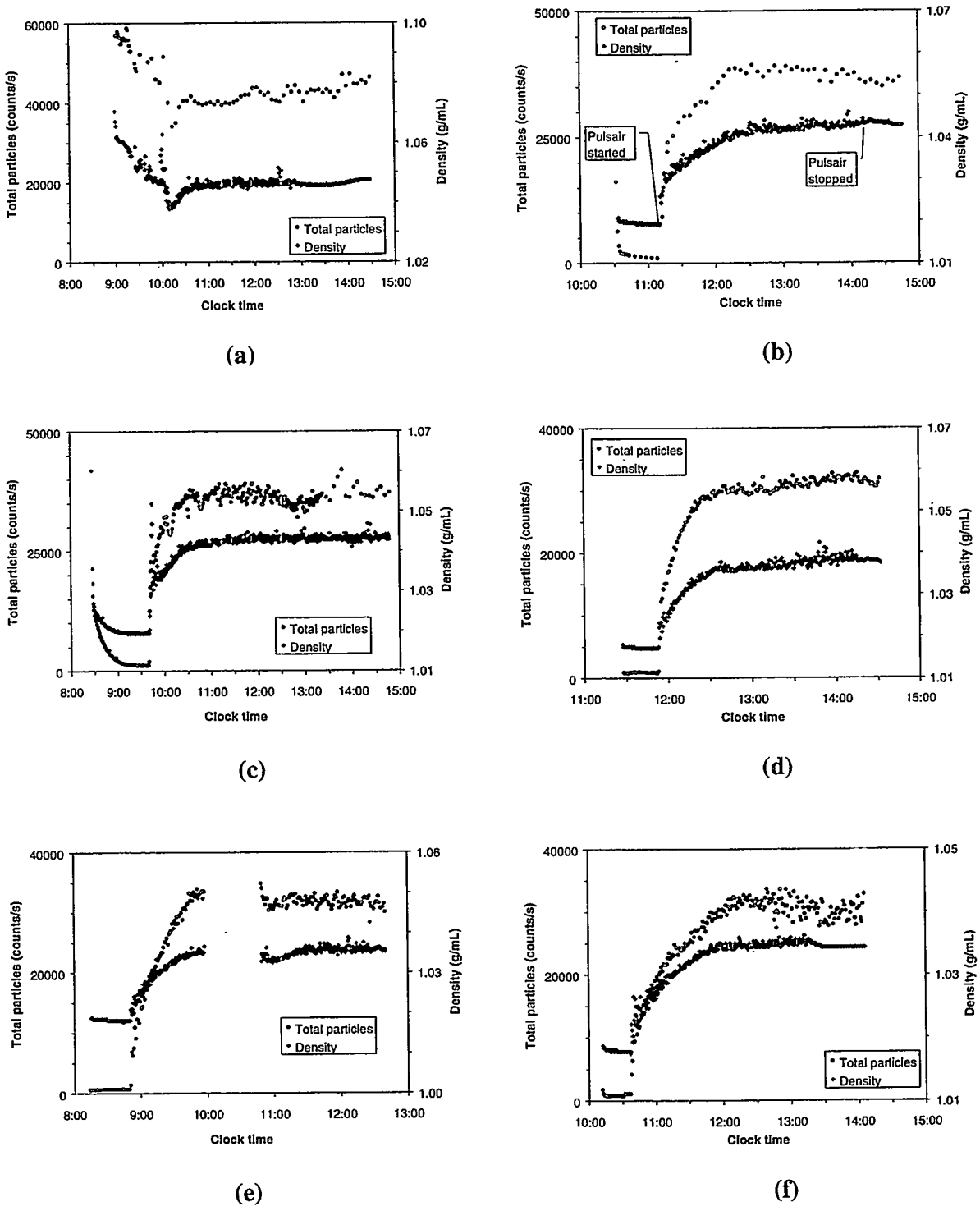

Figure 3.18. Total Particle Count as a Function of Time for the Radioactive Acceptance Testing of the Lasentec Monitor at ORNL. (a) Test 1, (b) Test 2, (c) Test 3, (d) Test 4, (e) Test 5, and (f) Test 6 (Hylton and Bayne 1999)

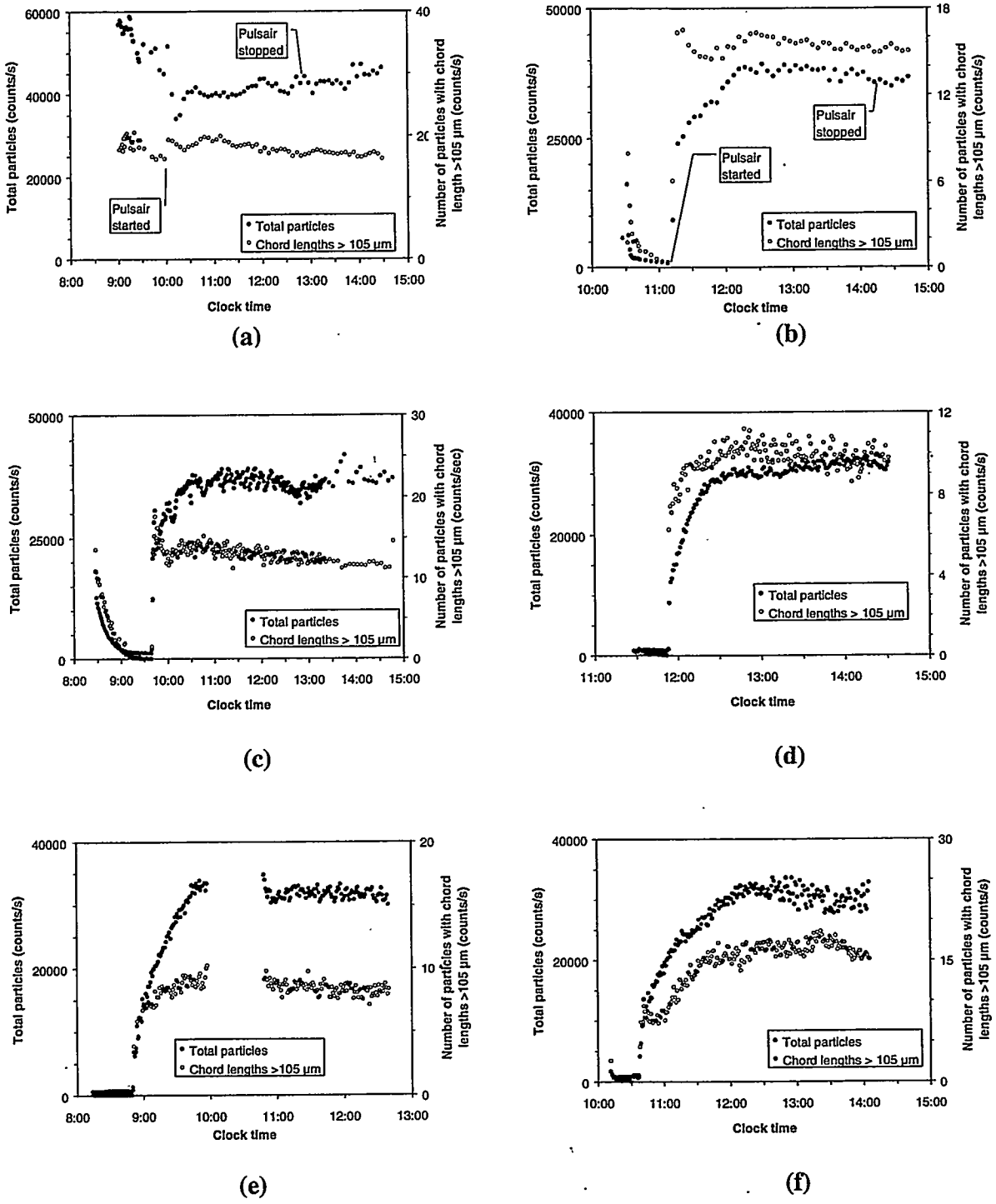

Figure 3.19. Particle Count >105 µm as a Function of Time for the Radioactive Acceptance Testing of the Lasentec Monitor at ORNL: (a) Test 1, (b) Test 2, (c) Test 3, (d) Test 4, (e) Test 5, and (f) Test 6 (Hylton and Bayne 1999)

3.3.2 Slurry Test 2

During Slurry Test 2, to start the run with the recirculation pump immersed in the supernatant, the contents of the slurry were allowed to settle for approximately 118 hrs after the termination of Slurry Test 1. The fluid was mixed for about 40 minutes after the fluid recirculation was initiated through the SMTS and the Pulsair system was started. Also, in this case, the Pulsair system was stopped after approximately 3 hrs while recirculation continued, and data were collected for another 45 minutes.

A graph of the time-dependent variation of the chord-length distribution and the total number of particles measured by the Lasentec probe is shown in Figures 3.17b and 3.18b, respectively, for the Slurry Test 2. Also shown in Figure 3.18b for comparison purposes are the density results for the same test as measured by the Promass 63M Coriolis meter. As observed with the Slurry Test 1, the chord-length distribution and the total particle count respond similarly to the density results. Since the Coriolis meter indicated that the density was low at the beginning of the test as the pump was only circulating the supernatant, one would expect that the Lasentec instrument would show a low particle count at the beginning of the test. Figure 3.18b shows that the particle count started out at the mid-range, but declined quickly. The mid-range count immediately at the start of the experiment was probably due to a dried film or particles that settled on the probe window from the previous testing. The particle count for the supernate before the start of the Pulsair system was < 1000 counts/s.

Figure 3.19b shows the total number of particles and the number of particles with chord length > 105 μm. The Lasentec results show that >99.8% of the particles have chord lengths < 105 μm. The graph also shows that the number of particles with chord lengths > 105 μm increased when the Pulsair system was started. This is another indication that the instrument is very sensitive to small changes in the system.

3.3.3 Slurry Test 3

Before starting Slurry Test 3, the contents of Tank W-9 were allowed to settle for approximately 67 hrs after the termination of the Slurry Test 2. The fluid was recirculated through the SMTS for about 45 minutes before starting the Pulsair system, and mixing was continued until the recirculation pump was stopped (approximately 5.5 hrs after the start of the Pulsair system).

A graph of the time-dependent variation of the chord-length distribution and the total number of particles measured by the Lasentec probe is shown in Figure 3.17c and 3.18c, respectively, for the Slurry Test 3. Also shown in Figure 3.18b for comparison purposes are the density results for the same test as measured by the Promass 63M Coriolis meter. The data in these graphs show that the change in the particle size distribution corresponds very well with the density data. Figure 3.19c compares the total number of particles and the number of particles with chord lengths > 105 μm. Similar to the findings of the previous tests, the results show that >99.9% of the particles have chord lengths < 105μm.

3.3.4 Slurry Test 4

Before starting Slurry Test 4, the contents of Tank W-9 were allowed to settle for approximately 117 hrs from the termination of Slurry Test 3. After approximately 30 minutes after the recirculation pump was turned on, the Pulsair system was started, and the mixing was continued until the recirculation pump was stopped (approximately 2.5 hrs after the start of the Pulsair system).

A graph of the time-dependent variation of the chord-length distribution and the total number of particles measured by the Lasentec probe is shown in Figure 3.17d and 3.18d, respectively, for Slurry Test 4. Figure 3.19d compares the total number of particles and the number of particles with chord lengths > 105 μm. As with previous tests, the variation in the chord-length distribution and the total particle count correspond very well with the density data. Similarly, the results also show that >99.9% of the particles have chord lengths < 105 μm.

3.3.5 Slurry Test 5

Before starting Slurry Test 5, the contents of Tank W-9 were allowed to settle for approximately 66 hrs from the termination of Slurry Test 4. After approximately 30 minutes after the recirculation pump was turned on, the Pulsair system was started. Both the recirculation pump and the Pulsair system were temporarily stopped for approximately 50 min after about 1 hr of initiating the mixing process to facilitate other site operations of the GAAT project. Both units were then restarted, and the mixing continued for approximately another 1.5 hrs.

A graph of the time-dependent variation of the chord-length distribution and the total number of particles measured by the Lasentec probe is shown in Figures 3.17e and 3.18e, respectively, for the Slurry Test 5. Figure 3.19e compares the total number of particles and the number of particles with chord lengths > 105 μm. As with previous tests, the variation in the chord-length distribution and the total particle count correspond very well with the density data. Similarly, the results also show that >99.9% of the particles have chord lengths < 105μm.

3.3.6 Slurry Test 6

Before starting Slurry Test 6, the contents of Tank W-9 were allowed to settle for approximately 6 days from the termination of Slurry Test 5. After approximately 30 minutes after the recirculation pump was turned on, the Pulsair system was started, and the mixing was continued until the recirculation pump was stopped (approximately 3.5 hrs after the start of the Pulsair system).

A graph of the time-dependent variation of the chord-length distribution and the total number of particles measured by the Lasentec probe is shown in Figures 3.17f and 3.18f, respectively, for Slurry Test 6. Figure 3.19f compares the total number of particles and the number of particles with chord lengths > 105 μm. As with previous tests, the variation in the

chord-length distribution and the total particle count correspond very well with the density data. Similarly, the results also show that >99.9% of the particles have chord lengths < 105μm.

3.3.7 Statistical Analysis of the Lasentec Performance

The software for the Lasentec M600P counts the number of particles that have chord lengths (measured in micrometers) in 38 intervals that range from (0.8, 1.9) to (1000, 4). The probability distribution of the chord lengths can be estimated by dividing the number of particles in each interval by the total number of counts for all intervals. The average and variance of the chord lengths for each of the six tests can be estimated from these probability distributions by

$$Average = \sum_{j=1}^{38} X_j \, x \, f_j \tag{3.4}$$

and

$$Variance = \sum_{j=1}^{38} (X_j - Average)^2 \, x \, f_j \tag{3.5}$$

where

X_j = midpoint chord length of an interval
f_j = frequency of the j^{th} interval; j = 1, 2, . . ., 38.

The standard deviation is calculated as the square root of the variance. Table 3.3 summarizes the estimated averages and standard deviations for the six tests. Averages and standard deviations in Table 3.3 indicate no effects that are due to either the pump position or the dwell time. An overall average for all six tests is 6.41, with a standard deviation of 7.45.

Table 3.3. Lasentec M600P Particle Distribution Averages and Standard Deviations (in parentheses) of Cord Lengths

Recirculation Pump Position[(a)]	Particle Chord Length (μm) for Different Pulsair Dwell Times		
	10 s	14 s	18 s
4 ft	6.30 (7.49)	6.42 (7.28)	6.43 (7.48)
6 ft	6.39 (7.24)	6.56 (7.13)	6.42 (8.04)

(a)Pump position is the distance from the bottom of the tank to the pump.

Figure 3.20 shows the frequency of the midpoint chord length for particles with chord lengths of 50 μm. The unusually large frequency at the beginning is due to the first interval (0.8, 1.9) that contains a large number of counts. A possible improvement in the distribution may be

achieved if the first interval is partitioned into smaller intervals. The first interval contains five channels worth of data on a log scale; therefore, a spike occurs. Earlier versions of the FBRM were not able to discriminate between a 0.8-μm count and a 1.9-μm count. The manufacturer now reports that the latest version of the FBRM can discriminate between 0.5 and 1000 μm in 0.25-μm increments. A theoretical statistical evaluation indicates the classical Fisher's F-distribution can model the Lasentec M600P frequency distribution.

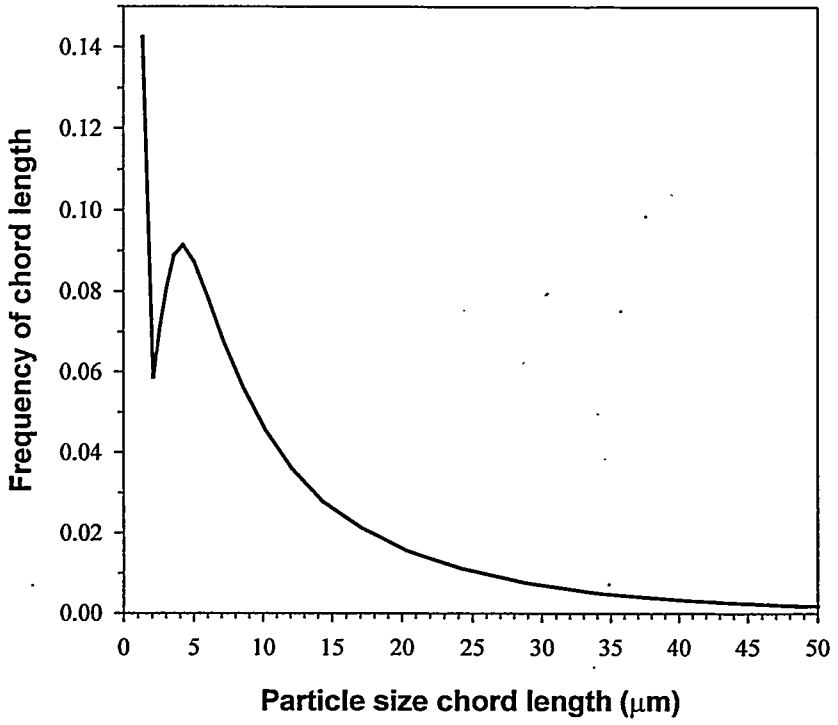

Figure 3.20. Overall Frequency Distribution of Particle Size Chord Lengths for the Six Tests at ORNL (Hylton and Bayne 1999)

3.3.8 Suspended Solids

The number of total counts per second measured by the Lasentec M600P should be directly related to the total suspended solids concentration in the slurry. Figure 4.4 shows the linear correlation between total counts per second with suspended solids (by laboratory analysis). The fitted line has a multiple correlation coefficient of 90.6%, showing good agreement between the two measurements. The linear relationship between the two variables can be expressed as:

$$Counts/s = 11{,}986 + 0.858x(Suspended\ Solids;\ mg/mL) \quad (3.6)$$

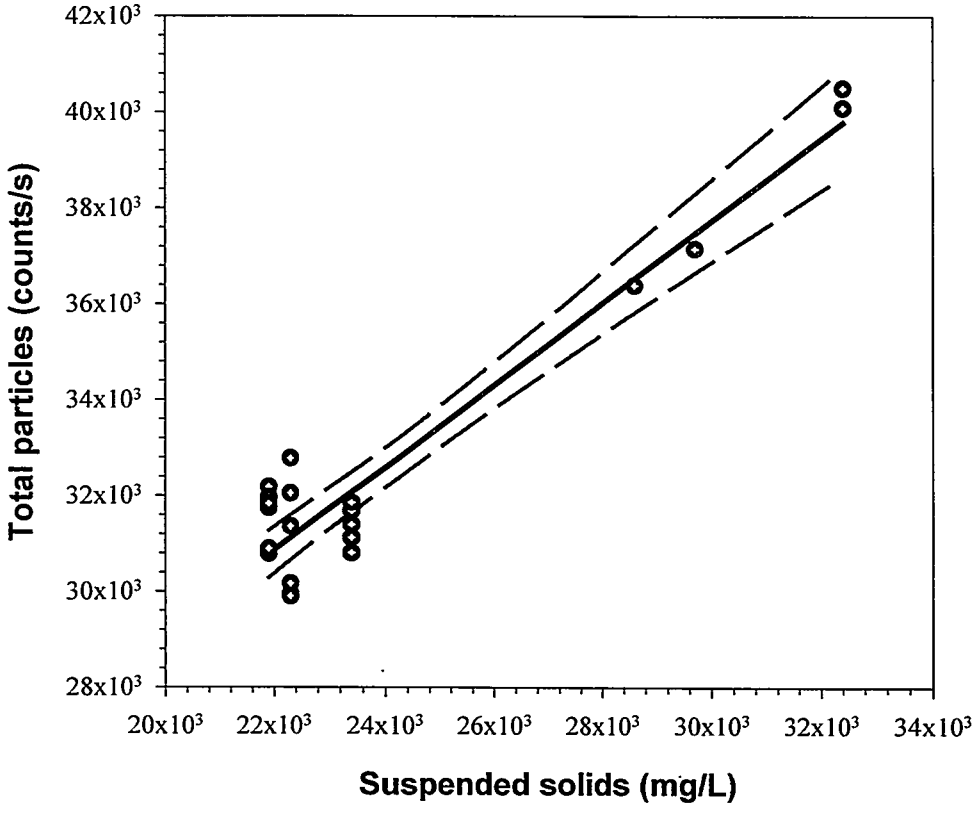

Figure 3.21. Line Fitted to the Lasentec's total particle count versus suspended solids concentration data with a 95% confidence interval (Hylton and Bayne 1999)

4.0 Red Valve Pressure Sensor

The Red Valve pressure transducer is widely used in the nuclear field. Unlike conventional pressure sensors where the slurry travels through a 1/4-inch Bourdon tube to act against the sensor's diaphragm, in the Red Valve pressure sensor a silicone fluid acts as an intermediate transmitting fluid so that the slurry never contacts the sensor's diaphragm. It should be noted that the Red Valve sensor unit is a sealed system. These sensors must be properly filled with the sensing fluid and sealed before any pressure is applied. If the sensor is dismantled or removed after installation, air could be introduced into the sensing fluid, which can cause inaccurate readings. Therefore great care must be taken to eliminate air in the system during installation. Also, users should be aware that the sensing fluid could enter the process stream if the elastomer supporting the sensing fluid should happen to breach.

The principle behind the Red Valve pressure sensor operation suggests that the transducer plugging and fouling issues can be eliminated. The instrument was endorsed at the Hanford Site following instrument validation tests at PNNL and is currently in operation in the Tank 241-C-106 pump pit. This type of pressure sensor could be configured to measure pressure drop over time. Information about the status of a slurry transfer could be inferred from a measured pressure drop. For example, as long as solids form a moving bed at the bottom of a pipe, it is unlikely that the onset of settling will be signaled by a pressure difference. However, if a stationary bed starts to build in a pipe due to gel formation or to some other increase in slurry viscosity, a gradual increase in the pressure difference (at a constant flow rate) would be observed.

The Red Valve Pressure Sensor, Model 1151, Smart Series 48, with Hypalon sleeve and silicon oil sensor fluid (Red Valve Company, Inc., Pittsburgh, PA) was validated during FY 1996 in the W-211 loop at the IVF (Reynolds et al. 1996). This instrument is certified to ± 1% of full scale or 1.0 psig on a 1-to-100 psig scale. Pressure-measurement data obtained during validation testing are compared with the Rosemount Model 3051CG (Rosemount Measurements, Eden Prairie, Minnesota) direct tap sensor in Figure 4.1. This figure shows that within the test range of 40 psi to 100 psi, the pressure measured by the Red Valve pressure sensor is within 1% of the actual direct pressure-tap readings obtained by the Rosemount sensor.

In 1998, four Red Valve pressure sensors (with Sensotech Model AE-213 pressure transducers) were installed before and after the booster pumps of the 4-in. slurry (SL-200) and supernatant (SN-200) transfer lines between Tank 241-C-106 and Tank 241-AY-102. Figures 4.2 and 4.3 illustrate the pressure sensor readings from one of the sensors installed in the discharge line of the booster pump in the SL-200 slurry transfer line. The data in these figures were obtained during two recent 12-hr operations of the slurry transfer line. These figures show that the sensor responds rapidly to changes in the booster pump discharge pressure. These figures also show that the Red Valve pressure sensor is extremely sensitive to variations in the discharge pressure. Note that the pressure fluctuations in these figures are most probably due to changes in the slurry flow to the booster pump and from nitrogen entering the line from the pump pit.

The pressure sensor components in the SL-200 and SN-200 transfer lines are exposed to a total radiation dosage on the order of 300 R/yr. These pressure sensors have been in operation for over 1 year, and to date, the sensors have been trouble-free according to the operators involved with slurry and supernatant transfer operations. Based on these observations, it is apparent that the Red Valve pressure sensors could be installed at the end of the slurry transfer lines and used to measure the pressure drop in the system.

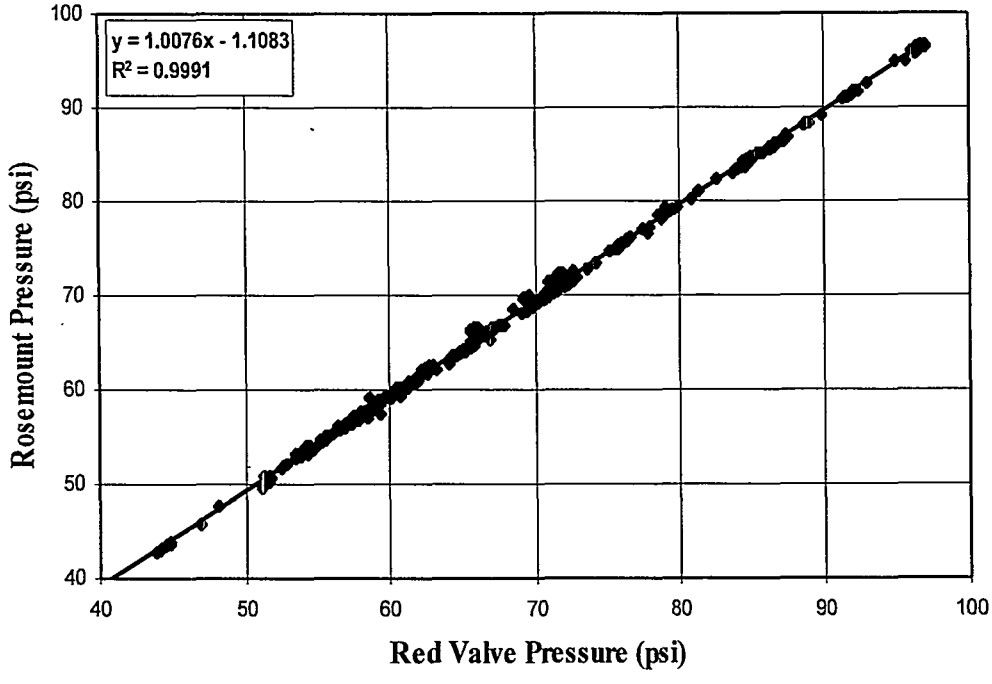

Figure 4.1. Comparison of the Pressure Measured by the Red Valve Pressure Sensor to that Measured by the Direct Tap Rosemount Pressure Sensor (Reynolds et al. 1996).

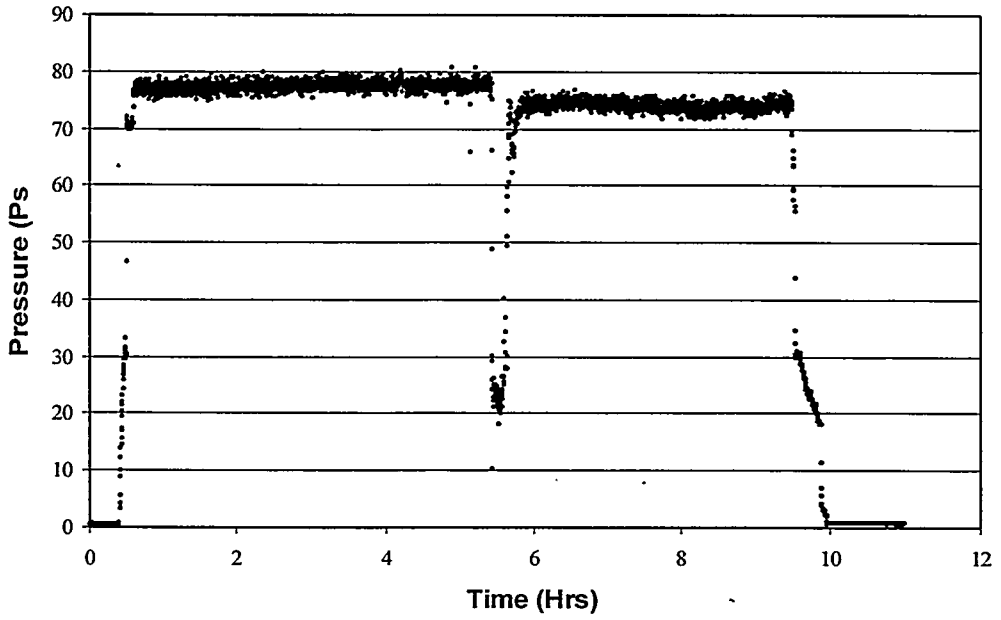

Figure 4.2. Pressure Measurement Data Obtained on September 24, 1999 from the Red Valve Pressure Sensor Installed in the 4-in. Discharge Line of the Booster Pump in SL-200 Slurry Transfer Line

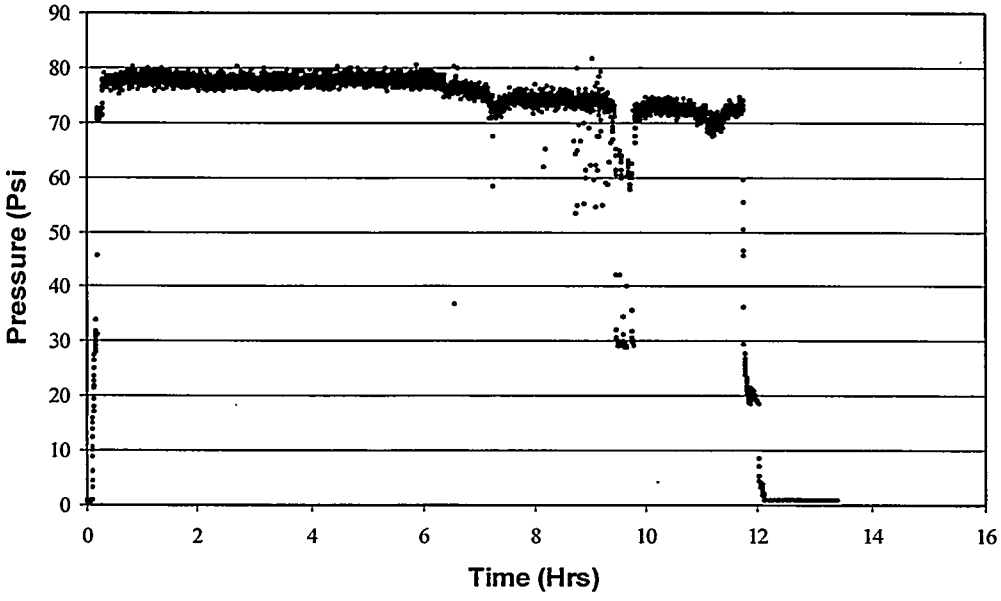

Figure 4.3. Pressure Measurement Data Obtained on September 26, 1999 from the Red Valve Pressure Sensor Installed in the 4-in. Discharge Line of the Booster Pump in SL-200 Slurry Transfer Line

5.0 References

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Appendix A
Background Information

Appendix A
Background Information

Under the current multi-year plan, the waste vitrification process will take place at a privately owned facility. The U.S. Department of Energy (DOE) will be responsible for the safe transfer of the waste from underground waste tanks to feed tanks for the private vitrification plant. To reduce costs and worker exposure to radiation, the sludge, salt cake, and liquid will be slurried and transferred via buried pipeline to the processing and vitrification facilities. The extreme complexity of the wastes stored in the underground storage tanks presents significant concern to the transfer operations. The following sections present a brief description of the issues associated with the slurry transfer operations at Hanford and Oak Ridge National Laboratory (ORNL).

Hanford's Cross-Site Slurry Transfer History

Cross-site transfer lines between the 200 East and 200 West Areas of the Hanford Site have existed for about 40 years.[1] A total of six lines have been built, and today four of the lines are permanently plugged. Because past plugging events were generally not thoroughly investigated, very little quantitative information is available to explain why the plugging events occurred. Portions of two remaining unplugged lines are to be restored, upgraded, and used to transfer waste only within the 200 East Area. A new cross-site transfer line is being designed under the Hanford Site Project W-058, Replacement of the Cross-Site Transfer System (RCTS).

During the first cross-site transfer operations conducted in the late 1950s and early 1960s, plugging was thought to occur because of insufficient heating of the waste during transfer (McKay et al. 1994). As the slurry cooled, it was believed that solids precipitated out of solution and began to accumulate on the pipe walls. Eventually, the solids would block the pipe. In some cases, lines were unplugged by back flushing with high-pressure warm water.

Adequate dilution of the waste was thought to be nearly as important as maintaining a high temperature during cross-site transfer.[2] It was found that the most convenient location to add dilution water was at the pump suction. At that time, the standard operating procedure required that all material be diluted 25% before cross-site transfer (e.g., 75 gallons of retrieved slurry diluted with 25 gallons of water; see Rockwell Hanford Operations 1978).

The most complete investigation of a line-plugging event occurred in 1978 (Washenfelder 1978). After 12 hours of normal waste transfer, a decrease in flow and a corresponding increase in pump discharge pressure was observed. The flow stabilized for 6 hours, then continued to decrease, indicating that the transfer line was plugging. Hot water dilution was started, and the flow rate stabilized and later increased. Later, on the same day,

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[1] Internal Letter Report by R. L. McKay, "TWRS Retrieval Technology Project, Slurry Transport Plugging Investigation," Pacific Northwest National Laboratory, Richland, Washington (1993).
[2] Internal Letter No. 60120-78-045-J by T. A. Lane, "Cross-country Transfer of 103/107-S Material," Rockwell Hanford Operations, Richland, Washington (1978).

power to the main transfer pump was lost. After the pump was repaired, the line was completely plugged. A high-pressure (200 psi) flush cleared the blockage one week later.

The investigation that followed revealed that a gel-like material had formed after the pump stopped and the temperature of the slurry dropped. This material also could have caused the flow rate to decrease after the first 12 hours of operation. The existence of the gel-like substance in the transfer line was confirmed by analyses of the plug material and by its appearance during unplugging operations. At the Hanford Site, this material has been commonly referred to as "green goo". The material is not in fact a gel, but rather a mass of interlocking Na_3PO_4 needle-like crystals that impeded the flow within the pipe. A micrograph of these crystals is shown in Figure 1.1. The formation of these phosphate crystals was studied extensively in the 1980s.[1,2] In general, the formation of phosphate crystals is inhibited at high temperatures and high dilution factors. However, the conditions that facilitate the formation of these crystals are not completely understood.

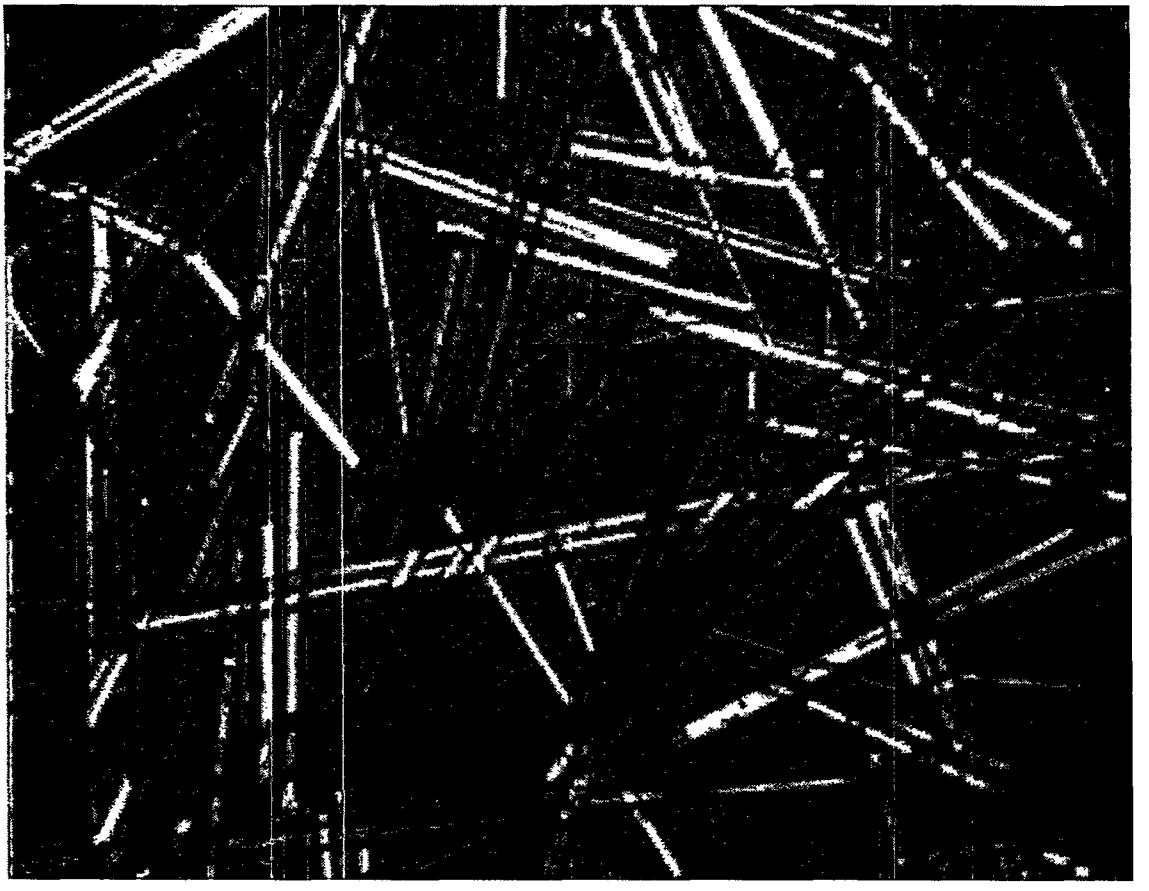

Figure A.1. Micrograph of the Na_3PO_4 $12H_2O$ crystals at 63× Magnification in Polarized Light[1]

[1] Internal Letter No. 65453-80-296 by D. L. Herting, "Evaporator Feeds High in Phosphate," Rockwell Hanford Operations, Richland, Washington (1980).

[2] Internal Memo No. 65124-068-80, by C. H. Delegard. "Viscosity/Cooling Data for Tanks 107-S and 105-BX Waste Liquors," Rockwell Hanford Operations, Richland, Washington (1985).

In addition to phosphate crystals, the precipitation of aluminates also has the potential to plug slurry transfer systems at the Hanford Site. In the highly alkaline tank waste (pH 14+), the soluble aluminum species are anionic. The dominant species thought to be present is $Al(OH)_4^-$, based on Raman spectroscopy and NMR evidence. Aluminates can precipitate by at least two mechanisms: the formation of amorphous or crystalline solid phases such as gibbsite (-$Al(OH)_3$), and the formation of sodium aluminate ($NaAlO_2$) ionic salts (McKay et al. 1994).

As pH is lowered, condensation reactions can result in the formation of amorphous or crystalline (gibbsite) $Al(OH)_3^-$. Below pH 12, aluminate solubility drops quickly. Furthermore, if tank waste containing $Al(OH)_4^-$ is cooled but the pH is not lowered, amorphous solid phases or crystalline gibbsite (-$Al(OH)_3$) precipitate. These precipitation reactions are kinetically slow, and the resulting solids are less likely to interfere with fluid flow.

In addition to condensation reactions that can occur below pH 12, counter-ion effects can reduce the solubility of aluminate at high sodium hydroxide concentrations. Although the decrease in aluminate solubility at high sodium hydroxide concentrations is not as dramatic as the decrease in aluminate solubility as the pH is reduced below pH 12, sodium aluminate precipitates form small crystals that rapidly increase the viscosity of the slurry (Reynolds and Herting 1984). In general, the concentration of the counter ion, Na^+ and the temperature dictate the solubility of aluminate at high pH. It has been shown by Reynolds and Herting (1984) that in 2 to 6 M NaOH solutions, the solubility of sodium aluminate decreases with increasing pH and NaOH concentration. Increasing temperature tends to increase the solubility of sodium aluminate, but there is a region at low NaOH concentration where increasing temperature actually decreases the solubility. The degree to which the solution is saturated in sodium nitrate and sodium nitrite also affects the solubility of sodium aluminate.

Thus, the chemistry of aluminates depends on the temperature, pH, concentration of aluminates, and the concentration Na^+. Lowering the pH or temperature increases the potential for generating insoluble aluminum hydroxides such as gibbsite. While at low pH the addition of sodium hydroxide increases solubility, the addition of sodium hydroxide at high pH can result in the precipitation of sodium aluminate.

Since the transfer lines at Hanford are designed to operate above the critical, or deposition, velocity of the slurry (the minimum velocity required to keep solids in the pipeline suspended; see Hudson [1996]), it is unlikely that the older slurry transfer pipelines were permanently blocked by the sedimentation of solids at the bottom of the pipe. Tank waste chemistry has therefore been suspected as the cause of the slurry pipeline plugging incidents.[1]

Previous slurry-transport experience has influenced the design of new slurry-transport systems at the Hanford Site. To reduce the probability of line blockage from the precipitation of solids due to cooling, slurry lines at the Hanford Site are now insulated. It is predicted that no more than a 20°F temperature drop will be experienced in the 6.5-mile pipeline at a flow rate of

[1] Internal Letter Report by R. L. McKay, "TWRS Retrieval Technology Project, Slurry Transport Plugging Investigation," Pacific Northwest National Laboratory, Richland, Washington (1993).

4.5 ft/sec, specific gravity of 1.5, and an initial temperature of 200°F (WHC 1993; WHC 1996). Even though Hanford slurry lines have always operated at or above the critical velocity, the minimum velocity for most slurry transfer systems has been increased to 6 ft/sec. This velocity is well above the minimum critical velocity for Hanford slurries, which is estimated at around 2 ft/sec (Hudson 1996).

Furthermore, the waste will be diluted with hot water (up to 200°F) to decrease the probability of precipitation or gel formation, and the pumps can provide greater pressure (up to 1000 psi) to dislodge any blockage that may form in the pipe (WHC 1996). The dilution water can be pH-controlled to theoretically prevent the precipitation of aluminates. However, a retrieved tank may not be homogenous, so sampling may not provide accurate or adequate information about the concentration of aluminates and other relevant species. Moreover, certain aspects of aluminate chemistry are not fully understood (e.g., kinetics, morphology of the precipitates, and the effect of other ionic species in the solution on the precipitation of aluminates). Therefore, pH control may not prevent the blockage of the pipeline with aluminate precipitates.

If the cross-site transfer line were to plug, long-term delays could be experienced as the replacement pipelines are designed, constructed, and tested. These delays would magnify the cost of pipeline replacement. For example, if slurry-transfer problems cause delays in the processing of Hanford tank waste by the private vendors, DOE would be subject to significant financial liabilities. DOE likely would be required to cover the costs of the private vendor on a daily basis if feed were not delivered as originally scheduled. Equally important is the loss of public and political trust that could occur with such a failure of the slurry-transfer system. Therefore, additional enhancements that improve the reliability of the slurry-transfer systems are desired.

Oak Ridge Gunite and Associated Tanks (GAAT)

ORNL was established in 1943 with the purpose to serve as a pilot facility for production operations at Hanford, Washington. Since then, six large underground concrete (Gunite) tanks, designated as tanks W-5 through W-10, were constructed to collect and store wastes that might be generated. These tanks have a volume capacity of 643,000 L (170,000 gal). They have an inside diameter of ~15.2 m (50 ft), a side-wall height of ~3.7 m (12 ft), and a dome height of ~5.5 m (18 ft) at the center. Six smaller Gunite tanks and four stainless steel tanks constitute the remainder of the GAAT. These tanks eventually became an integral part of the ORNL waste system (ORNL 1995).

The radioactive, hazardous, and other chemical wastes that have been stored in the tanks were routinely treated with caustic to adjust the pH to 10 or greater. Compounds that were insoluble in high-pH solutions precipitated and settled in the tanks. When the Gunite tanks were taken out of service in 1980, approximately 1.5 million L (400,000 gal) of sludge containing between 0.5 and 1 million curies had accumulated in the tanks (ORNL 1995).

A program to empty the six original tanks and dispose of the accumulated sludge began in 1977. Process development, system design, and facility construction were completed by June 1982. In approximately 18 months of operation, most of the sludge in five of the six tanks was removed. Approximately 90% of the sludge was resuspended and transferred to the Melton Valley Storage Tanks (MVSTs) (ORNL 1995).

The goal of the GAAT Remediation Project is to remove the remaining sludge and to permanently close the tanks. Currently, this sludge and the accumulated liquids are being removed from each tank and consolidated in GAAT Tank W-9. The tanks will be cleaned to the extent practical with existing technology, and the residual contamination will be characterized in preparation for final site closure under the Comprehensive Environmental Response, Compensation, and Liability Act (CERCLA) as part of a site-wide action. The waste consolidated in Tank W-9 will be resuspended, conditioned, and prepared for pipeline transfer to the MVSTs. When it is transferred to the MVSTs, the GAAT sludge will be consolidated with other transuranic sludges generated at ORNL. The sludges in the MVSTs will be mobilized, treated, and disposed of as part of a separate activity.

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