#### Continued Evaluation of the Pulse-Echo Ultrasonic Instrument for Critical Velocity Determination during Hanford Tank Waste Transfer Operations - 12518

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# ABSTRACT

The delivery of Hanford double-shell tank waste to the Hanford Tank Waste Treatment and Immobilization Plant (WTP) will be governed by specific Waste Acceptance Criteria that are identified in *ICD 19 - Interface Control Document for Waste Feed*. Waste must be certified as acceptable before it can be delivered to the WTP. The fluid transfer velocity at which solid particulate deposition occurs in waste slurry transport piping (critical velocity) is a key waste parameter that must be accurately characterized to determine if the waste is acceptable for transfer to the WTP. In 2010 Washington River Protection Solutions and the Pacific Northwest National Laboratory began evaluating the ultrasonic PulseEcho instrument to accurately identify critical velocities in a horizontal slurry transport pipeline for slurries containing particles with a mean particle diameter of >50 micrometers. In 2011 the PulseEcho instrument was further evaluated to identify critical velocities for slurries containing fast-settling, high-density particles with a mean particle diameter of <15 micrometers. This two-year evaluation has demonstrated the ability of the ultrasonic PulseEcho instrument to detect the onset of critical velocity for a broad range of physical and rheological slurry properties that are likely encountered during the waste feed transfer operations between the Hanford tank farms and the WTP.

### INTRODUCTION

212,000 m<sup>3</sup> (~56 million gallons) of radioactive and chemical waste are currently stored in 177 underground single- and double-shell tanks on the U.S. Department of Energy Hanford nuclear reservation located in southeastern Washington State. This high-level and low-activity waste is a byproduct of plutonium production efforts that supported America's defense program during World War II and throughout the Cold War. The Hanford underground storage tanks were not designed to store this waste indefinitely; the waste will ultimately be transferred to the Hanford Tank Waste Treatment and Immobilization Plant (WTP) that is being designed, constructed and commissioned to vitrify and transform the waste into solid glass logs for safe, long-term storage.

Washington River Protection Solutions (WRPS), the current U.S. Department of Energy contractor for Hanford tank farm operations, will be responsible for transferring waste from the Hanford tank farms to the WTP via slurry transport piping. WRPS must first certify the waste as acceptable per Waste Acceptance Criteria specified in *ICD 19 - Interface Control Document for Waste Feed* that were developed to ensure waste feeds can be successfully processed by the WTP [1]. Some of the specific Waste Acceptance Criteria pertaining to the waste feed physical and rheological properties are not easily measured with a small sample in an analytical laboratory environment. The critical velocity in slurry transport piping is a key waste acceptance parameter that falls into this category.

Critical velocity is defined as the fluid transfer velocity at which solid particles begin to deposit on the bottom of a straight horizontal pipe section during slurry transport. Critical velocity depends on the physical properties of the solid particles and carrier fluid, as well as the geometry of the slurry transport system [2]. Critical velocity is not a slurry property that can be directly measured. Instead, the symptoms of critical velocity, chiefly the settling and deposition of solid particles in a pipe, are detected and then correlated with the fluid transfer velocity that resulted in that condition – the critical velocity. The settling and deposition of solid particles in slurry transport piping at the critical velocity are undesirable phenomena during waste transfer operations to and within the WTP because they are precursors to pipeline plugging that is potentially irreversible. Therefore, the critical velocity of each batch of Hanford tank waste must be accurately identified in order to determine if the waste feed can be accepted by the WTP.

The current baseline plan of WRPS is to determine the critical velocity of each Hanford tank waste feed batch using a waste certification loop. The waste certification loop will be integrated into the WTP feed delivery systems and will allow real-time determination of the critical velocity as waste is being circulated through the transfer piping and back to the original source tank as illustrated in Fig 1. Once critical velocity and other analytically determined acceptance criteria have been shown to meet the *ICD19* Waste Acceptance Criteria, the waste feed will be certified as acceptable for transfer to the WTP receipt tank for further treatment.





Fig 1. Conceptual illustration of the double shell tank waste certification and transfer process.

The approach of using a waste certification loop to determine critical velocity will require realtime monitoring of the test loop piping for particle settling. A method that is sensitive to incipient settling of solid particles will be required to help pinpoint critical velocity and realistically determine if the waste feed can be safely processed by the WTP per *ICD19*. Identifying critical velocity with high accuracy will also allow WRPS to assign a proper fluid transfer velocity above the critical velocity during waste transfer to the WTP that will ensure the prevention of solid particulate settling and minimize wear on pumping equipment.

In response to the need for a method that accurately detects critical velocity, WRPS and the Pacific Northwest National Laboratory (PNNL) have conducted an extensive two-year evaluation

of an ultrasonic method and system, known as the PulseEcho instrument, for its ability to detect and report the onset of solid particle settling in a full-scale waste certification test loop at PNNL. The PulseEcho instrument was initially tested in 2010 using a range of Newtonian and non-Newtonian simulants that contained low and high concentrations of medium-density glass particles with a median diameter (d50) of >50 micrometers (µm) [3-5]. These tests established that the PulseEcho instrument's method and software can detect the onset of solid particle settling in slurry transport piping, thereby allowing the operator to identify critical velocity. The PulseEcho instrument was further evaluated during a second year of testing in 2011 with the focus on determining the instrument's particle size and concentration detection limits using simulants that contained relatively low concentrations of high-density stainless steel particles with a median diameter of  $<15 \,\mu m$ . The instrument was further challenged in 2011 by evaluating its performance based on ultrasonic measurements conducted through a full schedule 40 (Sch 40) 76.2-mm (3-inch) diameter slurry pipe wall. The cumulative two-year test campaign has demonstrated the ability of the PulseEcho instrument to non-invasively detect particle settling in slurry piping and identify critical velocity for a broad range of physical and rheological slurry properties that are likely encountered during the Hanford waste transfer operations [6].

### TEST PLATFORM, INSTRUMENTS AND SIMULANTS

2011 testing was conducted using the Multiphase Transport Evaluation Loop (MTEL) located in the Process Development Laboratory-East (PDLE) test facility at PNNL. The MTEL was originally designed and built in 2007 to evaluate the pipeline plugging issue during slurry transfer operations at the WTP, and later re-configured to represent a full-scale WRPS waste certification test loop for the 2010-2011 test campaign.

The waste certification test loop was used to test a variety of slurry simulants over a range of flow velocities. The critical velocity for each slurry simulant was accurately determined by incrementally decreasing the flow velocity and monitoring the test loop for particle settling using optical methods. The performance of the ultrasonic PulseEcho instrument was evaluated by comparing the fluid velocities at which the instrument ultrasonically detected particle settling with the optically-determined critical velocities.

### Waste Certification Test Loop

The waste certification test loop depicted in Fig. 2 has a 0.15 m<sup>3</sup> (40 gallon) volume capacity and was configured to maintain precise simulant particle inventory and reduce the duration of testing time required to achieve steady state at particular evaluation velocities.



Fig 2. Computer drawing of the waste certification test loop configuration at PNNL.

The 2011 test section configuration in the test loop is very similar to that used during the 2010 testing. Complete details and sketches of the waste certification test loop configurations and other instruments (i.e., Coriolis mass flow meters, differential pressure gauges, thermocouples) present in the loop for both years are presented in Bontha et al. [3] and Denslow et al. [6]. The 2011 configuration included a new PulseEcho test section (labeled in

Fig 2 as UT Test Sect.) and two transparent pipe sections (labeled in

Fig 2 as Upstream and Downstream Vis. Sect.), which were installed adjacent to each end of the stainless steel PulseEcho test section. The transparent sections facilitated visual observations and optical detection of particle settling with a high-resolution video camera system. It could be safely assumed that particle settling had occurred in the opaque PulseEcho test section at the transducer locations if optical detection methods yielded particle settling had occurred upstream and downstream of the PulseEcho test section. To promote preferential particle settling in the PulseEcho and visual test sections instead of elsewhere in the loop, the inside diameter of the hose associated with the recirculation leg of the loop was reduced to 60.2 mm (2.37 inches) while the primary section of the loop consisted of 76.2 mm diameter (3-inch) Sch 40 stainless steel piping. This resulted in a higher fluid velocity in the smaller diameter section of the loop.

# **Ultrasonic PulseEcho Test Section**

During the first year of testing, the PulseEcho instrument's ultrasonic transducers were installed on the outer diameter (OD) surface underneath the 2010 ultrasonic test section that contained a 0.3 m (12-inch) long flat area with an axial centerline pipe wall thickness of 2.5 mm (approximately half of a Sch 40 pipe wall). The purpose of including the flat area was to provide good surface area contact (coupling) between the test section pipe and the PulseEcho ultrasonic transducers. The purpose of making the wall thickness of the flat area as thin as possible was to provide advantageous ultrasonic conditions to establish the instrument's ability to detect particle settling during this first year.

A new challenge for the PulseEcho instrument during the second year of testing in 2011 was to detect particle settling through a true Sch 40 pipe wall, which required a new test section to be fabricated. The 2011 PulseEcho test section was constructed from a 76.2 mm (3-inch) inner diameter (ID) stainless steel tube with a 9.5 mm (0.375-inch) wall thickness constructed from the same stock tube that was used to fabricate the 2010 test section. The bottom of the PulseEcho test section was modified on its OD surface to contain several 25.4 mm (2-inch) long flat areas that served as installation locations for the ultrasonic transducers. Consistent with 2010 testing, the ID surface of the PulseEcho test section was preserved and not affected by the flat sections on the OD surface. The thinnest points between the OD flat areas and the ID of the pipe were along the axial centerlines of the flat areas. The thickness of each flat area centerline was made to equal an integral number of 1/2 wavelengths for optimal ultrasound transmission. The number of 1/2-wavelengths for each flat area were selected to be cumulatively equal to or greater than a Sch 40 pipe wall (i.e., ≥ 5.5 mm), or, equal to or greater than a Sch 40  $\frac{1}{2}$  pipe wall (i.e.,  $\geq$  2.7mm). The reasons for including flat areas with half- and full-Sch 40 pipe wall thicknesses were 1) to allow for verification of the instrument's measurement repeatability by repeating a pair of 2010 tests with a 2010 glass bead/water simulant and comparing the critical velocities determined in 2011 with those determined in 2010 as measured through the Sch 40 ½ pipe wall thickness; and 2) to evaluate the effect of a full Sch 40 pipe wall on the instrument's performance.

The center points of the ultrasonic transducers were aligned with the flat area centerlines during installation on the PulseEcho test section. The PulseEcho test section was then gravimetrically leveled during installation in the waste certification test loop to ensure perpendicularity between the flat areas and the direction of gravity. This was done to ensure the ultrasonic transducers were centered along the bottom-most points of the test section where particle settling was expected to occur first.





Fig 3. Photograph of two ultrasonic transducers installed on the flat areas located underneath the PulseEcho test section (left) and a computer simulation of the ultrasonic beam radiating from a transducer installed on a Sch 40 stainless steel pipe (right).

### **Reference Measurements for Identifying Critical Velocity**

The critical velocities for 12 different slurry simulants were determined during 2011 testing by detecting incipient particle settling with optical methods. The optically-determined critical velocities served as the reference data against which the ultrasonic PulseEcho data were compared. The performance of the PulseEcho instrument was evaluated by comparing the fluid velocities at which it ultrasonically detected particle settling with the optically-determined critical velocities. This approach is consistent with that reported in Bontha et al. for the first year of the test campaign in 2010 [3,5].

Critical velocity is commonly determined for slurries in transport pipes by optically detecting the settling of solid particles. 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. The solids behavior that was used to define and pinpoint critical velocity during the two-year test campaign was the formation of a stationary layer of solids.

The first year of the test campaign revealed the best technique for 1) observing solids behaviors characteristic of imminent critical velocity and 2) confirming the formation of a stationary layer of solids to identify critical velocity was to place a high-resolution video camera beneath the transparent pipe sections. Therefore, this technique was used again in the second year of the test campaign. The video camera employed was a Point Grey Research model Grasshopper–GRAS20S4M–monochrome (black/white) fit with a 1624 × 1224 pixel sensor, with each pixel representing a 4.4- $\mu$ m × 4.4- $\mu$ m square. The camera operates 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  $\mu$ m to 12800  $\mu$ m over the zoom range of the lens. As noted in Bontha et al. [3-4], this system is capable of detecting particle behavior from particle sizes ranging from 5 to 500  $\mu$ m in diameter.

Differential pressure ( $\Delta P$ ) measurements across a straight length of horizontal pipe under conditions of decreasing slurry flow velocity have been used in past studies to detect particle settling and identify critical velocity. This method relies on the development of an increasing pressure drop (rise in  $\Delta P$ ) across a given pipe length as solid particles accumulate at the bottom of the pipe. The assembly of this  $\Delta P$  vs. flow velocity data forms a "J-curve" where the critical velocity resides near the minimum  $\Delta P$  of the J-curve. However, a distinct drop in pressure that is expected to be characteristic of a pipe with settled solids is not always apparent in the  $\Delta P$  vs. flow velocity J-curve as reported by Poloski et al. [2] despite the presence of settled solids. Additionally, a substantial accumulation of solids could already have taken place by the time the inflection point is discovered. Low particle inventories available in the 2011 simulant slurries were expected to exacerbate the difficulty in detecting particle settling using  $\Delta P$  measurements. Settled particle volumes were not expected to drastically change the cross-section of the pipe and therefore the resultant  $\Delta P$  was expected to be small or nearly undetectable. However,  $\Delta P$ data were recorded throughout testing to validate visual observations and optical data if deemed necessary.

### Ultrasonically Identifying Critical Velocity with the PulseEcho Instrument

The ultrasonic PulseEcho instrument was developed at PNNL in 2007 specifically to address the need to detect the onset of solid particle settling and accumulation at the bottom of vessels and pipes during slurry mixing and transport. The instrument's ultrasonic transducer is noninvasively installed on the underside of a vessel or pipe (on the OD surface) as illustrated in Fig 3. The transducer sends pulses of ultrasonic energy through the vessel or pipe wall at wavelengths ( $\lambda$ ) that interact with the solid particles in the slurry that are on the same order as  $\lambda$ . These interactions result in scattering of the sound field energy, a portion of which is scattered back in the direction of the transducer. The non-coherent back-scattered energy is recorded in the form of amplitude vs. time signals, where time corresponds with depth in the slurry beyond the pipe or vessel wall via Equation 1.

$$d=c^{*}(t/2), \qquad (Equation 1)$$

where d=depth, *c*=speed of sound through the settled particles, and t=time. The user sets the range over which the instrument monitors particle behavior beyond the pipe or vessel wall. The range-gated back-scatter signals are then analyzed by the PulseEcho instrument's variance algorithm to determine if waveforms in the back-scatter signals are modulated, signifying particle motion, or not modulated, signifying no particle motion. This particle mobility information is used to determine if solids near the inside wall of the pipe or vessel are completely mobilized, beginning to settle, or settled/accumulated at the location where the transducer is installed. The PulseEcho instrument performs measurements at a rate up to 100 times per second (100 hertz) to keep pace with rapidly changing conditions during mixing or flow. The backscattered signals, such as that shown in

Fig 4 are analyzed immediately by the variance algorithm, and data on the state of the slurry are presented to the operator via the software user interface. Consequently, with these data, the operator can deduce critical flow velocities, characterize the effectiveness of mixing parameters, and quantify the thickness of a settled layer of solid particles in real time.



Fig 4. Example of a non-coherent ultrasonic backscattered signal.

One of the primary goals of 2011 testing was to evaluate the ability of the PulseEcho system to detect the settling of small, high-density particles (simulating plutonium partilces) through a pipe wall thickness that is equal to or greater than that of a Sch 40 stainless steel pipe wall. Another goal was to test the measurement repeatability of the PulseEcho instrument. Two ultrasonic transducer frequencies were used to accomplish these goals - 10 megahertz (MHz) and 5 MHz. The 10-MHz frequency was selected for its ability to detect the small particles with a median (d50) particle size of ~14 microns. The 5-MHz frequency was selected to 1) validate new 2011 results against those obtained during 2010 testing using the same simulant and a 5-MHz transducer installed on a Sch 40 ½ 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 Sch 40 stainless steel pipe. The 10-MHz and 5-MHz transducers were ordered in diameters of 6.4 mm (0.25 inch) for the same reason the 5-MHz transducer used for 2010 testing was 6.4 mm diameter, which was to maximize measurement accuracy by monitoring the behavior of solids over a small area. The transducers were purchased from NDT Systems, Inc. (Huntington Beach, CA), which is the same company that manufactured the 5-MHz transducer that was evaluated during 2010 testing. The transducers were interfaced with the system of PulseEcho electronics that currently include a waveform generator to provide system timing signals, an ultrasonic pulser/receiver unit to transmit and receive ultrasonic signals, and a high-speed analog-to-digital (A/D) card to convert analog ultrasonic signals to digital signals before sending data to the laptop computer for data analysis and reporting. The digital oscilloscope is used for continuous independent monitoring. A photograph of the hardware that comprises the PulseEcho electronics is shown in Fig 5. Additional discussion on wave-particle interactions and measurement requirements is provided in the Discussion section.





### Simulants

The slurry simulants used in the 2011 testing consisted of two primary components: solid particles that were the settling particles of interest and a carrier fluid. Two different types of settling particles were used for the continued evaluation of the PulseEcho instrument's performance: glass particles with a broad particle size distribution (PSD) and small, high-density stainless steel particles.

The broad PSD glass particle simulant was used during 2010 testing and selected for repeat testing in 2011 to 1) verify the repeatability of the PulseEcho instrument's measurements by comparing the critical velocities determined in 2011 with those determined in 2010 as measured through the Sch 40 ½ pipe wall thickness and 2) evaluate the effect of the full Sch 40 pipe wall on the instrument's ability to detect the onset of particle settling with two different ultrasonic transducers. The broad PSD of this simulant was achieved by preparing a mixture of glass particles of different sizes. The broad PSD formulation and the property and supplier information for the glass particle constituents are provided in Table I.

One of the primary goals of 2011 testing was to determine the sensitivity limits of the PulseEcho instrument in terms of particle size and concentration. Therefore, the stainless steel particles were the settling solids of primary interest during 2011 testing and were selected to evaluate the PulseEcho instrument's ability to detect the settling of these small, fast-settling, high-density particles in both simple and complex carrier fluids. This particle simulant is the same as that used by Poloski et al. during the WTP M1—"Plugging in Process Piping" issue resolution [2]. The stainless steel particle simulant had a density of ~8000 kg/m<sup>3</sup> and a broad particle size distribution with a significant portion of its particles falling in the range of 10 to 30  $\mu$ m. The property and supplier information for the stainless steel particles is provided in Table I.

Table I. Formulation for the Broad PSD Simulant and Property and Supplier Information for the Broad PSD Constituent Glass Particles, the Stainless Steel Simulant Particles and the Particles Comprising the Carrier Fluids.

Formulation for the Broad PSD Simulant						
Composition	Component (mass%)	Component Particle (mass%) Material		Particle Size (volume) d(50), μm <sup>a</sup>		
SPHERIGLASS <sup>®</sup> 5000	7					
SPHERIGLASS <sup>®</sup> 3000	14					
BALLOTINI Mil #13	29	Soda Lime	2500	03.8		
BALLOTINI Mil #10	29	Glass	2000	33.0		
BALLOTINI Mil #6	14					
BALLOTINI Mil #4	7					
	Broad PSD Gla	ass Particle Cons	tituents			
Constituent	Supplier/ Manufacturer	Product ID	Particle Material Density (kg/m³) <sup>ª</sup>	Particle Size (d50), μm <sup>ª</sup>		
SPHERIGLASS® 5000	Potters Industries	A Glass, 5000	2500	7.1		
SPHERIGLASS® 3000	Potters	A Glass, 3000	2500	34.0		

	Industries							
BALLOTINI Mil #13	Potters Industries	MIL-PRF- 9954D#13	2500	57.7				
BALLOTINI Mil #10	Potters Industries	MIL-PRF- 9954D#10	2500	114.9				
BALLOTINI Mil #6	Potters Industries	MIL-PRF- 9954D#6	2500	190.5				
BALLOTINI Mil #4 sieved <500 μm	Potters Industries	MIL-PRF- 9954D#4	2500	502.8				
Stainless Steel Simulant Particles								
Simulant Particle	Supplier/ Manufacturer	Product ID	Particle Material Density (kg/m³) <sup>a</sup>	Particle Size (d50), μm <sup>a</sup>				
Stainless Steel	Ametek	P316L	7950	13.9				
	Carrie	er Fluid Particles						
Carrier Fluid Particle	Supplier/ Manufacturer	Product ID	Particle Material Density (kg/m³) <sup>a</sup>	Particle Size (d50), μm <sup>ª</sup>				
Iron Oxide	Prince Minerals	3752 Red Iron Oxide	5200	2.0				
Gibbsite	Almatis	C-333	2500	7.9				
Kaolin	Feldspar Corporation	EPK Kaolin	2650	6.3 <sup>b</sup>				

<sup>a</sup> Material density of the particles presented are the nominal values and the d(50) particle size is based on the volume fraction.

<sup>b</sup> The kaolin PSD was measured using a well-hydrated kaolin slurry.

The carrier fluids used in 2011 were either water or emulsions of water and fine, non-settling mineral particles that were prepared to simulate more complex Newtonian and non-Newtonian waste feeds. Two non-Newtonian carrier fluids were prepared by mixing fine kaolin or iron oxide particles with water. The kaolin particles were selected for carrier fluid preparation to be consistent with previous 2010 tests. The high-density iron oxide particles were selected because iron oxide is known to be present in Hanford double-shell tank waste. Gibbsite particles were also selected for carrier fluid preparation because gibbsite is also a component

found in tank waste. The gibbsite particles had a PSD comparable to the kaolin particles, but, unlike kaolin, gibbsite does not yield a slurry with appreciable rheological properties at the concentrations used and consequently was considered a Newtonian fluid. The specifications of the carrier fluid particles along with the supplier/manufacturer details are presented in Table I.

A summary of the twelve 2011 waste feed slurry simulants, consisting of the previously described settling particles and carrier fluids, is provided in the test matrix in Table II. This test matrix was designed to evaluate the detection limits of the PulseEcho instrument in terms of settling particle size and concentration in water and more complex carrier fluids. The broad PSD glass particles were mixed with water only at two different particle concentrations. These two simulants were used to perform PulseEcho measurement repeatability testing and evaluate the effect of a full Sch 40 pipe wall on the instrument's performance. The stainless steel particles were mixed with water and with the more complex Newtonian and non-Newtonian carrier fluids to 1) evaluate the PulseEcho instrument's ability to detect the settling of the small, high-density stainless steel particles through a full Sch 40 pipe wall and 2) to evaluate its ability to detect settling of the stainless steel particles amid high concentrations of fine, non-settling carrier fluid particles.

Table II. Test Matrix comprised of 12 Slurry Simulants for which Critical Velocity was determined during 2011 Testing.

Test ID Number	Particle Simulant	Carrier Fluid	Particle Simulant Concentration	Test Purpose
1 (repeat test)	Glass, Broad PSD	Water	5 mass%	Evaluate repeatability of measurements performed
2 (repeat test)	Glass, Broad PSD	Water	20 mass%	through the ½ pipe wall. Test the ability to detect backscatter through the full pipe wall.
3	Stainless Steel	Water	2 mass%	Test the ability to detect small, high-density particles through a full pipe wall. Test the ability to detect settling of a low concentration of small, high-density particles through a full pipe wall.
4	Stainless Steel	Kaolin slurry (20 mass% kaolin)	1 mass%	Evaluate the lower concentration detection limit for small, high-density particles
5	Stainless Steel	Kaolin slurry (20 mass% kaolin)	2 mass%	fine, non-settling kaolin particles as measured through a full pipe wall.

6	Stainless Steel	Kaolin slurry (20 mass% kaolin)	4 mass%	
7	None	Kaolin slurry (20 mass% kaolin)	0 mass%	Test to determine if kaolin particles contribute to backscatter.
8	None	Gibbsite slurry (15 mass%)	0 mass%	Test to determine if gibbsite particles contribute to backscatter.
9	Stainless Steel	Gibbsite slurry (15 mass%)	1 mass%	Evaluate the lower concentration detection limit for small, high-density particles amid a high concentration of fine, non-settling gibbsite particles as measured through a full pipe wall.
10	Stainless Steel	Iron oxide slurry (15 mass%)	1 mass%	Evaluate the lower concentration detection limit for
11	Stainless Steel	Iron oxide slurry (15 mass%)	2 mass%	small, high-density particles amid a high concentration of fine, non-settling iron oxide
12	Stainless Steel	Iron oxide slurry (15 mass%)	4 mass%	particles as measured through a full pipe wall.

Slurry properties based on relative particle concentration and carrier fluid viscosity and yield stress are provided in Table III. The yield stress and carrier fluid viscosity were not controlled during 2011 testing; the carrier fluid particles were used simply as background particles. However, the rheological properties were measured along with the volume and mass fraction of the settling solids of interest, PSD and bulk density. For simplicity, these data are provided in separate tables for Newtonian and non-Newtonian slurry simulants in Table IV and Table V. Additional details on sample preparation and analysis for determining particle size, rheology and mass balance are provided in Denslow et al. [6].

Table III. Relative Concentration, Viscosity and Yield Stress for each Slurry Simulant.

Test ID Number	Slurry Properties (Acronym)	Solids Concentration <sup>a</sup>	Carrier Fluid Viscosity <sup>b</sup>	Carrier Fluid Yield Stress $^{\circ}$
1	LLL	L	L	L
2	HLL	Н	L	L
3	LLL	L	L	L
4	LMM	L	М	М

5	LMM	L	М	М
6	LMM	L	М	М
7	N/A	N/A	N/A	N/A
8	N/A	N/A	N/A	N/A
9	LLL	L	L	L
10	LLM	L	L	М
11	LLM	L	L	М
12	LLM	L	L	М

<sup>a</sup> For solids concentration, the low (L), mid (M) and high (H) concentrations correspond with ≤ 5 mass%, 10 mass% and 20 mass%, respectively. These values represent the concentrations of the glass or stainless steel particles only.

<sup>b</sup> For viscosity, the low (L) and high (H) values are 1 and 10 mPa·s, respectively. The viscosities of the non-Newtonian carrier fluids were driven by the kaolin or iron oxide concentrations that were necessary to achieve the target carrier fluid solids concentrations. This resulted in a mid (M) viscosity of ~4 mPa·s.

<sup>c</sup> For yield strength, the low (L), mid (M) and high (H) designations are consistent with those reported by Bontha et al. [3] and correspond with 0, 3 and 6 Pa, respectively. The yield strengths were driven by the carrier fluid solids concentrations.

N/A = not applicable.

Table IV. Properties of Newtonian Simulant Slurries.

Test Number	1	2	3	8	9			
Test Condition Acronym	LLL	HLL	LLL	N/A	LLL			
Volume Fraction (vol%)								
Simulant Particles (Total)	2.1%	9.1%	0.3%	0.0%	0.14%			
Carrier Fluid Particle	0.0%	0.0%	0.0%	6.7%	6.7%			
(None or Gibbsite)								
Water	97.9%	90.9%	99.7%	100.0%	99.9%			
Mass Fraction (mass%)								
Simulant Particles (Total)	5.0%	20.0%	2.0%	0.0%	1.0%			
Carrier Fluid Particle (None or Gibbsite)	0.0%	0.0%	0.0%	15.2%	15.1%			
Water	95.0%	80.0%	98.0%	100.0%	99.0%			
Component Density (kg/m <sup>3</sup> )								
Simulant Particles	2500	2500	7950	N/A	7950			
Carrier Fluid Particle (None or Gibbsite)	N/A	N/A	N/A	2500	2500			
Water or Gibbsite/Water Emulsion	1000	1000	1000	1000	1000			
Bulk	1031	1137	1018	1100	1110			
Particle Size Distribution (µm)								

d <sub>5</sub>	7.4	7.4	6.1	0.7	0.7			
d <sub>10</sub>	26.1	26.1	7.2	1.2	1.2			
d <sub>20</sub>	45.9	45.9	9.0	2.3	2.3			
d <sub>30</sub>	60.8	60.8	10.6	3.8	3.8			
d <sub>40</sub>	76.2	76.2	12.2	5.7	5.8			
d <sub>50</sub>	93.8	93.8	13.9	7.9	8.1			
d <sub>60</sub>	115.1	115.1	15.9	10.5	10.8			
d <sub>70</sub>	143.6	143.6	18.3	13.6	14.0			
d <sub>80</sub>	191.2	191.2	21.5	17.8	18.3			
d <sub>90</sub>	349.7	349.7	26.7	24.4	25.2			
d <sub>95</sub>	538.6	538.6	31.4	30.2	31.4			
d <sub>99</sub>	807.6	807.6	40.7	40.2	42.5			
Carrier Fluid Rheology: Flow Curve (0-600 s <sup>-1</sup> ) down								
Newtonian Viscosity <sup>a</sup> , mPa∙s	1	1	1	1	1			
r <sup>2</sup>	N/A	N/A	N/A	N/A	N/A			

<sup>a</sup> Newtonian viscosity of water is 1.

N/A = not applicable.

Table V. Properties of non-Newtonian Slurries.

Test Number	4	5	6	7	10	11	12		
Test Condition Acronym	LMM	LMM	LMM	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%		
Carrier Fluid Particle (Kaolin or Iron Oxide)	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%		
Carrier Fluid Particle (Kaolin or Iron Oxide)	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 (kg/m³)									
Simulant Particles	7950	7950	7950	0	7950	7950	7950		
Carrier Fluid Particle (Kaolin or Iron Oxide)	2650	2650	2650	2650	5200	5200	5200		

Water	1000	1000	1000	1000	1000	1000	1000		
Bulk	1152	1162	1183	1142	1149	1159	1180		
Particle Size Distribution (µm)									
d <sub>5</sub>	1.2	1.2	1.3	1.3	0.5	0.5	0.5		
d <sub>10</sub>	1.7	1.7	1.8	1.8	0.6	0.6	0.6		
d <sub>20</sub>	2.7	2.7	2.9	2.7	0.9	0.9	0.9		
d <sub>30</sub>	3.7	3.8	4.0	3.7	1.1	1.2	1.2		
d <sub>40</sub>	4.9	5.0	5.3	4.8	1.5	1.5	1.6		
d <sub>50</sub>	6.4	6.6	7.1	6.3	1.9	1.9	2.0		
d <sub>60</sub>	8.5	8.7	9.3	8.2	2.4	2.4	2.6		
d <sub>70</sub>	11.6	11.6	12.3	11.0	3.2	3.1	3.4		
d <sub>80</sub>	16.4	16.1	16.8	15.4	4.3	4.2	4.8		
d <sub>90</sub>	25.8	24.3	24.9	23.7	6.4	6.3	7.6		
d <sub>95</sub>	36.3	32.6	33.3	32.1	8.5	8.3	10.8		
d <sub>99</sub>	61.2	48.9	50.7	49.5	12.6	12.1	17.5		
E	Bingham Flo	ow Curve	(250-800	s⁻¹) dow	n				
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 <sup>2</sup>	0.999	0.9994	0.9992	0.9990	0.9875	0.9875	0.9953		
Casson Flow Curve (250-800 s <sup>-1</sup> ) 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		
<b>r</b> <sup>2</sup>	1.000	0.9994	0.9993	0.9997	0.9928	0.9928	0.9996		

### **Test Procedure**

The waste certification test loop was empty and dry at the start of each test. The loop was subsequently loaded with prepared simulant or pre-weighed simulant ingredients (stainless steel or glass particles and a carrier fluid) via the hopper at the top of the loop, as shown in

Fig 2, using one of three methods:

- 1. Mixing small amounts of particles with small volumes of carrier fluid and incrementally loading the loop. The mixtures were flushed into the loop by proper manipulation of manual valves and then residual material was rinsed into the loop using the carrier fluid.
- 2. Loading the dry simulant directly and following with the carrier fluid.

3. Preparing the simulant (particles and carrier fluid) in a separate, intermediate vessel (nominally 5 gallon batches) and then pumping the mixture into the loop.

A combination of these methods was used, depending on the carrier fluid and the amount of particles (weight percent) needed to meet the test requirements. In all cases, once the simulant was loaded, some fraction of the system volume remained empty. This fraction was filled by slowly adding carrier fluid directly into the loop while it was open to the atmosphere. The slurry pump was simultaneously operated to mobilize the particles and degas the fluid. Once air had been completely expelled from the system, the test loop was closed and brought to 80 psig to eliminate the formation of micro-bubbles and improve Coriolis flow meter performance.

Each simulant was loaded into the test loop the day prior to performing tests to determine critical velocity. After loading the non-Newtonian simulants, flow velocity was set to 8 ft/s and the throttle valve adjusted until the pump was operating at 60 Hz. These slurry simulants were circulated in the test loop for approximately 2 hours before a 300-500 mL sample was taken to measure the rheology of the slurry.

Prior to the beginning of each test to collect ultrasonic data, scoping tests were performed to bound the range containing critical velocity. This was accomplished by setting the flow velocity to 2.4 m/s (8 ft/s) and decreasing velocity in 0.3 m/s (1 ft/s) increments, allowing a steady state characterized by consistent mass flow and differential pressure measurements to be established at each setting. Flow velocity was reduced in this manner until a stationary bed of particles was observed in the visual test sections. This flow velocity was noted, but no ultrasonic data were collected. At the conclusion of the scoping tests flow velocity was increased again to 2.4 m/s to re-suspend the particles and mix the slurry. Flow velocity was then reduced to a value approximately 0.3 m/s above the point at which a settled bed of particles had been observed during the scoping tests. After a sufficient time period at steady state (minimum of 15 minutes), data were collected with the PulseEcho instrument and the highresolution camera. Ultrasonic data were collected at each flow velocity using a 10 MHz transducer at the full wall location, a 5 MHz transducer at the full wall location and a 5 MHz transducer at the ½ wall location. Data were collected over a period of 2-3 minutes at a measurement rate of 20 hertz for each transducer frequency. Flow velocity was decreased in increments of 0.03-0.06 m/s (0.1-0.2 ft/s) and data collected at each increment following steady state until the PulseEcho instrument detected a consistently settled bed of particles. The test loop chiller operated over the duration of testing to maintain a fluid temperature of 20-25°C.

Flow velocity was increased again to 2.4 m/s at the conclusion of each test to validate pressure data. Post-test rheology samples were collected for slurries prepared with the kaolin/water carrier fluids after shearing the slurry for approximately 1 hour in the test loop.

# RESULTS

The goals of 2011 testing included:

1. Verifying the PulseEcho instrument's measurement repeatability by repeating a pair of 2010 tests with the broad PSD glass particle simulants and comparing the critical

velocities determined in 2011 with those determined in 2010 as measured by the 5-MHz transducer through the Sch 40  $\frac{1}{2}$  pipe wall.

- 2. Evaluating the effect of the Sch 40 full pipe wall on the ability of the 5-MHz transducer to detect settling by comparing the critical velocities determined through the Sch 40 full pipe wall with those obtained through the Sch 40 ½ pipe wall for the broad PSD glass particle simulants.
- 3. Evaluating the particle size and concentration detection limits for the stainless steel (SS) particle simulant in Newtonian and non-Newtonian carrier fluids as measured through a Sch 40 full pipe wall thickness by the 10-MHz transducer.

A tabulated summary of the optically-determined reference critical velocities ( $V_{critical}$ ) and the ultrasonically-determined critical velocities through a Sch 40 full pipe wall using the 10-MHz transducer and through full Sch 40 and a Sch 40 ½ pipe walls using the 5-MHz transducers are provided in Table VI. Additional details on data analysis are provided in Denslow et al. [6].

Test ID Number	Simulant Slurry Description	V <sub>critical</sub> <sup>b, d</sup> Reference m/s [ft/s]	V <sub>critical</sub> <sup>c</sup> 10 MHz Full Wall m/s [ft/s]	V <sub>critical</sub> <sup>c</sup> 5 MHz Full Wall m/s [ft/s]	V <sub>critical</sub> <sup>c,d</sup> 5 MHz ½ Wall m/s [ft/s]
1	5 mass% Broad PSD in	0.98 (1.0)	1.0	1.0	1.0 (1.0)
•	water	[3.2 (3.3)]	[3.3]	[3.3]	[3.3 (3.3)]
2	20 mass% Broad PSD	1.2 (1.2)	1.2	1.2	1.2 (1.2)
2	in water	[4.0 (4.0)]	[3.9]	[4.0]	[4.0 (4.1)]
3	2 mass% SS	0.73	0.67	0.76	Not
5	in water	[2.4]	[2.2]	[2.5]	acquired
4	1 mass% SS	1.1	19	21	19
	in kaolin emulsion	[3.6]	10	2	10
5	2 mass% SS	1.1	19	19	19
5	in kaolin emulsion	[3.6]	10	2	10
6	4 mass% SS	1.1	1.1	19	19
0	in kaolin emulsion	[3.7]	[3.6]	10	10
7	Kaolin emulsion	N/A	N/A	N/A	N/A
8	Gibbsite emulsion	N/A	N/A	N/A	N/A
0	1 mass% SS	0.73	0.64	19	10
9	in gibbsite emulsion	[2.4]	[2.1]	15	15
10	1 mass% SS	0.79-0.82 <sup>a</sup>	IS: 0.79	10	10
10	in iron oxide emulsion	[2.6-2.7]	[2.6]	15	15
11	2 mass% SS	0.82	IS: 0.82	19	19
	in iron oxide emulsion	[2.7]	[2.7]	10	13
12	4 mass% SS	0.88	0.88	IS	IS

Table VI. Summary of Optically- and Ultrasonically-Determined Critical Velocity.

in iron oxide emulsion	[2.9]	[2.9]	

<sup>a</sup> Range reported due to uncertainty of settling in downstream Visualization Section (VS).

<sup>b</sup> Defined as a stationary bed of settled solids.

<sup>c</sup> IS: Insufficient Scatter at high flow velocities.

<sup>d</sup> Velocity in parentheses () indicates 2010 data.

N/A = not applicable, no settling solids to detect.

An ultrasonic frequency of 5 MHz is appropriate for the detection of particles >30 microns, as reported by Bontha, et al. [3]. However, to provide data to support this, data were collected using the 5-MHz transducers at the half- and full-Sch 40 pipe wall locations during tests that involved stainless steel particles with a d(50) of ~14  $\mu$ m. As shown in Table VI, there was insufficient scattering (IS) from the stainless steel particles at 5 MHz due to the small particle size-to-wavelength ratios.

Test 1 and Test 2 demonstrated the PulseEcho instrument has very good measurement repeatability. This is evidenced by the agreement between critical velocities determined in 2010 (provided in parenthesis) and the critical velocities determined in 2011, as measured by the 5 MHz transducers through the Sch 40 ½ pipe wall. Test 1 and Test 2 also demonstrated the full Sch 40 pipe wall does not have a negative effect on the instrument's ability to detect particle settling and determined critical velocity. This is evidenced by the agreement between critical velocities determined by the 5 MHz and 10 MHz transducers through the full Sch 40 pipe walls and those determined by the 5 MHz and 10 MHz transducers through the full Sch 40 pipe walls and those determined by the 5 MHz transducer through the Sch 40 ½ pipe wall. The critical velocities determined by the pulseEcho system using the 5 MHz and 10 MHz transducers were in very good agreement with the optically-determined critical velocities in Test 1 and Test 2 (i.e. within 0.03 m/s or 0.1 ft/s).

Test 3 established the PulseEcho instrument's ability to detect sufficient scattering from the small, high-density stainless steel particles in water using the 10 MHz transducer and to detect the settling of these particles through a Sch 40 full pipe wall. This test provided justification for evaluating the PulseEcho instrument using the more complex Newtonian and non-Newtonian carrier fluids and stainless steel particles. The critical velocities determined by the 10-MHz and 5-MHz transducers through the Sch 40 full pipe walls are in relatively good agreement with the optically-determined critical velocity (i.e. within 0.06 m/s or 0.2 ft/s). A possible explanation for the differences in critical velocities was a settling gradient in the PulseEcho test section. Settling was optically detected in the visual test section upstream of the PulseEcho test section before it was observed in the downstream visual test section. At the reported reference critical velocity, migrating dune structures had formed in the upstream section. However, in the downstream section, a band of particles approximately 6 mm wide comprised of mostly moving particles with some stationary particles present in the band was observed. Inconsistent particle settling inside the PulseEcho test section could explain the differences in the critical velocity determinations. The 5-MHz transducer at the Sch 40 ½ pipe wall location was not used to collect data during Test 3 because it was thought to be unnecessary; however, these data were collected for subsequent tests.

Tests 4, 5 and 6 were performed to evaluate the concentration detection limits of the 10-MHz transducer for slurry simulants composed of stainless steel particles and a non-Newtonian

carrier fluid prepared with water and 20% kaolin particles. This test demonstrated at least 4 mass% of the stainless steel particle simulant was required to obtain sufficient scattering from these particles amid the high background of the small, non-settling kaolin particles. A higher concentration of stainless steel particles was required in the kaolin-based carrier fluid than in water because of the higher attenuation (signal extinction) of the ultrasonic energy by the kaolin emulsion. Due to the insufficient concentration of detectable particles in Test 4 and Test 5, ultrasonic measurements were not obtainable. However, the optically-determined and ultrasonically-determined critical velocities for the Test 6 slurry with a 4 mass% particle concentration are in very good agreement (i.e. within 0.03 m/s or 0.1 ft/s).

Test 7 demonstrated a poor scattering contribution from the kaolin particles alone; however, Test 8 demonstrated a low degree of scattering was contributed by the larger fraction of the gibbsite particles. Critical velocities for Test 7 and Test 8 were not determined because these slurries only contained non-settling carrier fluid particles. The scattering contribution from the larger fraction of gibbsite particles created sufficient overall scattering with the 1 mass% stainless steel particles during Test 9, which allowed the 10-MHz transducer to detect the settling of the stainless steel particles and determine critical velocity for this lower concentration. The PulseEcho instrument detected particle settling at a lower flow velocity than the optical methods. Therefore, the critical velocity reported by the PulseEcho system is lower than that reported for the reference, but within 0.09 m/s (0.3 ft/s).

Tests 10, 11 and 12 were performed to evaluate the concentration detection limits of the 10-MHz transducer for slurry simulants composed of stainless steel particles and a non-Newtonian carrier fluid prepared with water and 15% iron oxide particles. This test demonstrated at least 4 mass% of the stainless steel particle simulant was required to obtain sufficient scattering from these particles amid the high background of small, non-settling iron oxide particles. Similar to kaolin, a higher concentration of stainless steel particles was required in the iron oxide carrier fluid than in water because of the higher attenuation of the ultrasonic energy by the iron oxide emulsion. The optically-determined and ultrasonically-determined critical velocities are in very good agreement for the Test 10, Test 11 and Test 12 slurry simulants (i.e. within 0.03 m/s or 0.1 ft/s).

A test was not performed with the iron oxide carrier fluid alone to determine if iron oxide particles contribute to ultrasonic backscatter because almost 100% of the iron oxide particles are below the detectable particle cut-off size of ~14  $\mu$ m and therefore essentially no iron oxide particles would be large enough to scatter the ultrasonic energy for the frequencies used. Additional discussion on particle size detection is provided in the Discussion section.

# DISCUSSION

2011 testing demonstrated the PulseEcho instrument can perform repeatable measurements and detect small, high-density, fast-settling particles in complex Newtonian and non-Newtonian slurries through a full Sch 40 pipe wall with good accuracy (i.e. within 0.03-0.09 m/s). The lower particle concentration detection limit for stainless steel particles in a carrier fluid ranged from 1-4 mass%, depending on the attenuation and scattering contributions from the carrier fluid. Although these are the apparent mass percentages required, the PulseEcho instrument was not sensitive to all the particles in the PSD of the stainless steel particle simulant. The PulseEcho method relies on obtaining back-scattered ultrasonic energy from solid particles in the slurry, which requires the instrument's transducer to operate at ultrasonic frequencies that will result in suitable wavelengths in the slurry that are on the same dimensional order as, and thus sensitive to, the solid particles in the slurry. The ka value that relates particle size to the ultrasonic wavelength can be used as an indicator for scattering strength for wave-particle interactions. In the equation  $ka = \pi^*(d\lambda)$ , d = particle diameter and  $\lambda$  is the energy wavelength in the bulk material, where  $\lambda = c/f$  (f is frequency and c = the longitudinal speed of sound in the slurry under test). Weak Rayleigh scattering occurs for ka values of <<1 (e.g. 0.1) where the particle size is much smaller than the wavelength (i.e.  $d < \lambda$ ) [8]. Intermediate stochastic scattering occurs for ka values of ~1 where the particle size becomes comparable to the wavelength (i.e.  $d \approx \lambda$ ) and strong geometrical scattering occurs for ka values of >1 where the particle size is larger than the wavelength (i.e.  $d > \lambda$ ). An ultrasonic operating frequency is selected that will allow for the highest ka values and be the least vulnerable to the combined effects of scattering and absorption (attenuation or signal extinction) in the slurry. Assuming a value of 1485 m/s for c, which is the speed of sound through water at 20°C, the calculated kavalues for candidate frequencies of 5 MHz, 10 MHz, and 15 MHz interacting with the d(50) (median) stainless steel particle diameter of 13.9 µm are 0.15, 0.29 and 0.44, respectively. Based on these ka values, weak to intermediate scattering was expected from the d(50) stainless steel particle size at all three candidate frequencies. Therefore, most of the scattering strength was expected to be contributed by particles above the 13.9 µm d(50) of the stainless steel simulant, which is only approximately half of the particles in the stainless steel PSD by volume.

With ~14-µm being the lower particle size cutoff for the 10-MHz transducer, the fraction of stainless steel particles that were detectable by the 10-MHz transducer was approximately only half of that which was added to any carrier fluid. Therefore, the lower particle concentration detection limits of the PulseEcho instrument are effectively only half of the prepared concentrations. Summaries of the percentages of detectable particles in each slurry simulant are provided in Table VII and Table VIII.

Test Number	1	2	3	8	9			
Test Condition Acronym	LLL	HLL	LLL	N/A	LLL			
Volume Fraction (vol%)								
Simulant Particles (Total)	2.1%	9.1%	0.3%	0.0%	0.14%			
Observable, 5 MHz <sup>a</sup>	0.13%	0.57%	0.02%	0.0%	0.01%			
Observable, 10 MHz <sup>b</sup>	1.9%	8.5%	0.13%	0.0%	0.07%			
Carrier Fluid Particle	0.0%	0.0%	0.0%	6.7%	6.7%			
(Gibbsite)								
Water	97.9%	90.9%	99.7%	100.0%	99.9%			
Mass Fraction (mass%)								
Simulant Particles (Total)	5.0%	20.0%	2.0%	0.0%	1.0%			

Table VII. Detectable Fraction of Particles in the Newtonian Simulant Slurries.

Observable, 5 MHz <sup>a</sup>	0.31%	1.24%	0.12%	0.0%	0.06%
Observable, 10 MHz <sup>b</sup>	4.6%	18.5%	1.0%	0.0%	0.5%
Carrier Fluid Particle	0.0%	0.0%	0.0%	15 2%	15 1%
(Gibbsite)	0.078	0.078	0.078	13.270	13.170
Water	95.0%	80.0%	98.0%	100.0%	99.0%

<sup>a</sup> Percentage of simulant particles >30  $\mu$ m observable by the 5-MHz transducer.

Percentage of simulant particles >15  $\mu$ m observable by the 10-MHz transducer.

Table VIII. Detectable Fraction of Particles in the non-Newtonian Simulant Slurries.

Test Number	4	5	6	7	10	11	12	
Acronym	LMM	LMM	LMM	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 <sup>(b)</sup>	0.07%	0.14%	0.29%	0.0%	0.07%	0.14%	0.29%	
Carrier Fluid Particle (Kaolin/Fe <sub>2</sub> O <sub>3</sub> )	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 <sup>(a)</sup>	0.06%	0.12%	0.24%	0.0%	0.06%	0.12%	0.25%	
Observable, 10 MHz <sup>(b)</sup>	0.5%	1.0%	1.9%	0.0%	0.5%	1.0%	2.0%	
Carrier Fluid Particle (Kaolin/Fe <sub>2</sub> O <sub>3</sub> )	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%	

<sup>a</sup> Percentage of simulant particles >30 µm observable by the 5-MHz transducer.

<sup>b</sup> Percentage of simulant particles >15 μm observable by the 10-MHz transducer.

The continued evaluation of the PulseEcho instrument during 2011 has determined the instrument's particle size and concentration detection limits in waste feed simulants containing low concentrations of high-density stainless steel particles with a median diameter of <15  $\mu$ m. These evaluations were successfully completed, based on measurements performed through a Sch 40 full pipe wall. The completion of 2011 testing marks the completion of a two-year test campaign. This campaign culminated in a PulseEcho instrument that has the demonstrated ability to non-invasively detect particle settling in slurry piping and identify critical velocity with good accuracy for a range of simulants that encompass the physical and rheological (i.e., Newtonian and non-Newtonian) properties that are likely encountered during Hanford nuclear waste transfer to the WTP.

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