U.S. DEPARTMENT OF
ENERGY

PNNL-20350 FINAL

Prepared for the U.S. Department of Energy
under Contract DE-AC05-76RL01830

Hanford Tank Farms Waste Certification Flow Loop Phase IV: PulseEcho Sensor Evaluation

KM Denslow
JR Bontha
CA Burns
NN Bauman
JJ Jenks
HE Adkins
PP Schonewill
DF Hopkins

May 2011

Pacific Northwest
NATIONAL LABORATORY

Proudly Operated by Battelle Since 1965

PACIFIC NORTHWEST NATIONAL LABORATORY
operated by
BATTELLE
for the
UNITED STATES DEPARTMENT OF ENERGY
under Contract DE-AC05-76RL01830

Printed in the United States of America

Available to DOE and DOE contractors from the
Office of Scientific and Technical Information,
P.O. Box 62, Oak Ridge, TN 37831-0062;
ph: (865) 576-8401
fax: (865) 576-5728
email: reports@adonis.osti.gov

Available to the public from the National Technical Information Service,
U.S. Department of Commerce, 5285 Port Royal Rd., Springfield, VA 22161
ph: (800) 553-6847
fax: (703) 605-6900
email: orders@ntis.fedworld.gov
online ordering: http://www.ntis.gov/ordering.htm

PNNL-20350 FINAL

Hanford Tank Farms Waste Certification Flow Loop Phase IV: PulseEcho Sensor Evaluation

KM Denslow
JR Bontha
CA Burns
NN Bauman
JJ Jenks
HE Adkins
PP Schonewill
DF Hopkins

May 2011

Prepared for
the U.S. Department of Energy
under Contract DE-AC05-76RL01830

Pacific Northwest National Laboratory
Richland, Washington 99352

Executive Summary

Phase III of the Washington River Protection Solutions (WRPS) certification loop testing indicated that the PulseEcho system is an excellent candidate for detecting settled bed formation in the waste certification test loop during slurry transfer operations between Hanford tank farms and the Hanford Tank Waste Treatment and Immobilization Plant (WTP). However, the Phase III work also identified some specific areas where further development was necessary. Some of these areas included using full schedule 40 SS pipe wall thickness to mount the transducers, repeatability of the measurements, ability of PulseEcho system to detect small high density particles, lower limit of the detectable solids concentration, and effect of commonly present non-settling components in Hanford tank wastes that can influence the attenuation of the PulseEcho signal. The Phase IV of the testing presented in this report was designed to address these gaps. Testing was conducted in the Multiphase Transport Evaluation Loop (MTEL) available in the Process Development Laboratory East (PDLE) test facility of PNNL. The evaluation included the 5-MHz transducer that was previously used in Phase III and a 10-MHz transducer specifically chosen for detecting critical velocities and settled bed formation with small (>20 µm) particles. Testing was conducted with simulants specifically chosen to enable comparison with the Phase III data and also to test the limits of detection with both the 5- and 10-MHz transducers. The results obtained lead to several conclusions and recommendations:

1. For >50µm particles, the performance of the 5-MHz transducer mounted at the half wall thickness was repeatable as compared with Phase III results. In addition, the performance of the 5-MHz transducers mounted at both the full and half wall thickness yielded the same results for critical velocity. Therefore, this transducer can be mounted on a near full Schedule 40 pipe wall thickness.

2. The 5-MHz transducer was not capable of detecting critical velocities and settling of particles less than 30 µm. However, the particle concentration threshold for this transducer for 30 µm particles was not evaluated.

3. The detection of critical velocity for particles in the WRPS desired range of 20 µm and greater requires a 10-MHz transducer.

4. It is recommended that the absolute threshold mode of operation be applied with the PulseEcho system when using the 10-MHz transducer. This will allow for detection of small particle settled beds even in the highly attenuated conditions expected with many small particles distributed in the slurry.

5. For highly attenuative carrier fluids, the 10-MHz transducer was capable of detecting settling of 15-30 µm sized stainless steel particles without false indications at a particle concentration of 2 wt% or higher in the detectable size range. NOTE - This minimum concentration is applicable to the small, high density stainless steel particles utilized during Phase IV testing. Additional testing is required to determine the minimum weight percent concentrations required for other particle sizes.

6. The 10-MHz transducer performed exceptionally well, considering the small volume fraction of detectable (>14 µm) particles within the carrier fluids. However, due to the limited testing performed with this transducer, more rigorous testing is recommended to validate the sensor performance over the range of expected waste conditions that will be encountered in the waste certification loop.

The Phase IV testing demonstrated the viability and sensitivity of the 10-MHz transducers to detect settled particles that are smaller and at lower concentrations than the 5-MHz transducer. It is recommended that future testing and field deployment include the 10-MHz transducers. Because of the added capability of the 5-MHz system to probe further into the pipe flow characteristics (i.e., measure settled bed depth via normalized threshold), complementing the 10-MHz system with 5-MHz transducers should be considered as an option for a more flexible and robust system. It should be cautioned that the PulseEcho system demonstrated in the Phase IV testing is primarily focused on measuring the bulk critical velocity properties of the significant slurry components. If detection of small particle accumulation of sparsely distributed slurry components becomes a priority, then alternate detection technologies should be considered.

Acronyms and Abbreviations

ASTM	American Society for Testing and Materials
CFM	Coriolis Flow Meter
D	diameter, distance
DAS	Data Acquisition System
DOE	U.S. Department of Energy
DS	Downstream
FOV	field of view
HDI	“How Do I” (PNNL’s Standards Based Management System)
ID	inside diameter
MTEL	Multiphase Transport Evaluation Loop
NQA	nuclear quality assurance
OD	outer diameter
PDL-E	Process Development Laboratory-East
PEEK	polyetheretherketone
PNNL	Pacific Northwest National Laboratory
PSD	particle-size distribution
PZT	lead zirconate titanate
QA	quality assurance
RSD	Remote Sampler Demonstration
SS	stainless steel
UDV	Ultrasonic Doppler Velocimeter
UPE	Ultrasonic Pulse Echo
UT	Ultrasonic Transducer
VS	Visualization Section
WAC	Waste Acceptance Criteria
WRPS	Washington River Protection Solutions
WTP	Hanford Tank Waste Treatment and Immobilization Plant

Contents

Figures

Tables

1.0 Introduction

This document presents the experimental results obtained to optimize the PulseEcho system to enable detection of small (20 to 50 µm), high-density (8 to 11 g/cm^3) particles using prototypic pipe wall thickness.

Section 1.1 describes the background associated with this project. Section 1.2 presents the justification for testing. Section 1.3 lists the overall objectives for this work. Section 1.4 defines the scope of the work for Phase IV. Section 1.5 describes quality assurance (QA) requirements. Section 1.6 lists success criteria.

1.1 Background

The current baseline plan of Washington River Protection Solutions (WRPS)[1] includes a waste certification test loop that will be integrated into the Hanford Tank Waste Treatment and Immobilization Plant (WTP) feed delivery systems and will allow real-time measurement of critical velocity and settling while waste is being circulated through the transfer piping and back to the original source tank. Critical velocity during slurry transfer operations is the minimum velocity below which solids settle to the bottom of a horizontal transfer line. Once critical velocity and other analytically determined acceptance criteria are shown to meet the Waste Acceptance Criteria (WAC), the feed will be certified as acceptable for transfer to the WTP receipt tank for further treatment.

In FY2009, researchers at Pacific Northwest National Laboratory (PNNL) conducted an extensive review and assessment of currently available instruments and sensors and selected three ultrasonic instruments—PulseEcho, Ultrasonic Attenuation, and Ultrasonic Doppler Velocimeter—as the most promising candidates for detecting critical velocity and settled bed formation in the field-deployed waste certification loop (Meyer et al. 2009). Meyer et al. (2009) included a recommendation for full-scale evaluation of these instruments to establish the reliability of these instruments to measure critical velocity and to select one or two of the instruments for further investigation.

In FY2010, Phase III testing was done to establish the reliability of these instruments to detect critical velocities (Bontha et al. 2010a and 2010b). All testing was performed using an existing pipe loop that was originally designed and built to evaluate the pipeline plugging issue during slurry transfer operations at the WTP. The Multiphase Transport Evaluation Loop (MTEL), previously referred to as the “M1-Pipe Loop” and currently available at the Process Development Laboratory–East (PDL-E) facility at PNNL, was modified to include a test section containing the three instruments being evaluated along with reference instrumentation to facilitate direct comparison of the instrument response with experimentally observed critical velocities. Testing of the ultrasonic sensors was conducted with 3-in., Schedule 40 piping that was operated under typical tank farm, waste-transfer conditions and for a variety of simulated waste streams that were selected to encompass the expected high-level waste feed properties. The results of Phase III testing indicated that both PulseEcho and Ultrasonic Doppler Velocimeter (UDV) are excellent candidates for use in the waste certification loop. The results also indicated that PulseEcho is the more suitable instrument for field deployment. This is because the PulseEcho system has a distinct advantage over the UDV system in terms of the simplicity in its mounting requirements; the PulseEcho

---

[1] WRPS is the current U.S. Department of Energy contractor for Hanford tank farm operations.

transducer can be mounted on the outside of pipe whereas the UDV system requires breaching the pipe to mount the sensor assembly that includes a material with a sound velocity of approximately 2500 m/s, such as Rexolite® or polyetheretherketone (PEEK).

1.2 Test Justification

Phase III test results identified several general issues that were recommended to be addressed before deployment in the highly radioactive environments typically encountered during waste transfer operations. Specifically, the items to be addressed include optimization of transducer frequency versus sensor resolution with respect to the capability to detect small high density particles, between 20 and 50 μm, and the capability to perform reliably using prototypic pipe wall thickness.

It should be cautioned that the PulseEcho system demonstrated in the Phase IV testing is primarily focused on measuring the bulk critical velocity of the significant slurry components. If detection of small particle accumulation of sparsely distributed slurry components becomes a priority, then alternate detection technologies should be considered.

1.3 Objectives

The objective of this work is to define the recommended transducer and instrument spool piece configuration to be further tested in the upcoming remote sampling demonstration (RSD) project. The RSD demonstration project will be performed by a separate WRPS sub-contractor. The tested spool piece (or spool piece design), transparent section, and sensors shall be provided to WRPS for use in RSD demonstration activities.

1.4 Scope

The Phase IV scope addresses the following four issues associated with the PulseEcho system:

- Expanding the sensitivity of the PulseEcho ultrasound instrument to detect particulates between 20 and 50 μm and with very high densities (8 to 11 g/cc)
- Evaluate the instruments' ability to perform reliably using prototypic pipe wall thickness (3-inch, Schedule 40)
- Evaluate the effect of carrier fluid density on the performance of the PulseEcho system
- Evaluate the detection sensitivity versus PulseEcho scan time.

Bench scale and flow loop testing were performed using the same MTEL (previously called the M-1 loop system located at PDL-E) that was used for Phase III testing. The loop was modified as necessary to perform the Phase IV investigation.

1.5 Quality Assurance Requirements

Under its prime contract with the U.S. Department of Energy (DOE), PNNL's QA Program implements DOE Order 414.1C, *Quality Assurance*, and 10 CFR 830, *Nuclear Safety Management*, Subpart A, *Quality Assurance Requirements*. PNNL has adopted Nuclear Quality Assurance (NQA)-1-2000 as its single consensus standard for implementing QA requirements. A graded approach is applied to quality in accordance with NQA-1 Subpart 4.2, *Guidance for Graded Application of Quality Assurance for Nuclear-Related Research and Development*. PNNL's standards-based management system "How Do I?" (HDI) is its web-based system for communicating the QA Program requirements through laboratory-wide procedures or subject areas. All work at PNNL is subject to the applicable requirements of HDI.

Two types of instruments were used. These are instruments that were part of the PulseEcho system, and instruments that were part of reference measurement used to compare with the PulseEcho data. Instruments that were a part of the PulseEcho system consisted of the transducers and data acquisition hardware. The PulseEcho transducers that were procured for Phase IV testing were evaluated per American Society for Testing and Materials (ASTM) E1065 *Standard Guide for Evaluating Characteristics of Ultrasonic Search Units*. The PulseEcho data acquisition hardware system was checked with emulated waveforms to verify that the system and algorithm would detect signal conditions that represent those that result from sediment formation. The reference instruments consisted of a coriolis mass flow meter to measure the slurry flow rates through the flow loop and visual observations coupled with a high resolution camera to observe settled bed formation in the upstream and downstream visualization sections. The former was calibrated to a NIST traceable calibration standard while the later does not require any calibrations.

1.6 Success Criteria

The success criterion is based on the scope of work listed in Section 1.4. The criterion is the completion of testing to evaluate PulseEcho instrument performance to detect the onset of critical velocity of small (20 to 50 µm), high-density (8 to 11 g/cm^3) particles via laboratory testing at full-scale flow conditions (full-scale pipe size, wall thickness, and flow rate) using simulated waste materials.

2.0 Background: PulseEcho Ultrasound

This section presents a background on the principles behind the PulseEcho technology, its past applications, results from the Phase III evaluation, and limitations and gaps in the Phase III research.

2.1 PulseEcho Ultrasound

The ultrasonic PulseEcho system was developed at PNNL to address the challenges faced by conventional PulseEcho measurement methods during dynamic sediment detection and monitoring. The PulseEcho system uses the single-transducer, PulseEcho measurement mode; however, the system does not require coherent signal returns in the form of echo patterns to detect and measure interfaces. Rather than relying on coherent echo returns to detect interfaces, the PulseEcho system relies on obtaining incoherent ultrasonic backscatter from an ensemble of sound-scattering particles (scatterers).

Ultrasonic backscatter is the portion of sound energy that is returned to the transducer after being scattered by reflectors (e.g., glass particles in water). The concept is illustrated in Figure 2.1.

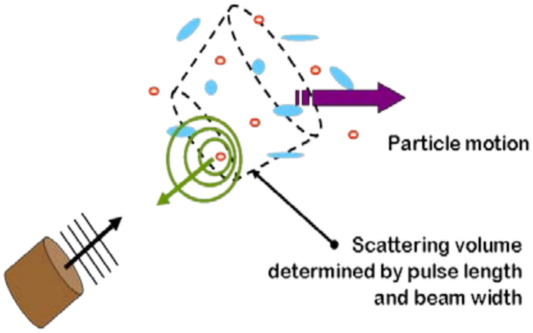

Figure 2.1. Concept of Ultrasonic Detection of Particle Motion

For backscattering to occur in a fluid, the fluid must contain materials (e.g., particles) that have acoustic impedances that are different from that of the surrounding fluid, and the wavelength of ultrasonic energy in the fluid mixture should be on the same order as the sound-scattering material. A minimum particle inventory must also exist in the sound field or insonified fluid volume to generate sufficient backscatter for a reliable measurement. The minimum number of required particles is dependent on the ultrasonic energy wavelength, the size of the sound field, and the size of the particles. When a sound field produced by an ultrasonic transducer of appropriate frequency interacts with a fluid that contains an ensemble of scatterers, backscattering occurs and is manifested in the form of amplitude-modulated signals in the time domain of real-time ultrasonic signals. Ultrasonic backscatter in the time domain is used to identify interfaces between non-moving and moving particles. Moving particles result in an amplitude-modulated ultrasonic signal, whereas stationary particles result in a non-modulated signal. The point in time where non-modulated backscatter meets modulated backscatter defines the interface between non-moving and moving particles, respectively.

2.1.1 Solids Mobility Detection

The PulseEcho system uses the backscatter measurement method to detect the onset of particle settling and subsequently the interface between settled and mobilized particles. The point in time between these regimes is not absolute because there naturally exists a modulation gradient, which represents the particle mobility gradient between non-moving and moving particles. A modulation threshold must be defined and set by the operator that defines the interface of interest. Using a variance algorithm and the user-defined threshold, the transition time between the non-modulated and modulated portions of the backscattered signals in time is defined. The simple detection of settled solids can be accomplished using this information alone; however, in combination with empirically derived *a priori* knowledge of speed-of-sound for the solids being monitored, the thickness of the settled solids can also be quantified in real time. Additional details on the PulseEcho algorithm can be found in Bontha et al. (2010a), Section 3.2.

The onset of solids settling detected by the ultrasonic transducer is typically evidenced by a fluctuation between zero and a small sediment depth value in the user readout. As solids continue to settle and increase in thickness, the transition point in time between non-modulated and modulated backscatter will continue to increase in time. This time value is continuously correlated with a user-defined speed-of-sound value to provide real-time values of sediment depth. The PulseEcho software automates the measurement process for an operator, providing a numerical readout at a rate of up to 10 per second and a graph of these data points (sediment depth vs. time) for simple visual data assimilation.

2.1.2 Prior Applications of PulseEcho

The ultrasonic PulseEcho system was developed at PNNL in 2007 and 2008 and was used on the WTP M1 and M3 projects. The purpose of the PulseEcho system was to perform non-invasive, real-time ultrasonic detection and measurement of sediment mobility and accumulation in pilot-scale pulse jet mixing vessels and the WTP M1 series initiative test loop (Poloski et al. 2009a and 2009b; Yokuda et al. 2009). The PulseEcho system was successful in detecting solids mobility in both applications.

2.2 Summary of Phase III Testing

The PulseEcho system was evaluated during Phase III of this project to establish whether this technique can be applied to detect the onset of critical velocity and settled bed formation of particles >50 µm during slurry transfer operations at Hanford. The technical basis for PulseEcho, its configuration, and the configuration of the flow loop are described in detail in Bontha et al. (2010a).

Twenty-five tests (one test was a duplicate) were conducted in Phase III testing. The simulants used in testing ranged from simple Newtonian (glass spheres in water) to complex non-Newtonian ($Zr(OH)_4$–$Al(OH)_3$–alumina mixture in kaolin-water slurries with yield stresses of up to 10 Pa). Since intermittent solids settling can occur near the critical velocity, the flow velocities at which the PulseEcho system was considered to have detected solids deposition represent those where at least 10% of the measured sediment depth values were greater than zero over the 1- to 2-minute measurement period. The results of the PulseEcho measurements were compared to the experimental measurements of the critical velocity. For the 24 unique tests, the Pulse Echo system was in excellent agreement (within the velocity range spanning Regime II (see definition of the critical velocity regimes in Table 2.1) with the experimentally observed critical velocity in 21 tests. In the other three tests, the agreement was still good (within ± 0.3 ft/s of the critical velocity), and in only one of these cases did the Pulse Echo system fail to detect solids settling before it was experimentally observed.[1] The full comparison is given in Table 2.1.

Table 2.1. Summary of Phase III Test Results: Critical Velocity Measurements

Phase III Test #	Simulant Type	Target Test Conditions(a)	Experimentally Observed Velocities (ft/s)(b)			PulseEcho Measurement (ft/s)
			R II	R III	$V_{critical}$	
1	Mono-dispersed	LLL	--	--	2.4	2.4
2	Mono-dispersed	MLL	2.75	2.65	2.55	2.65
3	Mono-dispersed	MLL	4.4	4.3	4.2	4.1
4	Broad PSD(c)	LLL	3.6	3.5	3.3	3.3
5	Broad PSD	HLL	4.2	4.1	4.0	4.1
6	Mono-dispersed	MLL	4.2	4.1	3.9	3.9
7	Mono-dispersed	MHL	2.9	2.8	2.7	3.0
8	Mono-dispersed	MLL	2.45	--	2.35	2.35
9	Binary size	LLL	3.3	3.2	3.1	3.5
10	Binary size	HLL	4.2	>4.1	4.1	4.5
11	Binary density	HLL	4.0	3.9	3.8	4.1
12	Broad PSD	HHL	~3.0	2.8	2.7	~2.6
13	Bi density Broad PSD	HLL	4.9	4.7	4.6	4.9
14	Carrier fluid-Kaolin	n/a	n/a	n/a	n/a	n/a
15	Mono-dispersed	MHH	--	--	2.1-2.3	2.1
16	Mono-dispersed	MHH	4.0	3.2	2.6-3.0	3.2
17	Broad PSD	LHH	4.2	--	3.6	4.1
18	Broad PSD	HHH	3.5	3.1	3.0	3.3
19	Mono-dispersed	LHH	--	0.3	0.2	0.5
20	Mono-dispersed	HHH	--	1.4	<1.0	1.5
21	Broad PSD	MMM	3.5	3.4	3.1-3.3	3.2
22	Complex simulant	LMM	3.5	3.4	3.1-3.3	3.3
23	Complex simulant	MHM	4.3	4.2	3.6-3.8	3.9
24	Complex simulant	MHH	5.0	4.2	4.1-4.7	4.3
25(d)	Broad PSD	HLL	4.2	4.0	3.7	4.0

(a) The test condition acronym specifies the solids concentration, viscosity, and yield stress, respectively. The target values were specified as Low (L), Medium (M), or High (H). For solids concentration, the targets were 5 wt% (L), 10 wt% (M), and 20 wt% (H), respectively (this does not include the mass of kaolin used to prepare the non-Newtonian slurries). For viscosity, the Low and High target values were 1 and 10 mPa·s, respectively. Additionally, when the non-Newtonian slurries were used, the viscosity of the carrier fluid was not controlled but was driven by the kaolin concentration necessary to achieve the target yield stress, resulting in a viscosity of ~5 mPa·s. For Yield strength, the target ranges were 0 (L), 3 (M), and 6 (H) Pa, respectively.

(b) R II and R III correspond to flow velocities where focused axial motion or a pulsating (stop/go) bed, respectively, was observed at the bottom of the pipe. $V_{critical}$ corresponds to the velocity at which a stationary bed was observed.

(c) PSD =particle size distribution.

(d) Test 25 was a repeat of Test 5.

---

[1] Phase III, Test #3: 10 wt% glass spheres (mono-disperse with d_{50} = 69.1 μm) in water; the experimental critical velocity was 4.2 ft/s; PulseEcho determined it to be 4.1 ft/s.

Other important results from Phase III testing of the PulseEcho system were the following:

- The 5-MHz frequency PulseEcho transducer used in the testing had a minimum particle size detection of 30 µm.
- The PulseEcho system typically detected settling before the observed critical velocity (15 of 24 tests), providing an “early warning” of the impending settled particle bed.
- When tested in the absence of other particles, the non-Newtonian carrier fluid (approximately 28 wt% kaolin in water) did not interfere with the capability of the system to detect settling for the particles of interest.
- For some tests with relatively low particle concentrations, some indications of settling were measured by the PulseEcho system at higher flow velocities (8, 7, and 6 ft/s) where particle settling was not observed. This occurred when insufficient particle concentrations were present in the ultrasonic transducer sound field at higher flow velocities when solids are well-dispersed. In general, these indications did not exceed the 10% detection threshold for tests when the carrier fluid was water, but did exceed it when the carrier fluid was a kaolin-water slurry.

2.3 Limitations and Gaps of Phase III Tests

The Phase III tests also identified some items that should be addressed before the PulseEcho system is deployed in the field. The objectives for the PulseEcho system during FY10 Phase III testing were limited to demonstrating the sensitivity of the 5-MHz inspection frequency to the selected particle-size range and evaluating the capability of the PulseEcho system to detect critical velocity and settled bed formation in the MTEL.

The limitations and gaps identified following Phase III testing include:

1. Phase III used a wide range of simulants, but did not specifically attempt to mimic the properties of expected WTP high-level waste feed batches. Using simulants that are more representative of the actual waste feeds would strengthen the case for using the PulseEcho system to detect particle settling. This includes particles of higher density ($> 4\ g/cm^3$).
2. Tests were not conducted at dilute solids concentrations to determine if the PulseEcho system has a significant concentration limit below which particle settling cannot be detected. The lowest concentration of any simulant testing in Phase III was 5 wt%. The detection threshold for solids concentration is a function of scattering strength, which is dependent upon the number of scatterers in the ultrasonic field as well as the attenuation of the carrier fluid. Some specific observations from Phase III testing are described in the next paragraph.

   Two Newtonian carrier fluids, water and a mixture of glycerin and water, were selected to change the viscosity from 1 to 10 millipascal seconds (mPa·s) during Phase III. Non-Newtonian mixtures of target yield strengths of 3 and 6 Pa were simulated using mixtures of kaolin in water. During Phase III Test 3 and Test 4, intermittent indications of settling were observed at 8 ft/s, 7 ft/s, 6 ft/s and 8 ft/s, 7 ft/s, 6 ft/s, and 5 ft/s, respectively. Indications of settling were observed due to the low particle concentrations near the bottom of the pipe at these high flow velocities. Ultrasonic signal modulation is absent or insufficient when either solids have settled, or there are no scatterers present in the insonified fluid volume under the test. The PulseEcho system cannot distinguish between these two conditions, and indications of settling result. For example, these requirements would not be met in clean water, such as during piping flush. During Phase III Test 3 and Test 4, indications at the high flow velocities occurred less than 10% of the time and thus did not meet the 10% criterion established for data reporting. Phase III Test 19 represented an extreme test case. The 5 wt% solids in kaolin slurry in Test 19 at 8 and 7 ft/s were challenging for the PulseEcho system as configured for Phase III. At these flow velocities, the scattering strength of the ultrasonic signal was weak due to the combined effect of low particle concentration and a highly attenuative medium (kaolin), resulting in indications of settling above the 10% criterion used for reporting. The false indication of critical velocity at 7 and 8 ft/s is due to a combination of two features of the Test 19 slurry. First, the d_{80} for this mixture is 26.6 μm. The majority of the particles in this slurry were below the ~30-μm cutoff size for the 5-MHz transducer used in this testing. This is magnified because the solids concentration for Test 19 was only 5 wt%, so at higher flow velocities, there were not enough particles present to produce detectable scattering. The particle concentration near the bottom of the pipe increased when the flow velocity reached 6 ft/s and was sufficient for enabling detection for the remainder of the low flow velocities during Test 19.

3. The 5-MHz transducer was not sensitive enough to detect particles smaller than ~30 microns. The scope of FY10 Phase III testing was limited to simulants with particle concentrations of 5 wt% or greater. The particle concentration threshold of detection for the PulseEcho system was not determined during Phase III testing in FY10. As discussed in Bontha et al. (2010a), the minimum particle concentration was not met at high flow velocities for simulants with particle concentrations of 5 wt%. However, given the 30-μm cutoff particle size, the detectable concentration by the PulseEcho system was considerably lower. The detection threshold for solids concentration is a function of scattering strength, which is dependent upon the number of scatterers in the ultrasonic field as well as the attenuation of the carrier fluid. One way to remedy this is to consider using a higher frequency transducer.

4. The PulseEcho transducer was not mounted on a pipe wall of full-thickness. To provide the most favorable measurement conditions for the Phase III test campaign, the flat on the Ultrasonic Transducer (UT) spool piece for the PulseEcho transducer was made as thin as possible. The flat was machined to a 2.5-mm (0.098-inch) pipe wall thickness to facilitate the highest sound transmittance and obtain the highest received backscatter amplitudes from the simulant particles. The PulseEcho system was evaluated during Phase III testing with this pipe wall thickness. Based on the signal strength obtained during Phase III full-scale testing, a greater number of half-wavelength pipe wall thicknesses would be permissible for future testing. It would be preferable to install non-invasive transducers, provided there was little to no loss in detection capability of the PulseEcho system.

5. The capability to reproduce results was not examined in detail. One test was repeated in Phase III (observed critical velocity and PulseEcho results were consistent between the duplicates); however, performing additional repeated tests will verify the reproducibility of the results.

6. Long-term coupling of the transducer to the pipe wall was not addressed. Epoxy was used in Phase III testing, but it is unknown if epoxy is acceptable for use in anything other than short-term testing.

7. Hardware and software improvements to reduce artificial jitter in the signal, eliminate signal saturation, provide a more stable pulse voltage to the transducer, and streamline data collection and file generation are needed.

Addressing the gaps listed above required more testing than was performed during Phase III. Some of the information is critical to better specify the performance range of the PulseEcho system and assess whether there are any slurries that are beyond the instrument’s capability to detect. Many of these issues drove the Phase IV testing discussed in this report.

3.0 PulseEcho System Adaptation to Phase IV Testing

The PulseEcho system was adapted to be sensitive to small particle sizes of 20 to 50 µm in diameter for FY11 Phase IV testing. In addition, the system was also evaluated with two pipe wall thicknesses: a pipe wall thickness equal to approximately half that of a Schedule 40 pipe and a pipe wall thickness equal to or greater than a full Schedule 40 pipe (0.216-inch). These details are presented in this section.

3.1 Transducer Frequency Selection

A higher frequency transducer is required to detect ultrasonic scattering by the 20-50 µm particle sizes of interest. The challenge for higher frequencies is to balance the attenuation components of ultrasonic scattering from particles (desired) and ultrasonic absorption by the carrier liquid (undesired). The selection of candidate frequencies for Phase IV testing was based on the originally anticipated SS particle size range of 20 to 50 µm.

The *ka* value that relates particle size to measurement wavelength can be used as an indicator for scattering strength for wavelength-particle combinations. In the following equation, $ka=\pi*(d/\lambda)$ where d = particle diameter, and λ is the wavelength in the bulk material, $\lambda = c/f$ where f is the frequency and c = the longitudinal speed of sound in the material (slurry in this case). Weak Rayleigh scattering occurs for *ka* values of <1, intermediate stochastic scattering occurs when the wavelength becomes comparable to the average particle size (*ka* ~ 1), and strong geometric scattering occurs for *ka* values of >1. During Phase III testing, scattering from all three regimes was detected for the calculated *ka* value range of approximately 0.5 to 5, which is summarized in Table 3.1.

Table 3.1. Calculated *ka* Values for Targeted Phase III Particles

Transducer Frequency f (MHz)	**Transducer Wavelength λ (µm)**	***Calculated ka value***	
		***d*=50 µm**	***d*=500 µm**
5	300	0.5	5

The calculated *ka* values for Phase IV are summarized in Table 3.2 for anticipated 20 to 50-µm particle sizes and initial candidate frequencies of 5 MHz, 10 MHz, and 15 MHz. Very weak scattering was expected for 5 MHz with *ka* values of 0.2 to 0.5, stronger scattering was anticipated for 10 MHz with *ka* values of 0.4 to 1, and good scattering was anticipated for 15 MHz with *ka* values of 0.6 to 2. However, contact transducer frequencies above 10 MHz are not practical to manufacture because the transducer face plate ultimately limits the transducer frequency to a frequency near 10 MHz. For this reason, 10 MHz was the highest frequency transducer that was procured and evaluated for Phase IV.

Table 3.2. Calculated *ka* Values for Targeted Phase IV Particles

Transducer Frequency f (MHz)	**Transducer Wavelength λ (µm)**	***Calculated ka value***	
		***d*=20 µm**	***d*=50 µm**
5 MHz	300	0.2	0.5
10 MHz	150	0.4	1.0
15 MHz	100	0.6	2.0

Modest sound field modeling and simulations were performed to support the key design for using a 10-MHz transducer on half and full schedule 40 pipe wall thicknesses. Simulations were performed using Imagine3D, Version 2.6. Imagine3D is a commercial ultrasonic ray tracing tool developed by UTEX Scientific Instrument, Inc. and was used to model the transducer and pipe for beam divergence simulations. These simulations were performed at an early stage in the project to determine how effectively a 10-MHz signal would propagate through half and full Schedule 40 pipe walls. Simulations for these scenarios are shown in Figure 3.1Figure 3.2. These simulations demonstrated that sufficient energy would propagate into a slurry contained in the pipe.

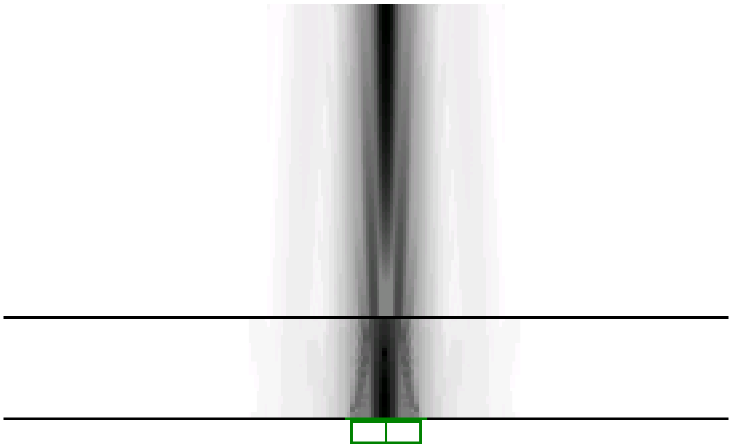

Figure 3.1. Side View of a 10-MHz, 0.25-in. Diameter Transducer on the Underside of a Water-Filled Schedule 40 SS Pipe. The sound field is represented by the black ray traces.

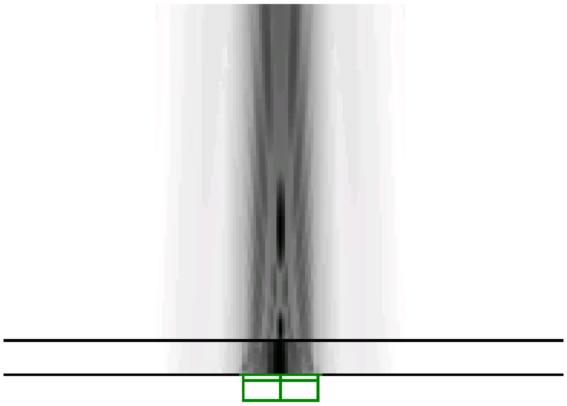

Figure 3.2. Side View of a 10-MHz, 0.25-in.-Diameter Transducer on the Underside of a Water-Filled Steel Pipe, with a Wall Thickness Half of that of a Schedule 40 Pipe. The sound field is represented by the black ray traces.

3.2 Pipe Wall Thickness

Two 5-MHz and two 10-MHz transducers were mounted onto the PulseEcho spool piece fabricated for Phase IV testing. One transducer of each frequency was mounted onto flat sections with pipe wall thicknesses that were approximately half the wall thickness of a Schedule 40 SS pipe. The other set were mounted onto flat sections that were slightly greater than the thickness of a Schedule 40 SS pipe. The actual wall thickness used was integral numbers of half- and full-wavelengths for each frequency.

3.3 PulseEcho Algorithm Thresholds

The PulseEcho software program has two thresholds for reporting conditions: a normalized variance threshold and an absolute variance threshold. The normalized threshold value that was used during Phase IV testing was equal to the normalized variance threshold that was used during Phase III testing (0.4558).

The normalized signal variance threshold is used to determine the point between non-modulated and modulated portions of a backscattered signal within a defined measurement window. This threshold is useful only when the measurement window captures both conditions. This threshold was developed to detect and measure the depth of settled solids.

The absolute variance threshold is used to determine if an ultrasonic signal is modulating above the baseline signal variance of the system. Once the absolute variance threshold is exceeded, the normalized variance threshold is activated to measure sediment thickness. During FY10 Phase III testing with the 5-MHz transducer, the absolute variance threshold was set to zero because modulated and non-modulated signal conditions were always obtainable and captured within the measurement window when solids began to settle. This can be attributed to the high scattering strength of the relatively large particles and the lower effects of carrier fluid attenuation on the 5-MHz measurement frequency.

With the introduction of the 10-MHz transducer for Phase IV testing, higher attenuation effects were observed at this measurement frequency. The presence of sediment often readily attenuated the 10-MHz signal completely, which resulted in only non-modulated conditions inside the measurement window. This became evident during validation testing with the glass bead simulants (Tests 2 and 3). Although this made the application of the normalized threshold alone a challenge, it provided an opportunity to easily distinguish between conditions of fully mobilized solids and settled/settling solids. To achieve this, the absolute threshold was set to match the baseline signal variance of the system, which is the intrinsic noise level of the system when there is no flow in the flow loop or when only water is flowing through the flow loop. For the 10-MHz signal, this baseline signal variance was a maximum of approximately $4 \cdot 10^{-5}$. Therefore, the absolute variance threshold was set to $4 \cdot 10^{-5}$. If the signal modulation exceeded this absolute threshold value during testing, then zero sediment would register. If the signal modulation was equal to or less than this value, then the presence of sediment would register. The conditions reported by the PulseEcho algorithm using this threshold could be reconciled with and verified against observations made on the system's digital oscilloscope. An example of the PulseEcho user interface is shown in Figure 3.3. The measurement window is defined in the top panel of the live ultrasonic signal. The second panel contains the averaged signal against which the individual live signals are compared to quantify signal variance. The third panel contains the signal variance profile for the defined measurement window. The absolute variance threshold is represented by the green horizontal line, and the normalized variance threshold is represented by the blue horizontal line, which correspond with values represented by the vertical scales on the left side and right side of the bottom panel, respectively. The bottom panel is a real-time readout of sediment bed thickness, which is zero for this example.

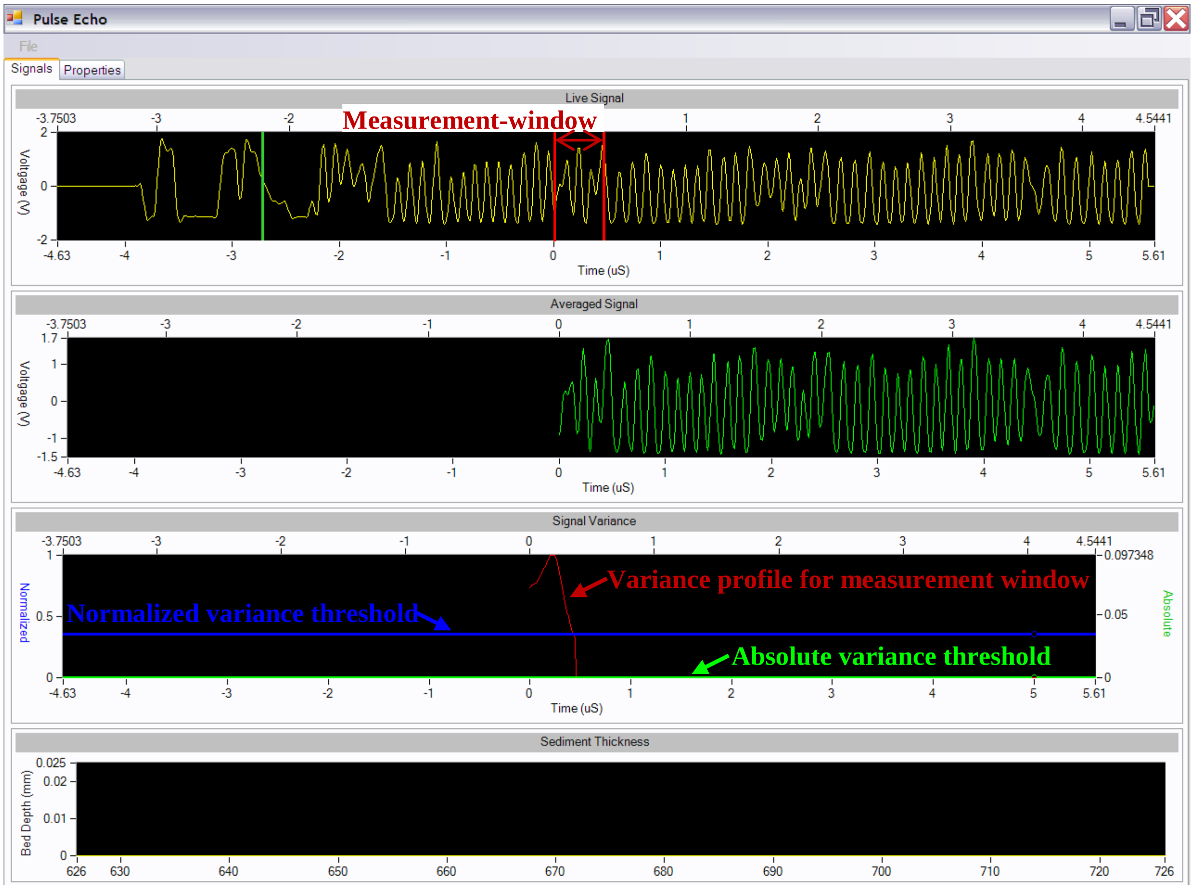

Figure 3.3. Example of PulseEcho Variance Analysis User Interface and Thresholds

Careful consideration should be give to the benefits and limitations of the PulseEcho thresholds when selecting thresholds for detecting settling/settled solids. There are benefits and risks of using each threshold individually. The system is most robust when both thresholds are used in conjunction.

The absolute variance threshold is used to determine if an ultrasonic signal is modulating above the baseline signal variance of the PulseEcho system, which is the intrinsic noise level of the system when there is no flow in the flow loop or when only water is flowing through the flow loop. This signal variance represents the total electronic noise of the system, which includes the transducer and data acquisition electronics. The use of the absolute threshold alone would require the measurement window, which is illustrated in the top panel of Figure 3.3, to be set to a small time value that represents a measurement range that is fractions of a millimeter beyond the pipe wall. A small measurement window is necessary in order to ensure that any modulation from mobilized solids above a settled layer of solids is not captured and cannot influence the signal variance. The use of the absolute threshold alone represents a detection-only, “red-light/green-light” approach to sediment detection. Quantification of the thickness of a settled solids layer would not be possible with the small measurement window required when the absolute threshold is used alone. However, this detection-only approach may be appropriate when the goal of testing is only to detect, not to measure. This approach may also be appropriate when high frequency ultrasonic transducers are used, where settled solids can result in completely attenuated signals.

The normalized variance threshold is used to determine the point between non-modulated and modulated portions of a backscattered signal within a defined measurement window. This threshold is useful only when the measurement window captures both conditions. The normalized threshold was developed to detect and measure/quantify the thickness of a settled solids layer. For the quantification of the thickness of a settled solids layer to be possible using the normalized threshold alone, the transducer has be able to detect scattering beyond a settled bed of solids, which Phase III testing showed the 5-MHz transducer to be capable of when the particles are greater than ~30 microns and are of sufficient concentration. To facilitate quantitative measurements of a settled solids layer, the measurement window also has to be set to a value that monitors an appropriate range beyond the pipe wall.

The primary risk of using the absolute threshold alone is the possibility of pipe wall scale. The secondary risk is the change in the baseline signal variance of the PulseEcho system over time. However, this can be addressed with periodic calibrations.

The primary risk of using the normalized threshold alone is the assumption that settled solids will not completely attenuate the signal and that backscatter from moving particles above a settled layer of solids can be detected. This will require that the solids moving above a settled bed be of a measurable size and concentration. This also requires that the measurement window be large enough to capture non-modulated and modulated signals that result from settled solids with overhead moving solids.

When both the absolute and normalized thresholds are used together, the PulseEcho system is prepared to handle the possible situations that may arise while monitoring for particle mobility and settling to determine critical velocity. A summary of different conditions that can arise and that the PulseEcho system would be prepared for when both thresholds are utilized are:

1. The measurement window contains only modulated signal. This represents conditions where all solids are mobilized inside the pipe at the transducer location. This condition could be detected with either of the thresholds alone.

2. The measurement window contains non-modulated and modulated signals resulting from moving particles above a settled layer of solids. The normalized threshold alone would detect the settled solids. The absolute threshold alone would not report settled solids since the moving particles would influence the total signal variance in the measurement window.

3. The measurement window contains only non-modulated signal. The absolute threshold alone would detect and report settled solids. No signal modulation correlates with several possible conditions:

    a. A very thick layer of solids is present in front of the transducer.

    b. A layer of solids is present that has totally attenuated the signal, even if the layer is thin.

    c. There is a layer of solids in front of the transducer and the moving solids above the settled layer are either too small in size to detect or are of insufficient concentration.

d. There are no particles or an insufficient concentration of particles in front of the transducer. This is possible for dilute slurries, especially at high flow velocities. As reported in Bontha et al. 2010a, the PulseEcho system cannot distinguish between non-modulated signals that result from lack of scattering and non-modulated signals that result from total attenuation due to a settled layer of solids. Logic and reasoning will have to be employed with the system. For example, if the PulseEcho system reports settled solids at 8 ft/s, the operator(s) may want to consider whether that is reasonable.

The use of the absolute threshold alone would allow the PulseEcho system to appropriate report most conditions; however, the use of both the absolute and normalized thresholds together would eliminate the primary risk of using the absolute threshold alone, which is in setting the appropriate measurement window size. Once the absolute variance threshold is exceeded, the normalized variance threshold is activated to measure sediment thickness.

3.4 Particle Concentration

Exploring methods to address the lower concentration limit of the PulseEcho system by evaluating alternative transducer sizes and geometries was not in the Phase IV scope of work. The lower particle concentration limit that is measurable by the PulseEcho system is reasonable for the intended application, measurement of bulk slurry critical velocity. If detection of small particle accumulation of sparsely distributed slurry components becomes a priority, then alternate detection technologies should be considered.

4.0 Test Facility

The PulseEcho system optimization was conducted with the MTEL configuration that was modified during the Phase III testing. Full details of this configuration, referred to as the short circuit loop, are presented in Bontha et al. (2010a and 2010b). This section briefly discusses the short circuit loop and the modifications that were made to evaluate this PulseEcho system. Other instruments (i.e., Coriolis mass flow meters, differential pressure gauges, thermocouples) are also present in the loop and are described in Bontha et al. (2010a and 2010b).

4.1 Short-Circuit Loop Configuration

Complete details and sketches of the Short-Circuit loop configuration are presented in Bontha et al. (2010a and 2010b), and only a brief summary is presented here. The loop configuration is shown in Figure 4.1. The Short-Circuit loop configuration maintains precise simulant particle inventory and reduces the duration of testing time required to achieve steady state at particular evaluation velocities. To promote preferential particle settling in the instrument test section instead of elsewhere in the loop, the inside diameter of the hose associated with the recirculation leg of the loop was reduced to 2.37 inches while the primary section consists of a 3-inch, Schedule 40 stainless steel (SS) piping. This results in a higher fluid velocity in the smaller diameter section of the loop. The Short-Circuit Loop volume is roughly 40 gallons.

The primary modification that was made to the existing short-circuit loop involved replacing the existing test sections (that contained the PulseEcho, UDV, and Ultrasonic Attenuation sensors) with a smaller section that contained only the PulseEcho transducers. This section contained several sets of PulseEcho transducers to investigate the capability of PulseEcho to detect small particles at the full wall thickness of a Schedule 40 SS pipe. The test section is described below.

4.2 Test Section

There were three reasons for replacing the existing test section with a new section: 1) to enable multiple frequency transducer evaluation during a single flow loop test to address the capability of PulseEcho to detect small particles, 2) to establish the effect of wall thickness on sensor sensitivity, and 3) to eliminate uncertainties regarding visual detection of settled bed by moving the transparent sections close to the test section.

The modified test section, shown in Figure 4.2, was designed to accommodate test three different transducers—the 10- and 15-MHz transducers to detect the small particles (20 to 50 µm) and the 5-MHz transducer to detect larger (>50 µm) particles. However due to manufacturing constraints and limited advantages over the 10-MHz transducer, the 15-MHz transducer was not procured.

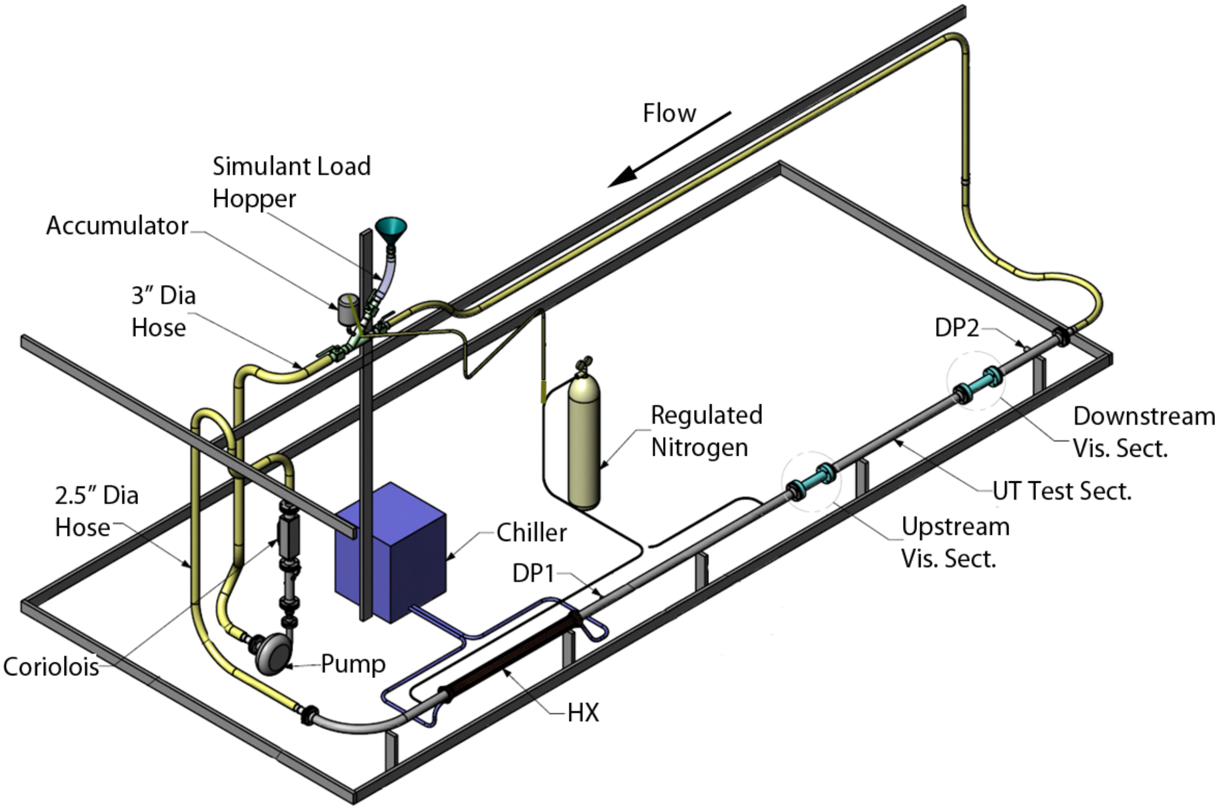

Figure 4.1. MTEL Test Facility

The test section was constructed from a 3-inch ID, 0.375-inch thickness SS tube (from the same SS piece that was used to fabricate the test section used in FY 2010 testing). For the 5- and 10-MHz frequency, two transducers were mounted on flats made along the bottom of the pipe. The wall thicknesses of the thinnest points of the flats were machined to be integral numbers of half-wavelengths through the steel. A wavelength in steel is defined as the speed of sound in steel divided by the measurement frequency. For each transducer frequency, one flat was machined such that the thickness was equal to or greater than that of a Schedule 40 SS pipe wall (i.e., ≥ 0.216-inches). The second flat was machined such that the thickness was equal to or greater than that of half a Schedule 40 SS pipe wall thickness (i.e., ≥ 0.108 inches). Each flat is 2-inches long, and the transducer is placed at the center of the flat; this eliminates any edge effects that may interfere with the path of the ultrasonic signal through the SS. It can be seen in Figure 4.2 that the 5-MHz transducer located at a thickness of 0.10 inches corresponds to that which was previously tested in FY2010 and, therefore, provides a reference point to compare the present test results to those performed in Phase III.

It can be seen from Figure 4.2 that the new test section is ~2 ft in length as compared to the original test section that was ~6 ft. This eliminates the extra 4 ft of section, brings the transparent sections closer together and minimizes differences in settling behavior that were observed upstream and downstream of the test section (Bontha et al. 2010a).

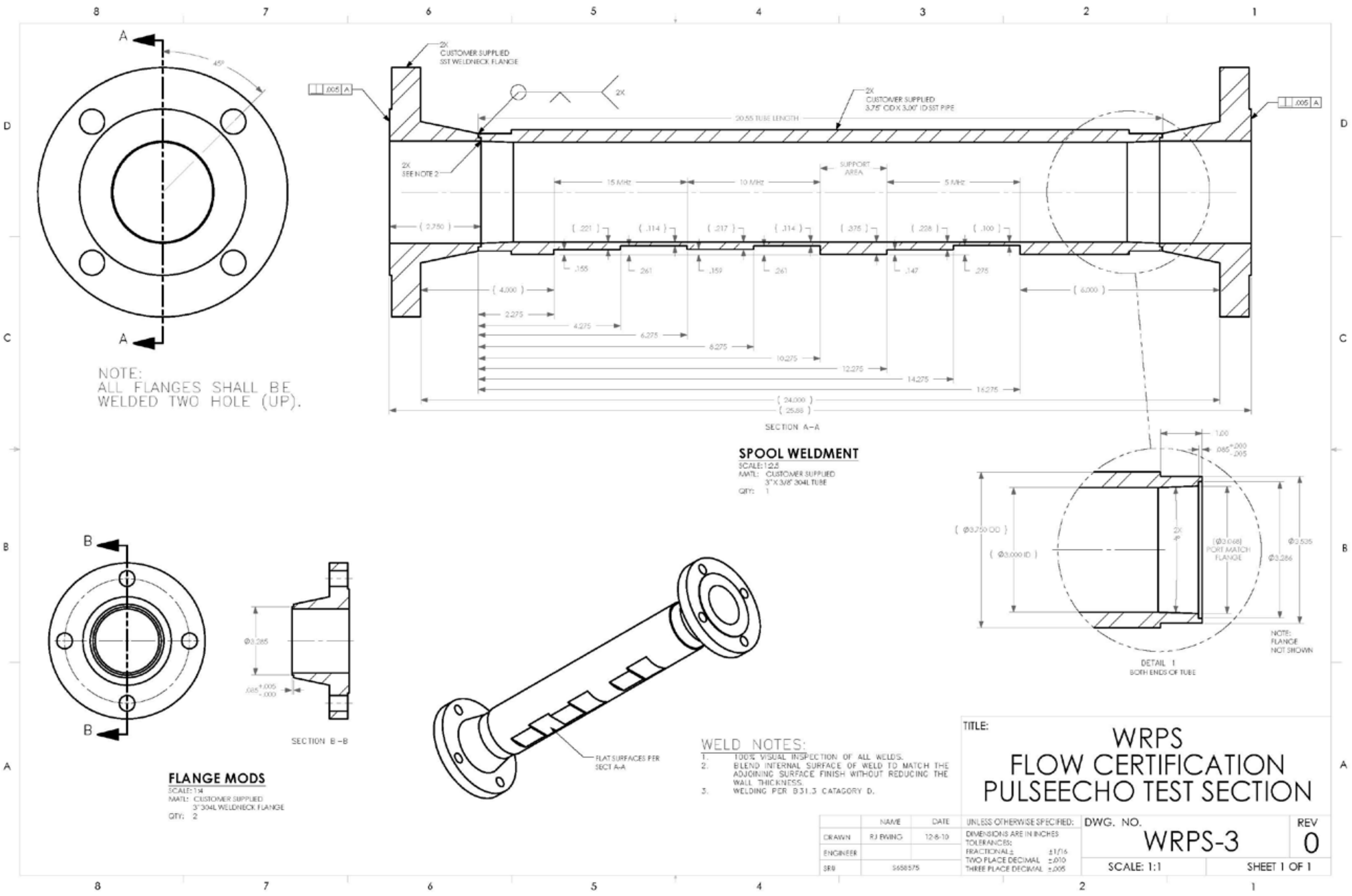

Figure 4.2. Section for Evaluating Different PulseEcho Transducers During Phase IV

4.2.1 Reference Instrumentation

The pipeline transport of solids suspended in a carrier liquid is considered “critical” when the flow velocity is just at the point where solids suspension becomes challenged. The behavior of the solids at this velocity depends on the specific properties of the solids and the carrier fluid and may exhibit conditions ranging from a solids concentration gradient, to “saltation,” to a “sliding bed,” or even a stationary layer of solids. During Phase III and this testing, critical velocity was reported at the velocity for which a stationary bed formed.

Phase III testing revealed that the best indication that flow is approaching the critical velocity can be made by using a high-resolution video camera mounted beneath one of the transparent pipe sections. The same approach was used during Phase IV testing. The camera is a Point Grey Research model Grasshopper–GRAS20S4M–monochrome (black/white). It has a 1624- × 1224-pixel sensor, with each pixel 4.4-μm × 4.4-μm square. The camera runs at 30 frames/second at full resolution (1600 × 1200 pixels). The camera lens is a Donder Zoom Module that provides a field of view (FOV) of 3200 μm to 12800 μm over the zoom range of the lens. As noted in Bontha et al. (2010a and 2010b), this system is capable of detecting particle behavior from particle sizes ranging from 5 to 500 μm in diameter.

4.2.2 PulseEcho Configuration

In this system, an ultrasonic transducer is attached to the outside surface beneath a horizontal section of pipe. Single-sided access to the measurement location is required. Ultrasonic pulses generated by the transducer penetrate the pipe wall and the fluid contents within the pipe. Ultrasonic backscatter signals from solid particles contained in the fluid are used to detect the presence of settled solids inside the pipe. Ultrasonic measurements are performed at microsecond time scales. Suspended, mobilized solids result in highly modulated ultrasonic backscatter signals with respect to time. Settled solids result in ultrasonic backscatter signals that are not modulated with respect to time. The ultrasonic signals received by the ultrasonic transducer are digitized by a digitizer card and analyzed real-time by a computer algorithm. A set of 10 signals is used by the algorithm to determine whether settled solids are present inside the pipe, therefore resulting in 10 read-outs per second. The depth of the settled solids layer is determined by measuring the point in time in the backscatter signal where the signal becomes modulated. This point represents an interface between a stationary and moving fluid. If the speed of sound through the slurry is known, which can be determined through routine laboratory measurements, the distance from the transducer to the moving interface can be calculated, thereby determining the thickness of the settled layer. The thickness of the settled bed is not required for detecting critical velocity.

Two PulseEcho transducer frequencies were evaluated during Phase IV testing: 5 MHz and 10 MHz. The 10-MHz frequency was selected for its capability to detect smaller particle sizes around 20-µm and greater. The 5-MHz frequency was selected to 1) validate Phase IV results against those obtained during Phase III testing with the same simulant and pipe wall thickness and 2) establish the sensitivity of the 5-MHz transducer with a pipe wall thickness that is equal to or greater than that of a Schedule 40 SS pipe.

Sound energy across transducer membranes (the pipe wall in this instance) that equal half-wavelength multiples in thickness allow maximum transmittance across the membrane. For example, a 10-MHz sound wave traversing SS has a calculated wavelength λ of approximately 600 μm based on the equation $\lambda = c / f$, where *c* is the speed of sound through the material under test, and *f* is the frequency of the sound energy. Therefore, the flat areas along the PulseEcho spool pipe were machined to 5 wavelengths and 9.5 wavelengths at the minimum points of the flats to achieve thicknesses that are close to half and full schedule, 40 pipe wall thickness. For a 10-MHz frequency in steel, 9.5 wavelengths correspond to 5.50 mm and 5 wavelengths correspond to 2.9 mm. The pipe wall thicknesses that were calculated for the 5-MHz and 10-MHz transducers are summarized in Table 4.1. Ultimately, the 15-MHz transducers were not ordered. Per the vendor, the actual frequencies generated by a contact transducer with a 15-MHz element would have been closer to 10 MHz because of the frequency filtering by the transducer face plate. Therefore, the 15-MHz transducer with frequency filtering would have provided no benefit over the 10-MHz transducer.

Table 4.1. Summary of Pipe Wall Thicknesses for Three Ultrasonic Frequencies

Proposed Pipe Wall Thickness (mm)	**5 MHz**			**10 MHz**		
	Calculated Number of wavelengths	**Next Whole λ or 1.5λ**	**Thickness to Machine (mm)**	**Calculated Number of Wavelengths**	**Next Whole λ or 1.5λ**	**Thickness to Machine (mm)**
5.49	4.74	5.0	5.79	9.48	9.5	5.50
2.74	2.37	2.5	2.90	4.73	5.0	2.90

The PulseEcho system electronics configuration for Phase IV testing was the same as that described in Bontha et al. (2010a) with the exception that the GaGe digitizer card was triggered by an Agilent 33250A waveform generator instead of the Olympus pulser/receiver unit. This was done to eliminate the GaGe digitizer jitter that was observed during Phase III testing. The waveform generator delivered a 50-Hz, 3.9-Volt square wave trigger to the GaGe digitizer. A photograph of the PulseEcho hardware is shown in Figure 4.3.

Figure 4.3. PulseEcho hardware configuration

5.0 Test Approach

The test approach consisted of bench scale and certification loop testing. Bench scale testing was performed to ensure that the transducers perform as anticipated and to permit definition of components and software configuration for evaluation in the certification loop. Certification loop testing was performed to assess the performance of the transducers to detect critical velocity and settled bed formation. This section presents the approaches used for the bench and certification loop testing as well as the simulants used during the testing.

5.1 Bench-Scale Evaluation

Piezo-composite ultrasonic transducers that were 5 MHz and 10 MHz and 0.25 inch in diameter were purchased from NDT Systems, Inc. (Huntington Beach, CA) for Phase IV testing. NDT Systems is the same transducer company that manufactured the 5-MHz transducer that was evaluated during Phase III testing.

Before starting Phase IV full-scale testing, the 5-MHz and 10-MHz ultrasonic transducers were evaluated with the SS particles in water. The transducers were initially evaluated in direct contact with a mixture of water and SS. Excellent backscatter was detected with all four transducers. The four transducers were subsequently mounted onto their respective flat sections as shown in Figure 5.1 on the PulseEcho spool piece using Hardman extra fast setting epoxy. The epoxy was allowed to cure overnight before performing bench scale testing.

Figure 5.1. PulseEcho Spool Piece with 5-MHz and 10-MHz Transducers Mounted onto Flat Sections

During bench scale testing, the spool piece was capped at one end, filled with water, and supported vertically at a slight angle with the transducers positioned on the underside of the spool piece as shown in Figure 5.2. SS particles were lightly dispersed in the water from the open end of the spool piece. The rapid settling rate of the SS particles resulted in the particles sliding along the side of the spool pipe containing the transducers. The backscatter detected by the transducers at all four locations was present and measurable. The results of bench scale testing warranted testing at full scale with the Multiphase Transport Evaluation Loop.

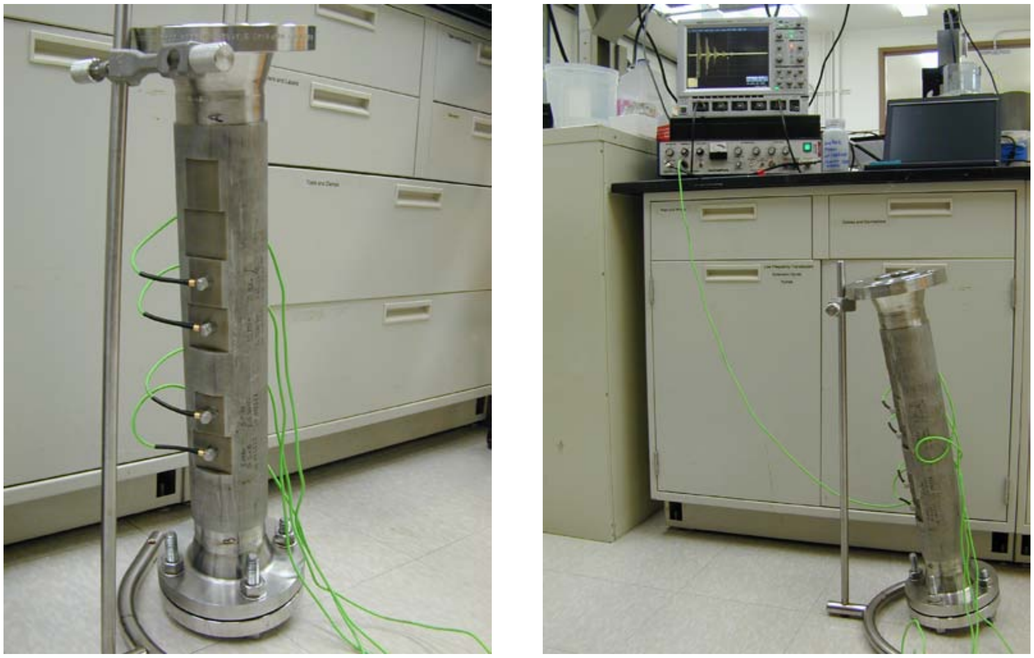

Figure 5.2. PulseEcho Spool Piece Configuration During Bench Scale Testing

The goal of testing for the PulseEcho system for this effort was to detect the onset of solids settling. The quantification of sediment thickness was not desired; therefore, sediment thickness values are not reported for PulseEcho. Values above zero thickness are referred to herein as “indications.” Although quantification is not desired, the PulseEcho algorithm is configured to receive a speed-of-sound value for the medium through which the ultrasound is propagating. A placeholder value of 1.62 millimeters per microsecond (mm/µs) was used for the duration of testing. Data were typically acquired for 1 to 2 minutes at a fixed measurement rate of 10 measurements per second.

Since the goal of testing for the PulseEcho system was to detect and not quantify, there will be no discussion of measurement accuracy. However, sensitivity and resolution can be discussed. Resolution is the smallest increment of the quantity that can be recognized. Here, that means the smallest possible change in the displayed reading. For a conventional PulseEcho mode flaw detector that requires coherent ultrasonic reflections in the form of signal echoes or peaks, resolution refers to the closest together that two echoes can occur and still be distinguished. This is determined by the wavelength in the material under test and the ultrasonic pulse duration. Because the PulseEcho system does not rely on coherent peaks to perform its measurements, but rather modulation due to scattering, only a fraction of the wavelength is required for analysis. The fraction required is estimated at one-half wavelength. The calculated wavelength in the slurry media at the 5-MHz PulseEcho measurement frequency is approximately 300 μm, meaning that the half-wavelength resolution is approximately 150 μm. The calculated wavelength in the slurry media at the 10-MHz PulseEcho measurement frequency is approximately 160 μm, meaning that the half-wavelength resolution is approximately 80 μm. The true speed of sound values would need to be measured to obtain a more accurate calculation of wavelength.

An important implication of the half-wavelength resolution is that the thickness of a settled solids layer must equal this half-wavelength value before it can be detected or quantified. For example, one layer of 14 micron particles would likely not be detected by a 10-MHz transducer; a small multiple of particle layers would need to be present, the exact number of which would be impacted by particle packing. This half-wavelength detection threshold is minimized when high frequency transducers, which have small wavelengths, are utilized.

5.2 Loop Evaluation

5.2.1 Shakedown Testing of Flow Loop

The purpose of shakedown testing was twofold: 1) verify that all systems operate as intended and 2) establish minimum concentration of the SS particles (see Table 5.1) that will be sufficient to produce a measureable response with the selected transducers.

Although the 10-MHz transducer at the flat section representing a half Schedule 40 pipe wall thickness operated well during bench scale testing, it failed to operate during shakedown testing. The transducer was later removed and inspected. A poor electrical ground was suspected as the cause.

The minimum concentration of SS particles in water was evaluated for the 5-MHz transducers at both the half and full schedule 40 pipe wall thickness and for the 10-MHz transducer at the full schedule 40 pipe wall thickness. This was accomplished by starting with ~2-wt% SS in water in the loop with the intention of gradually increasing the concentration until a measurable signal modulation was observed with the high-frequency transducers. However, 2-wt% SS in water was sufficient to obtain a measureable modulation for all three transducers.

5.2.2 Instrument Evaluation in Flow Loop

The purposes of these tests were to evaluate the capability of PulseEcho to detect the critical velocity of 20 to 50-μm sized, high-density (8 to 11 g/cm^3) particles and to establish the capability to use the full schedule 40 pipe wall thickness to detect settling or particles >20 μm. The tests that were performed to accomplish this are listed in Table 5.1.

5.2.3 Test Procedure

The test procedure was the same as that previously used during the Phase III testing and presented in Section 5.2 of Bontha et al. (2010a) with the following exceptions:

1. No Lasentec data were collected for analysis.

2. Data with only the PulseEcho system were collected. In this regard because there was only one data processor and multiple transducers, data collection at each flow velocity involved collecting separately from each transducer before moving to the next flow velocity (at each velocity, three measurements were made corresponding to two 5-MHz transducers at different pipe wall thicknesses and one 10-MHz transducer).
3. Observations regarding the critical velocity were made by visual observations for settling using the high-resolution video camera. Although differential pressure data were recorded throughout the tests, these data were only intended to be used to validate visual measurements if deemed necessary.

5.3 Matrix of Tests Performed

The various tests that were performed to establish the objectives discussed above are presented in Table 5.1.

Table 5.1. Test Matrix for PulseEcho System Evaluation in Phase IV

Test	Simulant[(a)]	Comments
1	2-wt % SS particles (with a d_{50} of ~14 µm) in water[(b)]	Established the capability of the high-frequency transducers to detect settling of high-density particles
2	Broad PSD (Test #4 of Phase III testing) at 5 wt% in water	First data point to establish the capability to use the previously tested 5-MHz transducer at/near the full wall thickness of a Schedule 40 SS pipe
3.	Broad PSD (Test #5 of Phase III testing) at 20 wt% in water	Second data point to establish the cap ability to use the previously tested 5-MHz transducer at/near the full wall thickness of a Schedule 40 SS pipe
4	1-wt% SS particles (with a d_{50} of ~14 µm) in 20-wt% Kaolin slurry	Established the influence of non-settling particles (i.e., attenuation of the PulseEcho signal) on the detection of small, high-density particle settling
5	2-wt% SS particles (with a d_{50} of ~14 µm) in 20-wt% Kaolin slurry	Same as test 4, with higher wt% SS to indentify threshold for transducers to achieve adequate modulation at higher flow velocities
6	4-wt% SS particles (with a d_{50} of ~14 µm) in 20-wt% Kaolin slurry	Same as test 5, with higher wt% SS
7	20-wt% Kaolin slurry	Baseline carrier fluid test required to understand transducer modulation in the absence of other simulant
8	15-wt% Gibbsite slurry	Baseline carrier fluid test required to understand transducer modulation in the absence of other simulant
9	1-wt% SS particles (with a d_{50} of ~14 µm) in 15-wt% Gibbsite slurry	Established the influence of non-settling particles (i.e., attenuation of the PulseEcho signal) that is more representative of actual waste on the detection of small, high-density particle settling
10	1-wt% SS particles (with a d_{50} of ~14 µm) in 15-wt% Fe_2O_3 slurry	Similar to test 9, but with iron oxide as the carrier fluid
11	2-wt% SS particles (with a d_{50} of ~14 µm) in 15-wt% Fe_2O_3 slurry	Same as test 10, but with increased wt% SS to indentify threshold for transducers to achieve adequate modulation at higher flow velocities
12	4-wt% SS particles (with a d_{50} of ~14 µm) in 15-wt% Fe_2O_3 slurry	Same as test 11, but with increased wt % SS

(a) See Section 5.4 for more details on the choice of simulants.
(b) Bench scale and/or loop shakedown tests were used to identify the initial concentration of the SS particles that are likely to yield good signal response from the PulseEcho transducers.

5.4 Simulant Test Matrix

Two different kinds of simulants were used to evaluate the performance of the PulseEcho tranducers during the Phase IV testing:

Broad PSD Particles: These particles were specifically chosen to evaluate the capability of the 5-MHz transducer to detect critical velocity at or near the full wall thickness of a Schedule 40 SS pipe. This simulant was previously used during Phase III with a goal here to establish repeatability of sensor performance as well as establish the effect of wall thickness on sensor sensitivity. In addition, since these particles have a PSD range from 7 to 500 µm, this simulant was used to simultaneously evaluate the capability of a higher frequency transducer to detect critical velocity. The formulation of the Broad PSD simulant used for two tests are listed in Test #2 and #3 of Table 5.4.

Stainless Steel Particles: These particles (AMETEK Product P316L-10M) were mainly chosen to establish the ability of the high-frequency PulseEcho transducers to detect the critical velocity of small, fast-settling, high-density particles. This simulant is the same as that used by Poloski et al. (2009a) during the M1 issue resolution. SS has a density of ~8 g/cm^3 and a broad distribution with a significant portion of the particles falling in the range of 10 to 30 microns. Gibbsite and iron oxide were chosen as carrier fluid particles because these components are representative of materials present in tank waste. The test matrix employed during testing is given in Table 5.2; it should be noted that tests 8 to 12 were added after the initially proposed tests were completed. Before starting these additional tests, the simulant test matrix was presented to the WRPS project management team for approval. The specifications of the various materials used during testing along with the supplier/manufacturer details are shown in Table 5.3.

Table 5.2. Test Matrix Employed During Phase IV Testing

Test Number	Simulant Type[a]	Target Test Conditions Acronym	Solids Concentration[b]	Carrier Fluid Viscosity[c]	Carrier Fluid Yield Stress[d]
1	Mono-dispersed	LLL	L	L	L
2	Broad PSD	LLL	L	L	L
3	Broad PSD	HLL	H	L	L
4	Mono-dispersed	LMM	L	M	M
5	Mono-dispersed	LMM	L	M	M
6	Mono-dispersed	LMM	L	M	M
7	Carrier Fluid-Kaolin	N/A	N/A	N/A	N/A
8	Carrier Fluid-Gibbsite	N/A	N/A	N/A	N/A
9	Mono-dispersed	LLL	L	L	L
10	Mono-dispersed	LLM	L	L	L
11	Mono-dispersed	LLM	L	L	M
12	Mono-dispersed	LLM	L	L	M

N/A= not applicable.

(a) See Table 5.3 and Table 5.4 for the various materials used to make up the different simulants and their formulation.

(b) For solids concentration, the Low (L), mid (M), and high (H) concentration ranges are ≤ 5 wt% (L), 10 wt% (M), and 20 wt% (H), respectively (this does not include the mass of kaolin or iron oxide used to prepare the non-Newtonian slurries or the gibbsite).

(c) For viscosity, the Low and High ranges are 1 and 10 mPa·s, respectively. Also, for the non-Newtonian slurries, the viscosity of the carrier fluid was not controlled but was driven by the kaolin/iron oxide concentration necessary to achieve the target carrier fluid solids concentrations resulting in a mid (M) viscosity ~4 mPa·s.

(d) For Yield strength, the Low (L), Mid (M), and High (H) designations are consistent with those reported by Bontha et al. (2010a), 0, 3, and 6 Pa, respectively. The yield strength was not controlled and was driven by the carrier fluid solids concentrations.

Table 5.3. Specifications of the Various Particles Used in the Simulant Formulation

Simulant name	**Supplier/Manufacturer**	**Product ID**	**Density (g/mL)**	**Particle Size d(50), µm**
		Glass		
SPHERIGLASS® 5000	Potters Industries	A Glass, 5000	2.50	7.1
SPHERIGLASS® 3000	Potters Industries	A Glass, 3000	2.50	34.0
BALLOTINI Mil #13	Potters Industries	MIL-PRF-9954D#13	2.50	57.7
BALLOTINI Mil #10	Potters Industries	MIL-PRF-9954D#10	2.50	114.9
BALLOTINI Mil#8	Potters Industries	MIL-PRF-9954D#11	2.50	177.4
BALLOTINI Mil #6	Potters Industries	MIL-PRF-9954D#6	2.50	190.5
BALLOTINI Mil #4 sieved <500 µm	Potters Industries	MIL-PRF-9954D#4	2.50	502.8
		Other		
Stainless Steel	Ametek	P316L	7.95	13.9
Fe_2O_3	Prince Minerals	3752 Red Iron Oxide	5.20	2.0
Gibbsite	Almatis	C-333	2.50	7.9
EPK Kaolin	Feldspar Corporation	EPK Kaolin	2.65	6.3[(a)]

(a) The density of the particles presented are the nominal values and the d(50)particle size is based on the volume fraction

(b) PSD measured on a well-hydrated kaolin slurry, post-test sample from Test 7.

Table 5.4. Broad PSD Simulant Formulation Used For Tests Two and Three

Simulant Name	Composition	Component (wt%)	Density (g/mL)	Particle Size (volume) d(50), µm
Broad PSD	SPHERIGLASS® 5000	7	2.50	93.8
	SPHERIGLASS® 3000	14		
	BALLOTINI Mil #13	29		
	BALLOTINI Mil #10	29		
	BALLOTINI Mil #6	14		
	BALLOTINI Mil #4 (<500 µm)	7		

The test matrix shown in Table 5.2 using the SS particles was designed to evaluate the detection limitations of the two transducers being evaluated. Two Non-Newtonian carrier fluids, kaolin and iron oxide, were selected to evaluate the capability of PulseEcho to detect the SS particles in a high background concentration of non-settling particles. The kaolin was used for consistency with previous Phase III testing while the iron oxide was chosen as a high-density, fine particle that is known to be present in tank waste. The yield stress and carrier fluid viscosity were not controlled during testing; the carrier fluid particles were used simply as background particles. The rheological properties were measured and are given in Table 5.5 and Table 5.6. Gibbsite was also used as a carried fluid particle because it is also a known component found in tank waste. The gibbsite used had a comparable PSD to the kaolin used, but unlike kaolin, gibbsite slurry does not have appreciable rheological properties at the concentrations used and was considered a Newtonian fluid.

5.5 Simulant Characterization

The physical properties (including volume and mass fraction of the solids, PSD, bulk density) of the various simulants used in the testing are presented in Table 5.5 and Table 5.6. For simplicity, the data in these tables are classified into Newtonian and non-Newtonian slurries. To facilitate data comparison, the data in these tables are presented in a format similar to that used previously (Poloski et al. 2009a and 2009b, Bontha et al. 2010a) to facilitate easy comparison of the data. Presented below is a description of the methods and approaches used to generate the data in these tables.

5.5.1 Particle-Size Measurement

Particle-size characterization was done with a Mastersizer 2000 (Malvern Instruments, Inc., Southborough, MA 01772 USA) with a Hydro G wet dispersion accessory (equipped with a continuously variable and independent pump, stirrer, and ultrasound). The Mastersizer has a nominal size measurement range of 0.02 to 2000 μm. The actual range is dependent on the accessory used as well as the properties of the solids being analyzed. When coupled with the Hydro G wet dispersion accessory the nominal measuring range is not typically reduced, allowing the 0.02 to 2000 μm range to be measured (depending on material density). A NIST-traceable particle size standard was measured before measuring the distribution of these simulants.

Small aliquots of the simulant samples (< 1 mL for slurries, ~ 0.2 to 1 g for dry simulants) were diluted in degassed, deionized water in the Hydro G dispersion unit with the pump and stirrer speed set at 2500 and 1000 rpm, respectively, for 60 seconds before making the particle-size measurements. The total volume of the dispersion unit is ~800 mL. Appropriate dilutions were determined by the amount of light passing through the diluted material (obscuration), which was measured by the particle-size analyzer. Samples were analyzed on the same aliquot initially without sonication and then during sonication (100%, 20 W) after an initial sonication period of 60 seconds. Both pre-test and post-test samples were analyzed for particle size. Post-test sample data with sonication are reported in Table 5.5 and Table 5.6.

All of the kaolin slurry samples were shaken before taking aliquots for PSD measurements. For the Broad PSD simulant mix, the dry components were weighed out and mixed with the same simulant ratio used in the test matrix; the sample was then riffle split to obtain a well-representative sub-sample. Duplicate samples were measured to confirm the mixing and sub-sampling technique.

5.5.2 Simulant Rheology Measurement

For test slurries using kaolin/water, iron oxide/water, and gibbsite/water carrier fluids, slurry rheology was characterized by measuring the slurry's flow curve (i.e., stress versus shear rate response). The flow curve was obtained on a Haake RS600 rheometer configured with a concentric-cylinder geometry with a recessed end. The shear rate was ramped from 0 to 1000 s^{-1} over a 5-minute period (Smith and Prindiville 2002). The shear rate was held at 1000s^{-1} for 1 minute and then ramped down from 1000 to 0 s^{-1} over another 5-minute period. This process was repeated twice on the same aliquot, and the Bingham plastic and Casson curve fits were obtained for the down-ramp portions of the curve over a typical range of 250 to 800 s^{-1} for the second measurement. The upper limit was established because of Taylor vortex formation at higher rotational rates for the lower rheology samples used. Note that the distance between the bottom of the rheometer cup and the bob was set to 1 cm to allow for coarse particles to settle without the bob increasing drag from being partially submerged in a sediment bed. A distance of 1 cm was sufficient to make sure that interference from sedimentation was unlikely for all test conditions used, and it permitted all measurements to be performed with the same experimental parameters. Post-analysis of the rheograms showed no signs of sediment bed interference. Due to the potential interference of sedimentation, only the viscosity/yield stress of the carrier fluid is given in Table 5.5 and Table 5.6. It should be noted that not all the simulant particles that are considered coarse particles for this study settled during rheological measurements and as such have become part of the carrier fluid.

The flow curves of the kaolin slurries were measured both pre- and post-test; the post-test yield stress obtained is reported in Table 5.5 and Table 5.6. Previously, during Phase III, the average of the pre- and post-test yield stress values were averaged, but this was not necessary during Phase IV testing. Given that only one type of simulant was used with the kaolin (iron oxide and Gibbsite) during Phase IV, and the simulant concentration ranged from 1 to 4 wt% (significantly less than in Phase III), the kaolin rheology was not as variable (see Table 5.5 and Table 5.6).

5.5.3 Slurry Mass Balance

For each of the 12 tests, a mass balance of simulant components (i.e., coarse particles) and carrier fluid components added (i.e., water, water/kaolin, water/iron oxide, or water/gibbsite mixtures) to the loop was used to calculate the volume and mass fraction of each component used for testing. The mass balance was facilitated by assuming that the components occupied the entire test loop volume, which was measured to be ~40 gallons. A computational mass balance of the carrier fluids and simulant mixes used during testing is given in Table 5.5 and Table 5.6 and is based on actual masses added to the test loop.

Table 5.5. Properties of Newtonian Slurries

Test Number	1	2	3	8	9
Acronym	LLL	LLL	HLL	N/A	LLL
Volume Fraction (vol%)					
Simulant Particles (Total)	0.3%	2.1%	9.1%	0.0%	0.14%
Observable, 5 MHz (a)	0.02%	0.13%	0.57%	0.0%	0.01%
Observable, 10 MHz (b)	0.13%	1.9%	8.5%	0.0%	0.07%
Carrier Fluid Particle (Gibbsite)	0.0%	0.0%	0.0%	6.7%	6.7%
Water	99.7%	97.9%	90.9%	100.0%	99.9%
Mass Fraction (mass%)					
Simulant Particles (Total)	2.0%	5.0%	20.0%	0.0%	1.0%
Observable, 5 MHz (a)	0.12%	0.31%	1.24%	0.0%	0.06%
Observable, 10 MHz (b)	1.0%	4.6%	18.5%	0.0%	0.5%
Carrier Fluid Particle (Gibbsite)	0.0%	0.0%	0.0%	15.2%	15.1%
Water	98.0%	95.0%	80.0%	100.0%	99.0%
Component Density (g/mL)					
Simulant Particles	7.95	2.5	2.5	n/a	7.95
Carrier Fluid Particle (Gibbsite)	n/a	n/a	n/a	2.5	2.5
Water (Gibbsite/water)	1	1	1	1	1
Bulk	1.018	1.031	1.137	1.100	1.110
Particle Size Distribution (µm)					
d_{5}	6.1	7.4	7.4	0.7	0.7
d_{10}	7.2	26.1	26.1	1.2	1.2
d_{20}	9.0	45.9	45.9	2.3	2.3
d_{30}	10.6	60.8	60.8	3.8	3.8
d_{40}	12.2	76.2	76.2	5.7	5.8
d_{50}	13.9	93.8	93.8	7.9	8.1
d_{60}	15.9	115.1	115.1	10.5	10.8
d_{70}	18.3	143.6	143.6	13.6	14.0
d_{80}	21.5	191.2	191.2	17.8	18.3
d_{90}	26.7	349.7	349.7	24.4	25.2
d_{95}	31.4	538.6	538.6	30.2	31.4
d_{99}	40.7	807.6	807.6	40.2	42.5
Carrier Fluid Rheology: Flow Curve (0-600 s^{-1}) down					
Newtonian Viscosity(c) , mPa.s	1	1	1	1	1
r^2	N/A	N/A	N/A	N/A	N/A

(a) Percentage of Simulant Particles >30 µm observable using the 5-MHz transducer
(b) Percentage of Simulant Particles >14 µm observable using the 10-MHz transducer
(c) Newtonian viscosity of water is 1.

Table 5.6. Properties of Non-Newtonian Slurries

Test Number	4	5	6	7	10	11	12
Acronym	LMM	HMM	MLL	N/A	LLM	LLM	LLM
Volume Fraction (vol%)							
Simulant Particles (Total)	0.14%	0.29%	0.58%	0.00%	0.14%	0.29%	0.59%
Observable, 5 MHz (a)	0.01%	0.02%	0.04%	0.0%	0.01%	0.02%	0.04%
Observable, 10 MHz (b)	0.07%	0.14%	0.29%	0.0%	0.07%	0.14%	0.29%
Carrier Fluid Particle (Kaolin/Fe_2O_3)	8.6%	8.7%	8.6%	8.6%	3.3%	3.3%	3.3%
Water	91.2%	91.0%	90.8%	91.4%	96.6%	96.4%	96.1%
Mass Fraction (mass%)							
Simulant Particles (Total)	1.0%	2.0%	3.9%	0.0%	1.0%	2.0%	4.0%
Observable, 5 MHz (a)	0.06%	0.12%	0.24%	0.0%	0.06%	0.12%	0.25%
Observable, 10 MHz (b)	0.5%	1.0%	1.9%	0.0%	0.5%	1.0%	2.0%
Carrier Fluid Particle (Kaolin/Fe_2O_3)	19.8%	19.7%	19.3%	20.0%	15.0%	14.9%	14.6%
Water	80.2%	78.4%	76.8%	80.0%	84.0%	83.1%	81.4%
Component Density (g/mL)							
Simulant Particles	7.95	7.95	7.95	0	7.95	7.95	7.95
Carrier Fluid Particle (Kaolin/Fe_2O_3)	2.65	2.65	2.65	2.65	5.2	5.2	5.2
Water	1	1	1	1	1	1	1
Bulk	1.152	1.162	1.183	1.142	1.149	1.159	1.180
Particle Size Distribution (µm)							
d_5	1.2	1.2	1.3	1.3	0.5	0.5	0.5
d_{10}	1.7	1.7	1.8	1.8	0.6	0.6	0.6
d_{20}	2.7	2.7	2.9	2.7	0.9	0.9	0.9
d_{30}	3.7	3.8	4.0	3.7	1.1	1.2	1.2
d_{40}	4.9	5.0	5.3	4.8	1.5	1.5	1.6
d_{50}	6.4	6.6	7.1	6.3	1.9	1.9	2.0
d_{60}	8.5	8.7	9.3	8.2	2.4	2.4	2.6
d_{70}	11.6	11.6	12.3	11.0	3.2	3.1	3.4
d_{80}	16.4	16.1	16.8	15.4	4.3	4.2	4.8
d_{90}	25.8	24.3	24.9	23.7	6.4	6.3	7.6
d_{95}	36.3	32.6	33.3	32.1	8.5	8.3	10.8
d_{99}	61.2	48.9	50.7	49.5	12.6	12.1	17.5
Bingham Flow Curve (250-800 s-1) down							
Bingham Yield Stress, Pa	2.8	2.3	1.9	3.0	0.5	0.5	0.5
Bingham Consistency, mPa·s	4.1	3.9	3.7	4.0	2.9	2.9	2.7
r^2	0.999	0.9994	0.9992	0.9990	0.9875	0.9875	0.9953
Casson Flow Curve (250-800 s-1) down							
Casson Yield Stress, Pa	1.6	1.2	1.0	1.9	0.3	0.3	0.2
Infinite Shear Viscosity, mPa·s	1.7	1.8	1.8	1.6	1.4	1.4	1.4
r^2	1.000	0.9994	0.9993	0.9997	0.9928	0.9928	0.9996

(a) Percentage of Simulant Particles >30 µm observable using the 5-MHz transducer
(b) Percentage of Simulant Particles >14 µm observable using the 10-MHz transducer

6.0 PulseEcho Results and Discussion

6.1 Summary of Results

The summary of detected critical velocities for the 10-MHz and 5-MHz transducers is provided in Table 6.1. Shown in the table are the visually observed regimes, the visually observed critical velocity, and the PulseEcho measured critical velocity for each test. Note that the three regimes were discussed in Bontha, et. al. (2010a) and represent fluid behavior that occurs just before a stationary bed forms (i.e., critical velocity). The tests are further discussed in Sections 6.2 and 6.3.

6.2 5-MHz Transducers Discussion

The primary objective of testing the 5-MHz transducer during Phase IV was to determine if the same critical velocities could be detected with the 5-MHz transducer at full Schedule 40 pipe wall thickness and also to validate the PulseEcho system against Phase III results obtained for the same simulant. The secondary objective was to evaluate how this transducer would perform in slurries where the PSD was at or below the theoretical measuring threshold of 30 µm. These results are presented in the next two sections.

6.2.1 Broad PSD Discussion

Test 2 and Test 3 with the broad PSD simulants were performed to validate the performance of the 5-MHz transducers at both the half-wall and full-wall locations against the results of Phase III testing with the broad PSD simulant (Phase III Tests 4 and 5). Figure 6.1 and Figure 6.2 show the response of the 5-MHz transducers, half and full wall thickness, respectively, for Test 2. It can be seen from these figures that the velocity at which the 10% criterion was first met for both transducers was 3.3 ft/s, corresponding to the experimentally determined critical velocity observed during the present and Phase III tests as indicated in Table 6.1.

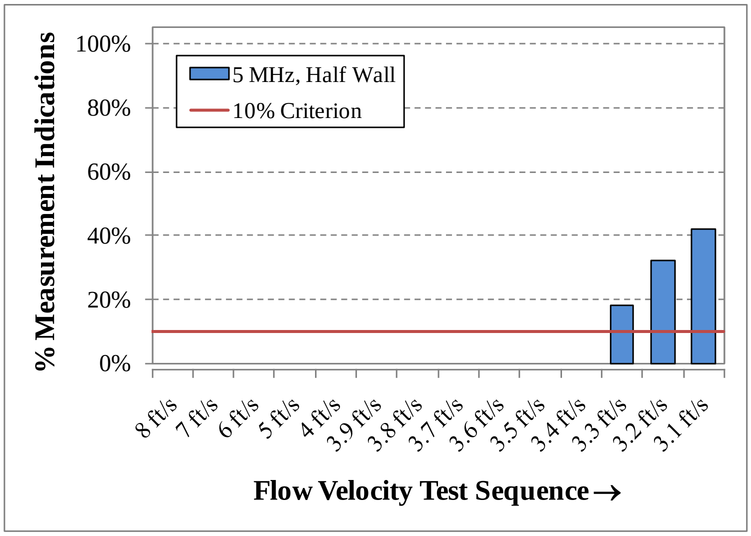

Figure 6.1. Test 2, 5-MHz Half Wall (5-wt% Broad PSD in water)

Table 6.1. Summary of Critical Velocity Detection

Test	Simulant Type	Regime I	Regime II	Regime III	$V_{critical}$ (c)(e)	$V_{critical}$ (c) (f) 10 MHz Full Wall	$V_{critical}$ (c) (f) 5 MHz Full Wall	$V_{critical}$ (c)(e)(f) 5 MHz Half Wall
1	2-wt % SS in Water	4	3	NO	2.4	2.2	2.5	Not acquired
2	5-wt% Broad PSD in water	3.8	3.5	3.5	3.2(3.3)	3.3	3.3	3.3(3.3)
3	20-wt% Broad PSD in water	4.5	4.3	4.2	4(4.0)	3.9	4.0	4.0(4.1)
4	1-wt % SS in Kaolin slurry	5	4	3.7	3.6	IS	IS	IS
5	2-wt % SS in Kaolin slurry	5	4	3.7	3.6	IS	IS	IS
6	4-wt % SS in Kaolin slurry	5	4.5	4.1	3.7	3.6	IS	IS
7	20 wt % Kaolin slurry	N/A	N/A	N/A	N/A	N/A	N/A	N/A
8	15 wt % Gibbsite slurry	N/A	N/A	N/A	N/A	N/A	N/A	N/A
9	1-wt % SS in Gibbsite slurry	2.5	NO	NO	2.4	2.1	IS	IS
10(a)	1-wt % SS in Fe_2O_3 slurry	NO	NO	NO	2.6-2.7(b)	IS: 2.6	IS	IS
11(a)	2-wt % SS in Fe_2O_3 slurry	NO	NO	NO	2.7	IS: 2.7	IS	IS
12(a)	4-wt % SS in Fe_2O_3 slurry	NO	NO	NO	2.9	2.9	IS	IS

(a) No distinct flow patterns were observed because of obscuration from the carrier fluid.
(b) Range reported due to uncertainty of settling in downstream Visualization Section (VS)
(c) Defined as a visually observed stationary bed
(d) IS: Insufficient Scatter at high flow velocities. NO: None Observed.
(e) Velocity in () indicates Phase III data.
(f) The normalized threshold was used for all tests for the 5-MHz transducers. The absolute threshold was used for the 10-MHz transducer for tests 3-12.

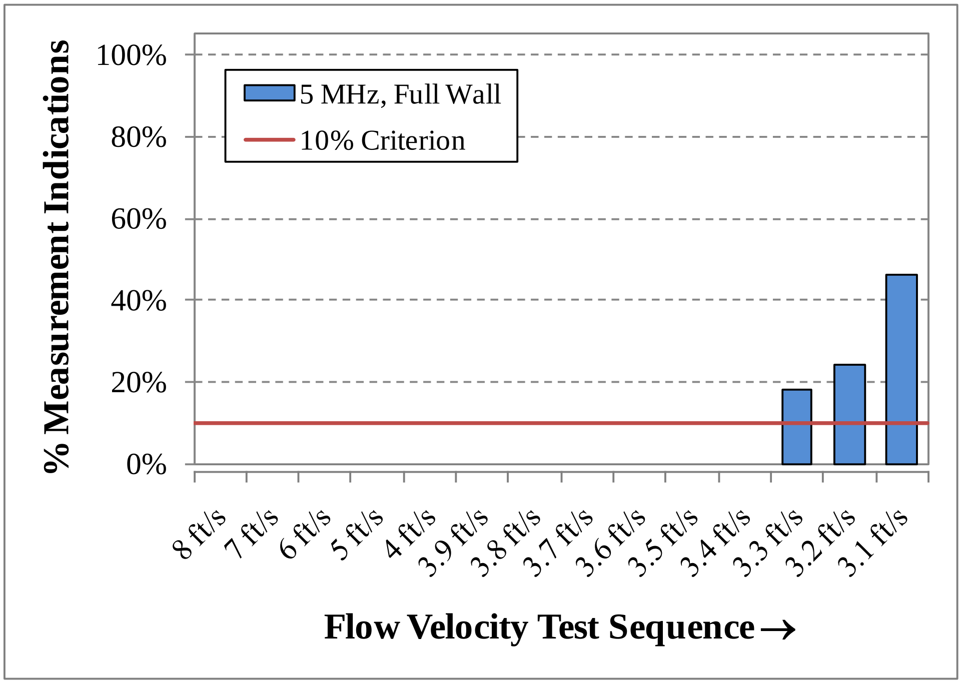

Figure 6.2. Test 2, 5-MHz Full Wall (5-wt% Broad PSD in water)

Similarly, Figure 6.3 and Figure 6.4 show the response of the 5-MHz transducer at half and full wall thickness for Test 3. Once again, it can be seen from these figures that the critical velocity determined is in excellent agreement with the experimentally determined critical velocity observed during Phase III tests as indicated in Table 6.1.

The results indicate that both the 5-MHz and experimentally observed results are repeatable. In addition, the data also indicate that wall thickness is not a factor. Thus, full Schedule 40 pipe wall thickness can be used with the PulseEcho.

An important observation during Test 3 was there were long periods of consistent indications followed by periods of no indications. This is consistent with the observation of migrating sediment dunes. Periodic measurements of shorter time duration would ensure positive indication of a settled bed in such cases.

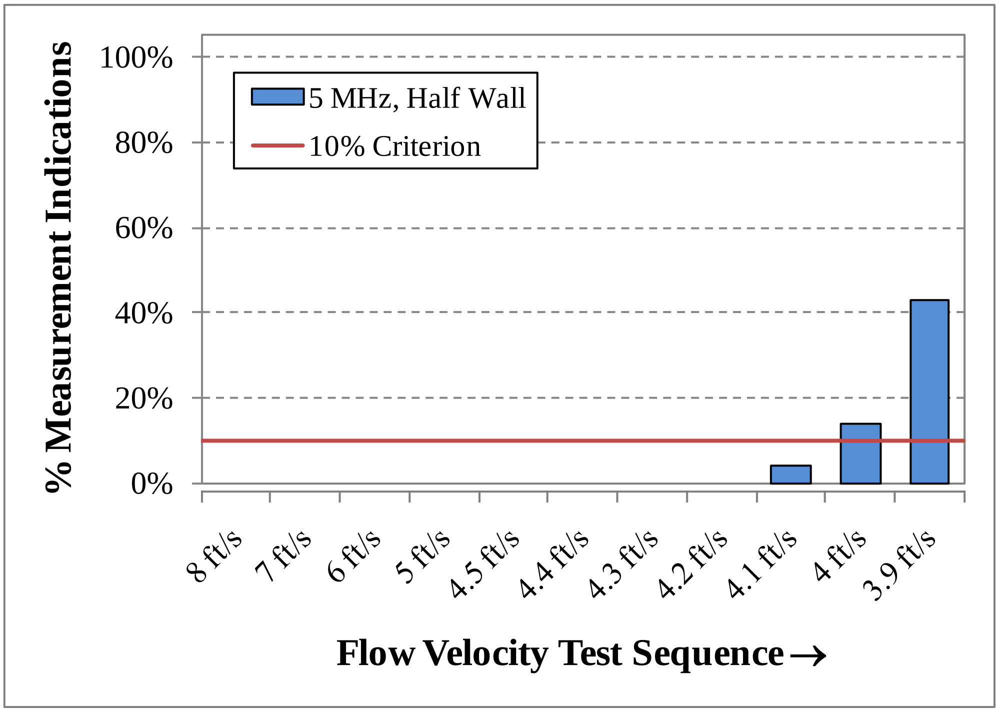

Figure 6.3. Test 3, 5-MHz Half Wall (20-wt% Broad PSD in water)

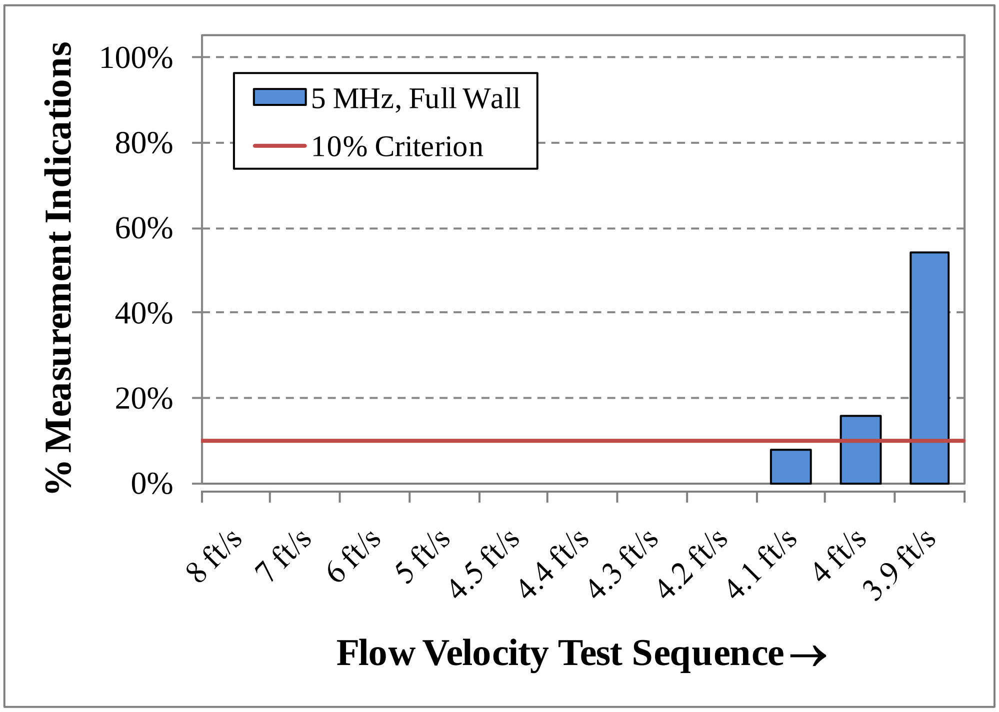

Figure 6.4. Test 3, 5-MHz Full Wall (20-wt% Broad PSD in water)

6.2.2 High Density, Small Particle Size Discussion

Based on the *ka* calculations discussed in Section 3.1, the 5-MHz transducers were not expected to perform well with the SS simulant. Due to the small particle size-to-wavelength ratio, insufficient scattering was anticipated with the 5-MHz transducers during the loop testing. Therefore, the objective of testing the 5-MHz transducer with the 6 to 31 μm (d_5 to d_{95}) SS particles was to evaluate the PulseEcho sensor performance for particles at or below the minimum detectable particle size. A sample set of these results is presented for the full-wall 5-MHz transducer in Figure 6.5 and Figure 6.6 for Test 1 (2 wt% SS in water) and Test 5 (2 wt% SS in kaolin), respectively.[1] The results in Figure 6.5 indicate that the critical velocity detected by the PulseEcho sensor is within 0.2 ft/s of the experimentally observed critical velocity from Test 1. However, the data in Figure 6.6 for Test 5 shows that the sensor gave false indications of settling from the beginning of the test at 8 ft/s. Similarly, false indications of critical velocity were also observed at high velocities for the remainder of SS particle tests listed in Table 6.1.

The reason that the 5-MHz transducer was able to detect a small fraction of the SS particles in water but resulted in false indications for all other carrier fluids can be explained by two factors: 1) there was a sufficient number of SS particles greater than 30 μm to provide backscatter, and 2) water has negligible attenuation to prevent backscatter compared to the other carrier fluids. The effect of the carrier fluid is discussed in more detail in Section 6.5.

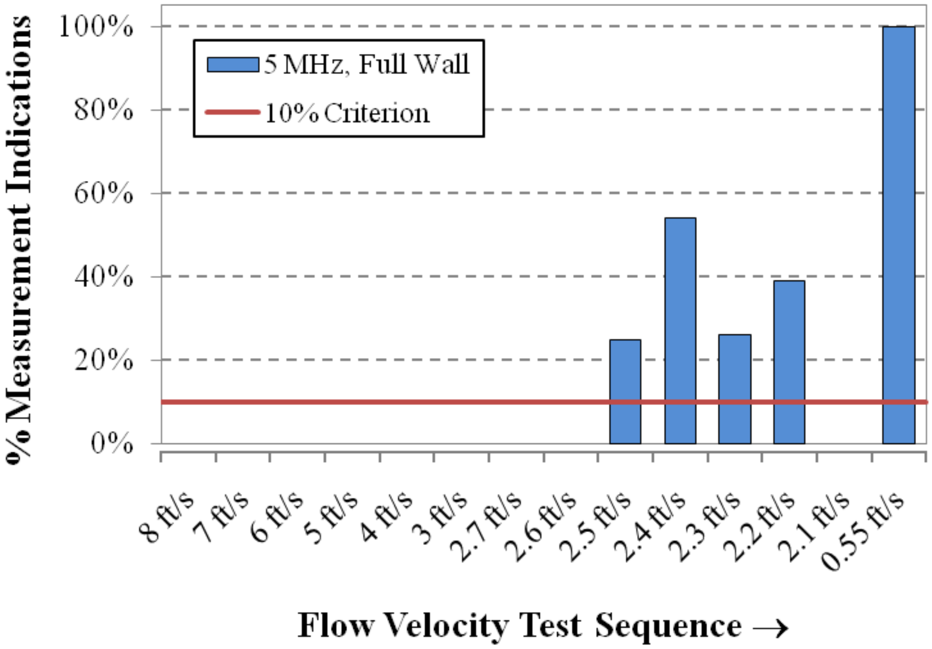

Figure 6.5. Test 1, 5-MHz Full Wall (2-wt % SS in Water)

---

[1] Data acquisition with the 5 MHz transducer located at the half-wall location was not performed because signals were obtainable at the 5 MHz full-wall location.

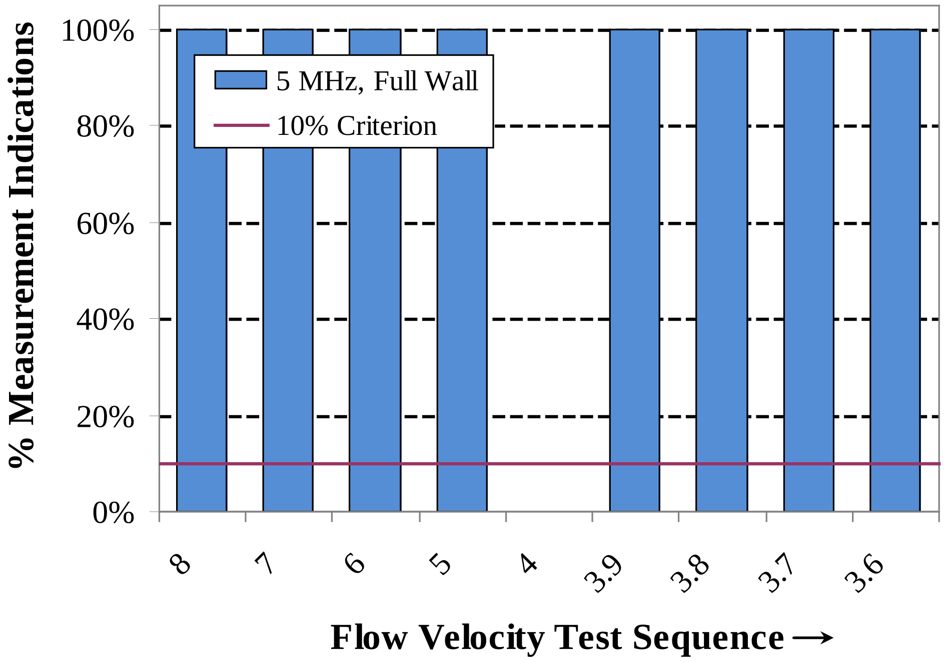

Figure 6.6. Test 5, 5-MHz Full Wall (2-wt % SS in Kaolin slurry)

6.3 10-MHz Transducer Discussion

The following subsections present the results for the full-wall, 10-MHz transducer. The primary objective was to evaluate how this transducer would perform in slurries where there is sufficient number of particles at or above the particle-size detection limit of 14 µm. The secondary objective was to determine the minimum solid concentration where there were no false indications.

6.3.1 Broad PSD Discussion

Figure 6.7 shows the percent indications using the normalized threshold versus the velocity sequence of Test 2. This figure shows that for the test sequence, a settled bed was not detected by the 10-MHz transducer using the standard 10% criterion. This is attributed to total attenuation of the signal from the settled bed, which rendered the normalized threshold ineffective because it relies on the backscatter of the particles in a moving fluid, i.e., the PulseEcho signal cannot penetrate through the settled bed.

During Test 2, the real-time oscilloscope measurements of the raw signal made simultaneously with the PulseEcho Data Aquistion System (DAS) produced a modulating signal that became stable at 3.3 ft/s and below. These observations suggested that using an absolute variance threshold instead of a normalized threshold is more appropriate for the 10-MHz transducer. As result, the absolute threshold was used for all subsequent tests for the 10-MHz transducer.

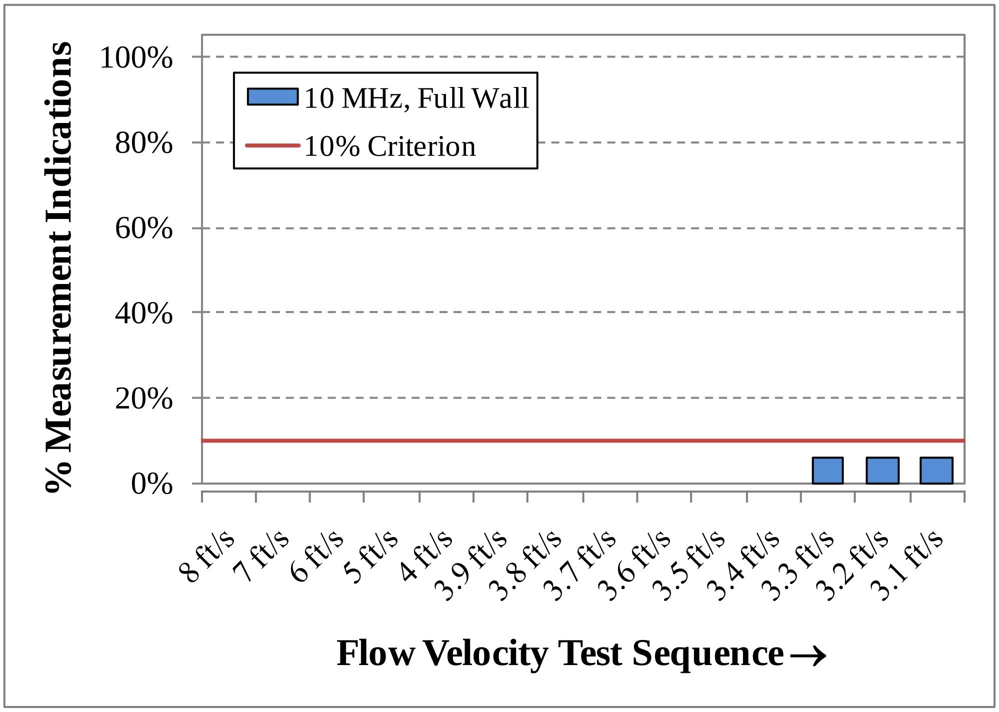

Figure 6.7. Test 2, 10-MHz Full Wall (5-wt% Broad PSD in water)

The results from Test 3 are shown in Figure 6.8. Note that for Test 3, the 10-MHz transducer exceeds the 10% criterion at 3.9 ft/s using the absolute threshold. This matches very well with the experimentally determined critical velocity of 4.0 ft/s.

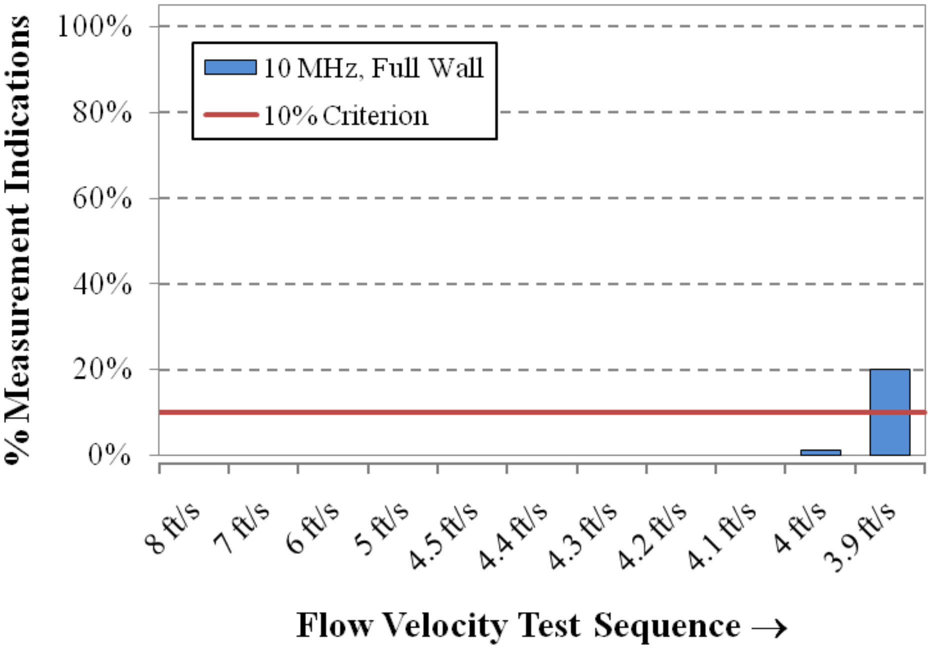

Figure 6.8. Test 3, 10-MHz Full Wall (20-wt% Broad PSD in water)

6.3.2 High-Density, Small Particle-Size Discussion

Based on the *ka* calculations performed in Section 3.1, the 10-MHz transducers were chosen specifically to detect settling of SS particles that were greater than 14 μm in size. The following section presents the results from evaluating the 10-MHz transducer for various carrier fluids and SS concentrations. SS particles were chosen to be a proxy for small, high-density particles, such as plutonium, in the actual wastes. Various carrier fluids were chosen to address the effects of attenuation of the 10-MHz signal. The carrier fluids tested included suspensions of kaolin in water, gibbsite in water, and iron oxide in water; two of the carrier fluids were chosen because they were representative of actual waste (suspensions of gibbsite and iron oxide in water).

Figure 6.9 shows the results for Test 1, which was performed with 2 wt% SS in water.[2] The 10-MHz transducer detected the critical velocity at 2.2 ft/s, which was within 0.2 ft/s of the experimentally observed critical velocity. As shown in Table 6.1, there is a 0.3 ft/s difference between the critical velocities detected by the 5- and 10-MHz transducers. A possible explanation for the difference is a settling gradient that was observed in the UT spool piece. Settling was observed in the upstream VS before the downstream VS. At the critical velocity reported, migrating dune structures had formed in the upstream section. However, in the downstream section, a ¼-inch wide band of mostly moving particles with some stationary particles present in the band was observed.

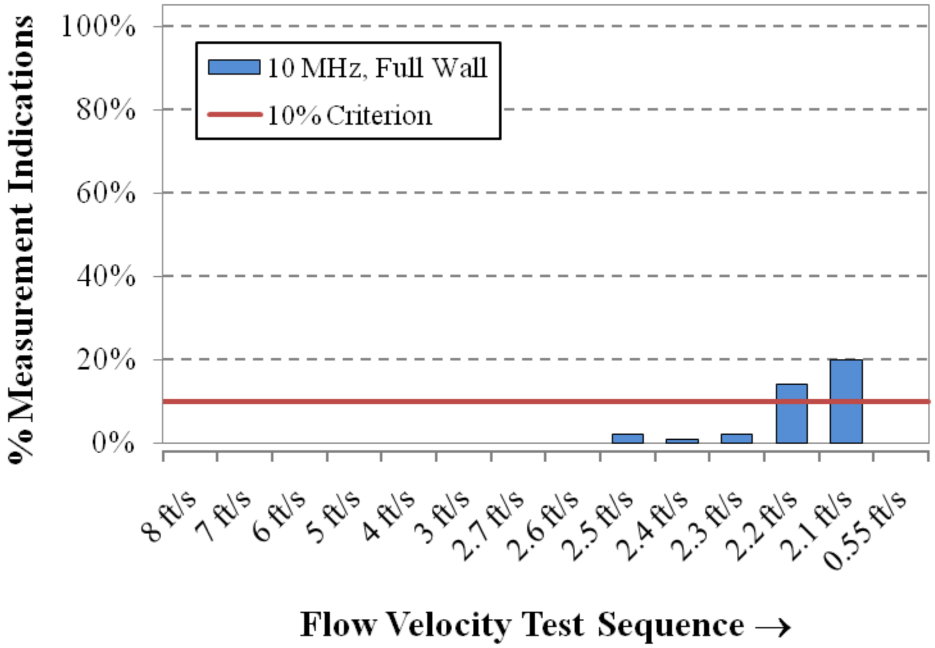

Figure 6.9. Test 1, 10-MHz Full Wall (2-wt % SS in Water)

The effect of changing the carrier fluid from a less attenuative medium (water) to a more attenuative medium, such as a kaolin slurry, for SS concentrations of 1 and 2 wt% resulted in false indications as detailed in Table 6.1. Kaolin was chosen as the initial carrier fluid in this study because it was previously used during Phase III testing, and the PSD overlaps that of the SS distribution. The 1 and 2 wt% were

---

[2] Note that the normalized threshold was used to generate the Test 1 results because the use of the absolute threshold was not warranted until Test 2 data was analyzed.

challenging for the 10-MHz transducer. At 1 wt% and 2 wt% SS in kaolin, the low concentration of detectable SS particles in the attenuative kaolin slurry resulted in very poor scattering and led to the false indications at velocities above the critical velocity.

Figure 6.10 shows the results of Test 6. At 4 wt% SS in kaolin, sufficient scattering could be obtained with the 10-MHz transducer at all flow velocities. The critical velocity was detected at 3.6 ft/s.

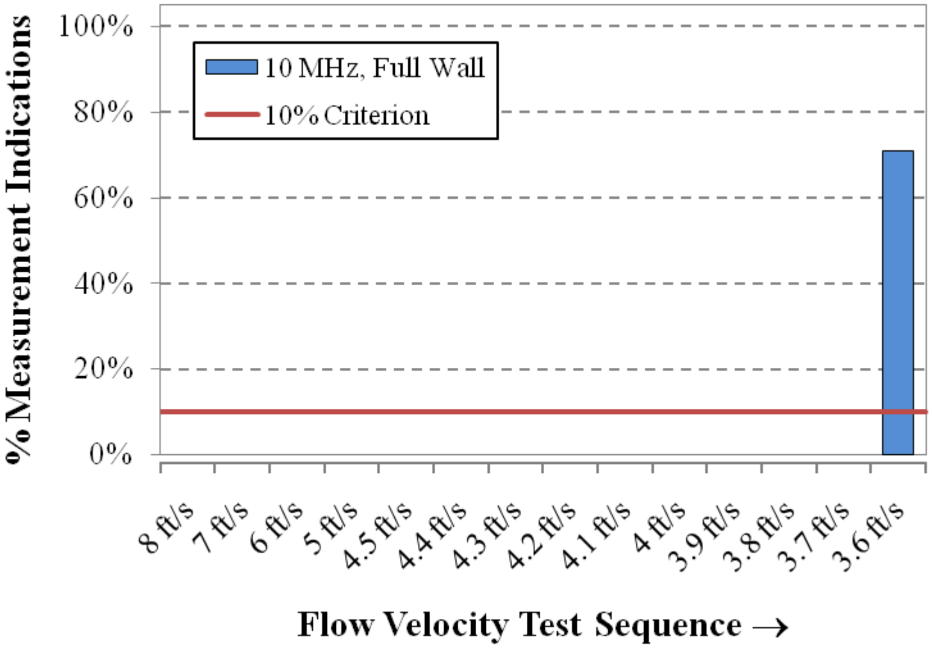

Figure 6.10. Test 6, 10-MHz Full Wall (4-wt % SS in Kaolin slurry)

Tests 7 and 8 were performed to determine whether scattering from the carrier fluids only, i.e., slurries of kaolin and gibbsite, could be detected by the 10-MHz and 5-MHz transducers. Different degrees of scattering were detectable with the 10-MHz transducer. A small amount of scattering was detected from the kaolin slurry, and appreciable scattering was detectable with the 10-MHz transducer for gibbsite. The scattering detected by the 10-MHz transducer in kaolin and gibbsite contributes to the detectable scattering in slurries that also contain SS particles. Testing to detect critical velocity was not performed for these simulants, although the velocity was reduced to 1 and 2 ft/s for the kaolin and gibbsite slurries, respectively. Settling was not observed at these velocities.

Due to the significantly higher scattering contribution from the gibbsite carrier fluid as compared to Kaolin, determination of the critical velocity with gibbsite in water was possible even at 1 wt% SS. This is illustrated in Figure 6.11. It can be seen from this figure that the critical velocity of 2.1 ft/s detected by the 10-MHz transducer matches within ±0.3 ft/s of the experimentally determined critical velocity of 2.4 ft/s (see Table 6.1).

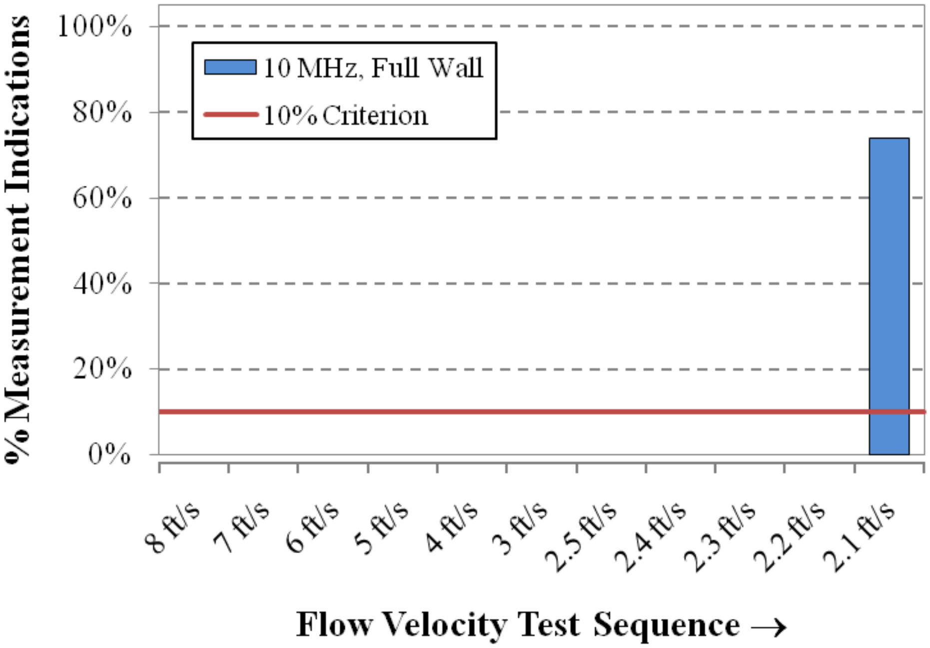

Figure 6.11. Test 9, 10-MHz Full Wall (1-wt % SS in Gibbsite slurry)

Figure 6.12 and Figure 6.13 show the results for tests 10 and 11 with SS in an iron oxide slurry. Tests 10 (1 wt% SS) and 11 (2 wt% SS) were difficult for the 10-MHz transducer to resolve. The low concentrations of SS in iron oxide did not provide sufficient scattering. Also, during both tests, no scattering contribution from the iron oxide was observed with the 10-MHz transducer. Lack of scattering led to false indications at velocities above the critical velocity. Despite the false indications at high flow velocities, the critical flow velocity was detected at 2.6 ft/s for Test 10 and 2.7 ft/s for Test 11. The ability of the transducers to give false indications at high velocities while still measuring the critical velocity at low velocities is attributed to the increasing concentration of the scatterers in the measuring volume near the transducer at lower velocities. This was discussed previously in Bontha et al. (2010a).

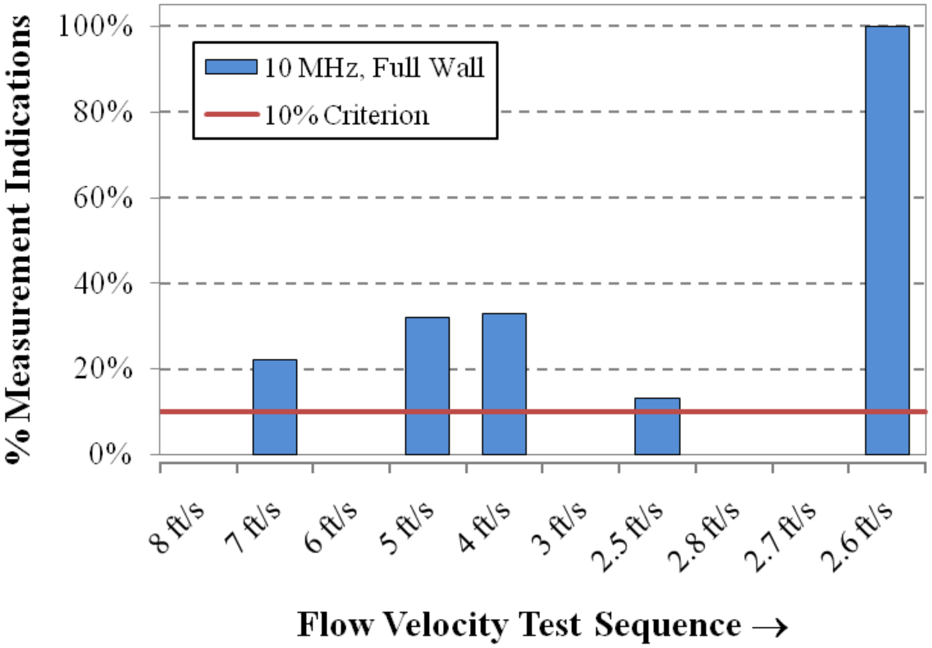

Figure 6.12. Test 10, 10-MHz Full Wall (1-wt % SS in Fe_2O_3 slurry)

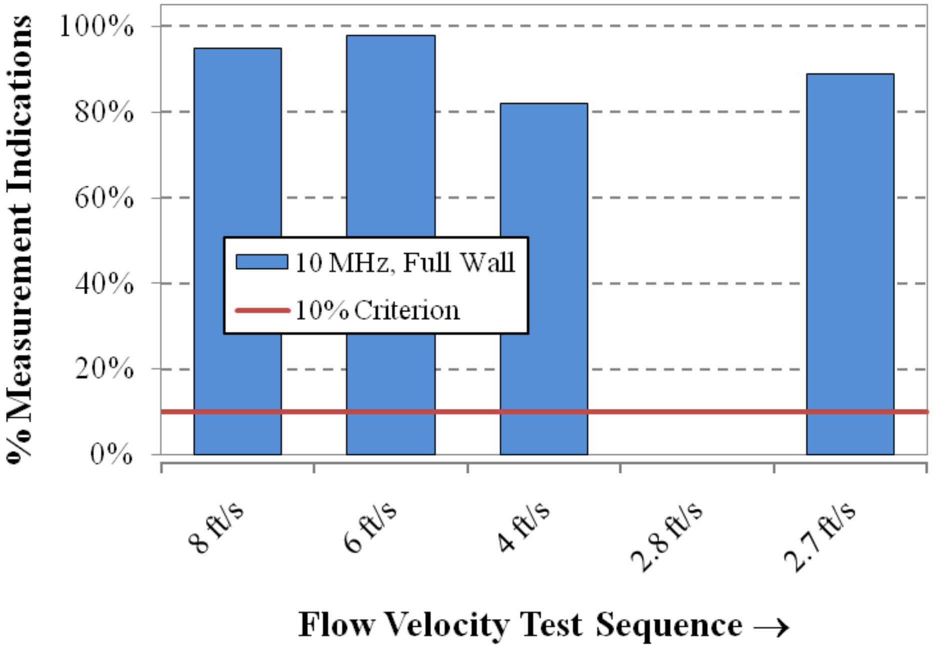

Figure 6.13. Test 11, 10-MHz Full Wall (2-wt % SS in Fe_2O_3 slurry)

Figure 6.14 shows the results for Test 12 with 4 wt% SS in iron oxide. It can be seen that sufficient scattering was obtained for the 10-MHz transducer at all flow velocities, and the critical velocity for this simulant could be measured. The velocity at which settling was detected was 2.9 ft/s. These results indicate that the SS concentration threshold for the 10-MHz transducer in a highly attenuative carrier fluid is ~4 wt%. Anything below this threshold will likely result in false indications at high flow velocities.

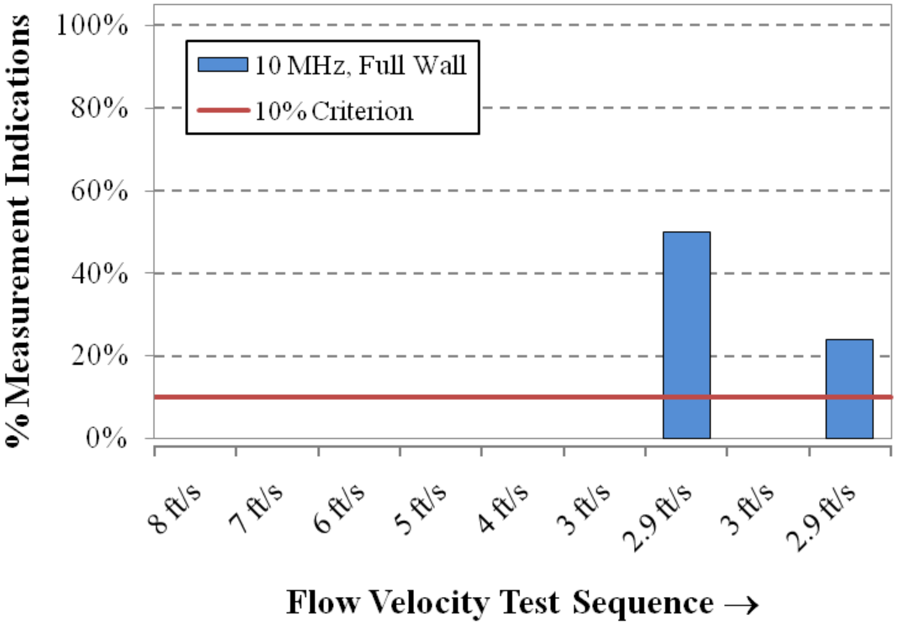

Figure 6.14. Test 12, 10-MHz Full Wall (4-wt % SS in Fe_2O_3 slurry)

6.4 Discussion of Small-Particle Effects

With ~14-µm being the lower particle size cutoff for the 10-MHz transducer, the fraction of SS particles that is detectable by the 10-MHz transducer is approximately only half of that which is added to any carrier fluid. This is evident in the cumulative plot provided in Figure 6.15. Therefore, the PulseEcho system was sensitive to approximately only half the SS particles in the prepared Phase IV simulants.

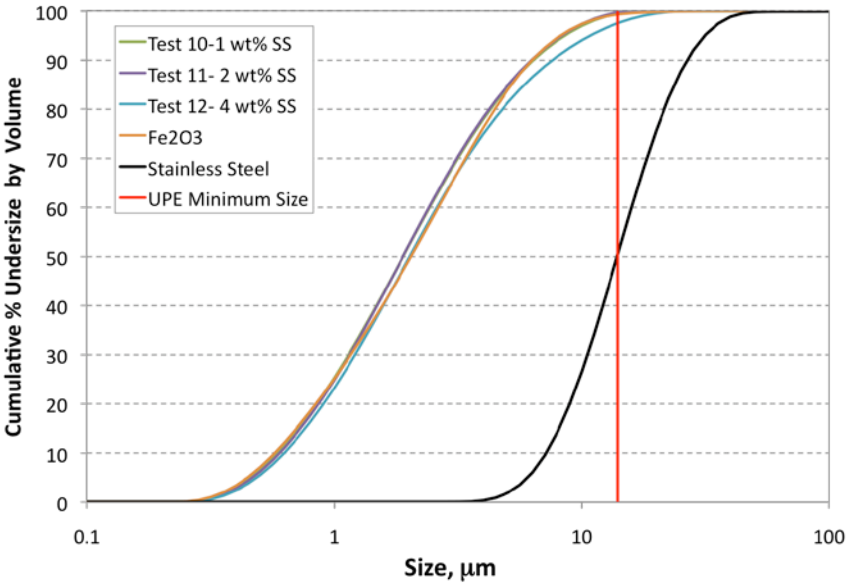

Figure 6.15. Cumulative Particle Size for SS and the Iron Oxide Simulants. The red vertical line represents the particle size cutoff for 10 MHz.

6.5 Scattering Contribution from Carrier Fluids

The lowest measurable solids concentration for the PulseEcho system is dependent upon the scattering strength that can be obtained from the measurable particle sizes amidst the background attenuation α of the carrier fluid. The scattering strength from particles in a carrier fluid is dependent upon the concentration of the detectable particle size, the difference in the acoustic impedances of the particle material and the carrier fluid, and the attenuation of the ultrasound signal by carrier fluid. These factors must be balanced to obtain sufficient scattering to perform a measurement.

Generally, attenuation means the loss of the signal's amplitude with increasing propagation distance. There are two major classes of attenuation mechanisms one should consider in ultrasonic materials characterization: absorption and scattering. The total attenuation can be written as the sum of the two components:

$$\alpha = \alpha_{scattering} + \alpha_{absorption}$$

Scattering converts the energy of the coherent, collimated beam into incoherent, divergent waves as a result of wave interaction with inhomogeneities (e.g., particles) in a material (e.g., carrier fluid). The scattered energy carried by the incoherent acoustic wave is not entirely lost due to scattering since a portion of it is detected by the ultrasonic transducer. The portion of scattered energy that is returned to the transducer is what the PulseEcho system uses to determine whether particles are mobile or stationary. Figure 6.16 illustrates the spherically diverging scattered wave from a small scatterer.

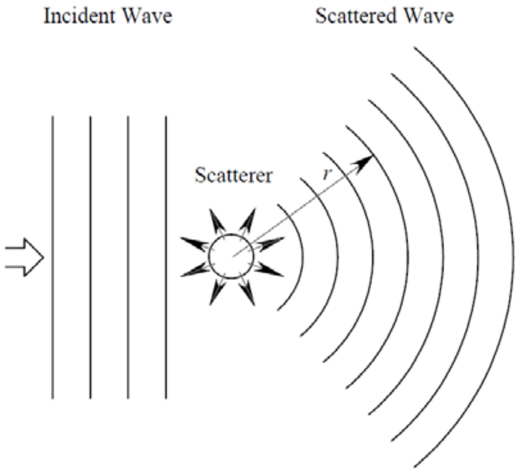

Figure 6.16. Spherically Diverging Scattered Wave from a Small Scatterer

Absorption, which converts acoustic energy into heat via viscosity, relaxation, heat conduction, elastic hysteresis, etc., is irreversibly lost from the acoustic field because it is dissipated in the medium (carrier fluid). Attenuation due to absorption is undesirable for the PulseEcho system because it decreases the strength of the scattered signals from the particles.

Water presents negligible ultrasonic attenuation for the 5-MHz and 10-MHz frequencies. Small fractions of SS particles are detectable in water because the backscatter signals from the particles are uninhibited by this carrier fluid.

The kaolin, iron oxide, and gibbsite carrier fluids presented higher attenuation (absorption) for the 5-MHz and 10-MHz frequencies. For the 5-MHz transducer, the fraction of the SS particles added to these carrier fluids that are detectable by the 5-MHz transducer was so small that scattering was completely attenuated by the carrier fluid. Therefore the 5-MHz transducer completely failed to detect settling in these carrier fluids.

Although the kaolin and gibbsite presented attenuation in the form of absorption, there was also favorable attenuation from these two carrier fluids in the form of scattering at 10 MHz. A relatively small amount of scattering was detected from particles in the kaolin carrier fluid, which contributed to the total detectable scattering from prepared simulants that used kaolin as the carrier fluid. This is explained by the PSD for kaolin, which contains a 22.7% fraction of particles that are greater than 14 microns, as shown in Figure 6.17. The absolute signal variance associated with the kaolin scattering contribution was measured at approximately $5 \cdot 10^{-5}$, which is 1.25 times that of the average signal variance of approximately $4 \cdot 10^{-5}$for a stable, non-modulating 10-MHz signal. However, even with the small scattering contribution from kaolin, a 4-wt% SS concentration was required to detect sufficient scattering at all flow velocities.

Scattering from particles in the iron oxide carrier fluid was not detected at 10 MHz because the size distribution of the iron oxide particles is below the ~14-μm cutoff particle size for 10 MHz. As shown in Figure 6.17, a negligible percentage (0.66%) of iron oxide particles is greater than 14 microns. Iron oxide represented the most challenging carrier fluid for the PulseEcho system because of the lack of scattering contribution and the high attenuation due to absorption. Due to the attenuation of the iron oxide carrier fluid, a 4-wt% SS concentration was required to detect sufficient scattering at all flow velocities at 10 MHz.

Appreciable scattering from gibbsite was detected at 10 MHz, which contributed to the total detectable scattering from the simulant prepared with gibbsite as the carrier fluid. This is explained by the PSD for gibbsite, which contains a 28.9% fraction of particles that are greater than the 14-μm cutoff as shown in Figure 6.17. The absolute signal variance associated with gibbsite alone was measured at 3e-03, which is 75 times that of the average signal variance for a stable, non-modulating 10-MHz signal. This significant scattering contribution from the gibbsite explains why scattering was obtained at all flow velocities for the simulant prepared with 1 wt% SS.

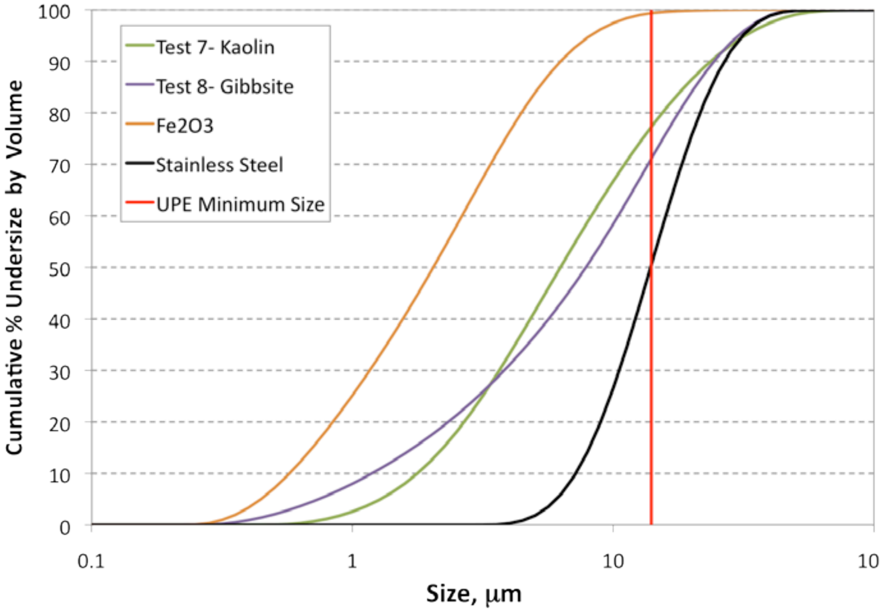

Figure 6.17. Cumulative Particle Size for SS and Carrier Fluids. The red vertical line represents the particle size cutoff for 10 MHz.

7.0 Considerations for Field Deployment

7.1 Multi-Transducer Configurations

The use of the 10-MHz and 5-MHz transducers to accommodate particle sizes of ~14 μm and greater is recommended. The 10-MHz transducer alone may be suitable to accommodate this range may be adequate; however, additional testing should be completed before making this decision.

7.2 Normalized Versus Absolute Threshold

It is recommended that both the absolute threshold and normalized threshold be applied with the PulseEcho system. The use of both thresholds together will report the presence of solids at the location of a transducer under conditions where 1) a signal modulation gradient is forming inside the defined measurement window and 2) signal modulation has stopped inside the defined measurement window as a result of total signal attenuation.

The use of the normalized threshold alone was found to be sufficient during Phase III testing because conditions of signal modulation and signal stability were captured inside the defined measurement window. This can be attributed to the capability of the 5-MHz transducer to detect moving solids above settled solids for the simulants tested. Therefore, the use of the absolute threshold was not necessary during Phase III testing. Conditions of total signal attenuation as a result of solids settling were observed inside the defined measurement window with the introduction of the 10-MHz transducer during Phase IV. This condition rendered the normalized threshold ineffective early on during testing and warranted the use of the absolute threshold. The absolute variance threshold allows the PulseEcho algorithm to report conditions of particle mobility and particle settling, but cannot measure the depth of sediment present at the location of the transducer.

It should be cautioned that the pulse-echo system demonstrated in the Phase IV testing is primarily focused on measuring the bulk critical velocity of the significant slurry components. If detection of small particle accumulation of sparsely distributed slurry components (i.e. less than 2 wt%) becomes a priority, then alternate detection technologies should be considered.

7.3 Transducer Bonding

The epoxy material that has been used to bond the PulseEcho transducers to the spool piece are appropriate only for testing in laboraotry environments. A sustainable material that can tolerate radiation environments and wider temperature ranges needs to be identified for field deployment.

7.4 Radiation Hardening

The radiation dosage tolerance of PulseEcho ultrasonic transducers, connectors, and cables needs to be evaluated to determine the expected lifetime of field deployed transducer(s). Ultrasonic technology is routinely used in industrial and nuclear environments. The ultrasonic transducer can be radiation hardened and shielded to operate in high/low temperature and low/high radiation environments. The

Applied Physics group at PNNL has recently ordered and received such transducers from U.S. transducer vendors.

A PNNL report by Griffin et al. (2009) provides a summary review of gamma and neutron radiation damage in piezoelectric materials. In general, inorganic piezoelectric materials (both crystalline and sintered) show minimal permanent damage due to gamma irradiation. The magnitude of gamma radiation effects on piezoelectric materials are material dependent. Lead zirconate titanate (PZT) is known to be quite radiation hard. Radiation effects are most easily observed in the hysteresis curve (i.e., the D-E curve) for ferroelectric (piezoelectric) materials. Previous work has shown that PZT films show more symmetric hysteresis curves after 5 Mrad(Si), regardless of their conditions during irradiation, different D-E curve distortions when different bias conditions were applied during irradiation, and radiation-induced D-E curve distortion that could be prevented by electrical cycling during irradiation and could be removed by post-irradiation electrical cycling. Lithium niobate shows no substantial changes in its piezoelectric properties even after exposure of 4×10^9 R of ^{60}Co irradiation at high temperatures. Because neutron damage in materials is due to elastic collisions and displacement of atoms in the material matrix, cumulative dose effects are anticipated. However, PZT shows no significant degradation due to neutron exposure event for fluencies at or below 10^{15} n/cm^2. Experimental data have not been found for neutron irradiation damage in lithium niobate.

Short, low-capacitance, coaxial, high-temperature, radiation-resistant cables are required. This type of cable is available commercially with a SiO_2 dielectric. Piezoelectric transducer impedance should be matched with a passive matching network to cable impedance to minimize spurious signals.

8.0 Conclusions and Recommendations

This report presents the configuration, methodology, and results of 12 tests performed at the MTEL. The tests were conducted to scrutinize the performance of three PulseEcho transducers. There were two 5-MHz transducers mounted at full and half Schedule 40 pipe wall thickness in addition to a 10-MHz transducer mounted at full wall thickness. Carrier fluids and simulant particles were specified to challenge the transducers and validate past performance of the 5-MHz transducer. The results obtained lead to several conclusions and recommendations, as follows:

1. For >50μm particles, the performance of the 5-MHz transducer mounted at the half wall thickness was repeatable as compared with Phase III results. In addition, the performance of the 5-MHz transducers mounted at both the full and half wall thickness yielded the same results for critical velocity. Therefore, this transducer can be mounted on a near full Schedule 40 pipe wall thickness.

2. The 5-MHz transducer was not capable of detecting critical velocities and settling of particles less than 30 µm. However, the concentration threshold for this transducer for 30 µm particles was not evaluated.

3. The detection of critical velocity for particles in the WRPS desired range of 20 μm and greater requires a 10-MHz transducer.

4. It is recommended that the absolute threshold mode of operation be applied with the PulseEcho system when using the 10-MHz tranducer. This will allow for detection of small particle settled beds even in highly attenuated conditions expected with many small particles distributed in the slurry. PulseEcho normalized thresholds were insufficient for detecting settled beds for the 10-MHz transducer. This is attributed to total attenuation of the signal, preventing it from penetrating through the bed. This condition rendered the normalized threshold ineffective early on during testing and warranted the use of the absolute threshold. The absolute variance threshold allows the PulseEcho algorithm to report conditions of particle mobility and particle settling, but cannot measure the depth of sediment present at the location of the transducer.

5. Changing the carrier fluid from water or gibbsite to kaolin or iron oxide increased the attenuation of the PulseEcho signal. The increased attenuation for these fluids resulted in decreased signal modulation and thus, false indications were given for low concentrations of small, high-density, SS particles at high flow velocities. For the tests with SS particles in highly attenuative carrier fluids, only the 10-MHz transducer was capable of detecting particle settling without false indications at a particle concentration of 2 wt% or higher in the detectable size range.

6. The 10-MHz transducer performed exceptionally well, considering the small volume fraction of detectable (>14 μm) particles within the carrier fluids. However, due to the limited testing performed on this transducer, additional rigorous testing is recommended to validate the sensor performance over the range of expected waste conditions that will be encountered in the waste certification loop.

The Phase IV testing demonstrated the viability and sensitivity of the 10-MHz transducers to detect settled particles that are smaller and at lower concentrations than the 5-MHz transducer. It is recommended that future testing and field deployment include the 10-MHz transducers. Because of the added capability of the 5-MHz system to probe further into the pipe flow characteristics (i.e, measure settled bed depth via normalized threshold), complimenting the 10-MHz system with 5-MHz transducers should be considered as an option for a more flexible and robust system. It should be cautioned that the

PulseEcho system demonstrated in the Phase IV testing is primarily focused on measuring the bulk critical velocity properties of the significant slurry components. If detection of small particle accumulation of sparsely distributed slurry components becomes a priority, alternate detection technologies should be considered.

9.0 References

Bontha JR, HE Adkins, KM Denslow, JJ Jenks, CA Burns, PP Schonewill, GP Morgen, MS Greenwood, J Blanchard, TJ Peters, PJ MacFarlan, EB Baer, and WA Wilcox. 2010a. *Test Loop Demonstration and Evaluation of Slurry Transfer Line Critical Velocity Measurement Instruments.* PNNL-19441, Rev.0, Pacific Northwest National Laboratory, Richland, Washington.

Bontha JR, HE Adkins, KM Denslow, JJ Jenks, CA Burns , PP Schonewill, GP Morgen, MS Greenwood, J Blanchard, TJ Peters, PJ MacFarlan, EB Baer, and WA Wilcox. 2010b. *Supplementary Information for Test Loop Demonstration and Evaluation of Slurry Transfer Line Critical Velocity Measurement Instruments.* PNNL-19560, Pacific Northwest National Laboratory, Richland, Washington.

Griffin JW, TJ Peters, GJ Posakony, HT Chien, LJ Bond, KM Denslow, SH Sheen, and P Raptis. 2009. Under-Sodium Viewing: A Review of Ultrasonic Imaging Technology for Liquid Metal Fast Reactors . PNNL-18292, Pacific Northwest National Laboratory, Richland, Washington.

Meyer PA, HE Adkins, KM Denslow, JA Bamberger, BE Wells, MS Greenwood, PA Scott, J Blanchard, GP Morgen. 2009. *Hanford Tank Farms Waste Certification Flow Loop Test Strategy.* PNNL-18688, Pacific Northwest National Laboratory, Richland, Washington.

Poloski AP, HE Adkins, J Abrefah, AM Casella, R Hohimer, F Nigl, MJ Minette, JJ Toth, JM Tingey, and SM Yokuda. 2009a. *Deposition Velocities of Non-Newtonian Slurries in Pipelines.* PNNL-17639, WTP-RPT-175 Rev. 0. Pacific Northwest National Laboratory, Richland, Washington.

Poloski AP, ML Bonebrake, AM Casella, MD Johnson, JJ Toth, HE Adkins, Jr, J Chun, KM Denslow, M Luna, and JM Tingey. 2009b. *Deposition Velocities of Non-Newtonian Slurries in Pipelines: Complex Simulant Testing* . PNNL-18316, WTP-RPT-189 Rev. 0. Pacific Northwest National Laboratory, Richland, Washington.

Smith GL and K Prindiville. 2002. *Guidelines for Performing Chemical, Physical, and Rheological Properties Measurements.* 24590-WTP-GPG-RTD-001 Rev. 0, Bechtel National, Inc., Richland, Washington.

Yokuda ST, AP Poloski, HE Adkins, AM Casella, RE Hohimer, NK Karri, M Luna, MJ Minette, and JM Tingey. 2009. *A Qualitative Investigation of Deposition Velocities of a Non-Newtonian Slurry in Complex Pipeline Geometries.* PNNL-17973, WTP-RPT-178, Rev. 0, Pacific Northwest National Laboratory, Richland, Washington.

Appendix

Test Summaries

Appendix

Test Summaries

Test Number/TI	Test #1 /WRPS-Phase4-TI-01
Test Date	01/20/2011
TE:	Jeromy Jenks
Carrier Fluid	Water
Simulant Description:	Stainless Steel (d_{50}~14μm)
Simulant Load:	2 wt%

Summary:

This test consisted of 2-wt% SS beads in water.

The test started at a velocity of 8 ft/s, and this was incrementally reduced to below the critical velocity of 2.4 ft/s. Two tests were performed, one for each PulseEcho frequency.

The test started at a velocity of 8 ft/s and was incrementally reduced by 1 ft/s until critical behavior was observed. No distinct stratification was evident until the velocity was 4 ft/s.

At 4 ft/s, the flow was beginning to stratify and come into focus. This was deemed Regime I. At 3 ft/s, a sediment bed was clearly visible and moving in-line with, but slower than, the main body of flow. This is indicative of Regime II. At 2.5 ft/s, a ¼-inch wide moving band had formed in the VS, with other particles streaking along the periphery of this band. At 2.4 ft/s, dune structures formed that were a ½ inch wide by 2 inches long and were spaced approximately 6 inches apart. These dune structures migrated in an erosion/deposition manner in which the bottom side of the dunes were stationary as viewed by the camera, before eroding away. Due to the stationary nature of these dune structures, this was considered critical velocity. In the Downstream (DS) VS, a ¼-in. band of moving particles was observed, with some particles "sticking" to the inside of the pipe at the strand's location. As the velocity was reduced further, the dunes grew larger and closer together. The dunes grew to 1 inch wide by 3 inches long at 2.1 ft/s. On an identical test performed the previous day, the velocity was throttled to 0.5 ft/s. At this velocity, and allowing over 30 minutes to achieve steady state, perfect dune structures formed and filled the bottom periphery of the pipe. The top portion of the pipe was translucent because all the simulant had settled on the pipe bottom.

Pressure differential data were verified at the end of the test by increasing the flow to 8 ft/s.

Test Number/TI	Test #2 /WRPS-Phase4-TI-02
Test Date	01/22/2011
TE:	Jeromy Jenks
Carrier Fluid	Water
Simulant Description:	Broad PSD, Potters glass, 2.50 g/mL
Simulant Load:	5 wt%

Summary:

This test consisted of 5-wt%, broad, PSD glass beads in water.

The test started at a velocity of 8 ft/s, and this was incrementally reduced to below the critical velocity of 3.1 ft/s.

The test started at a velocity of 8 ft/s that was incrementally reduced by 1 ft/s until critical behavior was observed. No distinct stratification was evident until the velocity was 6 ft/s.

At 5 ft/s, slightly more stratification was evident, but was not coming into focus on the pipe bottom as viewed using the camera. At 4 ft/s, the simulant was coming into sharper focus and some black “contaminant” particles were visible. These were believed to be an artifact of the manufacturing process of the glass beads. No distinct changes were noted at 3.9 ft/s. At 3.8 and 3.7 ft/s, Regime I was evident. At 3.6 ft/s, the particles slowed down, building up to a sliding bed before washing away. At 3.5 ft/s, the simulant had characteristics of Regime II and III. The bed would slow down, grow to a ¾ of an inch wide pulsatory bed and wash away to focused particles in-line with the flow field. At 3.4 ft/s, a starting/stopping bed was observed. The bed would stop for 15 to 20 seconds and then move for 15 to 20 seconds. As the bed grew, a different shade of moving simulant was visible on the periphery of the ½-inch-wide stopped bed, making the entire focused width 1 inch wide. At 3.3 dt/s, the phenomena was the same; however, the starting/stopping period was slightly longer, and the stopped bed width was ¾ of an inch. The critical velocity was reached at 3.2 ft/s, with a ½-inch wide bead of stationary particles. At 3.1 ft/s, the bed erosion/formation phenomena manifested itself on the DAS as inverse trends of oscillating fluid density (measured by the Coriolis Flow Meter [CFM]) and pressure drop across the test section.

Pressure differential data were verified at the end of the test by increasing the flow to 8 ft/s.

Test Number/TI	Test #3 /WRPS-Phase4-TI-03
Test Date	01/24/2011
TE:	Jeromy Jenks
Carrier Fluid	Water
Simulant Description:	Broad PSD, Potters glass, 2.50 g/mL
Simulant Load:	20 wt%

Summary:

This test consisted of 20 wt % broad PSD glass beads in water.

The test started at a velocity of 8 ft/s, and this was incrementally reduced to below the critical velocity of 3.1 ft/s.

The test started at a velocity of 8 ft/s that was incrementally reduced by 1 ft/s until critical behavior was observed. No distinct stratification was evident until the velocity was 6 ft/s.

At 5 ft/s, slightly more stratification was evident, but was not coming into focus on the pipe bottom as viewed using the camera. At 4.5 ft/s, the simulant was coming into sharper focus and moving in a chaotic manner indicative of Regime I. At 4.4 ft/s, a ½-inch wide band of particles moving in-line with the flow was visible before washing away in a chaotic manner. At 4.3 ft/s, Regime II was evident with a ¼- to ½-inch-wide band of individually moving particles forming on the pipe bottom. At 4.2 ft/s, a pulsating/eroding bed was observed indicative of Regime III. The bed varied, starting at 1 inch wide and eroding to ¼ inch wide. The bed erosion/formation phenomenon manifested itself on the DAS as inverse trends of oscillating fluid density (measured by the CFM) and pressure drop across the test section. At 4.1 ft/s, a starting/stopping bed formed with 15- to 20-second intervals growing to a maximum of 1.5 to 2 inches wide. At 4 ft/s, the crtical velocity was observed with a stationary bed measuring ½ of an inch wide. Note that a different shade of particles was forming/eroding away on top of this stationary bed. At 3.9 ft/s, the stationary bed grew to 1 inch wide.

Pressure differential data were verified at the end of the test by increasing the flow to 8 ft/s.

Test Number/TI	Test #4 /WRPS-Phase4-TI-04
Test Date	01/26/2011
TE:	Jeromy Jenks
Carrier Fluid	20 wt% Kaolin
Simulant Description:	Stainless Steel
Simulant Load:	1 wt% (SS)

Summary:

This test consisted of a 20-wt% Kaolin slurry with 1-wt% SS beads.

The test started at a velocity of 8 ft/s, and this was incrementally reduced to below the critical velocity of 3.6 ft/s. A rheology sample was taken.

The test started at a velocity of 8 ft/s that was incrementally reduced by 1 ft/s until critical behavior was observed. No distinct stratification was evident until the velocity was 7 ft/s.

At 7 and 6 ft/s, dark particle lines (SS) ~ $^{1}/_{16}$ inch wide were evident, streaking along the bottom of the pipe. At 5 ft/s, the SS lines began to coalesce near the pipe bottom, forming up to ¼-inch-wide bands at times and moving in a chaotic yet pulsatory manner indicative of Regime I. At 4 ft/s, the particle band width was between ¼ and ½ inch wide and in-line with the flow. Though the particles were pulsatory, this was more indicative of Regime II. At 3.7 ft/s, a ¼- to ½-inch wide, pulsatory, sliding bed formed, indicative of Regime III. At 3.6 ft/s, the critical velocity was reached with a ½-inch-wide, stationary band visible along the pipe bottom. At 3.5 ft/s, the stationary bed grew to 1 inch wide.

Pressure differential data were verified at the end of the test by increasing the flow to 8 ft/s. A rheology sample was taken.

Test Number/TI	Test #5 /WRPS-Phase4-TI-04
Test Date	01/27/2011
TE:	Jeromy Jenks
Carrier Fluid	20 wt% Kaolin
Simulant Description:	Stainless Steel
Simulant Load:	2 wt% (SS)

Summary:

This test consisted of a 20-wt% Kaolin slurry with 2-wt% SS beads.

The test started at a velocity of 8 ft/s, and this was incrementally reduced to below the critical velocity of 3.6 ft/s. A rheology sample was taken.

The test started at a velocity of 8 ft/s that was incrementally reduced by 1 ft/s until the critical behavior was observed. No distinct stratification was evident until the velocity was 7 ft/s.

At 7 and 6 ft/s, dark particle lines (SS) ~ $^1/_{16}$ inch wide were evident, streaking along the bottom of the pipe. At 5 ft/s, the SS lines began to coalesce near the pipe bottom, moving in a chaotic yet pulsatory manner indicative of Regime I. At 4 ft/s, the particle band width was from 1 to 1.5 inches wide in the upstream VS and ½ to 1 inch wide in the downstream VS and in-line with the flow. The bands on the periphery of the center band grew thicker. The flow was indicative of Regime II. At 3.9 ft/s, the band width in the upstream and downstream VS was 1.5 and 1 inch, respectively. At 3.8 ft/s, the band width in the upstream and downstream VS was 2 and 1.5 inches, respectively. At 3.7 ft/s, the bed widths were the same but had slowed considerably, indicative of Regime III. At 3.6 ft/s, the critical velocity was observed with a ½-inch-wide stationary bed in both VSs.

Pressure differential data were verified at the end of the test by increasing the flow to 8 ft/s. A rheology sample was taken.

Test Number/TI	Test #6 /WRPS-Phase4-TI-04
Test Date	01/28/2011
TE:	Jeromy Jenks
Carrier Fluid	20 wt% Kaolin
Simulant Description:	Stainless Steel
Simulant Load:	4 wt% (SS)

Summary:

This test consisted of a 20-wt% Kaolin slurry with 4 wt% SS beads.

The test started at a velocity of 8 ft/s, and this was incrementally reduced to below the critical velocity of 3.7 ft/s. A rheology sample was taken.

The test started at a velocity of 8 ft/s that was incrementally reduced by 1 ft/s until critical behavior was observed. No distinct stratification was evident until the velocity was 7 ft/s.

At 7 and 6 ft/s, dark particle lines (SS) ~ $^{1}/_{16}$ inch wide were evident, streaking along the bottom of the pipe. At 5 ft/s, the SS lines began to coalesce near the pipe bottom, moving in a chaotic yet pulsatory manner indicative of Regime I. At 4.5 ft/s, the particle band width was from 1 to 1.5 inches wide in the upstream VS and ½ to 1 inch wide in the downstream VS and in-line with the flow. The flow was indicative of Regime II. At 4.4 ft/s, no distinct changes were noticed from the previous velocity. At 4.3 ft/s, the band width in the upstream and downstream VS was 1.5 to 2 and ½ to 1 inch, respectively. At 4.2 ft/s, the band width in the upstream and downstream VS was 2 and 1.5 inches, respectively. At 4.1 ft/s, the bands slowed considerably and were pulsatory, indicative of Regime III. At the upstream VS, the particles mostly filled the bottom periphery of the pipe, with the downstream VS bed width varying between 1.5 and 2 inches. At 4 and 3.9 ft/s, the bottom centerline velocity appeared to slow, and the downstream VS band width was 2 inches wide. At 3.8 ft/s, a $^{3}/_{8}$-inch-wide, stationary bed was observed in the upstream VS, but the particles were still moving in the downstream VS. At 3.7 ft/s, a $^{3}/_{8}$ of an inch wide stationary bed had formed across the entire test section, and the critical velocity was observed. At 3.6 ft/s, the bed width grew to ½ of an inch.

Pressure differential data were verified at the end of the test by increasing the flow to 8 ft/s. A rheology sample was taken.

Test Number/TI	Test #7 /WRPS-Phase4-TI-05
Test Date	02/08/2011
TE:	Phil Schonewill
Carrier Fluid	20 wt% Kaolin
Simulant Description:	No additional simulant added
Simulant Load:	Kaolin only

Summary:

This test consisted of a 20-wt% Kaolin slurry. The purpose of the test was to verify that modulation was observed for the 10-MHz PulseEcho transducer.

The test started at a velocity of 8 ft/s, and this was incrementally reduced to a velocity of 1 ft/s. A rheology sample was taken.

The test started at a velocity of 8 ft/s that was incrementally reduced by 1 ft/s to 1 final velocity of 1 ft/s. No distinct stratification was evident except for an almost immeasurably small (~1 mm) wide band of SS that apparently did not exit the loop during the previous day’s flushing operations.

Pressure differential data were verified at the end of the test by increasing the flow to 8 ft/s. A rheology sample was taken.

Test Number/TI	Test #8 /WRPS-Phase4-TI-06
Test Date	02/17/2011
TE:	Jeromy Jenks
Carrier Fluid	15 wt% Gibbsite
Simulant Description:	No additional simulant added
Simulant Load:	Gibbsite only

Summary:

This test consisted of a 15-wt% Gibbsite slurry. The purpose of the test was to verify that modulation was observed for the 10-MHz PulseEcho transducer.

The test started at a velocity of 8 ft/s, and this was incrementally reduced to a velocity of 2 ft/s. A rheology sample was taken.

The test started at a velocity of 8 ft/s that was incrementally reduced to 5 ft/s and 2 ft/s. No distinct stratification was evident except for an almost immeasurably small (~1 mm) wide band of SS that apparently did not exit the loop during the previous day’s flushing operations.

Pressure differential data were verified at the end of the test by increasing the flow to 8 ft/s. A rheology sample was taken.

Test Number/TI	Test #9 /WRPS-Phase4-TI-06
Test Date	02/17/2011
TE:	Jeromy Jenks
Carrier Fluid	15 wt% Gibbsite
Simulant Description:	Stainless Steel
Simulant Load:	1 wt %

Summary:

This test consisted of a 15-wt% Gibbsite slurry with 1 wt% SS.

The test started at a velocity of 8 ft/s, and this was incrementally reduced to a velocity of 2.1 ft/s. A rheology sample was taken.

The test started at a velocity of 8 ft/s that was incrementally reduced by 1 ft/s to 3 ft/s. Smaller 0.1-ft/s increments were taken at 2.5 ft/s. No distinct stratification was evident until 5 ft/s, when SS was evident, flowing along the bottom portion of the pipe. More stratification was evident at 4 ft/s, whereupon distinct particle lines were visible. At 3 ft/s, the particle lines came into sharper focus. At 2.5 ft/s, the particle lines coalesced near the bottom of the pipe to form larger bands. The largest band was on the bottom center of the pipe and measured $^{1}/_{8}$ to ¼ inches wide, moving in a transverse chaotic manner indicative of Regime I. At 2.4 ft/s, the critical velocity was reached with a ¼ to $^{3}/_{8}$–inch-wide band formed in the upstream VS, and a $^{1}/_{8}$ inch band in the downstream VS. As the velocity was dropped, the bands grew in width until at 2.2 ft/s, the 1-inch-wide by 3-inch-long dunes began to form, spaced 3 to 4 inches apart and connected by stationary narrow bands of particles. At 2.1 ft/s, the dunes grew in length to 4 to 5 inches with some having free-flowing particles on the downstream side of the dune.

Pressure differential data were verified at the end of the test by increasing the flow to 8 ft/s. A rheology sample was taken.

Test Number/TI	Test #10 /WRPS-Phase4-TI-07
Test Date	03/02/2011
TE:	Jeromy Jenks
Carrier Fluid	15 wt% Iron Oxide
Simulant Description:	Stainless Steel
Simulant Load:	1 wt %

Summary:

This test consisted of a 15-wt% iron oxide slurry with 1 wt% SS.

The test started at a velocity of 8 ft/s, and this was incrementally reduced to a velocity of 2.6 ft/s. A rheology sample was taken.

The test started at a velocity of 8 ft/s that was incrementally reduced by 1 ft/s to 3 ft/s. Smaller 0.1-ft/s increments were taken at 2.8 ft/s. It was very difficult to see the SS on the bottom of the VS because of a thin layer of iron oxide forming around the pipe periphery. Although particle motion was discernable behind the film, flow (i.e., Regime) characterization was impossible. At 2.8 ft/s, slow particle motion was visible on the bottom of the VS using the camera. At 2.7 ft/s, an ~$^1/_8$-inch-wide stationary bed formed in the upstream visualization section. The camera was not moved to verify the DS visualization section because of the difficulty associated with refocusing on the particle bed through the iron oxide film. At 2.6 ft/s, the band grew to ~ ¼ inch. The critical velocity was determined to be between 2.6 and 2.7 ft/s.

Pressure differential data were verified at the end of the test by increasing the flow to 8 ft/s. A rheology sample was taken.

Test Number/TI	Test #11 /WRPS-Phase4-TI-07
Test Date	03/02/2011
TE:	Jeromy Jenks
Carrier Fluid	15 wt% Iron Oxide
Simulant Description:	Stainless Steel
Simulant Load:	2 wt %

Summary:

This test consisted of a 15-wt% iron oxide slurry with 2 wt% SS.

The test started at a velocity of 8 ft/s, and this was incrementally reduced to a velocity of 2.7 ft/s.

The test started at a velocity of 8 ft/s and was reduced to 6, 4, 2.8, and 2.7 ft/s. It was very difficult to see the SS on the bottom of the VS because of a thin layer of iron oxide forming around the pipe periphery. Although particle motion was discernable behind the film, flow (i.e., Regime) characterization was impossible. At 2.8 ft/s, slow particle motion was visible on the bottom of the VS using the camera. At 2.7 ft/s, an ~1-inch-wide stationary bed formed in the upstream visualization section. The camera was not moved to verify the DS visualization section because of the difficulty associated with refocusing on the particle bed through the iron oxide film. As such, the critical velocity was determined to be 2.7 ft/s.

Pressure differential data were verified at the end of the test by increasing the flow to 8 ft/s. A rheology sample was taken.

Test Number/TI	Test #12 /WRPS-Phase4-TI-07
Test Date	03/03/2011
TE:	Jeromy Jenks
Carrier Fluid	15 wt% Iron Oxide
Simulant Description:	Stainless Steel
Simulant Load:	4 wt %

Summary:

This test consisted of a 15-wt% iron oxide slurry with 4 wt% SS.

The test started at a velocity of 8 ft/s, and this was incrementally reduced to a velocity of 2.9 ft/s.

The test started at a velocity of 8 ft/s and was reduced by 1 ft/s down to 3 ft/s. It was very difficult to see the SS on the bottom of the VS because of a thin layer of iron oxide forming around the pipe periphery. Although particle motion was discernable behind the film, flow (i.e., Regime) characterization was impossible. At 3 ft/s, slow particle motion was visible on the bottom of the VS using the camera in the downstream VS. At the bottom center of the pipe, it appeared that a stationary bed of roughly $^1/_8$ inches wide had formed, although it was not easy to confirm and therefore deemed subjective. In the upstream VS, a ¾-inch-wide stationary bed had formed as viewed using the camera. At 2.9 ft/s, the downstream VS bed width was 3.4 to 1 inch. Thus, the critical velocity was confirmed at 2.9 ft/s

Pressure differential data were verified at the end of the test by increasing the flow to 8 ft/s. A rheology sample was taken.

Distribution

No. of
<u>Copies</u>

20	Pacific Northwest National Laboratory	
	JR Bontha (5)	P7-25
	CA Burns	P7-25
	HE Adkins	K7-15
	KA Denslow	K5-26
	JJ Jenks	K7-15
	P Schonewill	P7-25
	BE Wells	K7-15
6	Washington River Protection Solutions	
	M Thien (5)	B1-55
	T Wooley (1)	B1-55
2	Office of River Protection (DOE)	
	JS Shuen (2)	H6-60

Pacific Northwest
NATIONAL LABORATORY

U.S. DEPARTMENT OF
ENERGY

Proudly Operated by **Battelle** *Since 1965*

902 Battelle Boulevard
P.O. Box 999
Richland, WA 99352
1-888-375-PNNL (7665)
www.pnl.gov