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**Testing of In-Line Slurry Monitors
and Pulsair Mixers with
Radioactive Slurries**

**T. D. Hylton
C. K. Bayne**

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Chemical Technology Division

**TESTING OF IN-LINE SLURRY MONITORS AND PULSAIR MIXERS
WITH RADIOACTIVE SLURRIES**

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ACRONYMS

ANL	Argonne National Laboratory
ASTD	Accelerated Site Technology Deployment
ASTM	American Society for Testing and Materials
CERCLA	Comprehensive Environmental Response, Compensation, and Liability Act
CMST-CP	Characterization, Monitoring, and Sensor Technology Crosscutting Program
DOE	U.S. Department of Energy
EPA	Environmental Protection Agency
FBRM	Focused Beam Reflectance Measurement
GAAT	Gunitite and Associated Tanks
MVST	Melton Valley Storage Tank
ORNL	Oak Ridge National Laboratory
PNNL	Pacific Northwest National Laboratory
PVC	polyvinyl chloride
RSD	relative standard deviation
SD	standard deviation
SMTL	Slurry Monitoring Test Loop
SMTS	Slurry Monitoring Test System
SNL	Sandia National Laboratories
TFA	Tanks Focus Area
TOF	time of flight

TRADEMARKS

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BETE	Registered trademark of BETE Fog Nozzle, Inc., Greenfield, MA
Check-All	Registered trademark of Check-All Valve Mfg. Co., Inc., Des Moines, IA
FBRM	Pending trademark of Laser Sensor Technology, Inc., Redmond, WA
Flygt	Registered trademark of ITT Flygt Aktiebolag, Solna, Sweden
Intellution	Registered trademark of Intellution, Inc., Norwood, MA
ISOLOK	Registered trademark of Bristol Engineering Company, Yorkville, IL
Justrite	Registered trademark of Justrite Manufacturing Company, Des Plaines, IL
Lasentec	Registered trademark of Laser Sensor Technology, Inc., Redmond, WA
Omega	Registered trademark of Omega Engineering, Inc., Stamford, CT
Promass	Pending trademark of Endress + Hauser Flowtec AG, Reinach, Switzerland
Pulsair	Registered trademark of Pulsair Systems, Inc., Bellevue, WA
RTP	Registered trademark of RTP Corporation, Pompano Beach, FL

UNITS

ft	foot
g	gram
gal	gallon
h	hour
in.	inch
MHz	megahertz
min	minute
mL	milliliter
psig	pounds per square inch, gauge
W	watt
wt %	weight percent
°C	degree Celsius
μm	micrometer

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The Gunitite and Associated Tanks (GAAT) Remediation Project provided the process application for which the Slurry Monitoring Test System (SMTS) was installed. In addition to the GAAT project manager, S. D. Van Hoesen, the contributions of the following individuals associated with the GAAT Remediation Project are appreciated:

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D. Bolling	L. Holder	J. Platfoot	J. Rutenber
O. Hale	M. Johnson	D. Rice	G. Tubb

The project also received significant contributions from other individuals. J. R. Farmer of the Lockheed Martin Energy Systems Engineering Division performed the detail design of the SMTS. M. T. Hurst and D. G. Raby of the ORNL Instrumentation and Controls Division developed the data acquisition and control system for the SMTS. Sample analyses were performed by the ORNL Chemical and Analytical Sciences Division. M. K. Savage provided technical editing for this report.

EXECUTIVE SUMMARY

The U.S. Department of Energy has millions of gallons of radioactive liquid and sludge wastes that must be retrieved from underground storage tanks, transferred to treatment facilities, and processed to a final waste form. To remove the wastes from the current storage tanks, the sludge wastes will typically be mobilized and mixed with the liquid wastes to create a slurry. The slurry will then be transferred by pipeline to the desired destination. Since the slurries are radioactive, it is critically important that they are transferred safely and successfully.

To reduce the risk of plugging a pipeline, the transport properties of the slurry (e.g., density, suspended solids concentration, viscosity, particle size range) should be determined to be within acceptable limits prior to transfer and should be monitored and controlled within specified limits while the transfer is in progress. The baseline method for determining the transport properties of slurries is sampling and analysis. This method, which is very time-consuming, radiation exposure intensive, and costly, does not provide real-time information. In-line instrumentation that measures the transport properties of slurries and provides data in real time is a highly desirable option because it provides immediate feedback to controllers or operators, who can respond quickly to prevent conditions that could lead to pipeline plugging.

In FY 1997, a project was conducted to evaluate 12 in-line or in-tank slurry monitoring instruments with nonradioactive slurries for their ability to provide transport property data in real time. The transport properties that were measured included density, suspended solids concentration, viscosity, and particle size. Based on the results of the testing, the Endress + Hauser Promass 63M Coriolis meter (for measuring density) and a prototype ultrasonic instrument developed by Argonne National Laboratory (for measuring suspended solids concentration) were selected for additional testing with radioactive slurries.

The goal of this project was to design, install, demonstrate, test, and evaluate the two instruments from the FY 1997 study. In addition, the Accelerated Site Technology Deployment Program requested that the Lasentec M600P be included in the testing to measure particle size distribution. The Lasentec instrument had been cold tested by the Pacific Northwest National Laboratory. In addition, the power consumption by the recirculation pump was monitored to determine if there was a correlation between power consumption and suspended solids content.

A test loop system was designed, fabricated, and installed at the Gunitite and Associated Tanks (GAAT) Remediation Project. A Pulsair mixing system was used to create the slurries that were recirculated to and from the selected tank. During testing, samples were collected by an in-line sampling device and then analyzed in the laboratory to obtain baseline data for verifying the performance of the instruments. The testing was conducted with the recirculation pump positioned at two different depths in the tank. Three sets of operating parameters for the mixing system were tested at each pump position. After validation of the instruments, the GAAT project intends to use them as part of a waste conditioning and monitoring system for cross-site slurry transfers.

A statistical comparison of the sample results with the in-line slurry monitoring instruments was conducted to determine the bias and variance. An instrument's bias indicates how its output compares with the baseline method; however, an instrument can be calibrated to account for bias when it is known to exist. An instrument's variance indicates how it repeats the measurement for a mixture.

The results showed that the density results obtained with the Endress + Hauser Promass 63M Coriolis meter exhibited bias that was statistically indistinguishable from zero and very low variance compared with values for grab samples analyzed by the laboratory. The suspended solids concentration that was estimated from the density output from the Promass 63M Coriolis meter had some bias when compared with the laboratory analyses. The results did not show any degradation in the instrument's performance because of radiation damage.

The Lasentec M600P Focused Beam Reflectance Measurement (FBRM) could not be evaluated for bias because the laboratory's particle size analyzer was broken. An unsuccessful attempt was made to repair the instrument on-site by a manufacturer's service representative. Funds were not available to send the unit back to the manufacturer for additional maintenance or to purchase a new instrument. It was decided that the calibration data provided for the unit would serve as the criteria to show that it was performing correctly. The instrument exhibited some variance, but this could be expected for slurries in which the particles have irregular shapes. No degradation in the instrument's performance from radiation damage was observed.

The prototype ultrasonic suspended solids monitor that was developed by Argonne National Laboratory exhibited high bias and high variance, which were apparently caused by entrained air

introduced by the Pulsair mixing system. Based on the continuous data readings, the instrument appeared to work as expected until the mixing system was started. The instrument may be usable in applications that do not have entrained air. The data indicated that low-level radiation did not affect the instrument's performance.

An analysis of the recirculation pump's power consumption data versus flow rate indicated a high degree of variance. The results from this testing indicated that power consumption is not sensitive enough to the experimental parameters to be used for quality control monitoring.

No operational problems were observed with the Pulsair mixing system during the testing. The highest concentration of suspended solids obtained was ~3.1 wt % (laboratory analysis), when the recirculation pump was positioned approximately 4 ft from the bottom of the tank. Based on the concentration data obtained from the testing, it appears that a concentration gradient existed in the tank. Particle size data from the Lasentec FBRM indicated that particles with chord lengths <105 μm accounted for >99.9% (by number) of the particles. The waste transfer criteria for transporting the slurries through the cross-site pipeline require the concentration of suspended solids to be <5 wt % and the maximum particle size to be 100 μm . The Pulsair system appears to adequately satisfy the waste transfer criteria.

Based on the performance results obtained with the slurry monitoring instruments, the Endress + Hauser Promass 63M Coriolis meter and the Lasentec M600P FBRM instruments are expected to meet the requirements of the GAAT Remediation Project for measuring density and particle chord length, respectively. These instruments are expected to be deployed for process monitoring.

1. INTRODUCTION

1.1 BACKGROUND

The U.S. Department of Energy (DOE) has millions of gallons of radioactive liquid and sludge wastes that must be retrieved from underground storage tanks, transferred to treatment facilities, and processed to a final waste form. The wastes will be removed from the current storage tanks by mobilizing the sludge wastes and mixing them with the liquid wastes to create slurries. Each slurry would then be transferred by pipeline to the desired destination.

Since the wastes are radioactive, it is important that the slurries are transferred safely and successfully. If the transport properties of a given slurry increase beyond the design limits of the transfer system, a pipeline plug can occur. The consequences of plugging a pipeline with radioactive waste are unacceptable from safety, cost, and schedule perspectives. If conventional methods (e.g., water flushing) for removing pipeline plugs are not successful, either the entire pipeline must be replaced (and the plugged pipeline remediated at a later date) or the plug(s) must be located and the plugged section(s) excised and replaced. Either option would create radiation exposure to workers, and the costs and schedule delays could become untenable under these conditions. Even if the pipeline plug is removed by conventional means, delays in the schedule and additional costs to the project will still result. For example, flushing a plugged line creates additional cost due to the effort required to process the excess water.

To reduce the risk of plugging a pipeline, the transport properties of the slurry (e.g., density, suspended solids concentration, viscosity, particle size range) should be determined to be within acceptable limits prior to transfer. These properties should also be monitored and controlled within specified limits while the slurry transfer is in progress.

The baseline method for determining the transport properties of slurries is sampling and analysis. This method, which is time-consuming, exposure intensive, and costly, does not provide real-time information. Also, when dealing with solid-liquid samples, it is questionable whether a laboratory analyst can obtain a representative aliquot from the sample collection jar, particularly for those analyses that are restricted to small-volume aliquots.

An in-line instrument is one that is connected to the process, analyzes the slurry as it flows through or by the instrument, and provides the results within seconds. This type of instrumentation can be used to provide immediate feedback to operators, who can then respond

quickly to counteract conditions that could lead to pipeline plugging. Therefore, in-line instrumentation that measures the transport properties of slurries and provides data in real time is highly desired.

The problem with pumping slurries without in-line monitoring instrumentation can be illustrated by a situation at the Hanford Site, which has six cross-site slurry pipelines — approximately 6 miles long. Four of these six pipelines have been plugged, apparently due to crystallization and gelation of saturated supernates. All conventional means of unplugging them have been unsuccessful. Thus, the pipelines have been abandoned and will have to be remediated. The process is disruptive to the site, and the remediation effort will be very expensive. As a result of this problem, a new cross-site pipeline is currently being designed and built at Hanford for slurry transfers.¹

In 1995, DOE issued a call for proposals for developing in-line instrumentation to measure the transport properties of slurries. At that time, it was believed that either commercial instrumentation was not available or that the existing commercial instrumentation would not work properly in the needed applications. In response to the call for proposals, several researchers submitted proposals and were funded to develop slurry monitoring instruments. Before the instruments were installed in a radioactive application, DOE wanted to evaluate them under nonradioactive conditions to determine if they were accurate, reliable, and dependable. In FY 1997, nine pipeline and three in-tank instruments were evaluated for monitoring density, suspended solids concentration, viscosity, and particle size distribution.² Based on the results of the testing, the project sponsor selected the Endress + Hauser Promass 63M Coriolis meter (for measuring density) and the prototype ultrasonic monitor developed by Argonne National Laboratory (ANL) (for measuring suspended solids concentration) as the best prospects for demonstration with a radioactive application. The Advanced Site Technology Deployment (ASTD) Program requested that the Lasentec M600P instrument also be included in the testing. Although the Lasentec instrument was not included in the FY 1997 test program, it had previously been evaluated by the Pacific Northwest National Laboratory (PNNL).³

In order to retrieve the wastes from the tanks, the sludges will be suspended in liquid and transferred through a pipeline to the treatment facility. For the testing described in this report, a Pulsair mixing system was selected for a sludge mobilization and mixing demonstration. The

mixing system was designed by the Applied Physics Laboratory at the University of Washington in conjunction with Pulsair, Inc., under the direction of PNNL. This mixing system was cold tested at PNNL and at the Oak Ridge National Laboratory (ORNL) prior to its installation in the radioactive tank.

1.2 PROJECT OBJECTIVES

The objectives of this project were to demonstrate, test, and evaluate two factors: (1) the ability of the Pulsair mixing system to mobilize and mix the tank wastes in the selected tank and (2) the performance of selected in-line instruments for monitoring the transport properties of radioactive slurries. The performance of the mixing system was evaluated by results obtained from grab sampling and from data obtained from the in-line slurry monitoring instruments. The baseline method for comparing the performance of the instruments was grab sampling followed by laboratory analysis. Two measures of quantitative performance were used to evaluate slurry monitoring instruments:

- bias: the difference between the average of the measurement readings by a slurry monitoring instrument and the accepted reference value, and
- variance: a measure of dispersion of replicate measurement readings by a slurry monitoring instrument.

2. DESCRIPTIONS OF SITE, EQUIPMENT, AND INSTRUMENTATION

The Gunitite and Associated Tanks (GAAT) Remediation Project at ORNL was selected as the radioactive application for testing the selected instruments. The GAAT are being remediated as an action under the Comprehensive Environmental Response, Compensation, and Liability Act (CERCLA). The activities that are being performed as part of the remediation include the removal of sludge and supernate from the tanks and the subsequent cross-site transfer of these materials via pipeline.

The criteria for transferring slurries through ORNL's cross-site transfer pipeline are that the maximum concentration of suspended solids be 5 wt % and the particle size be no larger than 100 μm . The ORNL transfer specification is based on the transfer line's unique susceptibility to settling blockage in the event of loss of pumping power. This type of blockage is attributed to several deep dips in the elevation of the pipeline as it follows the topography on the way to the Melton Valley Storage Tanks (MVSTs) .

2.1 SITE DESCRIPTION

ORNL, formerly known as the Clinton Laboratories, originated in 1943. Its purpose was to serve as a pilot facility for production operations at Hanford, Washington. Six large underground concrete (Gunitite) tanks, designated as tanks W-5 through W-10, were constructed for the collection and storage of wastes that might be generated (see Fig. 2.1). These tanks have a volume capacity of 643,000 L (170,000 gal). They have an inside diameter of 50 ft, a sidewall height of 12 ft, and a dome height of 18 ft at the center. Six smaller Gunitite tanks and four stainless steel tanks constitute the remainder of the GAAT. These tanks eventually became an integral part of the ORNL waste system.⁴

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 Gunitite tanks were taken out of service in 1980, approximately 1.5 million liters (400,000 gal) of sludge containing between 0.5 and 1 million curies had accumulated in the tanks.⁴

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 170,000-gal tanks was removed. Approximately 90% of the sludge was resuspended and transferred to the MVSTs.⁴

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 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.

Fig. 2.1. Layout of the Gunite tanks, the Slurry Monitoring Test System, and the Pulsair mixing system.

2.1.1 Test System Configuration

The final slurry transfer configuration is illustrated in Fig. 2.2. The sludge will be resuspended in tank W-9, the slurry will be recirculated through the Slurry Monitoring Test System (SMTS), and the transport properties will be evaluated. When the transport properties are homogenous and in the correct range, the slurry will be diverted to the cross-site pipeline and transferred to the MVSTs. After a transfer is complete, the pipeline will be flushed with one full volume of water (~1000 gal). For the purposes of the testing described in this report, the discharge pipe from the SMTS was connected only to tank W-9.

Since storage space at the MVSTs is limited, the goal is to minimize the volume of waste generated. By consolidating the sludges into one tank, the quantity of slurry transfers to the MVSTs can be reduced, and consequently, the quantity of flush water is also reduced.

Fig. 2.2. Schematic illustrating the flow path for the Slurry Monitoring Test Loop.

2.1.2 Tank Mixing

The tank contents were mixed using a technology developed by Pulsair™ Systems, Inc. The description of the methodology for this system (contained in the following paragraph) summarizes information displayed at the Pulsair website (www.pulsair.com).

This technology uses accumulator plates installed in the bottom of the tank. Compressed air is pulsed from one of the accumulator plates, which 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 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 Pulsair mixing system was installed in GAAT tank W-9 as 3 separate assemblies with a total of 13 accumulator plates, as illustrated in Fig. 2.3. The mixer assemblies were installed through existing manholes with the accumulator plates folded upwarded. After installation, the accumulator plates were extended as shown in the photograph in Fig. 2.4. Because of off-gas ventilation restrictions, the accumulator plates were operated individually rather than simultaneously. The parameters that could be adjusted were the air supply pressure, injection time (the time that the valve opens to permit air to be injected), and dwell time (the time between the air injections). Because the dwell time was viewed as the process parameter most likely to have a pronounced effect on mixing, it was varied for the testing conducted in this study.

2.2 DESCRIPTIONS OF EQUIPMENT AND PROCESS MONITORING

The SMTS was designed for safe and remote operation with radioactive slurries. A piping and instrumentation diagram of the SMTS is shown in Fig. 2.5. The test loop was designed for operation with pressures up to 300 psig. All joints were welded except for locations where instrumentation was installed. The slurry monitoring instruments were installed in spool

Fig. 2.3. Layout of accumulator plates for the Pulsair mixing system in tank W-9.

Fig. 2.4. Photograph of Pulsair mixing assembly (provided by Mike Rinker, PNNL).

CV	Check valve
HV	Hand valve
LLLW	Liquid low-level waste
PI	Pressure indicator
PT	Pressure transmitter
PW	Process water
ROV	Remote operated valve
TE	Temperature element (thermocouple)
TW	Thermocouple well

Fig. 2.5. Schematic of the Slurry Monitoring Test System.

pieces with 300-lb flange connections to simplify operations such as installation, maintenance, and replacement of an instrument. The SMTS was designed so that any or all the slurry monitoring instruments could be used or bypassed. Photographs of the exterior and interior of the SMTS are shown in Fig. 2.6.

An equipment and instrumentation list for the SMTS, not including the instruments being evaluated, is shown in Table 2.1. The test loop was constructed from 2-in. Schedule 40 carbon steel pipe with air-actuated ball valves. A water back-flush was connected to the system by a 1-in. Schedule 40 carbon steel pipe. This pipe contained a check valve to prevent radioactive slurry from entering the process water supply.

The test loop was housed in a metal enclosure that was originally manufactured for storage of six hazardous waste drums but modified for this operation. The SMTS was designed to facilitate cleanup in the event of a containment breach. The enclosure, as manufactured, had a 80% plywood/ 20% steel grate floor over a built-in sump. The flooring was changed to 100% steel grate. A drain was installed in the sump so that any spills or leaks would automatically drain back to tank W-9. Conductivity sensors were positioned in the sump to detect the presence of liquid. For temperature control, a 3200-W heater was mounted in the enclosure to keep the minimum ambient air temperature at $\sim 10^{\circ}\text{C}$. The off-gas system for tank W-9 provided ventilation to the SMTS enclosure. A 6-in. neoprene seal was added to the inside door facing to prevent liquid sprays from escaping to the environment. Flush-down nozzles were installed in the ceiling of the enclosure for removing bulk contamination in the event of a leak inside the enclosure.

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 the 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 prior to beginning data collection activities.

Information about the equipment and instrumentation used for the Pulsair equipment was provided by M. A. Johnson and is shown in Table 2.2.

(a)

(b)

(c)

(d)

Fig. 2.6. Photographs of the Slurry Monitoring Test System. (a) Exterior view of the SMTS enclosure with the top of Pulsair air distribution unit in foreground, (b) interior view of east side, (c) interior view of south side, and (d) interior view of west side.

Table 2.1. SMTL equipment and instrumentation descriptions

Item	Description
Piping	Approximately 70 ft of 2-in. carbon steel pipe
Enclosure	Justrite® Mfg. Co., Model 28160; internal dimensions: 92 in. high (including 8 in. sump) × 86 in. wide × 62 in. deep
Pump	ITT Flygt® READY 8; submersible pump installed in tank W-9
Valves	Flow-Tek F30-WCB, 2-in., flanged, full-port ball valve with AirTech® PA115SR6 spring-return, fail-close pneumatic actuator (ROV-102, -104, -105, -107, -108, -110, -111, -113) Flow-Tek 8308BW, 2-in., butt-weld, full-port ball valve with AirTech 115SR6 spring-return, fail-open pneumatic actuator (ROV-101, -103, -106, -109, -112, -114)
Check valve	Check-All® UN-3
Pressure sensor/transmitter	Onyx Valve Co., Series 40 pressure sensor; Wika® 891.23.510 pressure transmitter
Thermocouples	Omega®, Type T, Model CPIN-116U-6
Heater	Fostoria Industries, Model 222-30-TH
Spray nozzle	BETE®, Model N3, 90° spray angle
Conductivity sensor	B/W Controls, 6013-MSA-1
Control software	Intellution® Fix 32
Data acquisition hardware	RTP Corporation

Table 2.2. Pulsair equipment and instrumentation descriptions

Item	Description
External piping, fittings, and miscellaneous equipment	1.5-in. carbon steel pipe and fittings; 1.5-in. rubber hose; pressure regulator, carbon steel valves; in-line air filter; 80-gal air receiver; Unistrut support frames
Flow meter	0–1000 scfm
Injection valves	MAC Valve Co. Model 58C-53-RA
Internal piping and hardware	1.25-in. Sched. 40, 304 stainless steel pipe and fittings; 304 stainless steel plate; aluminum silicate ceramic gaskets (fabricated by the Applied Physics Laboratory at the University of Washington)
Electropneumatic interface controller	Three pilot air-injection control interfaces, manufactured by Pulsair Systems, Inc.
Pulsair mixer controller	Pulsair Model PPC 1/13

2.3 DESCRIPTIONS OF SLURRY MONITORING INSTRUMENTS

Three in-line slurry monitoring instruments, which included the Endress + Hauser Promass 63M Coriolis meter (density), Lasentec M600P (chord length distribution), and a prototype ultrasonic suspended solids monitor that was developed by ANL, were evaluated. The power consumption of the recirculation pump was also monitored to determine if the power usage was related to the suspended solids concentration. Grab samples were collected with an ISOLOK sampler and analyzed in the laboratory to determine the baseline for comparing the instrumentation results. These items are described in more detail in Sects. 2.3.1 through 2.3.5.

2.3.1 Endress + Hauser Promass 63M Coriolis Meter

A photograph of the Promass 63M Coriolis meter is shown in Fig. 2.6(d). The Promass 63M is approximately 28 in. from flange to flange. The measuring tubes were straight and constructed of titanium, and the process connections were stainless steel 300-lb flanges. The straight tubes were considered an important feature to reduce the risk of plugging the pipeline. The transmitter was remotely mounted away from the Coriolis meter to reduce radiation exposure to the electronics and to personnel who may need access to it.

The Promass 63M measures the mass flow, density, and temperature of fluids or slurries simultaneously. By determining the mass flow and the density, other functions can be calculated (e.g., volumetric flow, suspended solids concentration). Measurement of the slurry density was the major parameter evaluated in this project; however, the suspended solids concentration, as determined from the slurry density, supernate density, and dry solids density, was also assessed. The mass flow rate was not evaluated in this study but was used to determine the flow rate of the slurry through the Slurry Monitoring Test Loop (SMTL). The methods for measuring these parameters are described in more detail in Sects. 2.3.1.1 through 2.3.1.4. The information in these sections was obtained from the Promass 63 Mass Flow Measuring System Operating Manual or from other material supplied by an Endress + Hauser company representative.

The Coriolis instrument provides the user with the option of providing several variables (e.g., mass flow, volume flow, density, temperature, solids concentration); however, the model used in this test program was limited to two outputs (mass flow and density). The manufacturer advises that it now offers an additional transmitter that can provide six outputs. The suspended solids concentration was calculated based on the method shown in Sect. 2.3.1.4.

2.3.1.1 Mass flow

The measuring principle is based on the controlled generation of Coriolis forces, which are always present when translational (straight-line) and angular (rotational) movements occur simultaneously (see Fig. 2.7):

$$\vec{F}_C = 2 \cdot \Delta m (\vec{\omega} \cdot \vec{v}), \quad (1)$$

where

$$\begin{aligned} \vec{F}_C &= \text{Coriolis force,} \\ \Delta m &= \text{mass of moving body,} \\ \vec{\omega} &= \text{angular velocity,} \\ \vec{v} &= \text{radial velocity in a rotating or oscillating system.} \end{aligned}$$

The amplitude of the Coriolis force depends on the moving mass; its velocity in the system; and, therefore, its mass flow.

Fig. 2.7. Coriolis forces in the measuring tubes.

The mass flow measurement method is further described in Fig. 2.8.

2.3.1.2 Density

The measuring pipes are always made to oscillate at their resonant frequency. This excitation frequency adjusts automatically as soon as the mass, and therefore the density, of the oscillating system changes (measuring tubes and medium). The resonant frequency is thus a function of the density of the medium and enables the microprocessor to produce a density signal.

Coriolis mass flow meters are normally operated at their natural or resonant frequency. For a resonant system, a very close relationship exists between this frequency and the moving mass. With good approximation, the natural frequency of a Coriolis mass flow meter viewed as a resonant system can be written as

$$f_R = \frac{1}{2\pi} \sqrt{\frac{C}{m}}, \quad (2)$$

with

$$m = m_t + m_n \quad (3)$$

and

$$m_n = \rho_n \times V_n, \quad (4)$$

where

f_R	=	resonant or natural frequency,
C	=	mechanical stiffness or spring constant of the measuring tube arrangement,
m	=	total oscillating mass,

The Promass uses an oscillation instead of a constant angular velocity $\vec{\omega}$ and two parallel measuring tubes (Promass M and F), with fluid flowing through them, are made to oscillate in antiphase so that they act like a tuning fork.

The Coriolis forces produced at the measuring tubes cause a phase shift in the tube oscillation (see Fig. 2):

- When there is zero flow, i.e. with the fluid standing still, both tubes oscillate in phase (1).
- When there is mass flow, the tube oscillation is decelerated at the inlet (2) and accelerated at the outlet (3).

As the mass flowrate increases, the phase difference also increases (A-B). The oscillations of the measuring tubes are determined using electrodynamic sensors at the inlet and outlet.

Unlike Promass M and F, Promass A and I only have a single measuring tube. However, the measuring principle and function of all sensors are identical.

The operating principle is independent of temperature, pressure, viscosity or flow profile.

Fig. 2.8. Principle of mass flow measurement.

m_t	=	oscillating mass of measuring tube(s),
m_{fl}	=	oscillating mass of fluid within the tube(s),
V_{fl}	=	volume of fluid within the tube(s),
ρ_{fl}	=	density of fluid.

The mechanical stiffness or spring constant of the measuring tube arrangement depends on the design of the meter and Young's modulus of elasticity of the tube material. Using Eqs. (3) and (4), Eq. (2) can be solved for ρ_{fl} as shown in Eq. (5):

$$\rho_{fl} = \frac{C}{V_{fl} \cdot (2\pi f_R)^2} - \frac{m_t}{V_{fl}}, \quad (5)$$

or shortened as shown in Eq. (6):

where C_1 and C_2 are coefficients determined during the calibration process.

$$\rho_{fl} = C_1 + \frac{C_2}{f_R^2}, \quad (6)$$

2.3.1.3 Temperature

The temperature of the measuring pipes is determined and used to compensate for temperature effects. The signal produced is a function of the product temperature and can be used for external purposes.

2.3.1.4 Suspended solids concentration

By using the functions for two-phase media, it is possible to calculate the percentage mass or volume contents of the carrier fluid or the target medium. The basic formulae (without temperature compensation) are

$$\text{Mass \%} = \frac{D2 \cdot (\rho - D1)}{\rho \cdot (D2 - D1)} \times 100 \quad (7)$$

and

$$\text{Volume \%} = \frac{(\rho - D1)}{(D2 - D1)} \times 100, \quad (8)$$

where

- ρ = measured density,
- D1 = density of carrier fluid,
- D2 = density of solid phase (i.e., material transported).

Equations that include temperature compensation are shown in Appendix A. The temperature-compensated equations require volume expansion coefficients, which are not typically known for the DOE wastes.

It should be emphasized that Eqs. (7) and (8) are valid only when the densities of the carrier phase and the solids phase are constant. For example, assume that a user is pumping a slurry with a density of 1.23 g/mL and has determined the values of D1 and D2 to be 1.20 and 2.50 g/mL, respectively. The corresponding suspended solids concentration, as calculated by Eq. (7), would be 4.7 wt %. Now assume that water had been added to the mixture, reducing the value of D1 to 1.17 g/mL. Although the carrier fluid density has changed by only 0.03 g/mL, the actual suspended solids concentration would be approximately 9.2 wt %, nearly 2 times the concentration calculated for the carrier fluid density of 1.20 g/mL. Therefore, it is important to know the densities of the carrier fluid and the solid particles when using this method.

The supernate density can be determined by grab sampling and laboratory analysis. It may also be determined with the in-line Coriolis meter by allowing the solid particles to separate from the carrier phase (e.g., settling) and then recirculating the supernate through the Coriolis meter. The supernate density could also be determined simultaneously with the slurry density by taking a sidestream of the slurry, filtering the stream to remove the solid particles, and analyzing the filtered liquid with another Coriolis meter.

2.3.2 Lasentec M600P

The Lasentec M600P uses a technique known as Focused Beam Reflectance Measurement (FBRM[®]), which involves focusing a laser to a fine point. The Lasentec FBRM provides a continuous, high-speed count of particle population by dimension, making it possible to track the rate and degree of change, on the basis of both particle count and particle dimension. FBRM is a real-time, in-process measure of particle count and dimension by chord length distribution. The chord length distribution is a function of the shape and dimension of the particles and particle structures as they exist in the process.

The most intense part of the focused beam (or beam waist) is approximately 2 μm in diameter and 10 μm in depth. The light intensity is distributed across the cross section of the beam spot in a gaussian fashion, with the center being far more intense than the edges. The focal point, which is just inside the window of the probe, is then rotated around the window with a linear velocity of 2 m/s. When the focal point intersects the edge of a particle, the particle begins to backscatter light. The time of backscatter off a particular particle or agglomerate of particles is measured and counted by the instrument. The chord length (c), which is defined as the straight-line distance from one edge of the particle to the other, is determined from the known scanning velocity (v) and the duration of reflection (t), as shown in Eq. (9):

$$c = v \times t. \quad (9)$$

Additional technical information about the operational methodology of the Lasentec M600P is reprinted (with permission) from Lasentec's web site (www.Lasentec.com) in Sect. 2.3.2.1.

2.3.2.1 Technical description

A FBRM probe is inserted into a flowing medium of any concentration or viscosity. A highly focused laser beam is projected through the probe window and, at a fixed velocity, rapidly scanned across particles and particle structures flowing past the probe window (see Fig. 2.9).

When the focused beam intersects the edge of a particle, the particle will begin to backscatter the laser light. The particle will continue to backscatter until the focused beam has reached the opposite edge of the particle. This backscatter is collected by the FBRM optics and then converted into an electronic signal. A unique discrimination circuit is used to isolate the time period of the backscatter from one edge of an individual particle to its opposite edge. This time period is multiplied by the scan speed and the result is a distance (see Fig. 2.10).

This distance is a chord length. A chord length is a straight line between any two points on the edge of the particle or particle structure (agglomerate). Hundreds of thousands of chords are typically measured per second. This results in a number by chord length distribution (number of counts per second sorted by chord length into 1000 linear size bins).

Fig. 2.9. FBRM probe tip enlargement.

Fig. 2.10. Enlargement of the focused laser beam scanned across each particle and the resulting chord length.

The chord length distribution is a highly precise and sensitive means to track change to both particle dimension and particle population. In addition, with a number/length/second distribution, specific regions of the distribution can be isolated to enhance resolution (i.e. number of fine particles or number of particles over a given dimension).

No particle shape is assumed. The chord length distribution is essentially unique for any given particle size and shape distribution. Assuming, over millions of particles, the average particle shape is constant, the changes to the chord length distribution are solely a function of the change in particle dimension and particle number. Where shape is also changing, this information can typically be filtered out or enhanced depending on the goal of the application.

FBRM can be used to dynamically quantify and control the effect of process variables (temperature, addition rates, residence time, mixing speed, etc.) on a particulate system as well as quantify the effect of the particulate system on downstream performance (separation, reactivity,

dispersability, etc.). FBRM can be correlated directly with any upstream or downstream process variable or final product specification (size, rheology, zeta potential, etc.) that is a function of the particle shape, dimension, and/or number of particles in the particle system.

It should be noted that FBRM is dependent on the optical properties of the product under investigation. For any given product, FBRM will provide a highly precise and sensitive measurement of change over time, from lab to production back to lab. Unless FBRM has been calibrated for a specific set of materials, it should be considered a relative measurement. It is not intended as an absolute measurement for the comparison of the physical properties of unrelated materials.

It should also be noted that materials that do not backscatter, such as optical-grade glass beads, optically clear polystyrene, and pure oils in pure water (without surfactant), cannot be measured with FBRM.

2.3.2.2 Lasentec installation

The Lasentec instrument was procured by the Hanford Tanks Initiative Project and evaluated at PNNL with nonradioactive slurries. Kaolin clay and silica were used to simulate the solid particles in the radioactive slurries. The clay was selected to provide particles of less than 100 μm (highest fraction at $\sim 20 \mu\text{m}$), and the silica had a mean particle size of 410 μm , as measured by sieve analysis. Water was used as the fluid phase. The testing was conducted with the suspended solids concentration at 5 and 10 wt %; however, the ratio of silica to kaolin clay was varied to evaluate the effect of changing particle size. As this ratio increased, it was expected that the average particle size would also increase because more large particles would be found in the slurry.⁵ After the evaluation, PNNL shipped the instrument to ORNL for installation in the SMTS.

The Lasentec probe was installed in the SMTS pipeline in a spool piece as illustrated in Fig. 2.11. A photograph of the probe in the SMTS is shown in Fig. 2.6(b). The probe was installed at a 45° angle per the manufacturer's recommendation. This helps make the probe self-cleaning: that is, the impinging slurry keeps the sludge from building up on the surface of the probe's window. Because the window is manufactured sapphire, it is very resistant to scratches. The Lasentec instrument does not account for the velocity of particles as they pass the

Fig. 2.11. As-built drawing for the Lasentec spool piece.

probe. To offset this effect, the focal point is scanned at 2 m/s. The manufacturer recommends that the slurry velocity be ≤ 1.8 m/s; however, the flow rate should be turbulent to mix the particles in the pipe and ensure that uniformly random material is presented to the probe window. The manufacturer advises that for constant flow speeds in excess of 1.8 m/s, a constant offset will occur in the measurement, caused by the slurry flowing faster than the scan rate used for the chord length calculation.

The nominal flow rate through the system was expected to be approximately 227 L/min (60 gal/min), which corresponded to a velocity of 1.9 m/s. As shown in Fig. 2.11, the Lasentec probe was installed in a section of 2.5-in.-diam pipe. The pipe expansion reduced the slurry velocity to the Lasentec probe to approximately 1.2 m/s and thus eliminated the offset error previously discussed.

The manufacturer also recommends an optimum configuration installation, which was employed for this test. The probe was installed at a 45° angle in a vertical upflow pipe, four to five pipe diameters after an elbow or other source of turbulence. As noted previously, the flow

should be turbulent. The 1-in. pipe, through which the probe entered the 2.5-in. pipe, extended into the slurry flow and acted as a shield for the probe's housing.

2.3.3 Ultrasonic Suspended Solids Monitor

The ultrasonic suspended solids monitor was a prototype instrument that was developed, fabricated, and provided to ORNL by S.-H. Sheen and H.-T. Chien of ANL. A photograph of the instrument installed in the SMTS with a protective shield around the waveguides is shown in Fig. 2.6(c). A technical description, which was prepared by Chien and Sheen, is provided in Sect. 2.3.3.1. The references for this material (placed in brackets within the text) are provided at the end of Sect. 2.3.3.1.

2.3.3.1 Technical description

The focus of this project in FY 1998 was to develop an ultrasonic instrument for measuring percent solids in waste slurry. Close monitoring of percent solids concentration is important to the transport of tank waste because it can provide a quantitative measure of solid wastes being transported and also an indication of flow conditions, particularly the extent of solids settling. Techniques applicable to in-line measurement of solids concentration are limited; most common are optical and ultrasonic techniques. Measurements of sound velocity and attenuation are typical ultrasonic methods for solids concentration monitoring. Attenuation generally provides a better correlation with solids concentration. Ultrasonic attenuation techniques for measuring solids concentration were examined at ANL for coal slurry monitoring [1,2] and were recently studied theoretically [3]. Results indicate that attenuation measurement can be used to monitor solids concentrations up to 40% by weight.

Acoustic attenuation in a solid/liquid slurry is caused by viscous, thermal, and scattering effects. Ideally, one prefers to design a percent solids monitor that operates in the frequency range where scattering dominates. In practice, however, because of the wide ranges of particle concentrations and particle size distribution, such an optimal frequency range cannot be realized. The alternative approach is to use a fixed frequency (1 MHz in the present case) that has a wavelength much longer than the average particle size and to measure the relative attenuation of slurry with respect to the suspending fluid. The relative attenuation, α_r , can be given as

$$\alpha_r = 20 \log \frac{P_s}{P_f} = \alpha_s - \alpha_f, \quad (10)$$

where α represents attenuation, P stands for acoustic pressure, and subscripts s and f represent slurry and fluid, respectively. For the hot test at ORNL, α_f represents the attenuation in supernate. Potential error may be introduced due to the uncertainty of the supernate composition. The relative attenuation defined in Eq. (10) is then directly related to absorption by and scattering from particles. However, to determine the relative attenuation, one must know the attenuation in the suspending fluid, and this is typically obtained from off-line calibration measurements. Error will be introduced if the actual fluid of the slurry differs from the calibration fluid. Therefore, the proposed technique requires additional measurements to provide real-time signals that relate to fluid properties. Figure 2.12 shows a diagram of the instrument, which consists of a pair of transducers bonded onto stainless steel waveguides. The instrument will operate in both pitch-catch and pulse-echo modes. The pitch-catch mode measures both sound velocity and attenuated signal amplitude, while the pulse-echo mode gives an estimate of the acoustic impedance of the slurry. The acoustic impedance and sound velocity measurements will be used to characterize slurry properties, from which a correct calibration constant may be applied to the percent solids measurement.

Sound velocity also varies with solid concentration but at a smaller scale, and the direction of the variation is not generally predictable. Simple phenomenological models predict that sound velocity increases with solid concentration. But, in many cases, measurements show the opposite. The ORNL waste slurry is an example. Because sound velocity can be measured with high accuracy, it may be used to verify the attenuation measurement.

Preliminary laboratory tests have proven the measurement concept. Figure 2.13 shows a typical correlation between attenuation and solids concentration. In general, the correlation can be fitted into a linear relationship.

Fig. 2.12. Diagram of the ANL solids concentration instrument.

Fig. 2.13. Amplitude variation as a function of percentage of kaolin in sugar water.

The instrument consists of a box of control electronics, a computer for data analysis, and a sensor spool piece. Figure 2.14 shows a photograph of the control electronics and the instrument spool piece, which is a 2-in. Schedule 80 pipe on which two ultrasonic transducers are installed. The transducers are bonded onto two stainless steel waveguides that are threaded into the pipe wall and kept flush with the pipe inner surface. Use of a threaded waveguide eliminates acoustic cross talks and thus improves signal-to-noise ratio. A thermocouple is attached to one of the waveguides for temperature measurement, which is used to correct the temperature effect on measured signals.

Fig. 2.14. Ultrasonic solids concentration monitor developed at ANL.

References for Section 2.3.3.1

1. A. C. Raptis and S. H. Sheen, "Ultrasonic Properties of Coal Slurries and Flow Measurements by Cross Correlation," *IEEE Trans. on Sonics and Ultrasonics*, Vol. SU-28, No. 4, pp. 248-256, 1981.
2. S. H. Sheen, A. C. Raptis, and W. P. Lawrence, "Advanced Research in Instrumentation and Diagnostics Technology," *Proc. 12th Annual Gasification and Gas Stream Cleanup System Contractors Review Meeting*, Vol. 1, pp. 180-188, 1992.
3. S. H. Sheen, H. T. Chien, and A. C. Raptis, *Ultrasonic Methods for Measuring Liquid Viscosity and Volume Percent of Solids*, Argonne National Laboratory report, ANL 97/4, April 1997.

2.3.4 Pump Power Monitor

The power consumption of the recirculation pump was monitored with a PH-2A Power Cell sensor manufactured by Load Controls, Inc. This instrument was used to determine if any correlation existed between the suspended solids concentration and the power consumed by the pump. The model PH-2A measures true power (volts $\times$ amps $\times$ power factor). Measuring power usage is considered better than measuring amperage usage because the power factor varies based on the load.

The pump that was used for the testing described in this report was employed only for this testing. The GAAT project will replace it with a different pump for cross-site slurry transfers.

2.3.5 In-Line Sampling

A Bristol Equipment Company ISOLOK® sampling device (Model SAA-19-09-44) was installed on the test loop to obtain representative grab samples from the pipeline. This device obtained samples in the following manner (illustrated in Fig. 2.15). When a sample was needed, the operator initiated a switch that caused a probe to extend into the pipeline. The probe contained an annulus that filled with slurry. The probe was retracted, and the sample drained from the annulus into a collection jar. The probe remained in the retracted position, except during sample collection. Each extension and retraction of the probe retrieved an aliquot of approximately 8 mL. A full sample (~300 mL) was obtained by repeatedly extending and retracting the probe. The grab samples were collected by ORNL staff and analyzed at the Radioactive Materials Analytical Laboratory at ORNL.

Figure 2.15, which is based on a drawing in the manufacturer's brochure (Bulletin 593-R1), represents a sampling device used in a 5-in. pipe; therefore, the probe does not extend across the pipe. In the ORNL test loop, 2-in. diam. pipe was used, which allowed the probe to extend almost completely across the pipe.

2.3.6 Accessory Instrumentation

The SMTS included instrumentation to monitor the pressure and temperature of the slurry in the pipeline. The pressure sensor was an Onyx Series 40 Isolator Ring. This type of pressure sensor is ideal for slurries because the assembly does not have any ports that are vulnerable to plugging. The isolator ring consists of an elastomeric membrane captured in a steel ring. The assembly was installed between flanges, and the elastomeric membrane senses the pressure 360° around the pipe. The inside diameter of the assembly matches the adjacent pipe, so the ring is continually cleaned by the motion of the process fluid. The elastomeric membrane transfers the pressure to a clear fluid, which transfers the pressure measurement to the pressure transmitter. The pressure transmitter was a WIKA Model 891.23.510, with a measuring range of 0 to 400 psig.

The temperature of the slurries was measured with an Omega type-T thermocouple. It was installed in a thermocouple well to protect the probe from abrasion.

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Fig. 2.15. Illustration of sample collection with an ISOLOK device.

3. TESTING OPERATIONS

3.1 TEST PLAN DESCRIPTION

The project test plan was designed to accomplish the objectives that were stated in Sect. 1.2. The operating parameters that were variable for the Pulsair mixing system were (1) the dwell time, the time between air injections; (2) the injection time, the amount of time that air is injected through a pulse plate; and (3) the air supply pressure. Of these parameters, the dwell time was considered to have the most influence on the mixing performance. Since a concentration gradient could possibly exist in the tank, it was also deemed important to perform some tests by positioning the recirculation pump at two different depths. The Pulsair mixing system was operated under three sets of operating parameters at each pump position. Table 3.1 summarizes the slurry testing that was performed.

Table 3.1. Conditions for testing the slurry monitors

Test	Date	Tank W-9 volume (gal)	Recirculation pump position ^a (ft)	Pulsair mixing parameters		
				Dwell time (s)	Injection time (s)	Air supply pressure (psig)
1	02/17/99	104,000	4	10	1	35
2	02/22/99	104,000	4	18	1	35
3	02/25/99	103,000	4	14	1	35
4	03/02/99	105,000	6	10	1	35
5	03/05/99	105,000	6	14	1	35
6	03/11/99	113,000	6	18	1	35

^aPump position is the distance from the bottom of the tank to the pump.

The slurry monitoring instruments that were evaluated included the following: (1) Endress + Hauser Promass 63M Coriolis meter for density (also evaluated for monitoring the suspended solids concentration via density correlation), (2) Lasentec M600P for particle size analysis (also evaluated for monitoring the suspended solids concentration by direct comparison), (3) a prototype ultrasonic instrument that was developed by ANL for suspended solids concentration, and (4) Load Controls PH-2A for measuring the power consumption of the recirculation pump.

3.2 INSTRUMENT CALIBRATIONS

3.2.1 Endress + Hauser Promass 63M Coriolis Meter

A special five-point density calibration and a flow calibration was performed on the Promass 63M by the manufacturer. Copies of the manufacturer's calibration sheets are provided in Appendix B.

3.2.2 Lasentec M600P

The Lasentec instrument was calibrated by the manufacturer prior to shipment. The instrument was first calibrated with a series of optical and electrical measurements. A calibration standard is prepared from polyvinyl chloride (PVC) beads, and the instrument is calibrated against a reference instrument. The calibration standard sample is shipped with the instrument. After the instrument was set up, it was tested with the calibration standard to ensure that it was still operating correctly. The manufacturer's calibration data and those obtained on-site with the PVC standard are shown in Appendix B.

For Tests 1 through 3, the M600P was configured to count and measure particles continuously for 5-min intervals. For Tests 4 through 6, the M600P was configured to count and measure particles continuously for 1-min intervals. The change in time interval was made to provide more frequent data updates to the computer monitor. It was also recognized that 5-min averages could be obtained later when analyzing the data in a spreadsheet. The measurements were acquired using the Lasentec Control Interface for Windows, Version 5.4, Build 58 software. The software was configured in such a manner that measurement-to-measurement averaging and the "curve smoothing" function were not used. The software saved every measurement, and the channel range used was the default, 0.8–1000+ μm , log scale.

3.2.3 Ultrasonic Suspended Solids Monitor

Ultrasonic attenuation in slurries is dependent on the composition of the carrier fluid and the solid particles in the mixture; therefore, the ultrasonic suspended solids monitor had to be calibrated with the actual waste. The instrument developer requested that the calibration include a test with supernate and a test with a slurry that contained a known amount of suspended solids, preferably the highest concentration expected. After the calibration data were collected, the data

files and analytical results were sent to the developer for evaluation. Because of the tight schedule, the test runs were continued before the revised version with the appropriate calibration data was received. The instrument was used to collect the raw data; they were then converted to suspended solids concentration data by a software program provided by the instrument developer. The software program provides the suspended solids concentration based on two measurements: (1) the ultrasonic amplitude signals and (2) the measured time of flight (TOF).

During cold testing, it was observed that the amplitude signals varied significantly as the temperature changed, indicating that the instrument had a temperature dependency. The instrument developer modified the software to incorporate temperature compensation; however, this modified version was not available for the testing performed for this report.

3.2.3.1 Supernate calibration data

Supernate for the calibration was obtained by allowing time for the undissolved particles to settle below the pump suction. Since the objective was to obtain data for a clear liquid, the Pulsair mixing system was not used during this data collection.

3.2.3.2 Slurry calibration data

On February 9, 1999, a run was performed with slurry to gain understanding of the effect of the Pulsair mixing system on transport properties. The collection of quantitative data was not an objective for this run. After the recirculation pump was started, the return stream from the SMTS to tank W-9 was viewed by video camera. It appeared very dark in color. The density, as measured by the Promass 63M, was ~1.07 g/mL. After the stream was recirculated for a few minutes, the density leveled out at 1.056 g/mL. The Pulsair mixing system was started, and the density began to decrease. This decrease was unexpected because the Pulsair mixing system was expected to stir up undissolved solids that had settled on the bottom of the tank and mix them with the supernate to increase the density. After reviewing the data, it was decided that the pump, at startup, had been suspended in a concentrated phase of slurry rather than supernate and that the density decreased because the slurry mixed with a clear layer of supernate above the slurry. It was concluded that the suspended solid particles settled more slowly than originally anticipated.

On February 11, 1999, the actual calibration sample run was performed. Upon startup of the pump, it was found that the fluid had a density of ~ 1.07 g/mL, which indicated that the pump was surrounded by slurry. The testing on February 9 indicated that the concentration would decrease when the Pulsair mixing was started. Since the developer wanted a high concentration of suspended solids, it was decided to obtain the calibration sample without using the Pulsair system. The slurry was recirculated ~ 30 min prior to initiating the sample collection.

3.2.4 Pump Power Monitor

The Load Controls Model PH-2A power monitor was calibrated by the manufacturer prior to shipment. After the instrument arrived, this calibration was verified by comparison with calibrated instruments traceable to standards of the National Institute of Standards and Technology. A graph of the verification data is shown in Appendix B (Fig. B.4). A linear-regressed line is shown in the graph, along with the equation for the line and the multiple correlation coefficient (R^2).

3.3 SLURRY TEST 1

The configuration for the first test included operating the Pulsair mixing system with the following parameters: dwell time = 10 s, injection time = 1 s, and air supply pressure = 35 psig. The recirculation pump, which was ~ 4 ft from the bottom of the tank, was operated for ~ 1 h before starting the mixing system. The tank was mixed for ~ 2.5 h before sample collection. The Pulsair system was stopped ~ 12 min after acquiring the sample; however, the recirculation through the SMTS was continued for another ~ 1.5 h. The performance of the various instruments is shown in Figs. 3.1 and 3.2.

Health Physics personnel reported that the radiation field outside the SMTS enclosure while the slurry was recirculating was ~ 100 mR/h. The radiation field from the sample jar (~ 300 mL of sample) was ~ 350 mR/h at contact. These values are typical for all the tests.

3.3.1 Endress + Hauser Promass 63M Coriolis Meter

The density output from the Promass 63M for the test is shown in Fig. 3.1(a). The density data show that the pump suction was initially in or near the suspended solids interface.

Fig. 3.1. Instrumentation results from Test 1. (a) Endress + Hauser Promass 63M density and suspended solids concentration for the full run, (b) Endress + Hauser Promass 63M density and suspended solids concentration for a 30-min segment including the sampling period, (c) Lasentec M600P total number of particles and Promass 63M density comparison for the full run, (d) Lasentec M600P total number of particles and number of particles with chord lengths >105 μm , (e) ultrasonic suspended solids monitor results for the full run, and (f) pump power consumption compared with the suspended solids concentration for the full run.

Fig. 3.2. Particle chord length distribution as a function of time. (a) Test 1, (b) Test 2, (c) Test 3, (d) Test 4, (e) Test 5, and (f) Test 6.

The density of the fluid was initially high (~ 1.07 g/mL) and decreased to ~ 1.046 g/mL just by recirculation. After the Pulsair mixing system was started, the slurry density decreased to ~ 1.038 g/mL, then began to increase, and eventually attained an approximate density of 1.047 g/mL. After the mixing was stopped, the density response continued for ~ 15 min. The density then began to respond with less variance, indicating that the Pulsair mixing system was entraining air into the slurry. Figure 3.1(a) also shows the suspended solids concentration (values are read from the right axis) as determined from the density correlation (see Sect. 2.3.1.4).

Figure 3.1(b) shows a 30-min segment of the data shown in Fig. 3.1(a) and includes the sampling period. This graph shows that the slurry density was at an equilibrium when the sample was collected. The variance in the density output was caused by the sampling activity. The ISOLOK sampler puts a slug of air (~ 8 mL) into the slurry with each piston stroke. Since the Promass 63M was downstream of the in-line sampler, it responded to the air bubbles.

3.3.2 Lasentec M600P

In essence, the Lasentec probe analyzes a continuously changing sample aliquot. The total number of particles measured by the Lasentec instrument includes only those particles that come close enough to the probe window to be counted. A graph of the total number of particles measured by the Lasentec probe is shown in Fig 3.1(c), as well as the density results from the Promass 63M Coriolis meter [Fig. 3.1(a)], which are included for direct comparison. As one would expect, the results show that the particle count responded in a similar fashion to the density. When the density decreased, the particle count decreased and vice versa.

The current acceptance criteria for transferring slurries through the ORNL cross-site pipeline is that the particles be less than $100\text{ }\mu\text{m}$. The Lasentec software divides the count data by chord length into 38 preselected channels. The channel closest to $100\text{ }\mu\text{m}$ is $105\text{ }\mu\text{m}$. Figure 3.1(d) is a plot of the total number of particles and the number of particles with chord lengths $>105\text{ }\mu\text{m}$ versus time. This plot shows that $>99.9\%$ of the particles measured by the Lasentec probe had chord lengths $<105\text{ }\mu\text{m}$. Although the number of particles that were $>105\text{ }\mu\text{m}$ was small, the graph shows that this number increased slightly after the Pulsair mixing system was started, indicating that the instrument can respond to small changes.

3.3.3 Ultrasonic Suspended Solids Monitor

A plot of the results from the ultrasonic suspended solids monitor is shown in Fig. 3.1(e). The instrument initially responded similarly to the Promass 63M: that is, the suspended solids concentration began high and slowly decreased. When the Pulsair mixing system was started, the instrument began to respond to the increase in suspended solids and entrained air in the slurry. After the suspended solids concentration began to level off, the output from the instrument became erratic, probably due to saturation of entrained air in the slurry. After the Pulsair mixing was stopped, the output from the instrument became more stable and began to show a decrease in the suspended solids concentration. This was probably a response to a decrease in the concentration of entrained air.

The change in the number of particles versus particle size distribution is shown in Fig. 3.2(a) as a function of time. The times shown in the graph were arbitrarily selected to show the progression of the changes in the particle size distribution. These graphs illustrate how the instrument can be used to track the chord length distribution with regard to the mixing process and time.

3.3.4 Pump Power Monitor

A graph of the power consumption by the recirculation pump is shown in Fig. 3.1(f). The power consumption decreased slightly when the Pulsair mixing system was started and the suspended solids concentration began to decrease. Note that there are two sharp increases in the power consumption, which are directly attributable to changes in the flow rates; these are identified in the figure.

3.4 SLURRY TEST 2

The configuration for the second test included operating the Pulsair mixing system with the following parameters: dwell time = 18 s, injection time = 1 s, and air supply pressure = 35 psig. The recirculation pump was positioned ~4 ft from the bottom of the tank. In order to allow a run to start with the recirculation pump submersed in supernate, the contents in tank W-9 were allowed to settle for a longer period of time (~118 h) since the previous use of the Pulsair system.

Fluid was recirculated for ~40 min prior to starting the mixing. Graphs that illustrate the performance of the various instruments are shown in Figs. 3.2(b) and 3.3.

3.4.1 Endress + Hauser Promass 63M Coriolis Meter

The Promass 63M density results for the full run are shown in Fig. 3.3(a). This graph shows that the pump was recirculating primarily supernate prior to starting the Pulsair mixing system. The graph also illustrates that the variance of the density value improved after the mixing was stopped. This again indicates that the instrument was affected to a small extent by entrained air in the slurry that was introduced by the mixing operation.

Figure 3.3(b) shows a 30-min interval that includes the sampling period for this run. The data before and after the sample collection period indicate that the tank contents were at an equilibrium. As previously discussed, the sampling activity had a slight effect on the density output. Figures 3.3(a) and 3.3(b) show the suspended solids concentration as determined by the density correlation (see Sect. 2.3.1.4).

3.4.2 Lasentec M600P

The total number of particles measured by the Lasentec instrument is shown in Fig. 3.3(c). The Promass 63M density results are also shown in the graph for comparison purposes. As observed for the first test, the data for the total number of particles responded similarly to the density results. Since the Coriolis meter indicated that the density was low and that the pump was primarily recirculating supernate, one would expect the Lasentec instrument to show a low particle count. Figure 3.3(c) shows that the particle count started out midrange but quickly declined. The midrange count was probably due to dried film or particles on the probe window from the previous testing. The particle count for the supernate prior to starting the Pulsair system was <1000 counts/s. Figure 3.2(b) shows the change in the particle size distribution as a function of time.

Figure 3.3(d) plots the total number of particles and the number of particles with chord lengths >105 μm . The Lasentec results show that >99.9% of the particles have chord lengths <105 μm . The graph also shows that the number of particles with chord lengths >105 μm

Fig. 3.3. Instrumentation results from Test 2. (a) Endress + Hauser Promass 63M density and suspended solids concentration for the full run, (b) Endress + Hauser Promass 63M density and suspended solids concentration for a 30-min segment including the sampling period, (c) Lasentec M600P total number of particles and Promass 63M density comparison for the full run, (d) Lasentec M600P total number of particles and number of particles with chord lengths >105 μm , (e) ultrasonic suspended solids monitor results for the full run, and (f) pump power consumption compared with the suspended solids concentration for the full run.

increased when the Pulsair mixing system was started. This is another indication that the instrument was responding to changes in the system.

3.4.3 Ultrasonic Suspended Solids Monitor

A graph of the suspended solids concentration, as measured by the ultrasonic suspended solids monitor for the full run, is shown in Fig. 3.3(e). While recirculating the supernate, the instrument showed a low concentration of suspended solids. When the Pulsair system was started, the instrument responded by showing an increase in the suspended solids concentration. After ~40 min of mixing, the instrument started to respond erratically, as seen in the previous test. When the mixing was stopped, this erratic behavior also stopped. The instrument continued to indicate a high concentration of suspended solids, but the solids concentration decreased with time, indicating that the entrained air was dissipating from the slurry.

3.4.4 Pump Power Monitor

The power consumption of the recirculation pump is shown in Fig. 3.3(f), along with the suspended solids concentration as determined from the density correlation (see Sect. 2.3.1.4). The graph shows that the power consumption began to decline slightly after the pump was started and to increase slightly after the mixing system was started; however, the magnitude of these changes was very small.

3.5 SLURRY TEST 3

The recirculation pump was located ~4 ft from the bottom of the tank for this run. The Pulsair mixing system was configured with a dwell time of 14 s, an injection time of 1 s, and an air supply pressure at 35 psig. Prior to the test, the contents of tank W-9 had settled for ~67 h since the previous Pulsair mixing activity. Fluid was recirculated for ~45 min before starting the Pulsair system. Mixing was continued until the recirculation pump was stopped. The performance of the slurry monitoring instruments is illustrated in Figs. 3.2(c) and 3.4 and is discussed in more detail in Sects. 3.5.1 through 3.5.4.

Fig. 3.4. Instrumentation results from Test 3. (a) Endress + Hauser Promass 63M density and suspended solids concentration for the full run, (b) Endress + Hauser Promass 63M density and suspended solids concentration for a 30-min segment including the sampling period, (c) Lasentec M600P total number of particles and Promass 63M density comparison for the full run, (d) Lasentec M600P total number of particles and number of particles with chord lengths >105 μm , (e) ultrasonic suspended solids monitor results for the full run, and (f) pump power consumption compared with the suspended solids concentration for the full run.

3.5.1 Endress + Hauser Promass 63M Coriolis Meter

The density results for the full run are shown in Fig. 3.4(a). The suspended solids concentration data, which were determined from the density correlation (see Sect. 2.3.1.4), are also shown in the figure. The instrument response was similar to that in the second test. The density of the fluid prior to starting the Pulsair mixing system indicated that the pump was recirculating supernate.

Figure 3.4(b) shows a 30-min segment that includes the sampling period. The data that were collected before and after this period show that the tank's contents were in equilibrium during the sampling activity. As discussed for previous tests, the Promass 63M responded slightly to the injection of air bubbles from the sampler.

3.5.2 Lasentec M600P

The particle concentration measured by the Lasentec instrument is plotted as a function of time in Fig. 3.4(c). This graph also indicates that the particle count data and the density data corresponded. Figure 3.2(c) shows the change in the particle size distribution as a function of time.

Figure 3.4(d) compares the total number of particles and the number of particles with chord lengths $>105\text{ }\mu\text{m}$. Similar to findings in previous tests, the results show that $>99.9\%$ of the particles had chord lengths $<105\text{ }\mu\text{m}$.

3.5.3 Ultrasonic Suspended Solids Monitor

The results from the ultrasonic suspended solids monitor are shown in Fig. 3.4(e). The data responded in a similar manner to those in the previous test condition. As expected, the instrument responded with a low concentration of suspended solids prior to starting the Pulsair mixing system and showed an increase in the suspended solids concentration after starting the mixing. However, after mixing for $\sim 25\text{ min}$, the instrument began to respond strongly to entrained air in the slurry and its response became erratic.

3.5.4 Pump Power Monitor

The power consumption by the recirculation pump is shown in Fig. 3.4(f), along with the suspended solids concentration determined from the density correlation. The power consumption appeared to be the same regardless of whether the recirculating fluid was supernate or slurry.

3.6 SLURRY TEST 4

The position of the recirculation pump was moved from ~4 ft above the tank bottom to ~6 ft above the tank bottom for slurry tests 4, 5, and 6. The Pulsair mixing system was configured for a dwell time of 10 s, an injection time of 1 s, and an air supply pressure of 35 psig. Approximately 117 h had passed since the previous use of the Pulsair mixing system. The performance of the instruments for slurry test 4 is shown in Figs. 3.2(d) and 3.5.

3.6.1 Endress + Hauser Promass 63M Coriolis Meter

Since the recirculation pump was located at a higher position in the tank and a long settling period had occurred, the inlet for the recirculation pump was expected to be surrounded by supernate. The density data in Fig. 3.5(a) confirm that expectation. In general terms, the density curve looks similar to that seen in previous test runs. The suspended solids concentration, which was determined from the density correlation (see Sect. 2.3.1.4), is also shown in Fig. 3.5(a).

Figure 3.5(b) shows a 30-min interval within the run and includes the sampling period. The data prior to and after the sampling period confirm that the slurry contents were at an equilibrium. As seen in previous tests, the operation of the sampler affected the density results because the sampler injects a slug of air when obtaining an aliquot.

3.6.2 Lasentec M600P

The total number of particles measured by the Lasentec instrument is shown in Fig. 3.5(c), along with the density data collected with the Promass 63M. As in previous tests, responses from the two instruments corresponded. The change in the particle size distribution as a function of time is illustrated in Fig. 3.2(d).

Fig. 3.5. Instrumentation results from Test 4. (a) Endress + Hauser Promass 63M density and suspended solids concentration for the full run, (b) Endress + Hauser Promass 63M density and suspended solids concentration for a 30-min segment including the sampling period, (c) Lasentec M600P total number of particles and Promass 63M density comparison for the full run, (d) Lasentec M600P total number of particles and number of particles with chord lengths >105 μm , (e) ultrasonic suspended solids monitor results for the full run, and (f) pump power consumption compared with the suspended solids concentration for the full run.

Figure 3.5(d) is a plot of the total number of particles and the number of particles with chord lengths $>105\text{ }\mu\text{m}$. As before, the majority ($>99.9\%$) of the particles were less than $105\text{ }\mu\text{m}$.

3.6.3 Ultrasonic Suspended Solids Monitor

The data from the ultrasonic suspended solids monitor are shown in Fig. 3.5(e). The instrument response was similar to that in previous test runs. The instrument showed an increase in suspended solids concentration after the Pulsair mixing system was started. After ~ 25 min of mixing, the instrument's response became erratic.

3.6.4 Pump Power Monitor

Power consumption data from the recirculation pump are shown in Fig. 3.5(f), along with the suspended solids concentration data that were determined from the density correlation (see Sect. 2.3.1.4). The data for the power consumption show a couple of minor changes, but they do not appear to be related to changes in the suspended solids concentration.

3.7 SLURRY TEST 5

The position of the recirculation pump was ~ 6 ft above the tank bottom. The Pulsair mixing system was configured for a dwell time of 14 s, an injection time of 1 s, and an air supply pressure of 35 psig. Approximately 66 h had passed since the previous use of the Pulsair mixing system. The recirculation pump and Pulsair system were temporarily stopped for ~ 50 min to support the site operations of the GAAT project; they were then restarted, and the test was completed as planned. The performance of the slurry monitoring instruments for this test is shown in Figs. 3.2(e) and 3.6. Additional discussion is presented in Sects. 3.7.1 through 3.7.4.

3.7.1 Endress + Hauser Promass 63M Coriolis Meter

The density results obtained from the Promass 63M instrument are shown in Fig. 3.6(a). The results are similar to those obtained in previous runs. The temporary shutdown of the recirculation pump and the Pulsair mixing system is seen to have a slight effect on the density data, which is believed to be due to settling. Figure 3.6(b) shows a 30-min interval of the run

Fig. 3.6. Instrumentation results from Test 5. (a) Endress + Hauser Promass 63M density and suspended solids concentration for the full run, (b) Endress + Hauser Promass 63M density and suspended solids concentration for a 30-min segment including the sampling period, (c) Lasentec M600P total number of particles and Promass 63M density comparison for the full run, (d) Lasentec M600P total number of particles and number of particles with chord lengths >105 μm , (e) ultrasonic suspended solids monitor results for the full run, and (f) pump power consumption compared with the suspended solids concentration for the full run.

that includes the sampling period. It indicates that the tank's contents had reached an equilibrium when the sample was collected. Because of air injection, the operation of the in-line sampler was seen to affect the density results, as previously discussed.

3.7.2 Lasentec M600P

Figure 3.6(c) shows the total number of particles, as measured by the Lasentec instrument. The density data are also shown in the figure. As seen in previous runs, the particle count data and density data are complementary. The temporary shutdown of the recirculation pump and Pulsair mixing system had little effect on the Lasentec data after the operations were restarted, but it indicates that the solids were settling slowly.

Figure 3.6(d) is a plot of the total number of particles and those with chord lengths $>105\text{ }\mu\text{m}$ counted by the Lasentec instrument. These results are similar to those from previous runs and indicate that $>99.9\%$ of the particles had chord lengths $<105\text{ }\mu\text{m}$.

3.7.3 Ultrasonic Suspended Solids Monitor

The data for the ultrasonic suspended solids monitoring instrument are shown in Fig. 3.6(e). The response was similar to that seen in previous runs, except for the amplitude data, which illustrated a sudden increase in the apparent suspended solids concentration while the supernate was recirculated. The suspended solids concentration based on the TOF data did not show the same response. After ~ 15 min of mixing, the responses of the amplitude and TOF correlations became erratic.

3.7.4 Pump Power Monitor

The pump power consumption data are plotted in Fig. 3.6(f), along with the suspended solids concentration that was determined from the density correlation. The power usage appeared to be slightly lower when pumping supernate than when pumping slurry; however, it appeared to be about the same after the mixing system was started.

3.8 SLURRY TEST 6

This test was conducted with the recirculation pump located ~6 ft from the bottom of the tank. The Pulsair mixing system was configured to provide an 18-s dwell time, a 1-s injection time, and a 35-psig air supply. Figures 3.2(f) and 3.7 illustrate the performance of the instruments for this run.

3.8.1 Endress + Hauser Promass 63M Coriolis Meter

The density data obtained from the Promass 63M are shown in Fig. 3.7(a). The test results appear similar to those of previous runs. The recirculation pump was operated for a short period after the Pulsair system was stopped. The change in density data after the mixing system was shut down is another indication of a slight effect of entrained air. The suspended solids concentration determined by the density correlation (see Sect. 2.3.1.4) is also shown in the graph.

A 30-min portion of the run that includes the sampling period is shown in Fig. 3.7(b). A comparison of the data before and after the sampling period shows that the tank's contents were in equilibrium when the sample was collected.

3.8.2 Lasentec M600P

Results collected with the Lasentec instrument are shown with the density data in Fig. 3.7(c). As noted for the previous runs, these data also show that the particle count data and the density data responded in a corresponding manner. Figure 3.7(d) is a plot of the total number of particles counted and the number of particles with chord lengths $>105\text{ }\mu\text{m}$. The results are similar to those of the previous tests. Particles with chord lengths $<105\text{ }\mu\text{m}$ made up $>99.9\%$ of the particles. The change in the particle size distribution is shown in Fig. 3.2(f) as a function of time for this run.

3.8.3 Ultrasonic Suspended Solids Monitor

The data from the ultrasonic suspended solids monitor is shown in Fig. 3.7(e). As previously discussed, the instrument operated normally until the Pulsair mixing system was started. The entrained air in the slurry apparently caused the instrument to respond erratically; however, it began to respond more normally after the Pulsair mixing system was stopped.

Fig. 3.7. Instrumentation results from Test 6. (a) Endress + Hauser Promass 63M density and suspended solids concentration for the full run, (b) Endress + Hauser Promass 63M density and suspended solids concentration for a 30-min segment including the sampling period, (c) Lasentec M600P total number of particles and Promass 63M density comparison for the full run, (d) Lasentec M600P total number of particles and number of particles with chord lengths >105 μm , (e) ultrasonic suspended solids monitor results for the full run, and (f) pump power consumption compared with the suspended solids concentration for the full run.

3.8.4 Pump Power Monitor

A plot of the pump's power consumption versus the suspended solids concentration determined by density correlation (see Sect. 2.3.1.4) is shown in Fig. 3.7(f). The data show that no discernible correlation between the two parameters. The change in the power consumption in the middle of the test can be attributed to a change in the slurry flow rate.

3.9. EFFECT OF MIXING CONDITIONS

As indicated in Table 3.1, the testing was performed with the recirculation pump positioned at two depths in the tank, and the Pulsair mixing system was operated under three sets of conditions. Figure 3.8(a) compares the density results for the mixing tests conducted with the pump positioned at 4 ft above the tank bottom, and Fig. 3.8(b) compares the density results for the mixing tests conducted with the pump at 6 ft above the tank bottom. As one would expect, the data in Figs. 3.8(a) and 3.8(b) indicated that the mixing time was dependent on the dwell time. The shorter dwell time (i.e., 10 s) mixed the tank more quickly than the longer dwell times (i.e., 14 and 18 s). A slight difference was observed in the density (attributable to the suspended solids concentration) for the three mixing rates. By comparing Figs. 3.8(a) and 3.8(b), it can be seen that the equilibrium density achieved at the 4-ft pump position was higher than that achieved at the 6-ft position. These data indicate that a concentration gradient existed in the tank.

Fig. 3.8. Comparison of mixing times. (a) Recirculation pump located ~4 ft from the tank bottom and (b) recirculation pump located ~6 ft from the tank bottom.

4. ANALYTICAL RESULTS AND STATISTICAL ANALYSIS

4.1 ANALYTICAL RESULTS

The density and suspended solids concentration results from the grab sample analyses are shown in Table 4.1. The radioactivity analyses, which are also included for information purposes, show that ^{137}Cs was the principal radioisotope. The analytical procedures for the density, solids concentration, and particle size distribution are discussed in Sects. 4.1.1 through 4.1.3.

Table 4.1. Analytical results for density, solids concentration, and radioactivity

Test	Density ^a (g/mL)		Solids concentration ^{b,c} (mg/L)			Radioactivity (Bq/mL)	
	Slurry	Supernate	Suspended	Dissolved	Total	^{137}Cs	Total
1	1.043	1.020	32,400	22,600	54,200	3.1E+5	4.7E+5
2	1.040	NR ^d	29,700	21,700 ^e	51,400	2.9E+5	4.3E+5
3	1.041	NR ^d	28,600	22,300 ^e	50,900	2.7E+5	4.1E+5
4	1.035	1.019	23,400	21,400	45,200	2.7E+5	3.9E+5
5	1.034	1.019	21,900	21,300	43,300	2.4E+5	3.6E+5
6	1.035	1.018	22,300	21,300	43,300	2.4E+5	3.4E+5

^aThe density results were reported with a confidence interval of ± 0.010 g/mL (1%).

^bThe solids concentration results were reported with a confidence interval of 10%.

^cTo convert the solids concentration units from mg/L to wt %, divide by (slurry density $\times$ 10,000).

^dNR = not requested.

^eThis value was determined from the difference between the total and suspended solids concentrations rather than by analytical determination.

4.1.1 Density

The density analyses were based on standard procedure D70-97, issued by the American Society for Testing and Materials.⁶ Modifications to the procedure included the following: (1) the use of radioactive slurry, instead of asphalt, samples; (2) not heating the samples as specified in Steps 10.1–10.4 of the procedure; and (3) analyzing each sample at the same temperature at which it was collected and analyzed by the Coriolis meter ($\pm 0.5^\circ\text{C}$).

The methodology for the density analysis begins with a special flask called a pycnometer. The tare weight and volume of the empty pycnometer are known (or determined) prior to beginning. An aliquot of sample is added to the pycnometer, which is reweighed to determine

the mass of the sample aliquot. The pycnometer, which is designed to facilitate removal of air, is then completely filled with a liquid of known density (e.g., water) and reweighed. The volume of liquid used is determined from its mass and its density. Since the volume of the pycnometer is known, the volume of sample is determined by subtraction. The density of the sample is determined by dividing its mass by its volume.

4.1.2 Solids Concentration

The total solids concentration in a sample is the sum of the total dissolved solids and the total suspended solids. The solids concentration analyses were performed by Environmental Protection Agency (EPA) standard methods.^{7,8,9} Because the samples were radioactive, the procedures were modified for small aliquot volumes to reduce radiation exposure to personnel.

The total solids concentration was determined by obtaining an aliquot of the well-mixed sample, which was then placed in a preweighed dish and reweighed. The dish was next placed in an oven and dried at 103–105°C. The sample was cooled in a desiccator and then reweighed. The drying, cooling, and reweighing cycle was repeated until a constant weight was obtained (less than 0.5-mg change). The total solids concentration was determined from the mass of the residue remaining after drying compared with that of the sample prior to drying.

The total suspended solids concentration was determined by obtaining a known-volume aliquot of the well-mixed sample. The sample was filtered to separate the dissolved and undissolved solids. The residue that was collected on the filter media was dried at 103–105°C and then cooled in a desiccator and reweighed. The drying, cooling, and reweighing cycle was repeated until a constant weight was obtained (less than 0.5-mg change). The total suspended solids concentration was determined by comparing the weight of the dried residue with that of the original sample volume.

The total dissolved solids concentration was determined by obtaining a known-volume aliquot of the well-mixed sample and filtering the sample to separate the dissolved and undissolved solids. An aliquot of the filtrate was then dried at 180°C, cooled in a desiccator, and reweighed. The drying, cooling, and reweighing cycle was repeated until a constant weight was obtained (less than 0.5-mg change). The ratio of the mass of the dried residue to the original volume of the aliquot was used to determine the concentration of total dissolved solids.

4.1.3 Particle Size

The original plan was to perform particle size analyses in the laboratory with the grab samples to compare results with the data obtained with the Lasentec FBRM; however, the laboratory particle size instrument was not functioning correctly. A manufacturer's service representative who attempted to repair the instrument on-site was unsuccessful and recommended returning the instrument to the manufacturer for repair. There were two problems with this recommendation: (1) the instrument had been contaminated with radioactive samples, and (2) funds were not available to pay for the repairs. After discussing this with the project facilitator, it was decided to rely on the calibration data supplied by Lasentec (prior to shipment) and those obtained by ORNL staff (after the unit's arrival) to qualify the data.

4.2 STATISTICAL ANALYSIS OF TEST RESULTS

The statistical method of hypothesis testing was used to compare measurements of the slurry monitoring instruments with results from the analytical laboratory. A null hypothesis is formulated that there is no difference between the expected values of the two measurements or, equivalently, that the difference between these two values is zero. A test statistic (e.g., t-statistic, F-ratio) based on averages and standard deviations of measured values is calculated. The test statistic has a theoretical probability distribution, such as a normal distribution, t-distribution, or F-distribution. If the calculated test statistic has a small probability of occurring, the null hypothesis is rejected. An alternative hypothesis—that a difference exists between the two expected values—is then accepted. For this study, a value of less than 0.05 is considered a small probability of occurring, and the difference is expressed as "significant (or not significant) at the 5% significance level." Frequently, the method of hypothesis testing is confused with the method of parameter estimation, which uses the phrase "a 95% confidence interval." Although the two methods are related, the parameter estimation terminology should not be used for results of hypothesis testing.

4.2.1 Endress + Hauser Promass 63M Coriolis Meter

The Endress + Hauser Promass 63M was positioned downstream from the sample collection device in the test loop. When samples were collected for the analytical laboratory, the operation

of the sampler introduced air bubbles into the test loop and affected the performance of the instrument. Because of this effect, the test results were examined for the 6-min period before sampling and the 6-min period after sampling, for a total of 24 data points. Table 4.2 summarizes the test results for density and suspended solids concentration recorded for these two time periods for each test run and compares them with the analytical results from the grab samples.

Table 4.2. Endress + Hauser's Promass 63M average test results for the 6 experiments, with 24 data points per experiment^a

Recirculation pump position ^b (ft)	Density (g/mL) for different Pulsair dwell times			Suspended solids (wt %) for different Pulsair dwell times		
	10 s	14 s	18 s	10 s	14 s	18 s
4	1.043	1.041	1.040	3.11	2.75	2.86
	1.0464	1.0433	1.0427	4.13	3.67	3.58
	(0.0004)	(0.0005)	(0.0004)	(0.06)	(0.07)	(0.06)
6	1.035	1.034	1.035	2.26	2.12	2.15
	1.0382	1.0356	1.0349	3.24	2.70	2.64
	(0.0008)	(0.0005)	(0.0003)	(0.13)	(0.07)	(0.05)

^aAnalytical results are in bold. Standard deviations are in parentheses.

^bPump position is the distance from the bottom of the tank to the pump.

4.2.1.1 Density bias and factor effects

The differences between the average Promass 63M density measurements and the analytical density results are estimates of the instrument bias. Both the measurement errors for the analytical results and the Promass 63M were used to determine if the instrument bias is statistically different than zero. The analytical density results were reported with a projected error of ± 0.010 g/mL. Table 4.2 shows that all average Promass 63M density measurements are greater than the analytical density results, except those for a dwell time of 18 s at a pump position of 6 ft; however, all the biases (-0.0001 to 0.003) are small. Considering both measurement errors, the biases are not significantly different from zero at the 5% significance levels.

Figure 4.1 shows the effects of dwell time and pump position on the density bias of the Promass 63M. The overall bias is lower for a pump position of 6 ft than for one of 4 ft. At 6 ft, the density bias decreases with increasing dwell times. This may be attributed to the amount of air entrained in the slurry from the Pulsair mixing system (i.e., longer dwell times result in less

entrained air). The Promass 63M had the smallest density bias for the pump position of 6 ft and the slowest mixing rate (i.e., dwell time = 18 s). For a pump position of 4 ft, the density bias decreased for a change in dwell time from 10 s to 14 s. Although the dwell times and pump positions have a statistically detectable effect on the Promass 63M density measurements, these two factors do not change the density measurements sufficiently to make the bias significantly different from zero.

The measurement of the suspended solids concentration using the density provided by the Promass 63M, the supernate density, and the dry solids density (see Sect. 2.3.1.4) follows a similar pattern to the density measurements. The analytical measurement error for suspended solids concentration was reported with a projected error of $\pm 10\%$. Table 4.1 shows that all average Promass 63M suspended solids concentration measurements are greater than the analytical results for suspended solids concentration. These averages are statistically different (5% significance level) from the analytical results. Both the analytical measurement errors and those for the Promass 63M are considered in the statistical evaluation.

Figure 4.2 shows the biases of the measurements of total suspended solids concentration. The best case is for a dwell time of 18 s at a pump position of 6 ft. In general, the bias of the suspended solids concentration decreases with increasing dwell time (i.e., slower mixing) but not

Fig. 4.1. Promass 63M biases for density measurements.

Fig. 4.2. Promass 63M biases for measurement of total suspended solids.

at the same rate for the two pump positions. Both the dwell times and pump positions have a significant effect on the suspended solids measurements obtained with the Promass 63M. However, the smallest bias for suspended solids concentration is still significantly greater than zero. As shown in Sect. 2.3.1.4, the equations for calculating the suspended solids concentration do not compensate for temperature variation, which is a potential explanation for the bias with this method. Also, the supernate and solids densities were assumed to be constant for the evaluation. This assumption was probably valid for the supernate density; however, because of the nature of the sludges, it is possible that the density of the solids varied somewhat and contributed to the variance.

4.2.1.2 Measurement variability

Table 4.1 indicates that the measurement variability for the Promass 63M is consistent for both density and suspended solids concentration over the six test conditions. The overall standard deviation (SD) for a single density measurement is 0.0005 g/mL, or a relative standard deviation (RSD) of 0.05%. This small SD indicates that density changes can be detected at the level of the third decimal point. The overall SD for a single suspended solids concentration measurement is 0.08 wt %, or an RSD of 9.8%. The precision of each of these measurements is better than the analytical laboratory's projected error.

4.2.2 Lasentec M600P

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, ∞). 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

$$Average = \sum_{j=1}^{38} X_j \times f_j$$

and

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

where

X_j = midpoint chord length of an interval,

f_j = frequency of the j^{th} interval; $j = 1, 2, \dots, 38$.

The standard deviation is calculated as the square root of the variance. Table 4.3 summarizes the estimated averages and standard deviations for the six tests. Averages and standard deviations in Table 4.3 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. Figure 4.3 shows the frequency of the midpoint chord length for particles with chord lengths $\leq 50 \mu\text{m}$. The unusually large frequency at the beginning is due to the first interval (0.8, 1.9), which contains a large number of counts.

Table 4.3. Lasentec M600P particle distribution averages and standard deviations (in parentheses) of chord lengths

Recirculation pump position ^a (ft)	Particle chord length (μm) for different Pulsair dwell times		
	10 s	14 s	18 s
4	6.30 (7.49)	6.42 (7.28)	6.43 (7.48)
6	6.39 (7.24)	6.56 (7.13)	6.42 (8.04)

^aPump position is the distance from the bottom of the tank to the pump.

Fig. 4.3. Lasentec M600P overall frequency distribution of particle size chord lengths for the six tests.

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 of the Lasentec M600P frequency distribution indicates that it can be modeled by the classical Fisher's F-distribution.¹⁰

Table 4.4 gives the cumulative distribution for the Lasentec M600P. This table shows that the median chord length is 4.25 μm and the 99.9% percentile point is about 81.5 μm . The interval containing 100 μm has a 99.962% percentile point, indicating that most of the chord lengths were below this critical value.

Table 4.4. Lasentec M600P cumulative distribution of particle size chord lengths

Channel number	Interval (μm)		Mid-chord length (μm)	Size frequency	Sum of frequency
	Low	High			
1	0.8	1.9	1.35	0.14255	0.14255
2	1.9	2.3	2.10	0.05868	0.20123
3	2.3	2.8	2.55	0.07064	0.27187
4	2.8	3.3	3.05	0.08100	0.35286
5	3.3	3.9	3.60	0.08892	0.44179
6	3.9	4.6	4.25	0.09137	0.53316
7	4.6	5.5	5.05	0.08715	0.62031
8	5.5	6.6	6.05	0.07840	0.69871
9	6.6	7.8	7.20	0.06729	0.76601
10	7.8	9.3	8.55	0.05638	0.82239
11	9.3	11.1	10.2	0.04551	0.86790
12	11.1	13.1	12.1	0.03596	0.90386
13	13.1	15.6	14.4	0.02784	0.93170
14	15.6	18.6	17.1	0.02115	0.95285
15	18.6	22.1	20.4	0.01562	0.96848
16	22.1	26.3	24.2	0.01117	0.97965
17	26.3	31.3	28.8	0.00759	0.98724
18	31.3	37.3	34.3	0.00492	0.99217
19	37.3	44.3	40.8	0.00308	0.99524
20	44.3	52.5	48.4	0.00188	0.99712
21	52.5	62.5	57.5	0.00114	0.99826
22	62.5	74.5	68.5	0.00069	0.99895
23	74.5	88.5	81.5	0.00042	0.99937
24	88.5	105	96.8	0.00025	0.99962
25	105	125	115	0.00016	0.99978
26	125	149	137	0.00009	0.99987
27	149	177	163	0.00006	0.99993
28	177	210	194	0.00003	0.99996
29	210	250	230	0.00002	0.99998
30	250	298	274	0.00001	0.99999
31	298	354	326	0.00001	0.99999
32	354	420	387	0.00000	1.00000
33	420	500	460	0.00000	1.00000
34	500	596	548	0.00000	1.00000
35	596	708	652	0.00000	1.00000
36	708	840	774	0.00000	1.00000
37	840	1000	920	0.00000	1.00000
38	1000	1000+		0.00000	1.00000

4.2.2.1 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

$$\text{Counts/s} = 11,986 + 0.858 \times (\text{Suspended Solids [mg/L]}) .$$

4.2.3 Ultrasonic Suspended Solids Monitor

The ultrasonic suspended solids monitor measures the concentration of suspended solids, in units of weight percent, by two methods: time-of-flight (TOF) and amplitude signal. Table 4.5 summarizes the average suspended solids concentration measurements on 18 data points for each of the 6 tests. These data were recorded during the period that samples were taken for analytical measurements.

Fig. 4.4. Line fitted to the Lasentec's total particle count versus suspended solids concentration data with a 95% confidence interval.

Table 4.5. Ultrasonic suspended solids average test results for the 6 experiments, with 18 data points per experiment^a

Recirculation Pump Position ^b (ft)	Conc. of suspended solids (wt %) for different Pulsair dwell times					
	Time-of-flight method			Amplitude method		
	10 s	14 s	18 s	10 s	14 s	18 s
4	3.11	2.75	2.86	3.11	2.75	2.86
	16.41	14.77	12.18	17.62	17.88	16.03
	(1.05)	(1.05)	(1.09)	(2.19)	(1.47)	(2.12)
6	2.26	2.12	2.15	2.26	2.12	2.15
	14.78	10.19	9.35	18.14	13.92	11.13
	(1.95)	(1.38)	(1.99)	(2.85)	(1.83)	(2.87)

^aAnalytical results are in bold and have a projected error of $\pm 10\%$. Standard deviations are in parentheses.

^bPump position is the distance from the bottom of the tank to the pump.

4.2.3.1 Suspended solids concentration and factor effects

The differences between the average measurements of suspended solids concentration measurements and the analytical suspended solids concentration are estimates of the instrument bias. Both the instrument measurement error and the analytical measurement error are used to determine if the instrument bias is significantly different from zero. There are large positive biases for both types of measurements, and all are significantly greater than zero. This high bias was attributed to entrained air in the slurry caused by the Pulsair mixing. This entrained-air effect could not be separated from instrument bias.

Figures 4.5 and 4.6 show the effects of dwell times and pump positions on the suspended solids concentration biases for the TOF and the amplitude measurements, respectively. For the TOF measurements, the bias decreases with both increasing dwell time and increasing pump height from the bottom of the tank (i.e., 6 ft). This decrease is more rapid with dwell time for the greater pump height. However, even when the bias is at the lowest value, it is still statistically significantly larger than zero.

The bias for suspended solids concentration by the amplitude method is larger than that for the TOF. The bias for the amplitude measurements decreases with dwell time but at different rates for the two pump positions. The bias decreases monotonically for the 6-ft pump position but decreases only slightly from a dwell time of 14 s to 18 s, which is not a statistically

significant change. Again, even the lowest bias for the amplitude weight percent measurements is statistically significantly larger than zero.

Fig. 4.5. Ultrasonic suspended solids monitor bias for TOF measurements.

Fig. 4.6. Ultrasonic suspended solids monitor bias for amplitude measurements.

4.2.3.2 Measurement variability

Table 4.5 shows the SD of the two methods of measuring the concentration of suspended solids. The overall SD for the TOF method of 1.48 wt % (RSD of 11.4%) is statistically significantly lower than the SD for the amplitude method of 2.28 wt % (RSD of 14.4%). The absolute SD for the TOF measurements is about 5 times larger than the analytical laboratory's projected error, and the absolute SD for the amplitude measurements is about 8 times larger than the analytical laboratory's projected error.

The precisions of the suspended solids concentration measurements are also affected by the pump position. For a fixed pump position, the SDs are relatively constant for the three dwell times. The SDs at the 4-ft pump position for TOF measurements are statistically significantly lower than those at the 6-ft position. The SD for the TOF method shows an overall relative increase of 59% from the 4-ft to the 6-ft pump position. The amplitude method shows an overall relative increase of 31% from the 4-ft to the 6-ft pump position, but this increase is not statistically detectable.

4.2.4 Pump Power Monitor

The power consumption (in watts) of the recirculation pump was investigated to identify any relationship with the experimental factors. Any strong correlation between power consumption and the experimental factors could be used as a quality control monitoring method. The first data set examined (1864 data points) was for the time between the start of Pulsair mixing and its termination.

The power consumption (e.g., 979 to 1121W) was fitted to the mass flow rate (457 to 534 lb/min) for the six different tests. Table 4.6 shows the intercept, slope, standard deviation, and multiple correlation coefficient ($\%R^2$) for the lines fitted to the data for each condition.

Table 4.6. Intercept, slope, standard deviation, and multiple correlation coefficient for lines for fitting power consumption (in watts) versus mass flow rate (in pounds per minute)

	<u>Pump position (relative to the tank bottom)</u>	
	4 ft	6 ft
Dwell time: 10 s		
Intercept	668.8	792.1
Slope	0.77	0.52
SD	16.3	10.4
$\%R^2$	16.6	13.9
Dwell time: 14 s		
Intercept	632.9	538.5
Slope	0.85	1.04
SD	14.7	8.0
$\%R^2$	17.0	36.9
Dwell time: 18 s		
Intercept	801.8	623.8
Slope	0.53	0.85
SD	14.5	12.2
$\%R^2$	7.8	55.1

A statistical test (5% significance level) of the equality of intercepts and slopes for the six test conditions indicates these two parameters are significantly different for the six tests. This means that different intercepts and slopes should be used for each combination of pump position and dwell time. The multiple correlation coefficient ($\%R^2$) is used to judge the amount of data

variation as explained by a fitted linear regression model of the data. Examination of the $\%R^2$ values for each combination of pump position and dwell time indicates that only a weak linear correlation exists between power consumption and mass flow rate. Figure 4.7 shows that the power consumption data has too much scatter to be a good predictor of mass flow rate.

The power consumption of the recirculation pump was also analyzed for the same sampling time period (144 data points) as the Promass 63M. This analysis examined the correlation between the power consumption (1030 to 1079 W) and the concentration of the suspended solids (2.12 to 3.11 wt %) as measured by the analytical laboratory. A line fitted to power consumption versus weight percent showed a very weak correlation ($\%R^2 = 2.9$). If the line is adjusted for both pump position and dwell time, the multiple correlation coefficient increases to only $\%R^2 = 31.4$. A better model for fitting the data is to relate power consumption ($\%R^2 = 42.9$) to the test factors (e.g., $\text{Power} = \text{Intercept} + \text{Dwell} + \text{Position} + \text{Dwell} \times \text{Position} + \text{Error}$).

This analysis-of-variance model shows that the Dwell factor and the interaction Dwell $\times$ Position are significant but that the Position factor is not significant at the 5% significance level. Figure 4.8 shows the average power values for the different combinations of the test factors. In general, power consumption is not sufficiently sensitive to the experimental parameters to be used for quality control monitoring.

Fig. 4.7. Power versus mass flow rate for the six tests. (a) Test 1, position = 4 ft, dwell time = 10 s; (b) Test 2, position = 4 ft, dwell time = 18 s; (c) Test 3, position = 4 ft, dwell time = 14 s; (d) Test 4, position = 6 ft, dwell time = 10 s; (e) Test 5, position = 6 ft, dwell time = 14 s; (f) Test 6, position = 6 ft, dwell time = 18 s.

Fig. 4.8. Average power consumption for the combinations of dwell time and pump positions. The concentrations of suspended solids (wt %) are shown in parentheses.

5. CONCLUSIONS AND RECOMMENDATIONS

Three in-line slurry monitoring instruments were demonstrated, tested, and evaluated for their capability to determine the transport properties of radioactive slurries. The instruments included the Endress + Hauser Promass 63M Coriolis meter for measuring density, the Lasentec M600P for measuring particle size distribution, and a prototype ultrasonic monitor that was developed by Argonne National Laboratory for measuring suspended solids concentration. In addition, the power consumption of the recirculation pump was monitored to determine whether this parameter could be used as a tool for in-line slurry monitoring. The Promass 63M and the M600P were also evaluated as potential indicators of suspended solids concentration. In order to use the Promass 63M as a suspended solids monitor, the densities of the fluid phase and the dry solid particle phase must be known. In addition, the fluid phase density and the dry solids density must remain constant, as any change will affect the correlation between the slurry density and the suspended solids concentration. For the M600P, the particle size distribution would need to remain relatively constant.

These instruments were demonstrated and tested at the Gunit and Associated Tanks Remediation Project at the Oak Ridge National Laboratory. The testing of the instruments was conducted in parallel with the testing of a Pulsair mixing system, which was used to mix the contents of the selected tank. A total of six tests were performed. A submersible pump was positioned at two depths, while the Pulsair system was operated at three mixing rates.

The Endress + Hauser Promass 63M Coriolis meter performed well. The statistical analysis of the results indicated the density measurement had zero bias and low variance, compared with results obtained from grab sample analyses. When evaluated as a suspended solids monitor, the instrument showed a small bias and relatively low variance. However, if the amount of bias is known, it can be corrected. The results also showed that the Promass 63M could be used as a suspended solids monitor with the given constraints.

A statistical analysis for bias could not be determined for the Lasentec M600P because the laboratory's particle size analyzer instrument was broken. An unsuccessful attempt was made by a manufacturer's service representative to repair the instrument on-site. Funding was not available to send it back to the manufacturer for repair, so a decision was made to rely on calibration data (provided by Lasentec prior to shipment and obtained by Oak Ridge National

Laboratory personnel with the same sample after the instrument arrived). From a qualitative point of view, it appears that the Lasentec instrument worked well. The data outputs from the Lasentec M600P and the Endress + Hauser Promass 63M corresponded well, meaning that when the Pulsair mixing system was started, both instruments responded to the increase or decrease in suspended solid particles in the fluid.

The prototype ultrasonic suspended solids monitor showed indications of responding to the increase of suspended solid particles in the fluid as well; however, it was strongly affected by entrained air that was introduced by the Pulsair mixing system. Although entrained air is a known weakness of ultrasonic systems, it was thought that this prototype instrument could be used because the Pulsair system introduces large air bubbles that rise to the surface quickly. Based on the results of this testing, the mixing system apparently entrains small air bubbles in the mixture as well; however, this may have been reduced if the submersible pump had been positioned further away from the accumulator plates. Before the Pulsair system was started and at the very beginning of the mixing operation, the instrument appeared to be responding correctly. It is possible that the ultrasonic suspended solids monitor could work well in applications without entrained air.

The analysis of the power consumption of the recirculation pump showed a weak correlation between this parameter and suspended solids concentration. The statistical analysis also showed a weak correlation between the flow rate and the power consumption of the pump; however, the amount of scatter in the power consumption data made it unreliable as a quality control tool.

The Pulsair mixing system operated without problem. The results show that the tank's contents reached equilibrium relatively quickly after the Pulsair system was started. The results that were obtained at the two pump depths (same Pulsair operating parameters) indicate that the equilibrium was relative to the pump position. The results imply that a concentration gradient existed in the tank. The Lasentec results also showed that >99.9% (by number) of the particles had chord lengths less than 105 μm . One of the reasons for selecting the Pulsair mixing system was that it could be operated so that it would help prevent large particles from being transferred through the cross-site slurry pipeline. The maximum concentration of suspended solids (based on analytical results) that was obtained during the testing was ~3.1 wt %, suggesting that the

Pulsair mixing system suspended only particles in the close vicinity of the accumulator plates and those particles that had not completely settled to the sludge layer.

The instrument manufacturers and developer were provided a draft copy of this report for review and comment. They were given the opportunity to correct any misstatements about their instruments. They were also requested to provide written responses to the results and comments presented in the report and informed that their responses would be included in this report. No format was given, except that they limit their response to six pages. The responses that were received from the manufacturers and the developer are included in Appendices C through E.

6. REFERENCES

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Appendix A

CALCULATION OF SUSPENDED SOLIDS FROM DENSITY DATA (TEMPERATURE COMPENSATED)

Suspended Solids Concentration Determined from Density Measurements and with Temperature Compensation

The equations shown below for determining suspended solids concentration with temperature compensation were provided by Marcel Woiton of Endress + Hauser Instruments. The equations are generic for two-phase mixtures and can be used for liquid-liquid mixtures (e.g., alcohol and water) as well as liquid-solid mixtures.

$$\text{Mass } [\%]_{\text{temp. comp.}} = \frac{\frac{D_2 \times \rho}{1 + (\alpha_2 \times \Delta t)} - \frac{D_2 \times D_1}{[1 + (\alpha_2 \times \Delta t)] \times [1 + (\alpha_1 \times \Delta t)]}}{\frac{D_2 \times \rho}{1 + (\alpha_2 \times \Delta t)} - \frac{D_1 \times \rho}{1 + (\alpha_1 \times \Delta t)}} \times 100$$

and

$$\text{Volume } [\%]_{\text{temp. comp.}} = \frac{\rho - \frac{D_1}{[1 + (\alpha_1 \times \Delta t)]}}{\frac{D_2}{1 + (\alpha_2 \times \Delta t)} - \frac{D_1}{1 + (\alpha_1 \times \Delta t)}} \times 100 ,$$

where

- ρ = measured density of medium (e.g., Promass 63 value);
- D_1 = density of carrier fluid (e.g., supernate);
- D_2 = density of target medium (e.g., dry solid particles);
- α_1 = volumetric expansion coefficient of carrier fluid, unit = K^{-1} (K = kelvin);
- α_2 = volumetric expansion coefficient of target medium, unit = K^{-1} (K = kelvin);
- Δt = $t - t_n$;
- t = actual measured temperature of medium;
- t_n = standard temperature for temperature compensation (e.g., 20°C).

Appendix B
CALIBRATION DATA

Calibration Protocol

Endress+Hauser
Flowtec

Promass 63 M

The instrument listed has been calibrated at our facility and is within the published tolerance limits. The calibration is traceable to the N.I.S.T. through standards certified at preset intervals

The calibration system in use conforms to the guidelines of MIL-STD-45662A

Device : Promass 63 M
Manufacturer : E+H Flowtec Division USA
Calibration Rig : FCP-6.C

Serial Number : 93716
Record Number : 1
Nominal Diameter : 2"
Flowrange : 700.000 lb/min
Frequency Output : 80000.00000 Hz
Water Temperature : 22.67 °C

flowrate [%]	measured mass [lb]	rated mass [lb]	deviation [%]	calibration factor	zero adjustment
10.1	35.949807	35.924833	0.07	1.5734	-98.0
49.6	176.282570	176.285240	0.00	1.5734	-98.0
74.6	264.861867	265.003796	-0.05	1.5734	-98.0
99.2	351.085517	351.228791	-0.04	1.5734	-98.0
25.0	89.651025	89.610149	0.05	1.5734	-98.0

Calibration Factor = 1.5734
Zero Adjustment = -98.0

Calibrated : 04/29/98

J. Baase

Endress+Hauser Flowtec AG - Division USA - 2350 Endress Place - Greenwood, IN 46143 - Phone 317/535-7133 - Fax 317/535-1485

Fig. B.1. Mass flow calibration data for the Promass 63M.

DENSITY CALIBRATION PROMASS M
PROTOCOL

Special calibration

Date : 24. 6.1998

Serial number : 93716

Tube number : 21047

Pipo water :

Material : Titan

Pipo air :

Nominal diameter : 50

C0 : -12469 E-1
C1 : 8152 E5
C2 : -8068 E2
C3 : 2998 E2
C2/C1 : -0.000990
C3/C1 : 0.000368
C4 : 0.0000000000E+00
C5 : 0.0000000000E+00
KM : 852.000000
KT : -276.000000

Tm [x°C]	Tt [x°C]	f1 [Hz]	Density [kg/m ³]	Deviation [kg/m ³]	p [bar]	Fluid
11.0	18.3	601.15	999.70	-0.34	2.00	water
11.2	18.2	601.10	999.68	-0.34	2.00	water
11.3	18.1	601.06	999.67	-0.36	2.00	water
4.7	14.5	602.49	1000.06	0.26	2.00	water
4.8	14.4	602.43	1000.06	0.34	2.00	water
5.1	14.4	602.36	1000.06	0.26	2.00	water
27.1	16.6	596.49	996.59	0.16	2.00	water
27.0	16.7	596.52	996.60	0.17	2.00	water
27.0	16.9	596.55	996.61	0.18	2.00	water
17.0	18.8	599.54	998.88	-0.33	2.00	water
17.1	18.8	599.50	998.86	-0.34	2.00	water
17.2	18.7	599.48	998.84	-0.34	2.00	water
23.3	19.0	597.86	997.56	-0.55	2.00	water
23.3	19.0	597.88	997.57	-0.56	2.00	water
23.2	19.1	597.89	997.58	-0.56	2.00	water
21.0	20.6	601.25	977.53	0.72	2.00	ACETONE
21.0	20.6	601.25	977.53	0.77	2.00	ACETONE
21.0	20.6	601.26	977.53	0.77	2.00	ACETONE
21.3	20.8	566.82	1256.14	0.09	2.00	CALCIUM CL
21.3	20.8	566.82	1256.14	0.04	2.00	CALCIUM CL
21.3	20.8	566.83	1256.14	0.05	2.00	CALCIUM CL

Display temperature : 70.3 °F

Display density : 1.2561 g/v

Date : 6-24-98

Visum :

Andy M. L.

Fig. B.2. Density calibration data for the Promass 63M.

Fig. B.3. Calibration data results for the Lasentec M600P.

Fig. B.4. Calibration verification data for the power monitor.

Appendix C

**RESPONSE FROM ENDRESS + HAUSER:
PROMASS 63M CORIOLIS METER (DENSITY)**

Endress + Hauser Instruments • Greenwood, Indiana

June 10, 1999

T.D. Hylton
Oak Ridge National Laboratory
Engineering Development Section
Chemical Technology Division
P.O. Box 2008
Oak Ridge, TN 37831-6330

Re: In-Line Testing of Slurry Monitors with Radioactive Slurries

Dear Mr. Hylton

With pleasure, we took notice of the "In-Line Testing of Slurry Monitors with Radioactive Slurries" evaluation report. We would like to express our thanks to you and all the people involved in the evaluation for their excellent work.

The professional way of evaluating and reporting makes this report a very valuable document.

Yours Sincerely

Marcel Woiton
Product Manager
Endress + Hauser

PS: Please allow us to attach to this letter a brief history of Endress+Hauser.

In The Beginning . . .

The group of companies known as Endress+Hauser was founded in 1953 by Georg H. Endress and Ludwig Hauser near the border region of Germany, France, and Switzerland.

It all began as a vision – the use of electronics for the remote measurement of level in tanks and silos on a reliable and accurate basis. While working as a journeyman, Georg Endress came up with the idea to change the whole level measurement industry with the use of sensors and electronics. Ludwig Hauser, a banker, helped with the finances because he was certain that the ideas of the young engineer would lead to a successful business enterprise.

Hauser's instincts were on target and the company grew and prospered in the coming decades under the leadership of Dr. Endress. At the beginning of 1995 Klaus Endress succeeded his father as CEO, but not before Klaus had served in various capacities within the organization. Dr. Endress emphasized that each of his four sons should apply for their jobs with Endress+Hauser in the normal procedure, gaining an overview and insight about the growing enterprise.

Rapid Growth Signifies Success

During the 1950s and 60s, Endress+Hauser companies were formed throughout Europe. In addition to the initial facility in Maulburg, Germany, entities were initiated or acquired in Switzerland, Holland, France, Belgium, Sweden, Austria, and England.

The first products manufactured were electronic level instruments, based on the "capacitance" principle. Additional level measurement technologies were formulated, along with the development of products to measure flow, pressure, moisture, and temperature, and a liquid analysis line. Through an acquisition in 1976, process recorders and indicators were added, further diversifying the company's product offerings.

Today, Endress+Hauser has grown to obtain worldwide status – ranking as either the market leader or among the top five companies in the measurement of level, flow, pressure, tank gauging, temperature, and industrial liquid analysis. Endress+Hauser serves a wide range of industrial sectors including the chemical, pharmaceutical, food, water, wastewater, utility, and consumer products industries.

Endress+Hauser is controlled by a holding company in Reinach, Switzerland, that serves as a coordinating structure. In addition to 16 manufacturing companies in nine countries, Endress+Hauser also maintains sales and service operations throughout the world in 33 countries. Worldwide, the company currently has more than 5,000 employees.

Entry Into The U.S. Market

In 1970 Endress+Hauser started up their U.S. operations, initially establishing headquarters in Beverly, Massachusetts. In January 1974 the U.S. facilities relocated to Greenwood, Indiana, where eight full-time employees went to work in a 5,000 square foot facility to manufacture and market level measurement systems to customers in North America.

With the successful introduction of our level measurement systems, in 1985 flow products were added, with U.S. production beginning in 1987. During that same time period, construction was completed on a 6,000 square foot flow lab that included an extremely precise flow calibration system. The north manufacturing building, encompassing nearly 46,000 square feet, was added in 1990, and in May 1991 moisture measurement operations were relocated from the West Coast to Greenwood.

To keep pace with the growing popularity of the products in the U.S., the facility itself has been the site of several expansion efforts. Since 1974, the Greenwood facility has grown from 5,000 square feet to 80,000, and most recently, with the opening of the new Sales Center, to more than 145,000 square feet. Currently, there are approximately 250 people employed at the Greenwood campus.

Maintaining Our Competitive Edge

As the world's largest independent process measurement instrumentation company, products from Endress+Hauser have earned a reputation for reliability, accuracy, and effectiveness. This reputation came about as the instruments excelled in test after test in the field.

Technological research and innovation, long the trademark, continue to be at the forefront of Endress+Hauser's goals. As part of the company's commitment to maintaining leadership status, each year 10 percent of the overall profits are reinvested in research and development. This leads to the new product developments that serve as cornerstones for the technological breakthroughs today's industrial customers require.

Each of Endress+Hauser's manufacturing facilities (known as Product Centers) focuses on a particular measuring technology. At the same time, the technology and expertise are shared among the centers. This unique philosophy gives rise to new ideas, designs, and products that are unsurpassed by any other measurement manufacturer.

The most recent addition to Endress+Hauser occurred with the 1997 acquisition of the Whessoe Group. Whessoe gives Endress+Hauser a much stronger presence in the oil and gas industries with a product line that includes vapor recovery, and tank and marine gauging, along with systems and software, and mechanical components.

"Whessoe is making Endress+Hauser stronger in areas where we have not been so strong previously," explains Dr. Endress in the current edition of *Changes*, the Endress+Hauser magazine and annual report. "The signs are that the Endress+Hauser group has indeed lastingly strengthened itself with Whessoe . . . There is still homework to be done . . ., but I am pleased to see that the people have come closer and there is a real will to move together in new directions. That is a very good precondition for success."

Endress+Hauser Today

Endress+Hauser provides the precision measuring instruments needed to accurately measure critical process parameters like level, flow, pressure, temperature, pH, and conductivity. We develop, manufacture, and sell sensors, transmitters, and systems that extract the precision information our customers require.

Today no other company supplies such a wide assortment of models, materials of construction, and electronic interfaces. We offer instruments available with a variety of digital interfaces, including HART®, RS 485, and PROFIBUS. Endress+Hauser is committed to providing the best possible solutions to the unique and challenging situations our customers encounter.

Endress+Hauser is committed to quality. Backed by unsurpassed research and development that is incorporated into every stage of design and manufacturing, our products deliver the quality our customer have come to expect today, and in the future. In the pursuit of innovation and excellence, we achieve a standard of quality without comparison. All of our instruments are manufactured to the highest standards. Our production facilities are certified to ISO 9001 and our US sales facility is certified to ISO 9002. Our customers are always assured of distinctive quality products that are tested to national and international standards.

Today's processes are becoming increasingly complex. With so many different variables in our customers process, it is important that an instrumentation supplier have a staff that provides knowledgeable technical support. Endress+Hauser has professionals committed to make sure our customers obtain the best solution possible.

Before we recommend a product for an application, we analyze the specific requirements of the process. Every detail is documented and evaluated. We also take into consideration the future needs of our customers such as expansion and growth, including these issues in our evaluation and subsequent recommendations. Endress+Hauser's wide range of measurement technologies means there is no need for us to force-fit a product to a Customer application. Every process is unique, and we want to find the most suitable fit for your process.

Endress+Hauser is committed to providing timely and competent service to our Customers. We have a complete service support staff ready to serve our customers for start-up, training, calibrations, and many other specialized services. Endress+Hauser emergency service is available to our customers 24 hours a day, seven days a week.

Appendix D

**RESPONSE FROM LASENTEC:
M600P FBRM (CHORD LENGTH DISTRIBUTION)**

July 19, 1999

Dear Readers:

We would like to thank Tom Hylton and his colleagues at ORNL for involving Lasentec in this project. ORNL has worked with us closely on the application of Lasentec FBRM technology at the Oak Ridge facility, as well as the 'Cold' Testing loop at Pacific Northwest National Labs in Hanford, Washington.

We appreciate the opportunity to comment on the technical details of the paper, as they relate to Lasentec FBRM. I believe all of the issues raised during the draft review will be addressed in the final publication.

I do wish to clarify the use of the term "Particle Size". For those in the Particle Size industry, the term Particle Size Analyzer has a very specific meaning. PSA implies that you are presenting your data as a Volume Spherical Equivalent Diameter Distribution. Also, When "Particle Size" is reported as a single scalar number, the underlying assumption is that you are reporting a "Mean Volume Spherical Equivalent Diameter". Usually the technique is assumed to be Laser Diffraction, although other techniques such as sieving and sedimentation exist.

Lasentec FBRM measures and counts chord lengths. The data is presented as a distribution- Number of counts per chord length channel per second. This chord length distribution is a function of the number, the shape, and the dimension of the particles in the system. **No shape is assumed**, and no Spherical Equivalent is calculated. From this Chord Length Distribution, we could calculate a Volume Spherical Equivalent Diameter, if we knew the average shape factor of the particles in the system. Typically we find that for applications where the particles are not spherical, this calculation would remove information on shape that is contained in the chord length distribution, effectively reducing the sensitivity of the instrument to parameters that may be of interest.

So, while the Lasentec FBRM measurement is a function of particle size, we are careful not to call it a Particle Size Analyzer. There is generally far more information in a Chord Length Distribution on the number of particles, their dimension, and their shape that we do not want to remove from the measurement to obtain a Spherical Equivalent Diameter.

It should be noted that the Lab Instrument that ORNL was planning to use to validate the Lasentec FBRM measurement is a Laser Diffraction Instrument. Unfortunately, the instrument was not available in time to complete this study. Laser Diffraction instruments measure a different physical parameter of the particles than FBRM, and as such will provide a different distribution and average size than FBRM. Laser Diffraction Data is generally presented as a Distribution of Volume Fractions, where FBRM is generally presented as Number Based distribution.

The previous work done at Hanford had compared the FBRM measurement to a Centrifugal Sedimentation technique, and to traditional Sieves. We would expect these results to be slightly different

as well, for the same reasons. All of these methods have their advantages and limitations. The method of data presentation, as well as the measuring ranges, should be noted when comparing each of these techniques.

I do believe that Tom Hylton and his colleagues have shown that Lasentec FBRM can be a precise and sensitive measurement of change in their particle system. They will be able to use the information provided by FBRM to reach the process goal. That is, to ensure that material entering the transport pipe meets certain number and size criteria. Specifically they will be able to show that there are very few particles (by number or by volume fraction) over 100um entering the transport pipe. This information could then be used to make process control decisions, such as when to recycle the slurry back to the source tank and initiate a flush of the transport pipe.

The relationships demonstrated between Lasentec FBRM and all of the other measurements in this investigation are encouraging. I look forward to working with DOE and the National Labs on the continuation of this project at ORNL and other locations.

Thank you again for the opportunity to comment on your work.

Best regards,

Jim Jernigan
Application Manager
Lasentec Corporate Headquarters, Redmond WA.
JimJ@Lasentec.com

Tel: 800-527-3683
Fax: 425-881-8964
Direct: 206-399-1036

Appendix E

**RESPONSE FROM ARGONNE NATIONAL LABORATORY:
ULTRASONIC MONITOR (SUSPENDED SOLIDS CONCENTRATION)**

Performance of Ultrasonic Suspended Solids Monitor (ANL Comments)

In this section, we would like to point out some of the characteristic features of the data and offer our interpretation and suggestions. The ANL ultrasonic instrument acquires continuously three ultrasonic signals and one temperature measurement. The three ultrasonic signals are echos reflected back from the transmitter-waveguide/slurry interface, caught signals that are transmitted through the slurry, and the time-of-flights (TOF) of the transmitted signals. Figure A-1 shows the ultrasonic signals of the six tests, from which the following distinct features can be observed.

1. Catch and TOF signals are affected by the Pulsair mixing and the severity is directly related to air-injection frequency.
2. Echo signals show negligible noise pickup from Pulsair mixing.
3. When mixing is on, TOF increases (i.e., sound velocity decreases), catch signal amplitude decreases, and echo signal amplitude increases slightly.
4. *Noise* peaks in the TOF curve appear primarily in the upper part (i.e., higher TOFs) of an apparent baseline but the opposite is observed in the catch amplitude curve.

Based on the observation, we believe that the *noise* is resulted from entrained air bubbles in the slurry because (a) the *noise* appears only in transmitted signals, (b) the noise shows higher values of TOF due to the fact that sound velocity in air is much slower, and (c) the *noise* peaks show additional attenuation in the caught signals. Neglecting the *noise* peaks, both TOF and catch-amplitude curves show baseline shifts during mixing. The shifts are directly related to the slurry properties. The TOF shift indicates a real change in slurry density because sound velocity is inversely proportional to density while the catch-amplitude change is closely related to solids concentration. If we accept the fact that the *noise* is caused by bubbles, the data also indicates that the mixed-phase flow is in the bubbly-flow region because the echo signals are relatively *noise* free.

Hot tests provided us for the first time the opportunity to evaluate the ultrasonic suspended solids monitor in an actual slurry transporting environment. Based on the test results, the following conclusions can be made.

1. Low level radiation does not affect the instrument performance.
2. Acoustic attenuation measurement can provide a linear correlation with solids concentration over the range of interest ($< 30\%$ by weight).
3. The instrument is not ready to handle gas/solid/liquid three-phase flows, however, the hot-test data suggest that the gas phase flow can be identified (e.g. TOF change) and analyzed separately.
4. The presence of air bubbles would introduce measurement errors in density and solids concentration measurements, but one cannot simply ignore the bubble effect because multiphase flows are different from simple solid/liquid two-phase flows.
5. Proposed modifications to the ultrasonic instrument are (a) development of a method to separate (or bypass) gas phase from the slurry stream and (b) development of a data analysis method to quantify the air-bubble flow and make corrections to the solid/liquid flow.

Fig. A-1 Real-time data of the six hot tests obtained by the ANL ultrasonic suspended solids monitor, TOF = time-of -flight of transmitted signal, catch amplitude = amplitude of transmitted signal, and echo amplitude = amplitude of reflected signal.

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