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<p>7. Abstract</p> <p>This document provides a report of results obtained from testing described in test plan WHC-SD-WM-TP-195. Speed of sound and rheology measurements were made in a sample of 101-SY tank waste. Also speed of sound measurements were obtained from a sample of LANL simulant for the tank waste. Test temperatures ranged from approximately 20 °C to 50 °C.</p>		
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REV. 0

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101-SY WASTE SAMPLE SPEED OF SOUND/RHEOLOGY TESTING FOR SONIC PROBE PROGRAM

1.0 EXECUTIVE SUMMARY

The sonic probe concept is to generate acoustic vibrations in the 101-SY tank waste at nominally 100 Hz, with sufficient amplitude to cause the controlled release of hydrogen bubbles trapped in the waste. The sonic probe may provide a potentially cost-effective alternative to large mixer pumps now used for hydrogen mitigation purposes.

Two important parameters needed to determine sonic probe effectiveness and design are the speed of sound and yield stress of the tank waste. Tests to determine these parameters in a 240 mL sample of 101-SY waste (obtained near the tank bottom) were performed, and the results are reported. The following conclusions were drawn from these results.

Qualitatively, the ability of the waste sample to flow significantly increased (from the as-received condition) when it was vibrated. After vibration at intensities well above the expected yield strength, no shear strength could be detected using the HAAKE shear vane test. This result was unexpected since the same device has been used in the past to measure the shear strength of similar 101-SY samples. More testing to investigate the full nature of this effect is recommended.

The waste sample did not segregate as a result of vibration.

The properties measured for the sonified (vibrated) waste sample are as summarized as follows:

The initial Cup and Spindle yield stress averaged 81.6 Pa. Pre-measurement high amplitude test cup vibration (to 20g) did not significantly affect the sonified waste sample's initial yield stress.

High shear rates (to 700 s^{-1}) significantly reduced the sonified waste's yield stress with the effect lasting for at least 300 hours. Lower shear rates (to 60 s^{-1}) also reduced the waste's yield stress; however, the waste fully recovered its initial yield stress after 18 hours.

The measured speed of sound ranged from 87 m/s to 153 m/s, depending on temperature and the vibration condition of the sample.

When testing was initiated, speed of sound data had a higher priority than rheology testing; consequently, the entire waste sample was placed in the test cup and eventually heated (to about 52°C) and vibrated in an effort to make special "tone burst" speed of sound measurements. This testing eliminated the opportunity to make rheology measurements with the waste in the as-received condition.

Qualitatively, it should be noted that the as-received waste was very thick and had to be scooped out of the sample containers; after the heating and vibration process, the waste flowed more easily, and dripped off a sample collecting spatula. This change in properties of the waste sample from the as-received condition is attributed to the low level vibration (to 4g) of the

sample test cup at temperatures to about 52°C, and also the mechanical mixing that resulted from removing the sample from its container and stirring it. At this point the waste sample was described as being in a "sonified" condition (distinguishing it from the as-received condition).

All of the speed of sound and rheology data were obtained with the waste sample in the sonified condition. The data obtained from the waste sample in the sonified condition are important to the sonic probe design, since operation of the probe would probably produce a nearly cylindrical region of sonified waste surrounding the probe. Since the probe's vibrational energy must pass through this region to reach undisturbed waste, the sonified waste properties will determine the probe's effective range.

The speed of sound measured in the 101-SY tank sample ranged from 87 m/s to 153 m/s, depending on temperature and the vibration condition of the sample. Vibration decreased the speed of sound by nearly 40 percent. Increasing the waste sample temperature from 32°C to 52°C reduced the speed of sound in the waste by nearly 30 percent. Allowing the waste to cool and remain undisturbed overnight restored its original speed of sound properties.

One concern that has been expressed about the use of a sonic probe is that it might cause settling or segregation of the waste, perhaps adversely affecting the tank equilibrium conditions. The observed reduction in sound velocity as a result of vibration is considered evidence that the waste sample did not segregate or densify as a result of the vibration process. This conclusion is further supported by the recovery of the speed of sound to previbration conditions after allowing the sample to remain undisturbed overnight.

Shear vane testing had been planned to determine the change in waste yield stress versus vibration amplitude; however, the vibration and mechanical mixing of the waste during speed of sound testing apparently reduced the waste yield stress below the sensitivity of the shear vane apparatus. The more sensitive Cup and Spindle (C&S) apparatus was used to measure the sample yield stress; however, this technique could not be used during sample vibration.

The Cup and Spindle rheology tests made on the waste sample during and after the speed of sound test series resulted in an average measured yield stress of 81.6 Pa. Higher amplitude vibration (to 20g, acoustic peak to peak amplitudes at the hydrophones in excess of 500 Pa) of the waste sample at 52°C did not reduce the sonified sample's initial C&S yield strength significantly.

The high shear rate (to 700 s⁻¹) C&S rheology tests decreased the shear strength of the sonified waste sample, so that the yield strength was significantly reduced for each C&S retest (following a 10 minute delay) of the same sample. Following these high shear rate tests, the waste sample it did not significantly recover its yield strength even after resting 310 hours. Low shear rate (to 60 s⁻¹) C&S testing also reduced the shear strength of the waste sample so that the yield stress decreased by about a factor of 3 when retested 10 minutes later. However, the sample fully recovered its original yield stress after remaining undisturbed for 18 hours.

2.0 INTRODUCTION

One problem faced in the clean-up operation at Hanford is that a number of radioactive waste storage tanks are experiencing a periodic buildup and release of potentially explosive gases. The best known example is Tank 241-SY-101 (commonly referred to as 101-SY) in which hydrogen gas periodically built up within the waste to the point that increased buoyancy caused a "roll-over" event, in which the gas was suddenly released in potentially explosive concentrations (if an ignition source were present). This problem has been mitigated using a "mixer pump" that periodically remixes the waste so that the hydrogen release is controlled, and gas release concentrations are kept well below the ignition threshold.

In September 1993, a modest development project was funded by the Department of Energy (DOE) to investigate a potentially cost-effective alternative to large mixer pumps now used for hydrogen mitigation purposes. This alternate concept, commonly referred to as the "sonic probe", utilizes low frequency sound vibration to mobilize the trapped gas bubbles, allowing a controlled release. The initial program scope directed Westinghouse Hanford Company (WHC) to design, fabricate and test a sonic probe device to be eventually installed in the 101-SY tank for further testing; if successful, the sonic probe design could be used for other waste tanks with similar gas release problems, and as a backup for the 101-SY mixer pump.

The sonic probe concept is to generate acoustic vibrations in the waste at nominally 100 Hz, with sufficient amplitude to cause the controlled release of hydrogen bubbles trapped in the waste. Low frequency sound waves are preferred for this application because of increased attenuation at higher frequencies. Sonic vibration has been used since the 1930's in the concrete construction industry to remove air bubbles trapped in freshly poured concrete.

A preliminary step in the sonic probe program is to obtain data allowing an estimation of the effective range in tank waste for a given probe design. Two important parameters required for this estimate are the speed of sound (SOS) in waste and the waste yield stress. Both of these parameters will affect the design of the sonic probe in terms of physical size and power requirements.

The Tank Waste Speed of Sound/Rheology Test Plan - Sonic Probe Program (WHC-SD-WM-TP-195)¹ describes tests planned for a sample of waste obtained from the 101-SY tank. The test plan describes making measurements of the speed of sound (SOS) in simulants and actual tank waste using a three hydrophone configuration in sample volumes as small as 200 mL. Also described are rheology tests to measure the yield stress (σ_y) of the sample, including σ_y as a function of vibrational amplitude. The results obtained are given in this report.

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3.0 TEST APPARATUS

Speed of sound and rheology measurements were performed on a waste simulant and on an actual waste sample from the 101-SY tank. The rheology testing can be subdivided into "cup and spindle" and "shear vane" tests.

3.1 Speed of Sound Measurements

The volume of 101-SY tank waste available for the sonic probe program was limited to about 240 mL. A speed of sound test system was designed and fabricated to measure the SOS in 200 mL of test fluid, allowing some of the available material to be used in rheology testing. Water and simulant tests were also performed with this apparatus at these volume levels to be consistent with the waste testing.

Figure 1 provides a sketch of a "test cup" which was included as part of the speed of sound test system to allow uniform heating of the 200 mL waste volume to 52°C; a water jacket was sandwiched between the cup's inner and outer walls to "smooth" the waste temperature profile, eliminating hot spots. Heater tape wrapped around the cup's outer wall allowed the waste temperature to be increased. Type K thermocouples were attached inside and outside the cup for temperature control purposes. This cup was attached to a Vibration Test Systems Model 100 (VTS-100) "shaker table", a closed loop control vibrator with a rated peak sine force of 100 lbs. The VTS-100 was used to simulate the action of a sonic probe and produced acoustic vibrations in the test cup sample.

The speed of sound measurement system included three hydrophones inserted in the waste with positions chosen so that no path (including those utilizing parts of the sample container or waste surface) between hydrophones results in a shorter distance through waste than the direct line of sight between the pickup hydrophones. This eliminates the possibility of the waste container conducting sound at a higher speed and "short circuiting" the measurement. One of the hydrophones was pulsed to produce a sound wave in the waste. The hydrophones were acoustically isolated from each other in the test cup lid. The time of flight to the other two hydrophones was measured with a two channel, 12-bit resolution digital oscilloscope capable of 10 MHz sampling rates. A sketch of the relative hydrophone positioning is given in Figure 2.

Since the distances between sound source and the pickup hydrophones are very short (between 5 and 20 mm), the response time of the hydrophones may be significant. The use of two pickup hydrophones allows the response time to be eliminated from the SOS calculation, assuming the hydrophones have similar response times.

Figure 1. Test Cup Sketch Including Water Jacket and VTS-100 Mounting Bracket. All dimensions are given in inches.

Figure 2. Hydrophone Positioning within Test Cup. Three hydrophones are shown in the top view: S is the source hydrophone and P_1 and P_2 are the pickup hydrophones. Dimensions are $D = 70 \text{ mm}$ (2.75 in); $D1 = l = 25.4 \text{ mm}$ (1.0 in); $D2 = 7.6 \text{ mm}$ (0.3 in); $h = b = s = 12.7 \text{ mm}$ (0.5 in).

Test Cup

Top View

Side View

39404009.3

This process is described in more detail in Appendix A. The resulting calculation of sound speed is

$$SOS = (d_1 - d_2) / (\tau_{t1} - \tau_{t2})$$

where $(d_1 - d_2)$ is the effective distance between the two pickup hydrophones, and $(\tau_{t1} - \tau_{t2})$ is the time difference between pickup at the two hydrophones.

A "tone" burst pulse consisting of three sine wave cycles at the drive frequency (to 20 KHz, depending on conditions) was utilized for water and simulant tests; however, insufficient amplitude was generated for pickup in the waste sample. It was necessary to mechanically stimulate the source hydrophone (tapped it with a screwdriver) to achieve sufficient amplitude for detection at the pickup hydrophones. The disadvantage of the mechanical stimulation method is that high frequency components of the resulting signal can be preferentially attenuated, "artificially" delaying the rise above background at the second pickup hydrophone; this can result in a measured speed that is lower than the actual SOS. When a tone burst can be successfully be used, signal pattern recognition assures the correct SOS measurement. Thus, a speed of sound reported for the waste sample represents a minimum speed; the actual SOS may be slightly higher. Tone burst versus mechanical stimulation of the source hydrophone is discussed in more detail in Appendix B.

A diagram of the electrical hookup for the speed of sound test system (setup for tone burst measurements) is given in Figure 3. This system was calibrated by measuring the speed of sound in water and comparing the data with published results; a 7.5 percent agreement or better was obtained, well within the needs for the sonic probe program.

3.2 Tank Waste Rheology Testing

Two types of rheology tests were planned for the waste sample; "cup and spindle" (C&S) and "shear vane". The C&S testing was used to make rheograms allowing comparisons with previous simulant and waste tests. The C&S test is more sensitive than the shear vane and also measures yield stress (tending to give a low value); however, because of the C&S test apparatus configuration, it was not possible to do the vibration dependence study with this technique.

The shear vane can be inserted directly into the vibration test cup. This allows the yield stress to be measured while the test cup is being vibrated. The shear vanes used were custom made in two sizes, the larger size giving greater sensitivity at the cost of disturbing a greater volume inside the test cup, limiting the number of tests performed for a given waste condition. A sketch of the shear vane is given in Figure 4.

Figure 3. Electronics for Performing Speed of Sound (SOS) Measurements

SOS Electronics

39404009.1

Figure 4. Two Sizes of Shear Vanes were Used: for the Small Size, $a = 0.1$ inch, $b = 0.25$ inch; for Large Size, $a = 0.14$ inch, $b = 0.625$ inch.

Shear Vane

The C&S and shear vane tests were both performed using a HAAKE Model RV100 with an M5 head which was installed in B-cell of the High Level Radiochemistry Facility (HLRF) in 325 Building. The C&S tests were performed with an MV-1 sensor (attachment); a shear vane could be installed directly into the M5 head. The difference in test types depended on the attachment and software used to perform the measurement.

The majority of the C&S tests were performed to a maximum shear rate of 700 s^{-1} , with a 5 minute ramp up to the maximum, a 6 second pause, followed by a 5 minute ramp down from the maximum shear rate. Thus, the test described took 10 minutes and 6 seconds to complete. Four C&S tests were performed with a lower maximum shear rate (60 s^{-1}) using the same ramp up/hold/ramp down time frames. Shear vane measurements were made in the same test cup used for SOS testing.

3.3 Calibration/Quality Assurance

Instruments recording data were calibrated in accordance with WHC-CM-4-2 (QR 12.0, Rev. 5) either by WHC Standards Laboratory or as certified by the vendor. An exception was the calibration of the hydrophones which was performed by the operator in compliance with WHC-CM-4-2 (QR 12.2, Rev. 2). Verification of the calibration and documentation of the individual instruments was made by a WHC Quality Assurance representative.

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4.0 TEST PROCEDURE AND RESULTS

A test procedure was written for these tests, and is reproduced in Appendix C. However, a number of the steps in the procedure were altered by the Test Cognizant Engineer (N. S. Cannon) as results were obtained, adjusting to the new information. The actual test steps and results are reported here.

4.1 SOS Calibration in Water

Over the course of testing, a number of calibration runs were made in water to verify the accuracy of the speed of sound system. The individual test sequences are reported in Table 1, including comparisons with literature values² for the SOS in water (given in Table 2). "Literature" values for the SOS at the intermediate temperatures reported in Table 1 were interpolated from the Table 2 values.

All SOS measurements made in water reported in Table 1 were made using an electronic stimulation of the piezoelectric crystals of the source hydrophone. A worst case error of 7.4 % was recorded through the entire series of tests performed in water. This error appeared to be reduced to less than 3.1% when the effective distance (D1-D2) was increased to 20.3 mm (0.800 inches). However, the very last test in water (at the completion of all the hot cell tests) gave an error of 6.1 percent. It is likely that the distance between the probes changed slightly as a result of the many removal/insertion operations of the hydrophone cap on the test cup, especially while obtaining samples for the rheology tests. Thus, conservatively, the worst case error during waste sample testing due to distance uncertainties and hydrophone response times is estimated at ± 7.5 percent.

4.2 LANL Simulant Testing

A newly mixed sample of a simulant for the 101-SY tank waste developed at Los Alamos Nation Laboratory (LANL) was provided to WMC by Steve Agnew. The LANL simulant closely matches the chemical composition of the 101-SY tank waste; a more detailed description of this simulant is given in Table 3.

As received from LANL, the simulant had "settled" in its shipping container with approximately 6 mm of relatively clear liquid on the top. The container was shaken vigorously by hand to mix the simulant, and then 200 mL was poured into the test cup. The hydrophone cap was installed, sealing the container, to prevent moisture loss from the simulant.

An attempt was made to measure the speed of sound in the simulant using a 20 KHz (3 cycle) tone burst. However, no signal was received at the "far" pickup hydrophone, indicating significant attenuation. The sample was allowed to sit at ambient temperature over the weekend; then the 20 KHz tone burst was repeated with the same result. Subsequently, the SOS was successfully measured by lowering the tone burst frequency to 10 KHz (3 cycle), which reduced the effective attenuation.

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**TABLE 1
Measured Speed of Sound in Water**

Date	Temp (°C)	D1-D2 (mm)	SOS Avg. (m/s)	Literature SOS (m/s)	Percent Error	Comments
1-12-94	22.2	9.35	1438	1488	-3.5	Temporary Fixture
2-01-94	21.1	17.53	1425	1485	-4.2	Test Cup - Henceforth
2-02-94	24.4	17.53	1414	1494	-5.7	Tone Burst
2-02-94	24.4	17.53	1453	1494	-2.9	Pulse Signal
2-02-94	51.1	17.53	1465	1544	-5.4	Pulse Signal
2-02-94	51.1	17.53	1437	1544	-7.4	Tone Burst
2-02-94	39.4	17.53	1465	1528	-4.3	Tone Burst
2-02-94	38.9	17.53	1465	1527	-4.2	Tone Burst
2-04-94	25.0	16.76	1571	1496	+5.0	Tone - D1/D2 Hydrophones reversed.
2-04-94	25.0	16.76	1574	1496	+5.2	Pulse - D1/D2 Hydrophones reversed.
2-04-94	25.0	20.32	1543	1496	+3.1	Pulse - Reversed
2-04-94	25.0	20.32	1531	1496	+2.4	Tone - Reversed
2-04-94	25.6	20.32	1520	1497	+1.5	Tone - Reversed: In-hood pre-LANL Simulant Tests
2-11-94	21.1	20.27	1505	1494	+0.7	Tone - Outside Hot Cell in Vibrator Cup.
2-24-94	31.1	20.27	1488	1511	-1.5	Tone - Hot Cell ⁺
3-02-94	32.2	20.27	1501	1513	-0.8	Tone - Hot Cell ⁺
3-21-94	30.0	20.27	1601	1509	+6.1	Tone - Hot Cell ⁺ ; after all SOS testing was completed.

* This test was performed inside the hot cell with the hydrophone cap placed in a metal beaker of water instead of the vibrator cup - the vibrator cup was filled with the 101-SY waste sample.

TABLE 2 Literature ¹ Values for Speed of Sound in Water			
Temp(°C)	Temp(°F)	SOS (m/s)	SOS (ft/s)
10	50	1447.2	4748
20	68	1482.3	4863
30	86	1509.0	4950
40	104	1528.8	5016
50	122	1542.5	5061
60	140	1550.9	5088

TABLE 3 Description of the LANL Simulant (1 liter.)			
Constituent Chemical	Molarity (M)	Mass (Grams)	Comment
NaOH	3.39	136	Sodium Hydroxide.
Na ₂ CO ₃	0.53	56	Sodium Carbonate.
NaNO ₂	3.55	245	Sodium Nitrite.
NaNO ₃	3.73	317	Sodium Nitrate.
NaAlO ₂ · xH ₂ O	2.27	186*	Sodium Aluminate. *Mass excludes hydration.
Na ₄ EDTA	0.12	50	Sodium ETHYLENEDIAMINETETRAACETATE
Na ₃ HEDTA	0.13	50	TRISODIUM HYDROGEN ETHYLENEDIAMINETETRAACETATE, HYDRATE
Density of the mixed LANL simulant was measured at 2.8 g/cm ³ . The bottle label indicated the ph is 13 to 14.			

Speed of sound test results from the unvibrated simulant are summarized in Table 4 for data obtained at three temperatures (approximately 22°C, 37°C and 53°C). The sample was then vibrated in the test cup at 53°C for 42 minutes with the VTS-100 set at an acceleration of 5 gravities (g). Measurements of SOS at 53°C were completed within 9 minutes of turning off the vibrator. The test cup temperature was lowered to 38°C in about an hour, and another series of SOS measurements were made. These vibrated simulant SOS measurements are recorded in Table 5.

After these tests were completed, the simulant was examined visually in the test cup; it had segregated, with a nearly clear layer on the top of the sample, about 5 mm deep. Using a small area stirring device, it was qualitatively noted that the simulant was more difficult to stir with increasing depth. This segregation is believed to be the result of the vibration operation; although the simulant did not appear changed after sitting in the test cup over the weekend, its SOS did change significantly after the 42 minute vibration. The SOS increase observed after vibration is probably due to more conductive (higher speed) layers forming in the simulant after vibration, which provided "short circuit" paths between the pickup hydrophones.

4.3 Hot Cell Testing of the 101-SY Waste Sample

The VTS-100 actuator and test cup were installed in B-Cell of the 325 Building. Water was placed in the test cup, the hydrophone cap installed and an SOS measurement performed to verify that the hydrophone distances remained unchanged during installation.

Waste samples were taken from Tank 241-SY-101 during Window C on May 22-26, 1991, and then stored in the 222-S laboratory hot cells. The lower sludge samples were taken using the "push mode" sampler. A portion of the samples were transported to the 325 Building in the 300 Area for rheology testing by J. M. Tingey. On June 4, 1991, three more samples were taken from the lowest three segments, again using the push mode sampler. Both sets of samples were taken from riser 22a as reported in the Window C core sample report⁵ WHC-SD-WM-DTR-024. Initial testing by Tingey was performed on an undisturbed portion of the May 1991 samples. Subsequently, both sets of samples were homogenized using a mixing blade which is a motorized stirring machine.

The sample used in these sonic probe hot cell experiments was from the June 4 batch segment 22a, and represented the lowest position in the tank for which a sample was obtained. This sample was selected as a conservative representation of the tank sludge content since it is known

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<p style="text-align: center;">TABLE 4 LANL Simulant SOS Test Results - Unvibrated</p>						
Test Temp(°C)	D1 - D2 (mm)	Delay Time (μs)	Velocity (m/s)	Ampl. @ D1 (mV)	Ampl. @ D2 (mV)	A2/A1 Ratio
21.7	20.32	113.1	179.7	32.0	227	7.09
22.2	20.32	109.1	186.3	46.1	233	5.05
22.2	20.32	108.0	188.1	35.1	226	6.44
22.2	20.32	106.7	190.4	35.5	230	6.48
22.2	20.32	107.7	188.6	35.6	225	6.32
22.2	20.32	106.7	190.4	35.8	220	6.15
AVERAGES @ 22.2°C		108.6	187.3			6.26
36.7	20.32	117.3	173.2	40.3	266	6.60
36.7	20.32	117.5	172.9	41.0	262	6.39
36.7	20.32	118.9	170.9	46.6	269	5.77
36.7	20.32	116.9	173.8	43.7	268	6.13
36.7	20.32	115.7	175.6	46.5	270	5.81
36.7	20.32	117.4	173.1	48.8	265	5.43
AVERAGES @ 36.7°C		117.3	173.3			6.02
52.8	20.32	125.5	161.9	38.3	256	6.68
52.8	20.32	125.5	161.9	41.5	247	5.95
52.8	20.32	122.4	166.0	40.3	264	6.55
52.8	20.32	124.0	163.9	40.1	257	6.41
52.8	20.32	122.5	165.9	45.9	253	5.51
52.8	20.32	124.8	162.8	41.0	265	6.02
AVERAGES @ 52.8°C		124.1	163.7			6.19

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TABLE 5 LANL Simulant SOS Test Results - Vibrated						
Test Temp(°C)	D1 - D2 (mm)	Delay Time (μs)	Velocity (m/s)	Ampl. @ D1 (mV)	Ampl. @ D2 (mV)	A2/A1 Ratio
52.8	20.32	15.6	1303	111.6	1168	10.47
52.8	20.32	14.9	1364	117.3	1176	10.03
52.8	20.32	15.3	1328	117.1	1186	10.13
52.8	20.32	14.9	1364	120.0	1189	9.91
52.8	20.32	15.5	1311	121.7	1197	9.84
52.8	20.32	15.0	1355	127.2	1208	9.50
AVERAGES @ 52.8°C		15.2	1338			9.98
38.3	20.32	24.8	819.4	124.6	1250	10.03
38.3	20.32	24.6	826.0	138.4	1289	9.31
38.3	20.32	24.1	843.2	130.3	1289	9.89
38.3	20.32	24.0	846.7	128.0	1293	10.10
38.3	20.32	25.2	806.3	120.9	1279	10.58
38.3	20.32	24.9	816.1	127.4	1287	10.10
AVERAGES @ 38.3°C		24.6	826.3			10.00

from core analysis that the sludge gets stronger toward the bottom of the tank. The serial numbers on the two waste transport containers were J-1453/CS22R1 and J-1454/CS22R2, with each container holding approximately 120 mL of segment 22a.

It should be noted at this point that when the hot cell testing of the waste sample was initiated, speed of sound data had a higher priority than rheology testing; consequently, the entire waste sample was placed into the test cup and eventually heated (to about 52°C) and vibrated in an effort to make the special "tone burst" speed of sound measurements. This testing eliminated the opportunity to make rheology measurements with the waste in the as-received condition.

As mentioned, the waste samples from both containers (about 240 mL) were scooped out and placed in the test cup; the hydrophone cap was installed to prevent moisture loss. It was necessary to scoop the waste from the containers because it would not pour out; the waste had the consistency of very thick mud, with a few drops of liquid on the surface. Shaking the container would deform the waste sample slightly, but it could not be removed from the container without scooping it out with a spatula.

Testing performed included SOS measurements at two temperatures (32 and 52°C) and two vibration conditions (unvibrated and vibrated, to be defined in Section 5). Standard cup and spindle rheology testing was also performed at the two temperatures and vibration conditions. Shear vane rheology was attempted, but yield stresses were too low to be measured. A complete chronological test summary is given in Appendix D.

The outside thermocouple (TC) on the test cup failed shortly after Sample 1 was taken for the C&S rheology testing. The TC failure was noticed when the remaining waste temperature measured with the inside TC was about 54°C; at this time power to the cup was turned off. However thermal inertia carried the waste temperature (at the cup inner wall) to nearly 65°C. This higher temperature level was maintained only a few minutes before it began to decrease. It is believed that the waste properties were not significantly affected since SOS and C&S rheology results (Samples 2-4) remained consistent with prior data.

At the completion of testing, SOS measurements were made in water with the hydrophone cap; results were within 6 percent of the literature values, indicating the system still functioned correctly at the end of testing. The density of the waste sample was then measured at 1.76 g/cm³.

4.3.1 Waste Speed of Sound Results

Attempts to measure the speed of sound in the waste sample using the tone burst technique failed. When the drive amplifier voltage was increased above the levels used in the LANL simulant testing, electrical cross-talk was observed, complicating the search for real acoustic signals at the pickup hydrophones. Even at the higher drive voltages, the waste apparently attenuated the acoustic signal before it could reach the nearest pickup hydrophone.

In order to make SOS measurements, it was necessary to mechanically stimulate the source hydrophone (hit it with a screw driver) in order to generate sufficient amplitude to reach both pickup hydrophones. This technique was successful, and the results obtained at different temperatures and vibration conditions are reported in Table 6. The disadvantage with the mechanical stimulation technique is that the speeds reported in Table 6 represent minimum velocities; actual SOS values may be slightly higher, as is discussed in Appendix B.

4.3.2 Waste Rheology Results

As mentioned previously, the two types of rheology tests planned for the 101-SY tank waste sample were cup and spindle and shear vane tests. C&S rheology samples were removed from the vibrator test cup at various stages of the hot cell test program. Results from the C&S tests are summarized in Table 7. Raw data plots and subsequent computer analysis plots for the C&S testing are given in Appendix E.

Shear vane tests were performed with two different sized vanes. In both cases, the waste shear stress was below the sensitivity of the instrument, and no meaningful data was obtained. For this reason, it was not possible to determine yield stress as a function of vibration amplitude, and this part of the test procedure (see Appendix C) was deleted.

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TABLE 6 SOS Dependence on Temperature and Vibration				
----- UNVIBRATED -----				
Test Group	Temp(°C)	SOS(m/s)	Hours Since Last Vibration	Comments
A	31.1	158.5	70	Test group A contains 31.1 and 51.7°C samples
B	32.2	119.5	19	Test group B contains 32.2 and 50.6°C samples
D	32.2	166.7	20	Single sample in Test Group D.
F	33.3	169.8	18	Single sample in Test Group F.
I	32.6	148.4	172	Test group I contains 32.6 and 51.7°C samples
AVERAGE SOS =		152.6	UNVIBRATED AT NOMINALLY 32°C	
A	51.7	109.4	94	Test group (TG) A contains 31.1 and 51.7°C samples
B	50.6	112.5	21	TG B contains 32.2 and 50.6°C samples
G	52.6	125.3	69	Single sample in TG G.
I	51.7	133.5	192	TG I contains 32.6 and 51.7°C samples
AVERAGE SOS =		120.2	UNVIBRATED AT NOMINALLY 52°C	
----- VIBRATED -----				
E	33.9	112.8	0	Two samples from TG E.
E	33.9	107.9	0	Two samples from TG E.
AVERAGE SOS =		110.4	VIBRATED AT NOMINALLY 34°C	
C	52.2	93.0	0	Single sample in TG C.
H	52.6	93.0	0	Single sample in TG H.
J	52.1	76.2	0	Single sample in TG J.
AVERAGE SOS =		87.4	VIBRATED AT NOMINALLY 52°C	

TABLE 7
Rheology Results

Test Type	Temp(°C)	Waste Condition	Yield Stress (Pa)	Comments
C&S	33.3	Sample 1	82	Mechanically stirred waste prior to taking sample for C&S. Three tests separated by about 10 minutes. Standard Test, see next block.
C&S	33.3	Sample 1	52	
C&S	33.3	Sample 1	15.4	
C&S	33.3	Sample 1	35	22 hours since previous C&S testing. 10 minutes between tests. Shear rate to 700 s^{-1} ; 5 min up, 6 s rest, 5 min down.
C&S	33.3	Sample 1	3.1	
C&S	33.3	Sample 1	1.7	
C&S	51.7	Sample 1	12.3	310 hours since last C&S; little recovery. Standard Test sequence.
C&S	51.7	Sample 1	2.6	
C&S	51.9	Sample 2	62	New C&S sample - vibrated at 52°C , about 2 hrs before sample taken. Standard Test sequence.
C&S	51.9	Sample 2	16.4	
C&S	51.9	Sample 2	16.5	
C&S	51.9	Sample 2	12.8	
C&S	31.7	Sample 3	75.3	New C&S sample: 71 hours since last vibration. Standard Test sequence.
C&S	31.7	Sample 3	9.5	
C&S	31.7	Sample 3	7.3	
C&S	29.5	Sample 4	107	New C&S sample: 95 hours since last vibration. Max. shear rate reduced to 60 s^{-1} ; same timing.
C&S	29.5	Sample 4	30.2	
C&S	30.1	Sample 4	140	Same sample and test sequence as previous 60 s^{-1} run. Rested 18 hours.
C&S	30.1	Sample 4	38.2	

5.0 DISCUSSION

A discussion of both the speed of sound and the rheology test results follows. The waste will be described as being in an "unvibrated" or "vibrated" condition; the definition used for unvibrated is that the waste has been undisturbed for at least 15 hours since it was last stirred or shaken. Testing in vibrated waste was performed within 15 minutes of the completion of the vibration cycle.

Note that the above definitions are different from the "sonified" condition of the waste sample defined earlier. The sonified waste sample did not recover its "as-received" properties even when allowed to rest undisturbed for over 300 hours. In contrast, waste allowed to rest for more than 15 hours (unvibrated) recovered its initial sonified speed of sound.

5.1 Speed of Sound

The speed of sound data obtained from the 101-SY tank waste sample is presented in Table 6. SOS averages for different temperature and vibration conditions are plotted in Figure 5. In all cases, vibration decreased the SOS in the waste; however, the SOS recovered once the waste was allowed to "rest" undisturbed overnight, as can be seen from Appendix D. Also, a decrease in SOS with increasing temperature was noted for the waste in both the unvibrated and the vibrated condition, and also was observed for the LANL simulant in the unvibrated condition.

For unvibrated waste testing, the average of the SOS measurements made at 32°C was 152.6 m/s; the average SOS for a waste temperature of 52°C was lower at 120.2 m/s. For the waste sample in the vibrated condition, the average sound velocities were 110.4 m/s and 87.4 m/s at 34°C and 52°C, respectively. It is noted that the ratio of the average velocities at the two temperatures is 1.27 for unvibrated waste, and 1.26 for vibrated waste. The velocity ratio between unvibrated and vibrated waste at both temperatures is 1.38; for this temperature range, the SOS is reduced more by vibration than by the temperature change.

The SOS measurements between the unvibrated LANL simulant (164 m/s to 187 m/s for the temperature range of 22°C to 53°C) and the 101-SY tank waste sample (120 m/s to 153 m/s between 32°C and 52°C) are relatively consistent. Comparisons between the vibrated simulant and waste are not reasonable because the simulant segregated with vibration, providing higher speed layers for sound transmission.

There is no evidence that the tank waste sample segregated as a result of being vibrated. Indeed, the decreasing SOS observed as a result of vibration is evidence that the waste sample did not segregate. If the waste sample had segregated, then the SOS would probably increase as segregation layers provided alternative higher SOS paths between hydrophones, as was observed in the LANL simulant. Also, segregated waste would probably not "desegregate" after resting overnight, as would be required to explain the reset of the SOS to its original value. Recovery of the SOS is more likely due to some type of weak chemical bond "restructuring" of the waste sample than to a physical remixing of segregated layers.

Figure 5. Speed of Sound Plotted versus Test Temperature, under different Vibration Conditions.

It was possible to measure the speed of sound in the LANL simulant using the tone burst method over a separation distance of about 2 cm, with attenuations measured as high as an order of magnitude. It was not feasible to determine attenuation in the waste from the mechanical stimulation technique results because of pattern recognition problems. Since the tone burst method did not work in the actual tank waste sample, it is probable that the attenuation factor over 2 cm was greater than 10. However, the acoustic coupling between the source hydrophone and the test sample may have been different between the LANL simulant and the tank waste; this possibility prevents drawing a conclusion about attenuation in the waste sample.

A plot of the individual test results for the waste in the unvibrated condition is made in Figure 6. Each data point is labeled according to its test group; often, a pair of tests were performed within 2 hours of each other, one at each temperature condition, and are considered to be in the same test group (as for groups A, B, and I). In each case where a data pair exists, an increase in temperature reduced the SOS.

A plot of SOS versus vibration acceleration levels is given in Figure 7 for waste tested at nominally 52°C; increased vibration amplitudes resulted in a greater difference between the unvibrated and vibrated SOS.

5.2 Cup and Spindle Rheology

Almost half of the C&S rheology presented in Table 7 deals with the first sample (Sample 1) taken from the waste. This sample was retested eight times, in a series of three test sequences. The initial yield stress was measured at 82 Pa; the yield stress decreased significantly with repeat testing after a 10 minute delay between tests. When the sample was allowed to rest overnight, a partial recovery of the yield stress was observed, followed by another more modest recovery when the sample was retested 310 hours later. The yield stress data obtained from Sample 1 is plotted in Figure 8.

Once SOS measurements were completed, rheology Samples 2 and 3 were obtained in a vibrated and unvibrated condition, respectively. A plot of the two test sequences performed on these samples is given in Figure 9. The vibrated sample (Sample 2) had a lower initial yield stress, but the yield stress for this sample decreased less on the 10 minute retest cycles than did the unvibrated sample (Sample 3); however, these differences are within the limits of experimental error, and may not be significant.

A last sample (Sample 4) of the waste was tested at a much lower maximum shear rate of 60 s^{-1} (but still using the same 5 minute ramp up/ramp down sequence). The initial yield stress was higher (107 Pa) than measured in previous testing, probably due to the slower rotational rates used. With the lower maximum shear rate, the sample had fully recovered when the sample was retested after 18 hours.

Figure 6. Individual SOS Test Results from Unvibrated Waste. Data with same labels resulted from tests performed within two hours of each other.

Figure 7. Change in Waste Speed of Sound at 52°C versus Vibrational Amplitude.

Figure 8. Tank Waste Cup and Spindle Rheology Results from Sample 1. Three C&S test sequences were performed on Sample 1, with each test (run) in a given sequence separated by about 10 minutes from the previous test.

Figure 9. Tank Waste Cup and Spindle Rheology Results from Samples 2 and 3 in the Unvibrated and Vibrated Condition, Respectively.

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6.0 CONCLUSIONS

Initially, the as-received waste sample was very thick and had to be scooped out of the sample containers; after being "sonified" by vibration and mixing, the waste flowed relatively easily. This qualitative change in the flow properties of the waste sample is attributed to the relatively low level vibration (4g) of the sample test cup at temperatures of about 52 °C, and also the mechanical mixing that resulted from removing the sample from its container and stirring it.

All of the SOS and rheology data was obtained after the waste sample had been sonified. The data obtained from the waste sample in this condition is important since the acoustic vibrations will need to propagate from the probe through vibrated waste to reach the unvibrated material. The properties of the vibrated waste will determine the effectiveness and range of the sonic probe for a given design configuration.

The following conclusions are made from data obtained from the sonified waste sample.

The speed of sound measured in the 101-SY tank sample ranged from 87 m/s to 153 m/s, depending on temperature and the vibration condition of the sample. Vibration decreased the speed of sound by nearly 40 percent. Increasing the waste sample temperature from 32°C to 52°C reduced the speed of sound in the waste by nearly 30 percent. These values are consistent with the speed of sound measured in the LANL simulant (which averaged 175 m/s) using the tone burst technique.

The reduction in sound velocity as a result of vibration is considered strong evidence that this waste sample did not segregate or densify as a result of the vibration process. The recovery of the SOS after allowing the sample to remain undisturbed overnight is considered further evidence for this conclusion.

Cup and Spindle rheology tests made on the waste sample during and after the speed of sound measurements resulted in an average yield stress of 81.6 Pa. Higher amplitude vibration (to 20g, acoustic peak to peak hydrophone amplitudes in excess of 500 Pa) of the waste sample at 52°C did not reduce the sample initial C&S yield strength significantly.

The high shear rate (to 700 s⁻¹) C&S rheology tests decreased the shear strength of the waste sample, so that the yield strength was significantly reduced for each C&S retest (following a 10 minute delay) of the same sample. At these shear rates, the waste sample did not significantly recover its yield strength even after resting 310 hours. Low shear rate (to 60 s⁻¹) C&S rheology also reduced the shear strength of the waste sample so that the yield stress decreased by about a factor of 3 when retested 10 minutes later. However, the sample fully recovered its original yield stress after remaining undisturbed for 18 hours.

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7.0 ACKNOWLEDGEMENTS

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The authors also appreciate the programmatic support of J. R. Frederickson of WHC.

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APPENDIX A
SMALL VOLUME SPEED OF SOUND MEASUREMENT

APPENDIX A

The purpose of the Speed of Sound Test System (SOSTS) is to measure the sound velocity in a sample of 101-SY tank waste. The design of the SOSTS will be based on testing a 200 mL volume of waste, although 240 mL may actually be available.

The SOSTS system includes three hydrophones inserted in the waste with positions chosen so that no path (including those utilizing parts of the sample container) between hydrophones results in a shorter distance through waste than the direct line of sight between the pickup hydrophones. This eliminates the possibility of the waste container conducting sound at a higher velocity and "short circuiting" the measurement. One of the hydrophones will be pulsed to produce a sound wave in the waste. The time of flight (τ_f) to the other two hydrophones will be measured with a digital oscilloscope capable of 10 MHz sampling rates. Several types of "pulses" may be used to drive the source hydrophone; but a "tone" burst (three sine wave cycles at 20 KHz) will be the primary type of pulse utilized. See Figure 2 for a sketch of this system.

Since the distances between sound source and the pickup hydrophones are very short (between 0.2 and 0.8 inches), the response time (τ_r) of the hydrophones may be significant. The use of two pickup hydrophones allows the response time to be eliminated from the velocity calculation as follows.

The total time (τ_t) measured by the scope between triggering and reception at a hydrophone will be the sum of τ_f and τ_r , giving

$$\tau_{t1} = \tau_{f1} + \tau_r$$

for the pickup hydrophone #1, separated from the source by a distance d_1 . For hydrophone #2, separated by distance d_2 ,

$$\tau_{t2} = \tau_{f2} + \tau_r$$

Subtracting one equation from the other eliminates the response time (which includes both source and pickup response times; it is assumed that both pickup hydrophone response times are nearly equal). Since the time of flight is the distance divided by the sound velocity (V), the sound velocity can be determined as

$$V = (d_1 - d_2) / (\tau_{t1} - \tau_{t2})$$

The system will be calibrated by measuring the speed of sound in water, and comparing the data with published results. Errors as high as 30% are acceptable for the sonic probe work, but the actual error is expected to be significantly lower.

APPENDIX B

ELECTRONIC VERSUS MECHANICAL EXCITATION OF SOURCE HYDROPHONE

APPENDIX B

Two general types of excitation were used to produce acoustic energy at a source (hydrophone) for the speed of sound measurements; electronic and mechanical. For electronic excitation, an electrical signal was sent to the source hydrophone. This signal was either a tone burst (3 complete sine waves, generally at 15 to 20 KHz), or a single step function pulse. For mechanical excitation, a screw driver was tapped on the steel guide shaft holding the source hydrophone; this shaft is acoustically isolated from the rest of the test cup top.

Electronic excitation has several advantages over mechanically generated signals, as will be discussed. However, when testing 101-SY waste, electrical excitation was not able to produce sufficient acoustic amplitude at the source hydrophone to allow signal detection at the other two pickup hydrophones. Mechanical excitation was necessary to generate sufficient amplitude for signal detection and subsequent determination of speed of sound in the waste.

B.1 Pattern Recognition

Tone bursts were used to enable pattern recognition at the two pickup hydrophones. Pattern recognition can improve the sound velocity determination where the transmission medium has significant attenuation properties. Attenuation will preferentially reduce the amplitude of higher frequency components of a signal over a fixed distance. Both the electronic step pulse and the mechanically generated acoustic signal consist of a fairly wide distribution of frequency components. Without the pattern recognition provided by the single frequency tone burst, it is possible to miss an attenuated high frequency component of the acoustic signal at the furthest hydrophone that was observed at the near pickup hydrophone. The observer may record a time difference between pickup hydrophones that is too long, yielding a speed of sound lower than the actual value. For this reason, speed of sounds recorded from the 101-SY tank waste using the mechanical excitation method are noted to be MINIMUM speeds; the actual SOS may be higher. Although attempted, pattern recognition based on the mechanically generated signals in the tank waste was not feasible.

B.2 Signal Generation Location and Alternate Path Errors

Electronic excitation of the source hydrophone also has the advantage of generating the acoustic wave at the right location - the piezoelectric active position of the hydrophone. For the SOSTS, the source and pickup hydrophones are at the same vertical level, assuring that the distances between acoustic generating and pickup portions of the hydrophones are those measured. For the mechanically generated wave, the acoustic paths between hydrophones can be more complicated.

The mechanically generated acoustic wave travels down the source hydrophone guide shaft (stainless steel) to the hydrophone. When it reaches the surface of the test liquid, it can begin to propagate toward the pickup hydrophones as sketched in Figure B1, along paths Z1 and Z2. This complication is significant only when the waste SOS is comparable to that in the stainless steel shaft (about 5000 m/s). From geometry it can be concluded that errors associated with alternate paths (Z1 and Z2) for the acoustic signal can only overpredict the SOS. Since the measured SOS in the waste was relatively low (100 to 200 m/s), it is concluded that the mechanical stimulation of the source hydrophone did not lead to alternate path errors.

Figure B1. Alternate Paths (Z1,Z2) for the Mechanically Generated Acoustic Signal to Reach the Pickup Hydrophones.

APPENDIX C
SPEED OF SOUND/YIELD STRESS MEASUREMENT PROCEDURE

Page 1 of 1

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SPEED OF SOUND/YIELD STRESS MEASUREMENT PROCEDURE

N. S. Cannon
January 27, 1994

1.0 INTRODUCTION

1.1 Purpose

This test procedure was written to direct tests described in the Tank Waste Speed of Sound/Rheology (TWSOSR) Test Plan (WHC-SD-WM-TP-195).

1.2 Scope of Test

The TWSOSR testing is designed to measure the speed of sound and yield stress in simulants and a small (200 mL) sample of 101-SY tank waste. Yield stress will be determined in the waste sample at five different vibration amplitudes using a shear vane technique described in the test plan.

1.3 Impact Level

This testing will be performed in accordance with Impact Level 3SQ, as specified by MRP 5.43.

1.4 Test Data

A test logbook will be maintained by the test performer(s). The Cognizant Engineer shall be the designated test custodian. All pertinent observations, off normal events, sketches, photographs etc. shall be logged. Chronological test conduct shall also be logged. All logged data entries shall be signed and dated. The test logbook assigned for this work is WHC-N-832 1, "Sonic Probe Verification".

2.0 TEST FACILITY and EQUIPMENT

The TWSOSR testing will be performed simulants in the 306E building highbay. A sketch of the TWSOSR test system is made in Figure 1.

2.1 Equipment

The primary equipment used as part of the TWSOSR system includes three Bruel & Kjaer model 8103 hydrophones, two BK model 2635 charge amplifiers (the third hydrophone is the source and is driven with a Wavetek Model 75 arbitrary function generator, Nicolet Model 4094B digital oscilloscope, and a Haake viscometer including both a cone and plate attachment for rheograms and a shear vane attachment for yield stress measurements (Haake model and ID information will be recorded at test initiation time). Other equipment may be specified for a given test by the Cognizant Engineer.

2.2 Calibration

Data acquisition hardware will be calibrated as recommended by the vendor. Appendix AZ includes a table where TWSOSR equipment is listed including vendor and calibration information.

2.3 Simulant

Any simulant used in 306E Building must be preapproved by the Building Management. MSDS(s) must be supplied and posted in the test area. Post-test disposal plans for the simulant must also be made and approved by the Building management.

3.0 SAFETY

- 3.1 A Job Hazard Analysis (JHA) for 306E Building operations is attached as Appendix BZ. The TWSOSR operator(s) will have read and be familiar with this JHA for any testing performed in the 306E Building. Testing performed in the 325 Building will be directed by Joel Tingey and PNL safety procedures will apply.
- 3.2 The applicable MSDSs for all simulants used in the TWSOSR testing will also be included in Appendix BZ. The 306E Building Management will review and approve any simulant PRIOR to its being brought into the building. The operator(s) of the TWSOSR system will read the MSDS of any simulant used prior to performing the test.
- 3.3 All applicable JHAs and MSDSs shall be posted or located in the 306E Building test area.
- 3.4 All test area personnel shall observe all safety precautions outlined in the MSDSs and JHAs thus posted.

4.0 TEST PROCEDURE

A general test procedure is given in Appendix CZ.

- 4.1 The Cognizant Engineer will assign an TWSOSR # for a specific test, identify the test material, list specific equipment to be used on an Appendix DZ equipment data sheet, and give written instructions to the test performer identifying any special requirements.
- 4.2 The test performer will conduct the TWSOSR test in accordance with this procedure. All test setup, special instructions, equipment and acquired data will be recorded in the logbook (WHC-N-832 1) as described in Section 1.4. The test performer will verify that the equipment used was properly described on the equipment data sheets (Appendix DZ), and that acquired data is recorded on data sheets (Appendix EZ) in addition to logbook entries.
- 4.3 At the completion of testing the test performer will arrange for disposal of the test simulant in accordance with the preapproved plan described in Section 2.4, and given in Appendix BZ.

Figure C1. Sketch of the TWSOSR Hardware.

APPENDIX AZ
TWSOSR DATA ACQUISITION EQUIPMENT LIST

TWSOSR TEST # _____

EQUIPMENT	DESCRIPTION	CALIBRATION INTERVAL	LAST CAL. DATE	CAL. Procedure
Bruer & Kjaer Model 2635	Charge Amplifier for B & J Model 8130	1 year	11-03-93	WHC Standards Lab
Bruer & Kjaer Model 8103	Hydrophone	1 year	12-22-93	Operator Calib.
Nicolet Scope	Model 4094B Digital Oscilloscope SN# 87B02650	1 year	01-19-94	WHC Standards Lab
Fluke 2166A	Temperature readout for Type K Thermocouples	1 year	01-12-94	WHC Standards Lab

PNL hardware used for rheology will be listed in Appendix DZ, with calibration information given there.

Bruer & Kjaer Hydrophones(8103)/Amplifiers(2635)

Equipment Item #	Description	SN#	Last Cal. Date	Next Cal. Date
✓ 1	Amplifier	1742663	11-03-93	11-03-94
✓ 2	Amplifier	1742664	11-03-93	11-03-94
3	Amplifier	1742674	11-03-93	11-03-94
✓ 4	Hydrophone	1672080	12-22-93	12-22-94*
✓ 5	Hydrophone	1672089	12-22-93	12-22-94*
6	Hydrophone	1672100	12-22-93	12-22-94*

* Calibrations were performed by N. S. Cannon, WHC, using Bruel and Kjaer model 4229 calibrator designed for this purpose. The BK 4229 calibrator (SN 1617574) was itself calibrated 12-07-93 by BK; calibration and certificates are given in logbook WHC-N-832 1 on pages 41 through 46. These hydrophones were found to still be within the BK specification.

APPENDIX BZ

JOB HAZARD ANALYSIS AND MATERIAL SAFETY DATA SHEETS

THE FOLLOWING JHAs and MSDSs ARE LOCATED IN THE TEST AREA
(List by title and number as applicable. This may be hand written entry.
initialed and dated)

TWSOR TEST # HOT STUFF

NUMBER	TITLE	DATE
Job Hazard Analysis	Attenuation Chamber Test System (ACTS)	11-11-93

~~SIMULANT DESCRIPTION:~~

NA

Building Management Approval for simulant use in 306E Building:

Date

DISPOSAL PLAN FOR SIMULANT:

NA

Building Management Approval for simulant disposal:

Date

Waste from Segment 2Z, Window "C" - 101-54 Tank
MS Cannon

APPENDIX CZ

TWSOSR Test Procedure

This Appendix really provides two procedures: 1) a procedure measuring the speed of sound (SOS GENERAL TEST PROCEDURE), and 2) a procedure for doing rheological measurements on tank waste (325 BUILDING RHEOLOGY GENERAL PROCEDURE - TANK WASTE). Note that the Cognizant Engineer is authorized to add or delete steps to these procedures as he deems necessary.

The speed of sound measurement procedure is given on pages 2 and 3 of this appendix. The 325 Building waste rheology procedure comprises pages 4 through 7 of this appendix. Portions of the waste rheology procedure may also be used to test simulants in 325 Building, as deemed necessary by the Cognizant Engineer.

Additionally, viscosity measurements may be made in 326 Building on simulants using a Nametre Model 1810NPTROD viscometer. If used, calibration of this machine will be performed by WHC "in-house" using calibration standards. A formal procedure for these tests will not be written, but the testing and calibration details and data will be recorded in the logbook WHC-N-832 1.

APPENDIX CZ(cont. page 2)

SOS GENERAL TEST PROCEDURE					
TEST DATE <u>2-23-94</u> TWSOSR # <u>Hot Stuff</u>			TEST MATERIAL <u>Tank Waste - Seg 22 from window "C"</u>		
COG ENGINEER <u>N S Cannon</u>			TEST PERFORMER		
STEP NUMBER	INSTRUCTION	REF. DWG. PROC. OR SPEC.	COMPLETION SIG/INIT.	DATE	NOTES/DATA/COMMENTS
1.0	Record Instruments/Equipment to be used on the Appendix D data sheet, and verify calibration dates.	Appendix D	Q <u>NSC</u> T <u>NSC</u> E <u>NSC</u> OXXXXXXXXX	<u>5/9/94</u> 2-23-94	QA verified instrument calibration after tests were completed
2.0	Verify that the test material information has been entered into Appendix B, and that proper approvals have been obtained as required. Then load the test material into test cup.	Appendix B	QXXXXXXXXX T <u>NSC</u> E <u>NSC</u> OXXXXXXXXX	2-24-94	Tank waste loaded into cup at 9:35 AM 9:05 AM Approvals not needed
3.0	Install hydrophone array cap, placing hydrophones in proper position in the sample cup. For "viscus" test material, allow to sit at least 1/2 hour before testing.	Figure 1	QXXXXXXXXX T <u>NSC</u> E <u>NSC</u> OXXXXXXXXX		Hydrophone Top loaded by 9:35 AM
4.0	Turn on all instrument electronics (record time of completed turn on in logbook) and allow to warm up at least 1 hour prior to testing.	Appendix D	QXXXXXXXXX T <u>NSC</u> E <u>NSC</u> OXXXXXXXXX	2-24-94	Turn on electronics at 8:05 AM

Q = QUALITY T = TEST PERFORMER E = COG ENGINEER O.= OTHER; DEFINED _____

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APPENDIX CZ(cont. page 3)

SOS GENERAL TEST PROCEDURE					
TEST DATE			TWSOSR # <i>Hot Stuff</i>		
COG ENGINEER <i>N. S. Cannon</i>			TEST MATERIAL		
TEST PERFORMER					
STEP NUMBER	INSTRUCTION	REF. DWG. PROC. OR SPEC.	COMPLETION SIG/INIT.	DATE	NOTES/DATA/COMMENTS
5.0	Bring test cup to target temperature, as directed by the Cog. Engineer. Cup temperature is based on the inner TC. Allow to remain at this temperature at least 30 minutes before testing.		QXXXXXXXXXX T X EXXXXXXXXXX OXXXXXXXXXX	3-2-94	Tests were run at ambient (90°F) and ~125°F <i>N.S. Cannon</i>
6.0	Choose a pulse type, and perform at least three SOS measurements for a given pulse type and save data on disk files (Nicolet 4094). Record the data on the Data Sheet (Appendix E) for SOS.	Appendix E	QXXXXXXXXXX T X E X OXXXXXXXXXX	3-2-94	Tone burst testing did not work - it was necessary to excite the S transducer with a screw driver. Data recorded in logbook, but not always on App. E forms. <i>N.S. Cannon</i>
7.0	Repeat steps 5.0 and 6.0 as required by the Cog. Engineer.	NA	QXXXXXXXXXX T X E X OXXXXXXXXXX	3-2-94	A complete set of SOS measurements are recorded on pages 84-96 of WMC-N-8321 logbooks. <i>N.S. Cannon</i>
8.0	Record all data sheets in logbook. QA verification of test completion.	Logbook WMC-N-8321	Q X T X E X OXXXXXXXXXX	4-20-94 3-2-94	QA Not applicable for hot cell testing. <i>N.S. Cannon</i>

Q = QUALITY T = TEST PERFORMER E = COG ENGINEER O = OTHER; DEFINED _____

* SOS also recorded in logbook on pages 100-101, 106-108, 110-111, 118. *N.S. Cannon (4-20-94)*

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APPENDIX CZ(cont. page 4)

325 BUILDING RHEOLOGY GENERAL PROCEDURE - TANK WASTE					
TEST DATE		TWSOSR # <i>Not Stuff</i>		TEST MATERIAL	
COG ENGINEER		<i>N.S. Cannon</i>		TEST PERFORMER	
STEP NUMBER	INSTRUCTION	REF. DWG. PROC. OR SPEC.	COMPLETION SIG/INIT.	DATE	NOTES/DATA/COMMENTS
1.0	Record Instruments/Equipment to be used on the Appendix D data sheet, and verify calibration dates.	NA	Q <i>(initials)</i> TXXXXXXXXX E <i>NAC</i> O	<i>5/9/94</i> <i>3-2-94</i>	<i>Cup & Spindle Rheology instead of Core & Plate NAC</i> <i>QA verified calibration of instrumentation after testing complete</i>
2.0	Verify that the test material information has been entered into Appendix B.	NA	QXXXXXXXXX TXXXXXXXXX E <i>NAC</i> OXXXXXXXXX	<i>3-2-94</i>	
3.0	Perform a Haake <i>Sup</i> and <i>MX</i> <i>Spindle</i> plate rheogram at 125 °F on waste in the "as received" condition according to PNL procedures. Shear rates from 0 to 400 s ⁻¹ should be used with ramp up/ramp down completed over a 4 minute cycle.	NA	QXXXXXXXXX TXXXXXXXXX E XXXXXXXXXX O <i>NAC</i>	<i>3-3-94</i>	<i>Shear rates 0-700 s⁻¹, 5 minute ramp up, 6 sec hold, 5 minute ramp down. NAC.</i>
4.0	Place 200 ml of waste in the test cup. Allow to sit for at least 30 minutes before performing first SOS test.	Figure 1	QXXXXXXXXX TXXXXXXXXX E XXXXXXXXXX O <i>NAC</i>	<i>3-2-94</i>	<i>This was done after SOS before C&S testing at my discretion NACannon</i>

Q = QUALITY T = TEST PERFORMER E = COG ENGINEER O = OTHER; DEFINED - PNL Scientist/Engineer/Technician

Shear rate testing was not possible because the waste yield stress was below the sensitivity of the equipment to accurately measure. Additional cup & spindle tests were performed at my discretion, and are recorded in logbook WNC-N-832-1. NAC 4-20-9

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APPENDIX CZ(cont. page 5)

325 BUILDING RHEOLOGY GENERAL PROCEDURE - TANK WASTE					
TEST DATE		TWSOSR #	TEST MATERIAL		
COG ENGINEER		TEST PERFORMER			
STEP NUMBER	INSTRUCTION	REF. DWG. PROC. OR SPEC.	COMPLETION SIG/INIT.	DATE	NOTES/DATA/COMMENTS
5.0	Perform speed of sound tests on waste at approximately 70, 100 and 125 °F using SOS GENERAL TEST PROCEDURE.	Appendix E	QXXXXXXXXX TXXXXXXXXX E OXXXXXXXXX		NA
6.0	With the waste still at 125 °F, vibrate the cup at maximum amplitude (as determined by Cog. Engineer) for at least 30 minutes.	NA	QXXXXXXXXX TXXXXXXXXX E OXXXXXXXXX		NA } M.J. Carmona
7.0	Perform speed of sound tests on waste at approximately 125, 100 and 70 °F using the SOS GENERAL TEST PROCEDURE.	Appendix E	QXXXXXXXXX TXXXXXXXXX E OXXXXXXXXX		NA
8.0	Remove SOS top, and insert the shear vane hardware, with torque set to 0.	NA	QXXXXXXXXX TXXXXXXXXX E OXXXXXXXXX		Shear vane testing was not performed
9.0	Raise the waste temperature back to 125 °F, and hold for about 8 hours.	NA	QXXXXXXXXX TXXXXXXXXX E OXXXXXXXXX		because yield stress was below instrument
10.0	After 8 hours, gradually increase the torque until the waste yields - record the yield torque (σ_0)		QXXXXXXXXX TXXXXXXXXX E OXXXXXXXXX		Sensitivity M.J. Carmona

Q = QUALITY T = TEST PERFORMER E = COG ENGINEER O = OTHER; DEFINED - PNL Scientist/Engineer/Technician

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APPENDIX CZ(cont. page 6)

325 BUILDING RHEOLOGY GENERAL PROCEDURE - TANK WASTE					
TEST DATE		TWSOSR #	TEST MATERIAL		
COG ENGINEER		TEST PERFORMER			
STEP NUMBER	INSTRUCTION	REF. DWG. PROC. OR SPEC.	COMPLETION SIG/INIT.	DATE	NOTES/DATA/COMMENTS
11.0	With waste temperature maintained at 125 °F, leave waste undisturbed for 16 hours, then measure σ_0 again.		QXXXXXXXXX TXXXXXXXXX EXXXXXXXXX 0 _____		
12.0	The goal for this Step is to determine the minimum time t_0 for which the σ_s will be about the same: i.e., the "reset" time. If σ_s is about the same for Steps 10 and 11, then use a 4 hour wait; if different pick a longer time (Cog. Engineer). Then measure σ_0 again. Continue until t_0 (≥ 2 hours) has been determined. See the Test Plan for more details.	Test Plan WHC-SD- WM-TP- 195	QXXXXXXXXX TXXXXXXXXX E _____ 0 _____		Cog. Engineer will have to make "judgement calls" on the "sameness" of σ_0 and wait times during the determination of t_0 .
13.0	After waiting t_0 since the waste was last disturbed, set the torque to 0.8 σ_0 , start vibrations at lowest setting, and gradually increase the measured vibrational amplitude until the waste yields. Record the amplitude and torque settings.	Test Plan WHC-SD- WM-TP-195	QXXXXXXXXX TXXXXXXXXX E _____ 0 _____		<i>M. J. Cannon</i>

Q = QUALITY T = TEST PERFORMER E = COG ENGINEER O = OTHER; DEFINED - PNL Scientist/Engineer/Technician

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APPENDIX CZ(cont. page 7)

325 BUILDING RHEOLOGY GENERAL PROCEDURE - TANK WASTE					
TEST DATE		TWSOSR #	TEST MATERIAL		
COG ENGINEER		TEST PERFORMER			
STEP NUMBER	INSTRUCTION	REF. DWG. PROC. OR SPEC.	COMPLETION SIG/INIT.	DATE	NOTES/DATA/COMMENTS
14.0	Repeat Step 13.0 for torques of 0.65, 0.5, 0.35, 0.2 of σ_p . Record data on data sheet and in logbook.	Test Plan WHC-SD-WM-TP-195	QXXXXXXXXX TXXXXXXXXX E O		
15.0	Perform a Haake cone and plate rheogram at 125 °F on waste in its final post-test state according to PNL procedures. Shear rates from 0 to 400 s^{-1} should be used with ramp up/ramp down completed over a 4 minute cycle.	NA	QXXXXXXXXX TXXXXXXXXX E O		
16.0	Record all data sheets in the Logbook. QA verification of test completion.	Logbook WHC-N-832 1	Q TXXXXXXXXX E O	5/9/94 4-20-94	Rheology test data recorded in LOG BOOK WHC-N-8321 on pages 97-99, 102-120

Q = QUALITY T = TEST PERFORMER E = COG ENGINEER O = OTHER; DEFINED - PNL Scientist/Engineer/Technician

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APPENDIX DZ

Data Acquisition Equipment Included for TWSOSR #HOTSTUFF

EQUIPMENT (SN#)	CALIBRATION EXPIRATION DATE	COMMENTS
NICOLET 4094B DIGITAL OSC. SCOPE	1-19-95	Basic Acquisition for SOS WHC STD LAB 679-51-01-005
Hydrophone (D1) SN 1672089	12-22-94	Pickup for SOS measurements. Calibrated with B&K 4229. See WHC-N- 8321 logbook pages 41-46.
Hydrophone (D2) SN 1672080	12-22-94	
Hydrophone SN 1672092	Not Calibrated Source Only	This is a projector - calibration is not necessary
OMEGA MODEL DQ41 SN B141527	9-94	PNL Instrument at B - reads multiple channels of Type K TCS
HAAKE Model RV100 with M5 head	4-17-94	PNL's instrument installed inside hot cell (sensor head). Calibration performed using A19, 110 ± 50 CP standard (3-15-94 and 3-17-94) 2-18-94

Effective Distance (D1-D2) = 0.800 in

Conditioner #2 Settings:

[illegible]

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APPENDIX D
HOT CELL TEST SUMMARY

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HOT CELL TEST SUMMARY

(DATE) TIME	STEP	TEMP (°F)	DESCRIPTION	RESULT	COMMENT
2-11-94 (NA)	SOS Water	- 70°F	SOS measured outside cell in water (in Test Cup)	V=4938 ft/s	Excellent agreement with literature
2-24-94 (08:18)	SOS Water	88.2°F	SOS measured inside Test Cup in-cell in water	V=4842 ft/s	Still good agreement with lit.
2-24-94 (9:12)	SOS Water	88.1°F	SOS in water - external cup (not test cup)	V=4882.5 ft/s	Consistent data
2-24-94 (11:09)	SOS Waste (Tone)	- 89°F	SOS in waste - tone burst. Doesn't work	NA	Electrical Cross Talk Only
2-24-94 (15:55)	SOS Waste (Tone)	- 125°F	SOS in waste - tone burst. Doesn't work	NA	Electrical Cross Talk Only. Square pulse tried also.
2-25-94 (11:00)	SOS	125°F	More attempts with tone burst/square wave for SOS	NA	Electrical Cross Talk Only. Square pulse tried also.
2-25-94 (11:48)	Vibrate	125°F	Vibrator run at several settings from 0.5 to 4 g - on about 10 minutes max.	Shook off heater tape	Control loop inconsistent. Heater tape re- installed
2-25-94 (16:00)	Freq. Scan	125°F	Tone Burst at 500Hz, 1KHz, 2.5KHz, 5KHz, 10KHz, 12.5KHz, 15KHz, 20KHz	NA	Electrical Cross- talk only.
2-28-94 (09:20)	SOS Mech.	88°F	SOS - Mechanical excitation.	V=520 ft/s	Overnight set: Test Group A
2-28-94 (11:07)	SOS Mech.	125°F	SOS - Mechanical excitation.	V=359 ft/s	Overnight set: Test Group A
2-28-94 (14:00)	Vibrated	124°F	Vibrated about 40 minutes with hydrophones at about 200 mV (Pa).	NA	No tests performed until next Monday
3-01-94 (09:38)	SOS Mech.	90°F	SOS - Mechanical excitation.	V=407 ft/s	Weekend set: Test Group B
3-01-94 (11:40)	SOS Mech.	123°F	SOS - Mechanical excitation.	V=369 ft/s	Weekend set: Test Group B
3-01-94 (13:00)	Vibrated	125°F	Vibrated at 3 g for 71 minutes. D1=57mV, D2=41.6mV @ 10mV/Unit setting	NA	Low amplitude vibration.
3-01-94 (13:08)	SOS Mech.	126°F	SOS - Mechanical excitation.	V=305 ft/s	After vibration: Test Group C
3-02-94 (08:30)	SOS Mech.	90°F	SOS - Mechanical excitation.	V=547 ft/s	After overnight set: Test Group D
3-02-94	SOS Tone	90°F	SOS in WATER - Calibration (external cup)	V=4926 ft/s	Excellent match with literature

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(DATE) TIME	STEP	TEMP (°F)	DESCRIPTION	RESULT	COMMENT
3-02-94 (10:05)	SOS Mech.	90°F	SOS in water - mechanical excitation.	V=5969 ft/s	Speed advantage significant in water - not waste.
3-03-94	STIR	92°F	Mechanically stirred waste in test cup prior to taking sample for Cup & Spindle Rheology: SAMPLE 1		Stirring at ambient temperature.
3-03-94 (11:02)	Cup & Spindle	92°F	Three Cup and Spindle Rheology runs: SAMPLE 1	YS = 82, 52, 15.4 Pa	Shear Thinning
3-03-94 (13:57)	SOS Tone	94°F	SOS in water - Tone burst	V=4926 ft/s	Excellent Calibration
3-03-94 (15:03)	SOS Mech.	93°F	SOS in Waste - Mechanical excitation	V=400 ft/s	Hydrophones replaced in waste 1 hour earlier.
3-03-94 (15:15)	Vibrated	93°F	10 g for 5 minutes. D1=546 mV, D2=468 mV; 10mV/Unit Scale		Above measured yield stress, but at 93°F
3-03-94 (15:17)	SOS Mech.	93°F	SOS mechanical excit.	V=370 ft/s	Test Group E
3-03-94 (15:57)	STIR & SOS	93°F	Mechanically stirred waste with spatula; measured SOS	V=354 ft/s	Test Group E (but note stirring)
3-04-94 (09:29)	SOS - Mech.	92°F	SOS mechanical excitation	V=557 ft/s	Overnight reset: Test Group F
3-04-94 (09:29)	Cup & Spindle	92°F	Rheology - Cup and Spindle - Same SAMPLE 1	YS = 35, 3.1, 1.7 Pa	Overnight reset
3-07-94	Overtemp	<150°F	Loss of outer TC resulted in overtemp event. Inner cup temperature never more than 150°F		Up - 108°F @ 9:38 Down - 144°F @ 10:18
3-07-94 (11:52)	Vibrate	128°F	Vibrated at 10g setting for 31 minutes. No hydrophones recorded - assumed same as before. Temperature finished at 126.3°F	Preparation for Shear vane testing.	
3-07-94 (15:40)	Shear Vane Densifi- cation	122.8°F	Densification test using small shear vane at top of cup.	No yield stress detected.	Goop engulfed shear vane at the 1/4" depth.
3-08-94	Shear Vane Densifi- cation	122.0°F	Three densification tests at bottom of cup.	No yield stress detected.	Small shear vane.

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(DATE) TIME	STEP	TEMP (°F)	DESCRIPTION	RESULT	COMMENT
3-08-94	Replace Shear Vane	126.1°F	Replaced small shear vane with larger one.	Test Tomorrow	Stirred thoroughly
3-09-94 (08:10) (09:00)	Shear Vane	123.7°F 122.0°F	Two shear vane tests run centered vertically, different cup locations.	No yield stress measured.	Can't measure any stress with either shear vane.
3-10-94 (08:34)	SOS	126.7°F	SOS measured after overnight reset	V=411 ft/s	Next to vibrate: Test Group G
3-10-94 (10:29)	Vibrate	126.5°F	Vibrated at hydrophone amplitudes consistent with previous 10g levels		Difficulty keeping vibrator on
3-10-94 (10:35)	SOS	126.6°F	SOS measured after vibration	V=305 ft/s	Same trend as lower temperatures: Test Group H
3-17-94 (11:04)	Cup & Spindle	125.1°F	Original SAMPLE 1 retested (twice)	YS=12.3 Pa YS= 2.6 Pa	Not much recovery over last 310 hours
3-17-94 (14:15)	SOS	90.6°F	Test Cup sample undisturbed for last 172 hours	V=487 ft/s	Back up from 305 ft/s: Test Group I
3-18-94 (09:51)	SOS	125.0°F	Other than SOS, sample undisturbed for last 192 hours	V=438 ft/s	Test Group I
3-18-94 (10:30)	Vibrated	125.6°F	20g vibration, but hydrophones @ low amp. most (20 min.) of time.	D1= 84 Pa D2=108 Pa	Amplitudes jumped around early on to D1=752Pa, D2=1120Pa
3-18-94 (10:34)	SOS	125.7°F	Right after vibration.	V=250 ft/s	Lowest SOS yet: Test Group J
3-18-94 (12:34)	Cup & Spindle	125.4°F	NEW SAMPLE 2 from vibrated cup. Four runs - 1st aborted @ over-range, last not stored on computer.	YS1= 62 Pa YS2= 16.4 Pa YS3= 16.5 Pa YS4= 12.8 Pa	YS1, YS4 are extrapolated from pen plots.
3-21-94 (09:30)	Cup & Spindle	89.1°F	Another NEW SAMPLE 3 from vibrated cup. Three runs.	YS1= 75.3 Pa YS2= 9.5 Pa YS3= 7.3 Pa	YS1 extrapolated from pen plot.
3-21-94 (12:30)	Cup & Spindle	85.1°F	Another NEW SAMPLE 4 (Last) for lower maximum shear rate test. Two runs.	YS1= 107 Pa YS2= 30.2 Pa	Lower shear rates probably reason for higher YS1
3-21-94 (14:55)	SOS Water	83.6°F	Final calibration.	V=5253 ft/s	Distance may have shortened, but still within 5%
3-22-94 (09:00)	Cup & Spindle	86.2°F	Last SAMPLE 4 retested for recovery. Two runs, low shear rate max.	YS1= 140 Pa YS2= 38.2 Pa	Total recover inferred.

WHC-SD-WM-DTR-034
Rev. 0

APPENDIX E

CUP AND SPINDLE RHEOLOGY - RAW DATA

APPENDIX E

Plots of the raw data obtained from the C&S testing are given in this appendix, along with computer generated "fits" to these tests. Yield strength data was often obtained from the computer plots. The order of the plots in this appendix is the same as the order of results given in Table 7.

All of the 240 mL of waste sample from the 101-SY tank were initially placed in the vibrator test cup. During the course of testing, Samples 1 - 4 were removed from the test cup for C&S rheology testing. The last three samples were taken after SOS measurements were completed. Each sample was weighed at about 70 grams; the density of the waste was measured at 1.76 g/cm^3 . Thus, the approximate volume of each sample was about 40 mL.

ROTOVISCO

Fließkurve
Flow curve

SAMPLE 1

HAAKE

$$\tau = A \cdot \% \tau \cdot S \text{ [Pa]}$$

$$D = M \cdot \% D \cdot S_D \text{ [s}^{-1}\text{]}$$

$$\eta = \tau / D \text{ [Pa} \cdot \text{s]}$$

Datum
Date 3-3-94
3-4-94

Nr.
No 1

Substanz
Substance 91015 2

Temperatur
Temperature 33.4

ROTOVISCO RV 100

System
System M3

Meßeinrichtung
Sensor system MV-DIR-034

A.

M.

Programmzeit
Program time

t₁ 0

t₂ 5

t₃ 1

Unterschrift
Signature

Sample 1

TAU (Pa)

TAU/D - CURVE

H A A K E

Test of
03-03-1994

Substance
101sys1

Sample No
1

Temp (C)
33.6

Meas. Drive
500

Meas. System
MV1

Linear

A= 8.255E 1

B= 3.009E-2

R2= .3686

D (1/s)

MHC-SD MM-DTR-034
REV. 0

67

ROTOVISCO

Fließkurve
Flow curve

Sample 1

HAAKE

60 % τ
Pa

$$\tau = A \cdot \% \tau \cdot S_{\tau} \text{ [Pa]}$$

$$D = M \cdot \% D \cdot S_D \text{ [s}^{-1}\text{]}$$

$$\eta = \tau / D \text{ [Pa} \cdot \text{s]}$$

S_{τ}

Datum
Date

3-3-94
3-9-94

Nr.
No

2

Substanz
Substance

sylo 5-2

Temperatur
Temperature

33.6

ROTOVISCO RV

100

System
System

MS

Meßeinrichtung
Sensor system

MH-1

A

M

Programmzeit
Program time

t₁ 0

t₂ 5

t₃ 1

Unterschrift
Signature

40 % D
s⁻¹

S_D

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③

SAMPLE 1

TAU/D - CURVE

TAU (Pa)

H A A K E

Test of
03-03-1994

Substance
101sy1

Sample No
2

Temp (C)
33.6

Meas. Drive
500

Meas. System
MV1

Linear

A= 5.239E 1

B= 1.504E-1

R2= .7839

D (1/s)

MHC-SD-WM-DTR-034
REV. 0

HAAKE

SAMPLE 1

ROTOVISCO Fließkurve
Flow curve

CO % τ
Pa

$$\tau = A \cdot \% \tau \cdot S \cdot S_D$$

$$D = M \cdot \% D \cdot S_D$$

$$\eta = \tau / D$$

S_τ

40 % D
S'

Datum
Date 3-3-94

Nr.
No. 3

Substanz
Substance Sydris 22

Temperatur
Temperature 33.73

ROTOVISCO RV 100

System
System RV

Meßeinrichtung
Sensor system MV 10

A M

Programmzeit
Program time

t_1

t_2

t_3

Unterschrift
Signature

SAMPLE 1

TAU/D - CURVE

TAU (Pa)

H A A K E

Test of
03-03-1994

Substance
101sys1

Sample No
3

Temp (C)
33.6

Meas. Drive
500

Meas. System
MV1

Linear

A= 1.488E 1

B= 2.403E-1

R2= .9947

D (1/s)

MHC-SD-MM-DTR-034
REV. 0

②

HAAKE

ROTOVISCO

Fließkurve
Flow curve

SAMPLE 1

CO % T
Pa

$$\tau = A \cdot \dot{\gamma}^n \cdot S_z \text{ [Pa]}$$

$$D = M \cdot \% D \cdot S_D \text{ [s}^{-1}\text{]}$$

$$\eta = \tau / D \text{ [Pa} \cdot \text{s]}$$

Datum
Date

Nr.
No.

Substanz
Substance

Temperatur
Temperature

ROTOVISCO RV

System
System

Messanordnung
Sensor system

A
M

Programmzeit
Program time

t₁
0

t₂
5

t₃
1

Unterschrift
Signature

40 % D
S

SAMPLE 1

TAU (Pa)

TAU/D - CURVE

H A A K E

Test of
03-04-1994

Substance
sy101s22

Sample No
1

Temp (C)
32.3

Meas. Drive
500

Meas. System
MV1

Linear
A= 3.468E-1
B=-2.532E-2

R2= .071

D (1/s)

MHC-SD-MM-DTR-034
REV. 0

(8)

SAMPLE 1

SAMPLE 1

TAU/D - CURVE

ROTOVISCO

Fließkurve
Flow curve

SAMPLE 1

HAAR

$\gamma' \cdot \% \tau$
Pa

S_{τ}

0.9

0.8

0.7

0.6

0.5

0.4

0.3

0.2

0.1

0

0.1

0.2

0.3

0.4

0.5

0.6

0.7

0.8

0.9

0

$$\tau = A \cdot \% \tau \cdot S_D$$

$$D = M \cdot \% D \cdot S_D$$

$$\eta = \tau / D \text{ [Pa} \cdot \text{s]}$$

Datum
Date 3-17-94

Nr
No

Substanz
Substance 51.7°C

Temperatur
Temperature

ROTOVISCO
REV. 0

System
System

Meßeinrichtung
Sensor system

A

M

Programmzeit
Program time

t₁ 0

t₂ 5

t₃ 1

Unterschrift
Signature

60 % D
S'

Bestell Nr. 222 0187
Order No. 222 0187

Druck Nr. 5721-10 87
Printing No. 5721-10 87

File: ① 1015702 ② 1015702
③

SAMPLE 1

TAU/D - CURVE

TAU (Pa)

SAMPLE 1

TAU/D - CURVE

TAU (Pa)

H A A K E

Test of
03-17-1994

Substance
101sys22

Sample No
2

Temp (C)
51.7

Meas. Drive
500

Meas. System
MV1

Linear

A= 2.552E 0

B= 5.067E-2

R2= .9993

D (1/s)

MHC-SD-M-DTR-034
REV. 0

(6)

ROTOVISCO

Fließkurve
Flow curve

SAMPLE 2

HAAKE

$\tau = A \cdot \dot{\gamma} \cdot S_1 \cdot S_2 \cdot S_3$ [Pa]
 $D = M \cdot \dot{\gamma} \cdot S_4 \cdot S_5$ [s⁻¹]
 $\eta = \tau / D$ [Pa · s]

Datum
Date 3-18-74

Nr.
No.

Substanz/
Substance POLY ST 22

Temperatur
Temperature 51.9

ROTOVISCO RV MV 100

System
System MS

Meßanordnung
Sensor system MVI

Λ

M

Programmierte
Program time

1. 0

2. 5

3. 1

Unterschrift
Signature

REP

$\dot{\gamma}$ [s⁻¹]
D

S_D

Bestell-Nr. 222-0187 Druck Nr. 7 5721-10 87
 Order No. Printing No.

SAMPLE 2

TAU/D - CURVE

TAU (Pa)

H A A K E

Test of
03-18-1994

Substance
101sys22.

Sample No
2

Temp (C)
51.9

Meas. Drive
500

Meas. System
MV1

Linear

A= 1.636E 1

B= 1.626E-1

R2= .9912

D (1/s)

$\tau = A \cdot \dot{\gamma} \cdot S_{\tau} \text{ [Pa]}$
 $D = M \cdot \dot{\gamma} \cdot S_D \text{ [s}^{-1}\text{]}$
 $\eta = \tau / D \text{ [Pa} \cdot \text{s]}$

Datum
Date 3-18-94

Nr.
No.

Substanz
Substance 101555932

Temperatur
Temperature 51.5

NOIOWISCO RV HV 100

System
System MS

Meßeinrichtung
Sensor System HV 1

A

M

Programmiertes
Program line

1. 0

2. S

3. 1

Signature

Unterschrift

60 % D
S

SAMPLE 2

(17)

SAMPLE 2

TAU/D - CURVE

TAU (Pa)

120

96

72

48

24

0

140

280

420

560

700

H A A K E

Test of
03-18-1994

Substance
101sys22

Sample No
3

Temp (C)
51.9

Meas. Drive
500

Meas. System
MV1

Linear
A= 1.645E 1
B= 1.424E-1

R2= .996

D (1/s)

HAAKE

SAMPLE 3

ROTOVISCO

Fließkurve
Flow curve

100 % τ
Pa

$\tau = A \cdot \% \tau \cdot S_D$ [Pa]
 $D = M \cdot \% D \cdot S_D$ [s⁻¹]
 $\eta = \tau / D$ [Pa · s]

S_D

Date
3-21-74

No.

Substance
POLY 5-7-77

Temperature
31.7

ROTOVISCO IV
770100

System
MIS

Medium/medium
Sensor System
MIV1

A

M

Programmer/
Program name

1, 0

1, 5

1, .1

208.0

Unterzeichnet
Signature

100 % D
S⁻¹

S_D

Revised 722 0187 Date & File
Under the Printing No. F 5121-10 87

WHC-SD-WM-DTR-034
REV. 0

18

SAMPLE 3

TAU/D - CURVE

TAU (Pa) .

H A A K E

Test of

03-18-1994

03-21-1994

Substance

101sys22

Sample No

1

Temp (C)

31.7

Meas. Drive

500

Meas. System

MV1

Linear

A= 1.174E 2

B=-8.339E-2

R2= .2186

D (1/s)

MHC-SD-MM-DTR-034
REV. 0

SAMPLE 3

TAU/D - CURVE

TAU (Pa)
71.67
43

57.33
34.4

43.0
25.8

28.67
17.2

14.333
8.600001

0

0

140

280

420

560

700

H A A K E

Test of
03-21-1994

Substance
101sys22

Sample No
2

Temp (C)
31.7

Meas. Drive
500

Meas. System
MV1

Linear

A= 5.707E 0

B= 5.434E-2

R2= .9859

D (1/s)

WHC-SD-WM-DTR-034
REV. 0

multiply by
1.667

20

SAMPLE 3

TAU/D - CURVE

TAU (Pa)

H A A K E

Test of
03-21-1994

Substance
101sys22

Sample No
3

Temp (C)
31.7

Meas. Drive
500

Meas. System
MV1

Linear
A= 7.284E 0
B= 8.253E-2

R2= .9948

D (1/s)

MHC-SD-WM-DTR-034
REV. 0

ROTOVISCO

Fließkurve
Flow curve

SAMPLE 4

HAAKE

$$\tau = A \cdot \dot{\gamma}^n \cdot S_t \text{ [Pa]}$$

$$D = M \cdot \dot{\gamma} \cdot S_D \text{ [s}^{-1}\text{]}$$

$$\eta = \tau / D \text{ [Pa} \cdot \text{s]}$$

WHC-SD-WM-DTR-034

Datum
Date 3.21.94

Nr
No 1

Substanz
Substance R1515

Temperatur
Temperature 29.5 °C

ROTOVISCO RV HV100

System HS

Meßeinrichtung
Sensor system MVI

A

M

Programmzeit
Program time

t₁ 0

t₂ 5

t₃ 1

Unterschrift
Signature

22

SAMPLE 4

TAU/D - CURVE

TAU (Pa)

SAMPLE 4

TAU/D - CURVE

TAU (Pa)

H A A K E

Test of
03-21-1994

Substance
101sys22

Sample No
2

Temp (C)
29.5

Meas. Drive
500

Meas. System
MV1

Linear
 $A = 3.02E 1$
 $B = 1.024E 0$

$R^2 = .7741$

D (1/s)

MHC-SD-MM-DTR-034
REV. 0

ROTOVISCO

Fließkurve
Flow curve

SAMPLE 4

HAAKE

$$\tau = A \cdot \% \tau \cdot S_t \text{ [Pa]}$$

$$D = M \cdot \% D \cdot S_D \text{ [s}^{-1}\text{]}$$

$$\eta = \tau / D \text{ [Pa} \cdot \text{s]}$$

WHC-SD-WM-DTR-034
REV. 00

Datum
Date 3.22.94

Nr
No 1

Substanz
Substance 1015Y 55 2120

Temperatur
Temperature

ROTOVISCO IV MV 100

System
System H5

Meßanordnung
Sensor system MVI

A

M

Programmiert
Program time

t₁ 0

t₂ 5

t₃ 1

Unterschrift
Signature

DEL

SAMPLE 4

TAU (Pa)

TAU/D - CURVE

H A A K E

Test of
03-22-1994

Substance
101sys22

Sample No
1

Temp (C)
30.1

Meas. Drive
500

Meas. System
MV1

Linear

A= 1.395E 2

B=-1.729E -1

R2= .0052

D (1/s)

MHC-SD-MM-DTR-034
REV. 0

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Inches

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2 of 2

ROTOVISCO

Fließkurve
Flow curve

SAMPLE 4

HAAKE

100 % τ
Pa

0.9
0.8
0.7
0.6
0.5
0.4
0.3
0.2
0.1

5 % D
s⁻¹

WHC-SD-WM-DTR-034

REV. 0

Datum Date: 3.22.84

Nr. No.: 2

Substanz Substance: 10157 5922

Temperatur Temperature: 17V100

System System: M5

Meßeinrichtung Sensor system: M4

Programmiert Program time: t₁ 0, t₂ 5, t₃ 1

Unterschrift Signature: R. G.

SAMPLE 4

TAU (Pa)

TAU/D - CURVE

H A A K E

Test of
03-22-1994

Substance
101sys22

Sample No
2

Temp (C)
30.1

Meas. Drive
500

Meas. System
MV1

MHC-SD-MH-DTR-034
REV. 0

**DATE
FILMED**

10/ 4 / 94

END