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FINAL REPORT-
GAS RETENTION AND RELEASE TESTS SUPPORTING THE CONCENTRATE RECEIPT VESSEL (CRV-VSL-00002A/2B) CONFIGURATION (U)

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ENGINEERING DEVELOPMENT LABORATORY
HANFORD RIVER PROTECTION PROJECT - WTP
SAVANNAH RIVER NATIONAL LABORATORY

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Prepared for the U.S. Department of Energy Under Contract Number DEAC09-96SR18500

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Test Exceptions
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LIST OF ACRONYMS

BNI	Bechtel National, Inc.
CCN	correspondence control number
CRV	concentrate receipt vessel
DACS	data acquisition and control software
DBE	design basis event
GR&R	gas retention and release
H/D	ratio of slurry height to vessel diameter
HLW	high-level waste
ID	inner diameter
JPP	jet pump pair
OD	outer diameter
PJM	pulse jet mixer
R&D	research and development
R&T	research and technology
RD	rheological additive
RFD	reverse flow diverter
ROB	region of bubbles
scfm	standard cubic feet per minute
VVV	Validate, Verify, nozzle Velocity
WTP	Waste Treatment Plant
ZOI	zone of influence

1.0 TESTING SUMMARY

Gas Retention and Release (GR&R) tests were performed in the scaled Concentrate Receipt Vessel (CRV) Test Stand at the Savannah River National Laboratory to validate the capability of candidate Hybrid-Mixing systems for the CRV to safely release hydrogen during normal and upset conditions. Hydrogen is generated in the radioactive waste as a result of natural and plant processes and must not be allowed to accumulate above flammability limits. Two types of tests were conducted. Gas holdup tests determined the steady state amount of gas accumulated in the simulant under normal PJM only or PJM plus sparging conditions. Gas release tests determined what operating conditions are necessary to fully release gas after a steady state gas fraction of 4% tank volume or more was reached in the simulant.

Three CRV configurations were tested:

1. 3 PJMs with 1.5-inch upward facing (135^{o}) nozzles, 2 PJMs with downward-facing 45^{o} nozzles, 1 center downward nozzle (no spargers)
2. 5 PJMs with 1-inch nozzles facing downward and 1 center downward nozzle (5 spargers)
3. 5 PJMs with 1-inch nozzles facing downward and 1 center downward nozzle (no spargers)

For test purposes, hydrogen gas generation was simulated by the breakdown of hydrogen peroxide into oxygen gas and water in the kaolin:bentonite simulant. First, a steady state gas fraction simulating accumulated gas after an extended period of plant shutdown was achieved by mixing in a batch injection of 30 wt% hydrogen peroxide (**Sec. 3.5.1**). After an overnight growth period, the PJMs/spargers were restarted and the change in level measured by the use of laser sensors. Second, the rate of change and steady state value of the gas fraction during continuous injection of hydrogen peroxide was measured with the PJMs/spargers operating (**Sec. 3.5.2**). Gas release tests were also performed to determine the rate of release of gas after the batch and continuous injections (**Sec. 3.5.3**) with PJMs and sparging operating singly or combined.

The tests on Configuration 1 showed that the maximum holdup after batch injection of 1325 gm of H_2O_2 solution was 16.3%. After slow and fast (continuous) injection of hydrogen peroxide, the maximum holdup was 13.6% and 23.6%, respectively, which was due to the total amount of H_2O_2 (1325 and 2800 gms, respectively) injected. During continuous H_2O_2 injection of 8 ml/min and 16 ml/min of H_2O_2 solution, corresponding to the slow and fast injection rates above, a maximum holdup of 0.5% and rates of increase of 0.005%/min were observed for both cases. Upon restarting the PJMs/spargers, 95% of the accumulated gas was released after 20 cycles.

The tests on Configuration 2 exhibited a 5.5% maximum holdup after batch injection of 664 gm H_2O_2 solution. All of the gas accumulated was released after 20 cycles of PJM/sparger operation. During continuous injection simulating normal operating conditions, the rate of rise of the gas fraction was 0.028%/min and the maximum holdup was 2%. After the PJMs/spargers were turned off, a maximum holdup of 7.5% developed. This decreased to 1.25% after restarting the spargers only. The gas was completely released only when the PJMs were also restarted.

The tests on Configuration 3 determined a maximum holdup of 4.9% after batch injection of 664 gm of H_2O_2 solution. The accumulated gas was released after 20 cycles of PJM/sparger operation. Holdup testing simulating normal operating conditions (28 ml/min of 30 wt% H_2O_2 solution) showed a rate of increase of the gas fraction of 0.2%/min and a maximum holdup of 1%. The gas accumulated after the PJMs/spargers were turned off amounted to 7.5%, which was completely released after 20 cycles (8 minutes) when the PJMs/spargers were restarted.

1.1 OBJECTIVES

The test objectives from Test Specification 24590-WTP-TSP-RT-03-011, Rev. 0 are shown in Table 1-1:

Table 1-1. Test Objectives from Test Specification 24590-WTP-TSP-RT-03-011, Rev. 0

Test Objective	Objective Met (Y/N)	Discussion
1. Measure gas hold-up levels during steady state operation using vessels that have demonstrated sufficient mobilization of the simulant(s)	Y	Three sets of GR&R tests were performed on three different CRV mixing systems that simulated gas holdup during steady state operation. For all test sets, the gas holdup was measured while PJMs and spargers were operating. The maximum steady state holdup did not exceed 2%.
2. Measure gas release characteristics to allow Engineering to define PJM operating parameters that can be adopted into the WTP design basis to assure that the PJMs and vessel ventilation systems can sustain safe operation.	Y	Three sets of GR&R tests were performed on three different CRV mixing systems that simulated gas release after loss-of-power events. For all tests, the accumulated gas was released within 40 cycles of PJM and sparger restart operation.

1.2 TEST EXCEPTION

The test exception is shown in Table 1-2:

Table 1-2. Test Exception

List Test Exceptions	Describe Test Exceptions
1. 24590-WTP-TEF-RT-04-00006 [3]	Provided test conditions for two PJM/sparger configurations to be tested. No impact on the objectives.

1.3 RESULTS AND PERFORMANCE AGAINST SUCCESS CRITERIA

Test results are compared to expected plant conditions in Table 1-3.

Table 1-3. Test Results Compared to Expected Plant Conditions

List Success Criteria	Explain How the Tests Did or Did Not Meet the Success Criteria
1. These tests will be deemed successful if gas hold-up levels are measured during steady state PJM operation using vessels that have demonstrated sufficient mobilization of the simulant(s) under prototypic PJM operating conditions.	A set of GR&R tests was performed on each of three different CRV mixing systems that simulated gas hold up during steady state operation. For all test sets, the gas holdup was measured while PJMs and spargers were operating.
2. These tests will be deemed successful if applicable gas release characteristics are measured after loss-of-power events.	A set of GR&R tests was performed on each of three different CRV mixing systems, respectively, that simulated gas release after loss-of-power events.

1.4 QUALITY REQUIREMENTS

This work was conducted in accordance with the RPP-WTP QA requirements specified for work conducted by SRTC as identified in DOE IWO M0SRLE60. SRTC has provided matrices to WTP demonstrating compliance of the SRTC QA program with the requirements specified by WTP. Specific information regarding the compliance of the SRTC QA program with RW-0333P, Revision 10, NQA-1 1989, Part 1, Basic and Supplementary Requirements and NQA-2a 1990, Subpart 2.7 is contained in these matrices.

1.5 R&T TEST CONDITIONS

The Test Specification establishes conditions to ensure that results are valid for project needs. This section lists those conditions and indicates whether they were followed. It describes the circumstances and consequences where deviations may have been necessary.

List R&T Test Conditions	Were Test Conditions Followed?
1. Holdup tests involve the generation of gas bubbles in simulant waste in scaled vessels during prototypic PJM operation. The holdup (fraction of gas retained in simulant) will be measured as a function of generation rate in prototypic vessels or a single representative vessel, as required to meet the testing schedule, (e.g., the gas release and retention vessel). The gas hold-up will be determined by means of surface level changes. A minimum of four scaled gas holdup tests are planned, one using a representative particulate simulant and three using Laponite simulants based on two gas generation rates and two consistencies.	Only the particulate simulant (kaolin:bentonite) tests were performed since the method of generating hydrogen relied on hydrogen peroxide on the clay particles. Three CRV mixing systems were evalutated. In one mixing system, three gas generation rates were tested. Two rheologies were tested, but in different mixing systems.
2. Tests will involve generating bubbles in stationary, unmixed simulant, and then measuring the release rate by means of either surface level changes (primary candidate) or a controlled ventilation of headspace gas and associated gas composition determinations (secondary alternative) after restart of the PJMs in normal pulsed operating mode. A minimum of four scaled gas holdup tests are planned, one using a representative particulate simulant and three using Laponite simulants based on two gas generation rates and two consistencies.	Only the particulate simulant (kaolin:bentonite) tests were performed since the method of generating hydrogen relied on hydrogen peroxide on the clay particles. Three CRV mixing systems were evalutated. In all of the mixing systems, two gas release rates were tested. Two rheologies were tested (different mixing systems).

Table 1-4 lists the specific test conditions communicated to SRNL (orally) and via Test Exception 24590-WTP-TEF-RT-04-00006, Rev. 0.

Table 1-4. CRV Mixing System Configurations and Test Conditions for GR&R Testing

Test Group	Test Sequence	Test type	Nozzle Configuration			Nozzle I.D., inches	Nominal Nozzle Velocity m/s	# (Spargers) @ scfm tot.	Nominal Yield Stress (Pa)
			# 45 deg.	# 135 deg.	# Vertical				
B	7	PJM only	2	3	1	1.5	8	N/A	20
E	2	PJM + sparging	5	N/A	1	1	12	(3) 6	30
E	4	PJM + sparging	2	3	1	1	12	(5) 9	30

1.6 SIMULANT USE

1.6.1 Plant Bounding Conditions

For the CRV vessel which will contain non-Newtonian fluids, it was assumed that the HLW pretreated sludge bounding physical and rheological properties would hold (CCNs 069099, 065607, and 082255).

1.6.1.1 Normal Plant Operation Rheological Bound

Data from actual radioactive and simulant waste rheograms combined with general engineering principles were used to define a set of bounding physical and rheological properties that agree well with actual data (Poloski et al. 2003). The non-Newtonian HLW pretreated sludge rheological properties were fit using a linear Bingham plastic model. The bounding conditions were used to develop the waste simulants used in the PJM program. Figure 1-1 is a plot of actual pretreated waste rheograms and the upper bounding rheological properties curve. The linear Bingham plastic model fit parameters are yield stress (y-axis intercept) of 30 Pa and consistency (slope) of 30 cP. Table 1-5 contains a summary of expected physical and rheological properties.

Because the rheological window is based on only four samples from three tanks, it is possible that slurries from other tanks could exceed the rheological boundary. It has been estimated that 20 to 30% of HLW tanks may have rheological properties higher (yield stress and consistency higher than 30 Pa and 30 cP, respectively) than those documented in the three active tank samples analyzed to date (CCN 082255). This uncertainty will be addressed by laboratory testing prior to receipt of the waste at the WTP to define the extent to which the slurry may be concentrated and stay below the rheological boundary.

Table 1-5. Physical and Rheological Properties that Help Define Simulants for Rating or Qualifying Fluidic Mixing Systems

Property	HLW Pretreated Sludge
pH	≈ 12[(a)]–14
Particle size distribution (D_{50})[(b)]	2 µm
Particle size distribution (D_{95})[(c)]	20 µm
Bulk density	1.1–1.6
Supernatant liquid density	≈1.0
Vol% settled solids	10%–90%
Wt% total dried solids	5%–25%
Wt% total oxide	7%–15%[(d)]
Shear stress versus shear rate (ambient and 40º C)	Bingham Plastic

(a) Expected pH after washing leaching in 0.01 M NaOH.
(b) 50% of particles are smaller than the indicated value.
(c) 95% of particles are smaller than the indicated value.
(d) Based on simulant data.

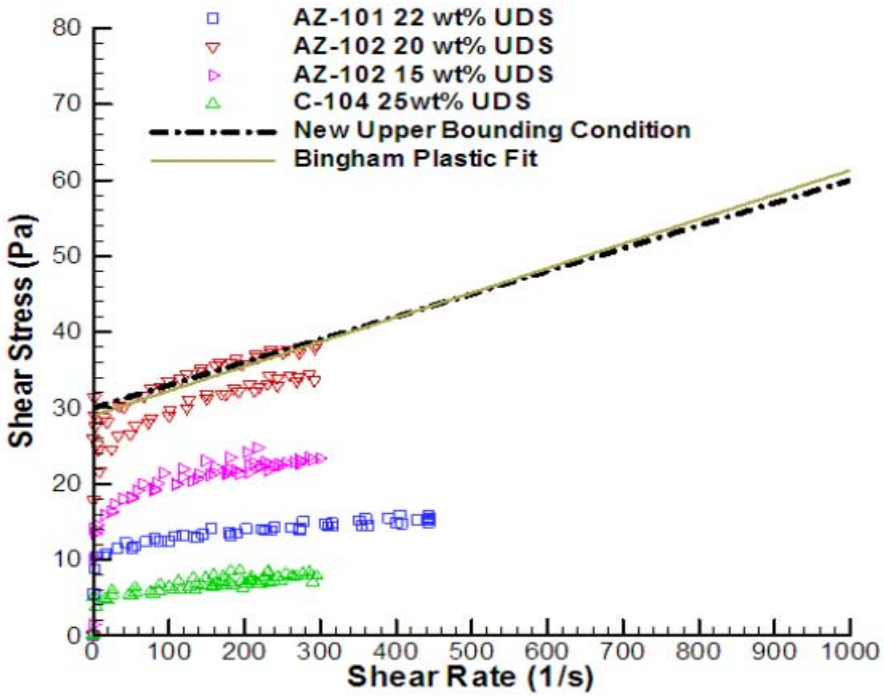

Figure 1-1. Rheogram of Actual HLW Pretreated Sludge Samples with Upper Bound Rheological Curve

1.6.1.2 Plant Upset Operation Rheological Bound

It is important to note that for actual HLW pretreated sludge samples when allowed to stand in an unmixed condition, that is, post-DBE, the waste will gel and reach maximum shear strength values greater than 30 Pa. For this reason, a bounding shear strength value of 70 Pa should be used (CCN 065607). In addition, the "gel" time (time required for the actual waste to reach its maximum shear strength) must be taken into account along with the maximum shear strength for plant operation considerations.

1.6.2 Simulants

One transparent simulant and one opaque simulant were used in the PJM program. The transparent simulant was Laponite RD (Southwestern Clay Products), a thixotropic colloidal synthetic clay that forms stable gel networks when unsheared. Due to the thixotropic nature of Laponite, the flow behavior of the simulant is dynamic, and it was allowed to gel and reach a target shear strength. Speers et al. (1987) demonstrated that the shear strength of clay drilling muds increases over time following first-order rate kinetics. Laponite shear strength behavior was observed to agree with the Speers et al. (1987) correlation for drilling muds. At this point the PJM system was started and a mixing cavern formed as defined by the gel's shear strength. After constant shearing, a steady-state flow behavior was approached. Unfortunately, this flow behavior was lower than the bounding rheology of WTP waste streams. This is illustrated in Figure 1-2, where actual HLW pretreated sludge rheograms are compared with PJM simulants. The bounding rheological parameters of the HLW pretreated sludge (Poloski et al. 2003) are defined as Bingham plastic consistency of 30 cP and yield stress of 30 Pa.

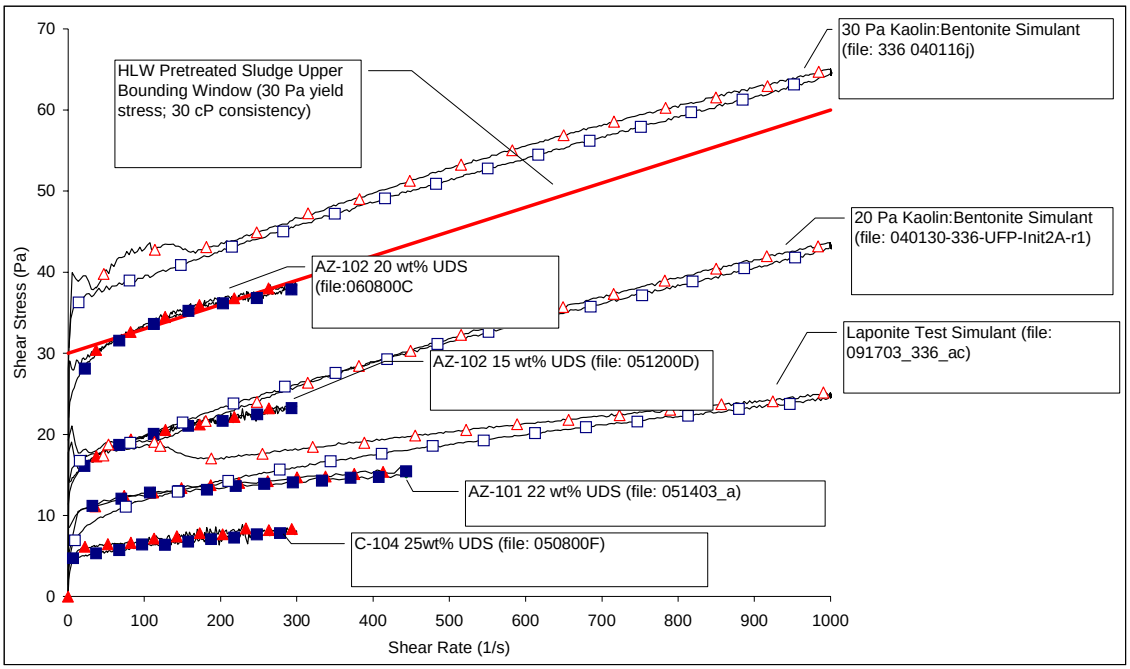

Figure 1-2. Flow Behavior Comparison of PJM Simulants and Actual HLW Pretreated Sludge

In addition to not possessing the target rheological parameters desired for PJM testing, the Laponite composition also does not match other target values given in Table 1-5. The Laponite recipe calls for 1-2 wt% Laponite RD in water where the actual waste is in the 15 to 25 wt% undissolved solids range. And the Laponite simulant consists of particles on the order of tens of nanometers, whereas the actual waste consists of particles in the tens of microns range. These differences may result in varying turbulent flow behavior in the PJM mixing cavern. For these reasons, a more representative particulate slurry was developed to enhance confidence in the PJM testing results. Unfortunately, this simulant is opaque.

The particulate simulant developed consists of a mixture of kaolin clay (EPK Feldspar Pulverized) and bentonite clay (WYO-Ben Big Horn CH-200) in water. To meet the WTP bounding parameters of Bingham plastic consistency of 30 cP and yield stress 30 Pa, a recipe was developed using these two clays. The recipe calls for a composite of 80% kaolin and 20% bentonite mixed with water to a loading of approximately 27 wt%. Water is then added to the simulant to adjust the rheological parameters to other target values. Table 1-6 compares these simulants with actual waste at various solids loadings to target 30+ and 20 Pa yield stress. A summary of the measured rheological parameters for significant CRV prototype tests and sparging tests is shown in Table 1-4. In addition, the bentonite/kaolin simulant shear strength behavior was observed to agree with Speers et al. (1987) correlation for drilling muds.

Table 1-6. Rheological Model Fits for CRV Prototype PJM Simulants at Ambient Temperature

Test Group-Test Sequence	B-7	E-2	E-4
Date	**12/11/03**	**2/11/04**	**2/17/04**
Bingham Plastic:			
- Bingham yield stress (Pa)	17.5	32.9	32.8
k - Bingham consistency coefficient (cP)	20.2	17.8	17.2
Herschel-Bulkley:			
- yield stress (Pa)	15	28	26.4
k - Herschel-Bulkley consistency coeff. ($Pa \cdot s^{-b}$)	0.274	0.35	0.522
b - Herschel-Bulkley power law exponent	0.7026	0.615	0.544

1.7 DISCREPANCIES AND FOLLOW-ON TESTS

While the Test Specification required tests with Laponite as simulant to visualize the gas holdup and release process, testing was done only with kaolin:bentonite simulant. This was necessary because the method of simulating gas generation used depended on catalyctic reaction between hydrogen peroxide and particles in the kaolin:bentonite simulant. No other discrepancies have been observed and no follow-on tests are planned.

2.0 DISCUSSION

2.1 INTRODUCTION

The Waste Treatment Plant (WTP) project plans to utilize Pulse Jet Mixer (PJM) technology for tank mixing applications requiring solids mixing, solids suspension, fluid blending, and release of hydrogen gas (H_2). PJM mixing tests have been initiated to provide design information on the operating parameters critical for the uniform movement (total mobilization) of non-Newtonian tank contents. While some mixing designs that rely on PJMs only have been demonstrated to be successful in mixing the vessel contents, due to impact on plant design and operating costs, hybrid-mixing designs utilizing PJMs and air spargers have also been investigated and shown to fully mobilize vessel contents. These mixing designs must also be shown to adequately release hydrogen generated during steady state operation and after loss-of-power effects to ensure plant safety.

BNI Process Engineering is utilizing a parametric model approach for providing design guidance for controllable release of flammable gas. Two key inputs to the model are gas holdup (how much gas is retained in the mixed waste during normal, continuous PJM operation) and gas release rate (how quickly gas is released upon PJM restart after a period of no mixing). Estimates of these two key parameters are being obtained from theoretical models and available data; however, these sources are limited. Scaled testing is required to validate models and obtain qualitative and quantitative data that will greatly strengthen the technical basis and defensibility of the parametric model inputs. This data will be used by Engineering to generate a defensible design basis for a selected PJM operating mode that will effectively assure the release of H_2 from the waste.

Test Specification 24590-TSP-RT-03-011, Rev. 0 [1] provided requirements for performing GR&R tests in the scaled Concentrate Receipt Vessel (CRV) mockup at Savannah River National Laboratory. These tests were to assess the volume fraction of gas retained in the simulant during continuous gas generation and steady state PJM operation (i.e., gas holdup tests), and the gas release characteristics (volume and rate) after the restart of mixing following a stoppage (i.e., gas release tests). The Test Plan is provided in Ref. [2]. This report specifically summarizes kaolin:bentonite clay simulant gas holdup and gas release tests completed in the CRV prototype vessel using near-final design configurations and operating conditions.

Testing beyond the scope of this task, i.e., other activities in the PJM area, includes bench-scale development activities and experiments in PJM vessels covering a range of configurations and scales, all using non-Newtonian waste simulant. The basis for scale-up of the GR&R results is not fully reviewed and could not be included in this document.

2.2 PRINCIPLE AND APPROACH

To assess gas holdup and gas release in PJM tanks, gas bubbles are generated *in situ* in the simulant. The gas bubble generation technique is based on the decomposition of hydrogen peroxide (H_2O_2) on catalytic surfaces according to the following reaction:

Equation 2-1 $$2H_2O_2 \leftrightarrow 2H_2O + O_2$$

Once sufficient H_2O_2 has decomposed to supersaturate the simulant in O_2, bubbles nucleate and existing bubbles grow. Further decomposition of H_2O_2 leads to additional bubble nucleation and/or bubble growth as O_2 diffuses through the simulant to the bubbles. Generated gas will be retained or released depending on many factors, including the degree of mixing in the system, the retained gas volume fraction, the size of bubbles, and simulant rheology.

In gas holdup tests, H_2O_2 solution is added continuously for a period of time while the PJM system is operated normally to establish a constant gas generation rate. At steady state, the rate of gas generation equals the gas release rate (e.g., from bubbles migrating to the surface), and the steady-state gas volume fraction is termed the gas holdup. In gas release tests, the mixing system is shut down after an amount of H_2O_2 solution is added to allow gas bubbles to be retained in the quiescent simulant. The release of gas upon restart of the mixing system is tracked to assess gas release volumes and rates.

The primary data obtained in gas holdup and gas release tests are measurements of the simulant surface level as a function of time. Through independently established correlations, the level measurements are used to calculate retained gas volume and gas volume fractions. The gas volume fraction α referenced to the initial simulant volume is defined as

Equation 2-2 $$\alpha = \frac{V_{gas}}{V_o} = \frac{V_{gas}}{V_{sim} + V_{sol}}$$

where V_{gas} is the volume of retained gas (e.g., O_2 bubbles), and the total initial slurry volume V_o includes the bubble-free simulant volume V_{sim} and the volume of H_2O_2 solution V_{sol}. In many cases V_{sol} is negligible compared with the large volume of gas-free simulant. However, in gas holdup experiments where H_2O_2 solution is added continuously for an extended period of time, a correction is made for the added solution volume.

According to the expected reaction stoichiometry (shown in Equation 2-1), two moles of H_2O_2 decompose to produce 1 mole of O_2 and 2 moles of H_2O. Using this relationship, the nominal H_2O_2 solution concentration (30 wt%), and ideal gas law considerations, the equivalent volumetric rate of O_2 gas generation can be determined for a given rate of H_2O_2 decomposition. Assuming instantaneous H_2O_2 decomposition or a steady process where a steady-state concentration of H_2O_2 is established in the slurry, O_2 gas is generated at a rate equivalent to H_2O_2 introduction. The latter is assumed to occur in gas holdup experiments, and reported steady-state volumetric gas generation rates (at 22º C and 1 atm) are calculated from measured H_2O_2 injection rates. Normalizing the gas volume generation rate by the volume of simulant in the vessel gives the specific volumetric gas generation rate (volume of O_2 gas/volume of simulant/time).

2.3 QA PROGRAM

This work was conducted in accordance with the RPP-WTP QA requirements specified for work conducted by SRNL as identified in DOE IWO M0SRLE60. All instrumentation used in this test program was calibrated according to 1Q Manual, Sec. 12-1, "Control of Measuring and Test Equipment."

2.3.1 CRV Vessel and PJM Configuration

This section describes the scaled CRV vessel and the final PJM configurations. The 168-inch-diameter, full-scale CRV tank was represented by a 40.125 -inch-ID clear acrylic vessel. The geometric scale factor was ~ 4.0. The scaled CRV prototypic test vessel was 76 ±1 inches tall with a ~2:1 elliptical dish head made out of stainless steel. The final, selected PJM arrangement is the so called "Chandelier" arrangement, Figure 2-1. Here, the charge vessels are positioned along the vessel wall along radial centerlines between PJMs. This is to ensure a flow distribution as close to symmetrical as possible.

All of the PJMs for the final, selected CRV prototype of the "Chandelier" arrangement were constructed from 8-inch-diameter (8.329-inch ID) schedule 10 stainless steel pipes with the end connected to an approximately 60° angle cone truncated to a 1.5-inch-diameter collar to which the nozzles were fitted. Figure 2-2 is a drawing of the PJM assembly. The cylindrical section of the PJMs was 37 ±1 inches tall; this corresponds to a PJM height scale factor of ~ 4.32. The difference between the CRV tank dimension scale factor and the pulse tube dimension scale factor was due to the need to use standard pipe sizes for procurement expediency. However, the volume expelled from the PJMs was consistent with the CRV vessel scale factor of ~ 4.0.

The center PJM nozzle (Figure 2-3) was constructed from a drilled stainless steel pipe cap attached to a 60° cone and was pointed straight down toward the center of the tank bottom and raised approximately 2 inches off the bottom. Two types of perimeter PJM nozzles were used. One, (Figure 2-4 – 1.05-inch ID shown) was angled 45° (using welded pipe sections) from the vertical; and the other (Figure 2-5) was angled 135° from the vertical. Both were directed radially outward from the tank center and raised approximately 2 inches off the tank floor.

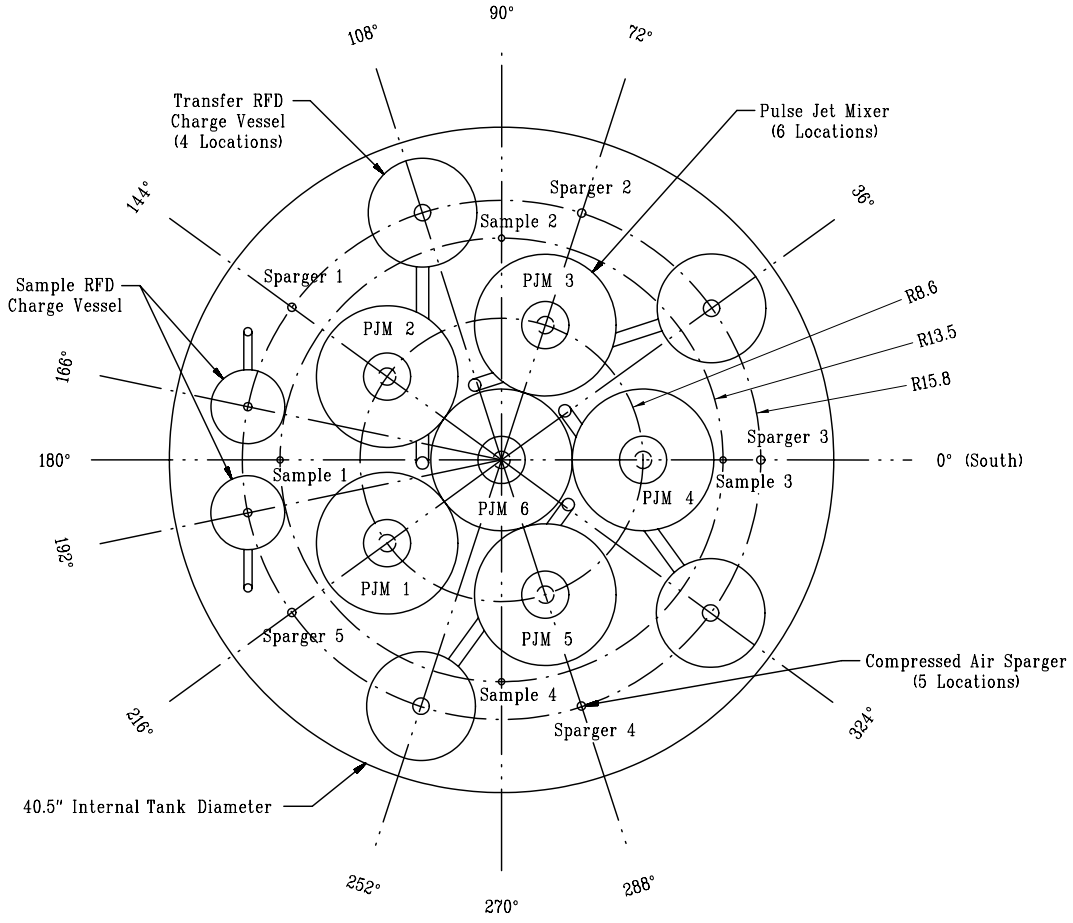

Figure 2-1. Top View of the CRV Prototypic Test Stand Showing Nominal Dimensions

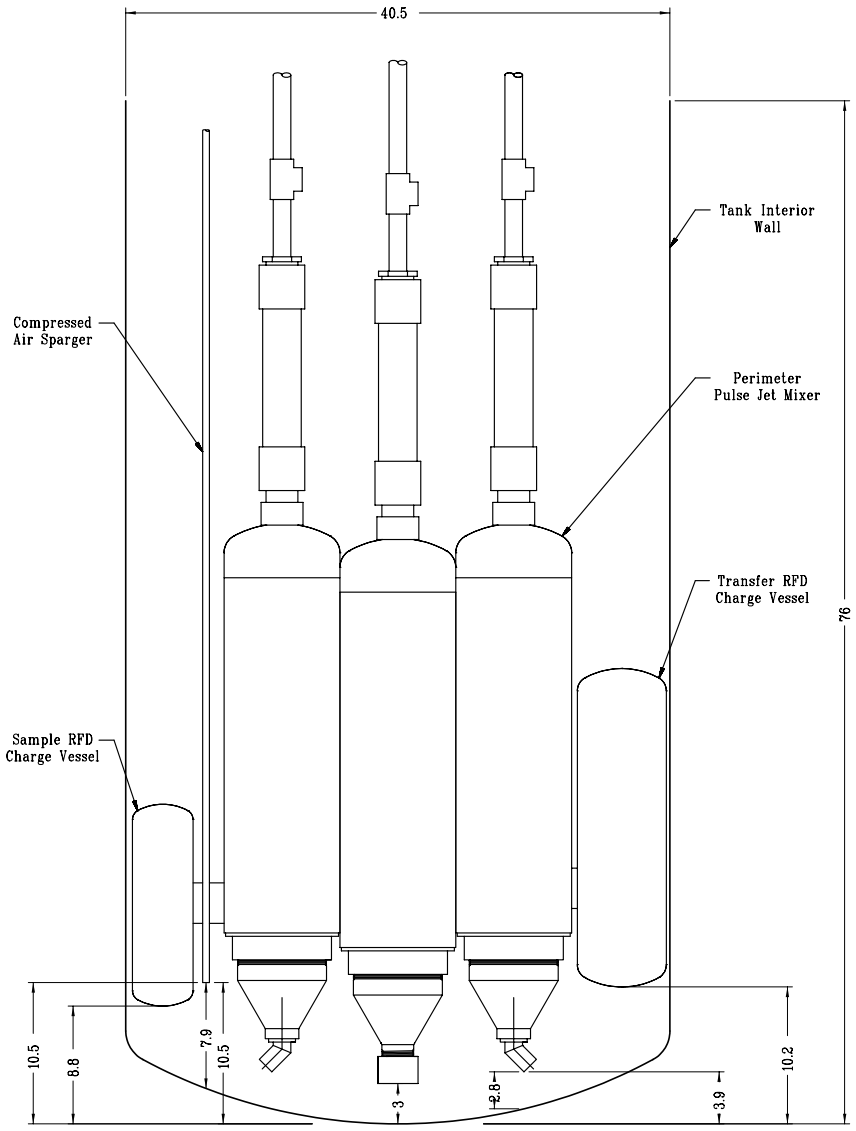

Figure 2-2. Plan View of the CRV Test Stand Showing Nominal Dimensions

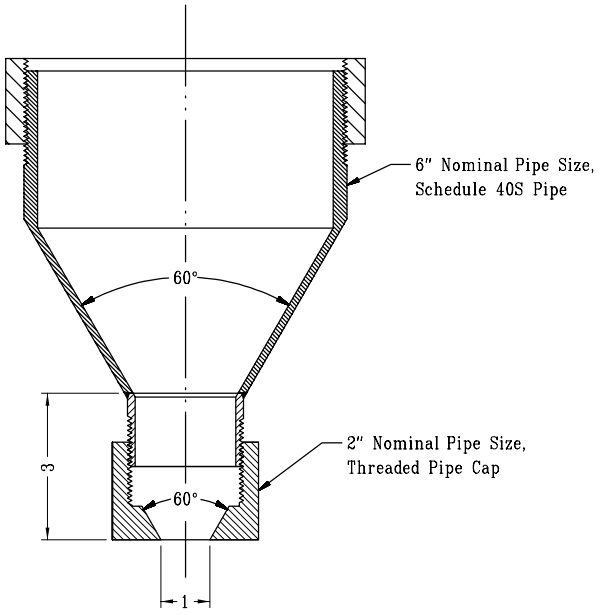

Figure 2-3. Center Nozzle Showing Nominal Dimensions for 1-inch Nozzle

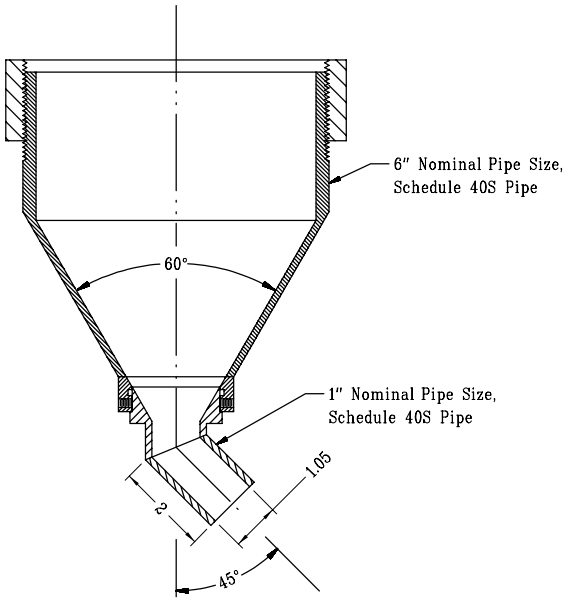

Figure 2-4. 45° Nozzle; Showing Nominal Dimensions for I-inch Nozzle

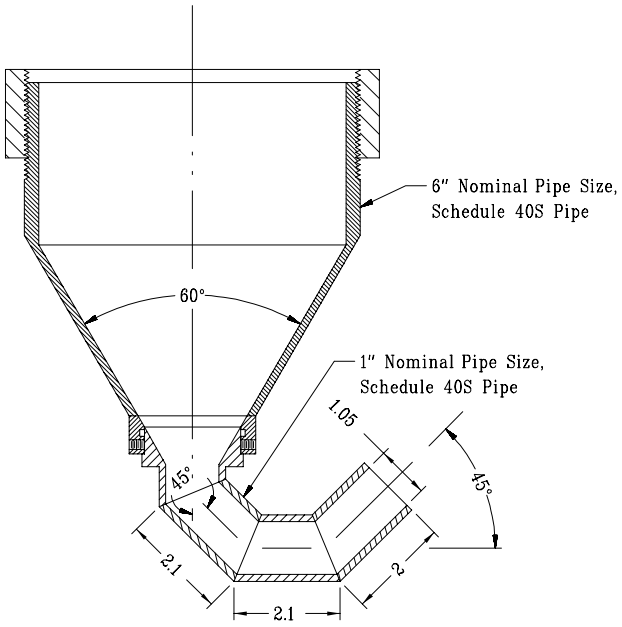

Figure 2-5. 135° Nozzle Showing Nominal Dimensions for 1-inch Nozzle

Only two combinations of nozzles were used. The first, which will be called the Down Nozzle Configuration, consists of five 45º downward facing nozzles and one center down nozzle. The second, which will be called the Up/Down Nozzle Configuration, consists of three 135º Nozzles at PJMs 1, 2, and 4, two 45º down nozzles, and one center down nozzle.

Tests using spargers were performed using an array of 5 spargers at a pitch circle of 31.6 in. The spargers were located approximately at the center of the open regions between the charge vessels, as shown in Figure 2-6. All sparger tubes were made from 0.5-inch-OD (0.37 inch ID) stainless steel tubing, and the lower ends of the sparger tubes were approximately 10.5 inches above the bottom of the tank as measured from the tank bottom. The sparger flow rates were individually controlled with throttle valves and measured with rotameters for equal flows. The total air flow was measured with a Kurz mass air flowmeter and recorded on the data acquisition system.

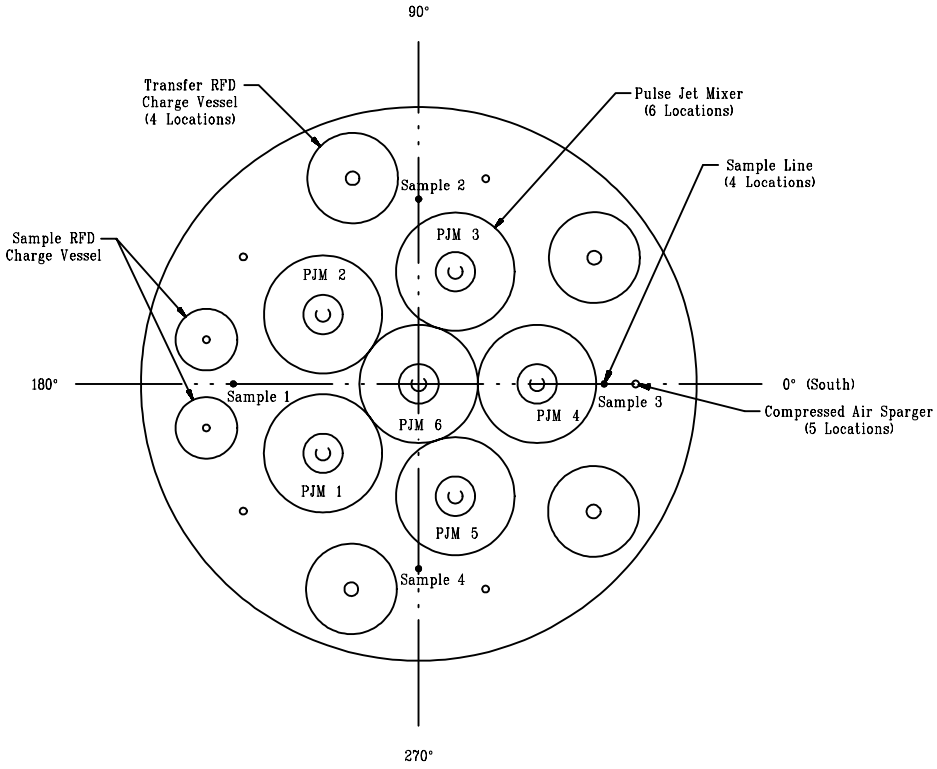

Figure 2-6. Sparger and Sample Line Locations in Final "Chandelier" Arrangement

2.3.2 System Operation and Data Acquisition System

Unlike conventional PJMs, whose operation is regulated by JPPs driven by compressed air, the prototype test systems used a series of solenoid valves and a combination of an air compressor and a vacuum pump to simulate the drive and suction phases of PJM operation. These operations were controlled through a control logic program using Labview software that turns the appropriate solenoid valves on and off at specified time intervals. The duration of each phase, the applied pressure, and the vacuum are all variables that can be independently varied to simulate the operation of the PJMs. The PJMs were operated at a specific average nozzle velocity ($\bar{u}_{disch}$), which is defined as

Equation 2-3

$$\bar{u}_{disch} = \frac{\Delta H}{\Delta t} * AR$$

where ΔH is the length of the PJM stroke, Δt is the time for achieving the stroke, and AR is the area ratio of the PJM to the nozzle.

The drive distance was based on volume scaling to the plant, given the linearly scaled PJM diameter. The drive distance was approximately 27-inches for Test B-7 and E-4. When the 8-inch PJM was used to simulate the 6-inch PJM as in Test E-2, the drive distance was reduced to 16-inches. During the drive portion of the cycle, the drive time was set so that the nominal velocity was achieved, knowing the initial and final simulant levels inside the PJM. The cycle time was controlled to be one over the scale factor of the plant cycle time and includes times for venting and quiescent periods.

During each mixing test, several variables such as PJM liquid levels, pressures, tank temperatures, air supply pressure, and total sparger air flow rate were monitored continuously and recorded digitally on a computer. The liquid/slurry level inside each of the PJMs was measured using Drexelbook capacitance level probes and transmitters. The level probes were calibrated against a tape measure on the side of the tank whenever there was a change of simulant (slurry yield stress changed). Compressor and vacuum supply pressures and the pressure inside each PJM were monitored using flush-diaphragm Endress+Hauser ceramic pressure transducers, installed at a pipe Tee fitting near the top of the PJM. Data from these sensors were recorded on a laboratory computer, running Labview software at sampling times of approximately 0.1 seconds.

All instrumentation used in this experiment were calibrated and conformed to QA procedures for Measuring and Test Equipment, as required by the QA program in the Task Plan.

2.3.3 Tank Level Measurement

Measurement of the tank level by means of a DISTO PRO laser sensor was the primary means of determining the gas fraction. The laser sensor was positioned above the tank and the laser light was directed at the middle of a annular sector between the PJM and the tank wall. During quiescent periods (no PJM or sparger operation), the sensor reading represented the average tank level. However, during PJM operation, the minimum level (suction phase) was recorded to track the gas release rate. The pulsing was also stopped at regular intervals to establish a quiescent tank level. The laser sensor readings were checked against readings taken with measuring tapes.

2.4 TEST METHODS

2.4.1 Gas Holdup Tests

Two types of holdup tests were run. In the overnight growth or batch injection test, a given mass of 30 wt% hydrogen peroxide was injected at a high rate through a sampling line near the bottom of the tank, while the PJMs and spargers were operating, and after completion of injection, the PJMs and spargers continued for 15 minutes to ensure full mixing, and then stopped. The tank level was then monitored overnight. In the steady state gas generation rate test, a given hydrogen peroxide injection rate is maintained for approximately 100 minutes to determine a steady state gas fraction. The injection was stopped and the PJMs and spargers continued to operate for another 100 minutes and then stopped. The gas fraction buildup was then monitored. Table 2-1 provides the amount and injection rates of hydrogen peroxide for the three CRV mixing test configurations.

Table 2-1. Hydrogen Peroxide Injection Rates for Gas Holdup Tests

Test Group	Test Sequence	Test type	Total H_2O_2 mass injected, gm			Injection rate, ml/min		
			Batch inj.	Slow rate	Fast rate	Batch inj.	Slow rate	Fast rate
B	7	Gas holdup	1323	1365	2877	220	8.4	23
E	2	Gas holdup	663	N/A	2180	220	N/A	22.8
E	4	Gas holdup	663	N/A	2000	220	N/A	22.8

2.4.2 Gas Release Tests

After the holdup tests reached a steady state gas fraction, the mixing systems were started and stopped to determine the quiescent tank level, until the initial gas free tank level was reached.

2.5 TEST RESULTS

2.5.1 Gas Retention Under Various H_2O_2 Injection Rates

This section illustrates the rate of increase of gas holdup and the maximum holdup attained in the event that the CRV PJMs and spargers are out of operation for an extended period of time. In the overnight growth test, a given mass of H_2O_2 was injected into the simulant at a high rate, typically 220 ml/min, mixed well with PJMs and spargers, and then the gas is allowed to expand overnight. This represents the upper end of the gas retention since no gas was released during the batch injection. The results of tests on the three CRV mixing configurations are as follows:

In Test B-7 ,with PJMs only configuration (1.5" nozzles, upward/downward facing nozzles), 1327 gm of 30 wt. % H_2O_2 solution was injected into the simulant at a rate of 220 ml/min, with the PJMs running for 15 minutes before they were stopped. The rate of increase in gas fraction is shown in Figure 2-7. A maximum gas fraction of 16.3% was reached in about 3 hours.

In Test E-2, with a hybrid-mixing system (1" downward facing nozzles, 3 spargers @ 1.9 scfm ea.), 663 gms of H_2O_2 solution was used in the batch mode. A maximum gas fraction of 5.5% was reached in about 60 minutes.

In Test E-4, also with a hybrid mixing system (1" upward/downward facing nozzles, 5 spargers @ 1.8 scfm ea.), the same amount of H_2O_2 solution was injected as in Test E-2. A closely similar gas fraction (4.9%) was reached in Test E-4 in 50 minutes.

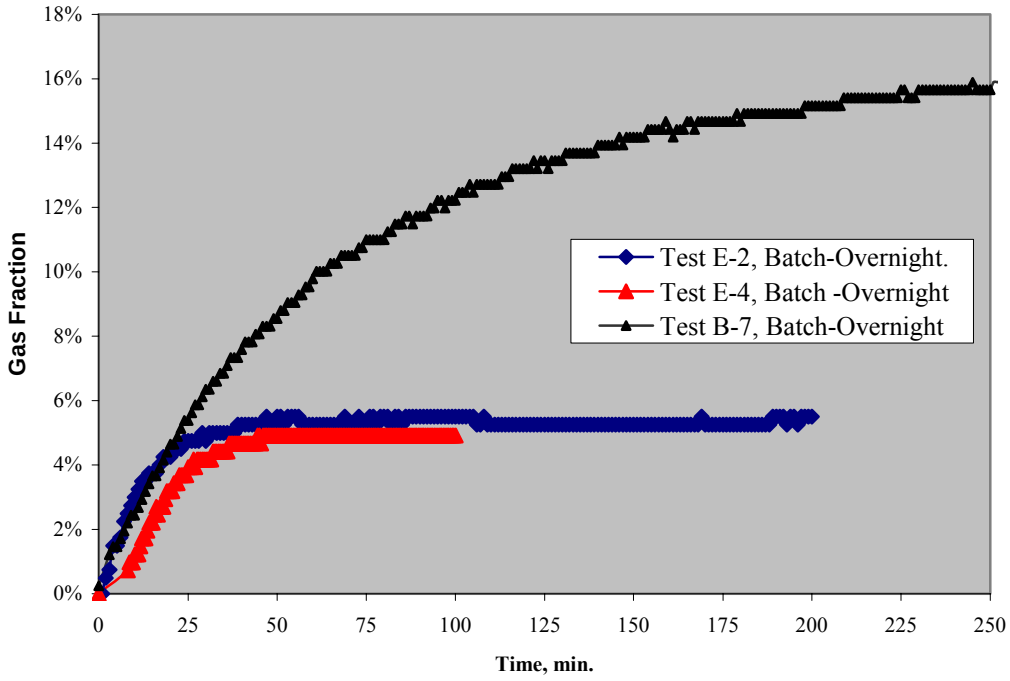

Figure 2-7. Gas Fraction as a Function of Time after Batch Injection of H_2O_2 in Three Gas Holdup Tests in the CRV

2.5.2 Gas Holdup in Normal Operations

This section demonstrates that gas is released regularly and controllably in normal operation of the CRV prototype system, resulting in relatively low gas holdup.

In Test B-7, two rates of H_2O_2 injection were used. In the slow injection test, 1365 gm of 30 wt % H_2O_2 was injected at a rate of 8 ml/min, while for the fast injection test, 2860 gms of H_2O_2 solution was injected at twice the rate of the slow rate (or 16 ml/min). For both slow and fast injection tests, the PJMs were operated continuously during the approximately 100 minutes of injection and 100 minutes after the end of the injection. Figure 2-8 shows that during operation of the PJM/spargers, the steady state gas fraction did not exceed 0.5%, which was reached in 100 min. or a gas generation rate of 0.005%/min. After the PJMs and spargers were turned off, the gas fraction started to rise again. The rate of rise of the gas fraction was higher for the slow injection test (17.6% in 4.6 hrs) than for fast injection test 23.6% in 13 hrs). This was probably due to the kinetics of gas generation in the particulate simulant. However it is the total amount of H_2O_2 injected which determines the maximum gas holdup value.

Figure 2-9 plots the measured gas volume fraction as a function of time during and after a gas holdup test in the CRV scaled prototype with a hybrid-mixing system (Test E-2). At elapsed time 0, a hydrogen peroxide addition rate (22.5 ml/min of 30 wt % hydrogen peroxide) was established to provide an effective O_2 gas generation rate of 0.028 vol%/min (normalized to atmospheric pressure and 22ºC) and a maximum steady state gas fraction of 2%. The specific gas generation rates used in the prototype experiments exceed the expected maximum actual waste gas generation rates (e.g., 2-4 vol%/day) by a factor of ~10 or more.

A steady-state gas fraction of ~2 vol% was attained after ~70 minutes. After an elapsed period of 103 minutes, the PJMs and spargers were turned off. Figure 2-9 shows the gas holdup increasing to 7.5% after about 27 minutes.

In Test E-4, a continuous injection of 27 ml/min of 30 wt% solution was used with the PJMs and spargers on for 108 minutes (Figure 2-10). A rate of gas fraction increase of 0.02%/min. was observed with a maximum gas fraction of 1%. After the PJMs and spargers were turned off, a maximum gas fraction of 5.9% was reached.

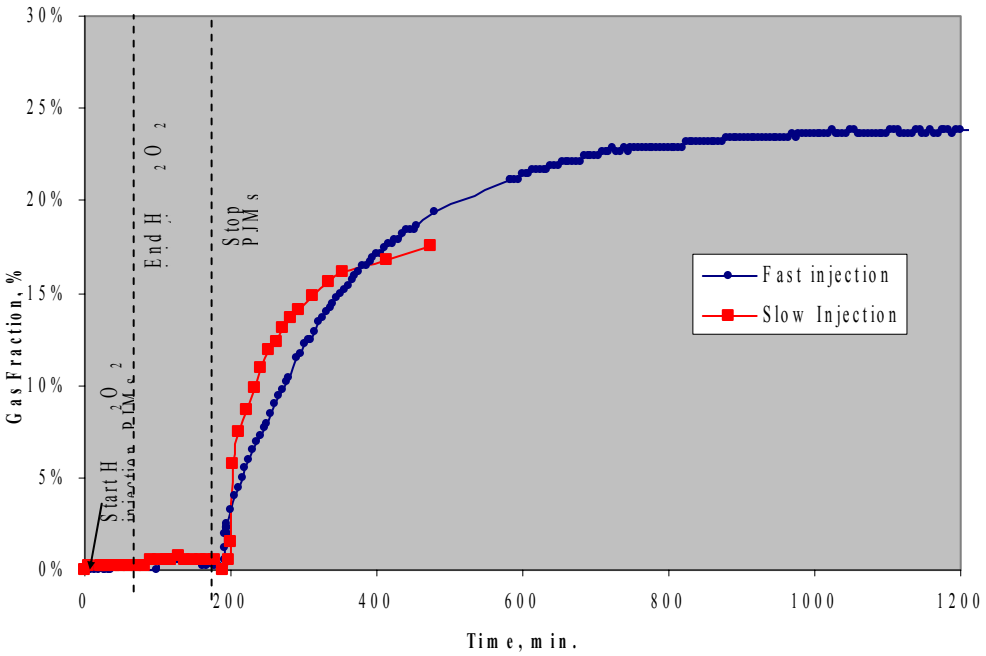

Figure 2-8. Gas Fraction as a Function of Time During and After Continuous H_2O_2 Injection in the CRV Prototype (Test B-7).

Events are marked on the plot by vertical lines.

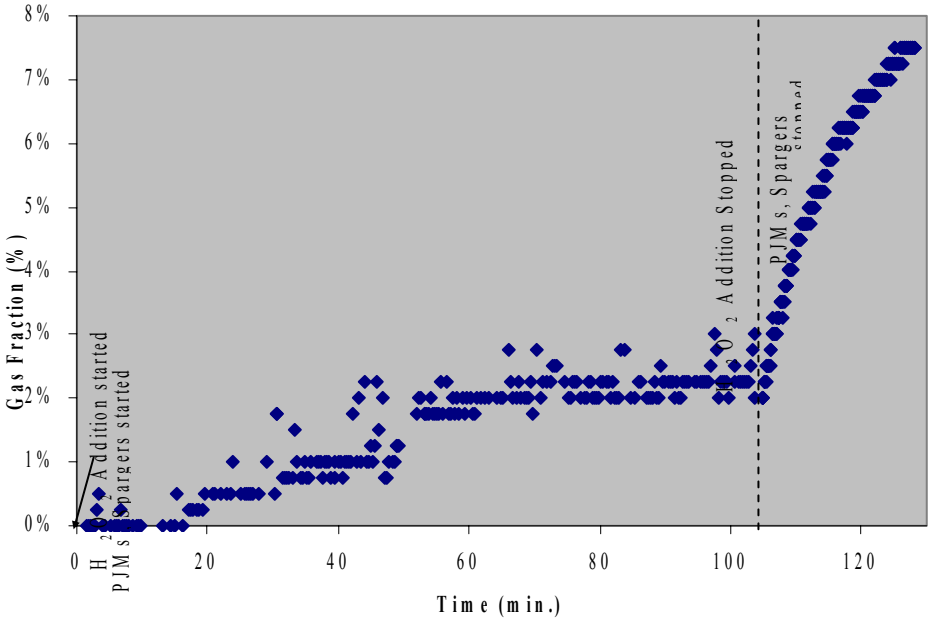

Figure 2-9. Gas Fraction as a Function of Time During and After Continuous H_2O_2 Injection in the CRV Prototype (Test E-2).

Events are marked on the plot by vertical lines.

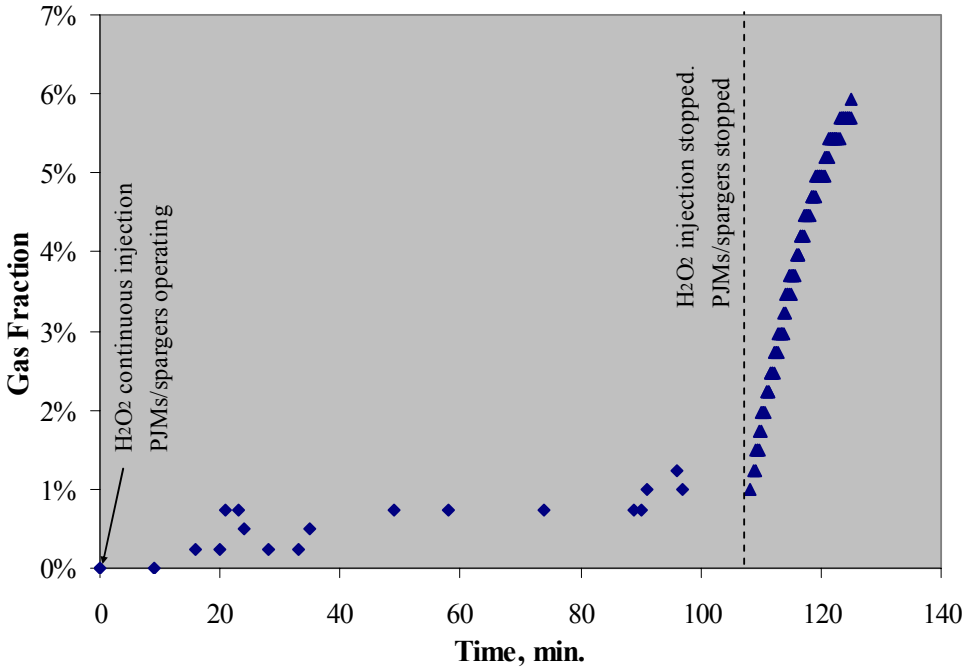

Figure 2-10. Gas Fraction as a Function of Time During and After a Gas Holdup Test in the CRV Prototype (Test E-4).

Events are marked on the plot by vertical lines.

Table 2-2 provides the experimental gas release and holdup values in the CRV test stand under normal operating conditions.

Table 2-2. Experimental Gas Release and Holdup in the CRV Test Stand Under Normal Operating Conditions

Configuration	Experimental Gas Generation Rate (vol%/min)	Measured Gas Holdup (vol%)
Test B-7: 6 PJMs with three 1.5" upward nozzles and three 1" downward nozzles	0.005	0.5
Test E-2: 6 PJMs @ 12 m/s nozzle vel., downward nozzles, 3 spargers at 1.9 scfm ea.	0.028	2.0
Test E-4: 6 PJMs with three 1" upward nozzles and three 1" downward nozzles, 5 spargers at 1.8 scfm ea.	0.02	1

2.5.3 Gas Release after Mixing System Restart

During a plant shutdown in which the air supply to PJMs and spargers is interrupted, generated gas is expected to accumulate in the quiescent waste. In the extreme, all gas generated during the outage will be retained in the waste slurry. Upon restart of the mixing apparatus, accumulated gas is likely to be released. The release rate is dependent on many factors including waste rheology and mixing energy. Examples of gas release from gelled clay resulting from the restart of PJMs and spargers in the CRV prototype are provided.

In Figure 2-11, gas accumulated in Figure 2-7 (overnight growth test), Test B-7, of up to 18% was released by operating the PJMs. The tank level was tracked by measuring the lowest tank level during the suction phase of the PJM cycle. After 10 cycles, the PJMs were stopped and the tank level stabilized at a value of 2.7%. After another 20 cycles, the PJMs were stopped and the gas fraction reached 1.5%.

In Figure 2-12, the gas fraction at the end of the overnight growth holdup test, Test E-2 (Figure 2-8) of 5.5% decreased to 0.7% after 10 cycles and then to 0.25% after 20 cycles of PJM and sparger operation. In Figure 2-13, the gas accumulated in overnight growth Test E-4 (Figure 2-8) of 4.7% was totally released after 20 cycles (8 min.) of PJM and sparger operation.

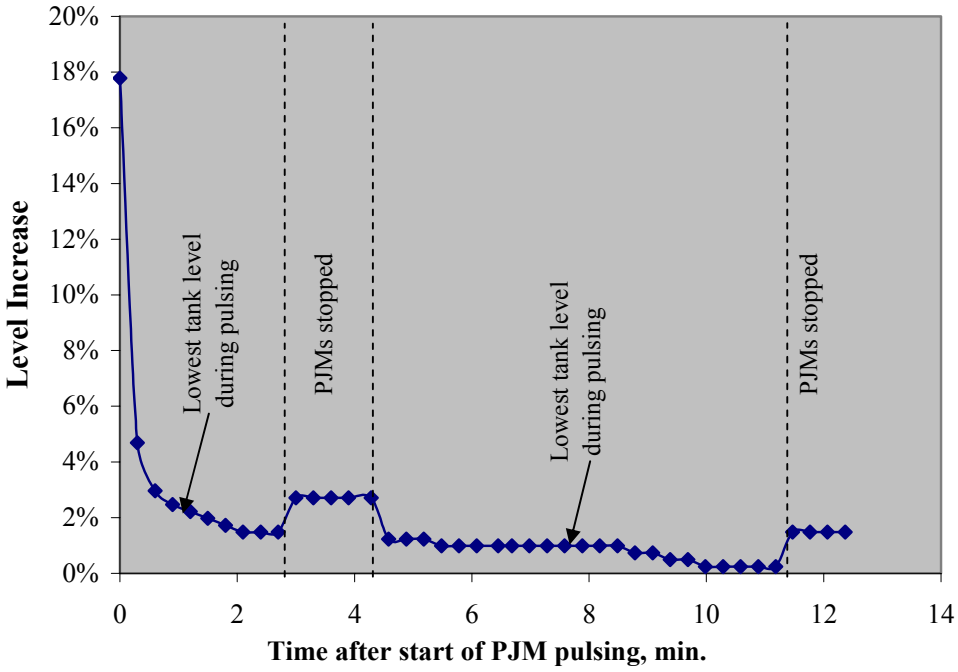

Figure 2-11. Gas Release from Gelled Clay in the CRV Prototype after Gas Holdup in Figure 2-7 (Test B-7, six PJMs, 3 upwards 1.5" nozzles and 3 downwards 1.5" nozzles)

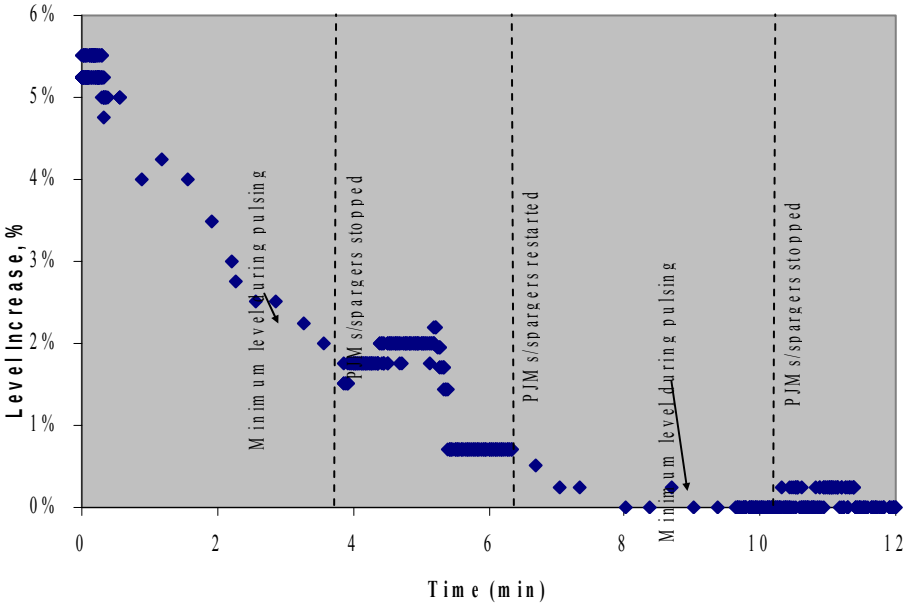

Figure 2-12. Gas Release from Gelled Clay in the CRV Prototype after Gas Holdup in Figure 2-7 (Test E-2, six PJMs, 1" downward nozzles+ 3 spargers @ 1.9 scfm ea.)

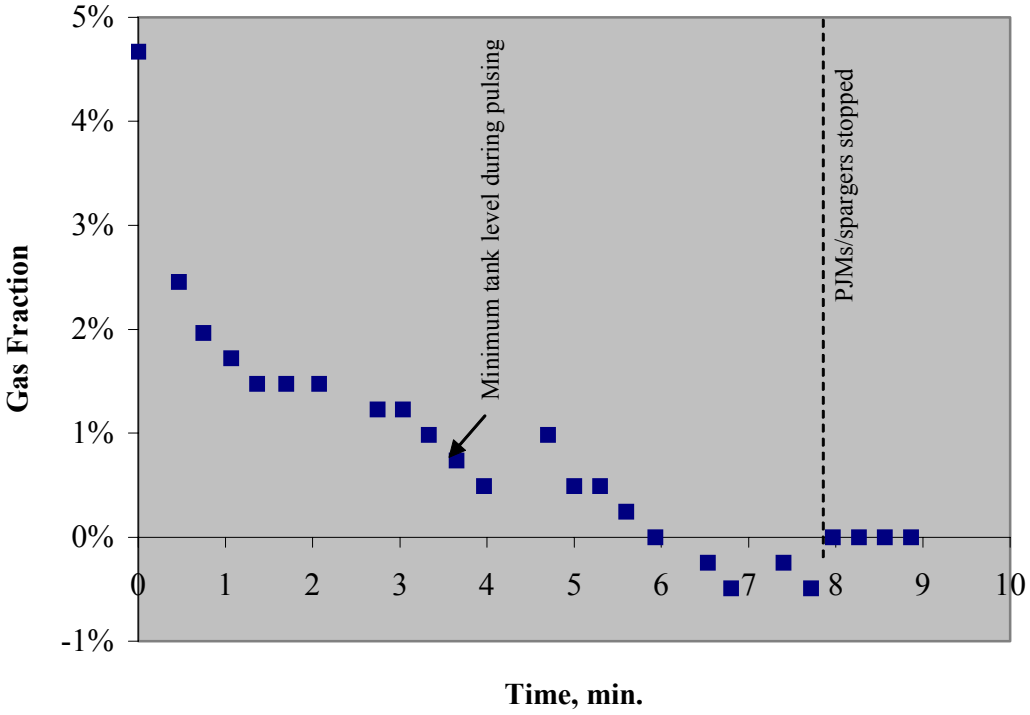

Figure 2-13. Gas Release from Gelled Clay in the CRV Prototype after Gas Holdup in Figure 2-7 (Test E-4, six PJMs, 3 upwards 1.5" nozzles and 3 downwards 1.5" nozzles)

In Figure 2-14, starting at an initial gas fraction (~7.5 vol% after the continuous injection test, Figure 2-9) with the PJMs and spargers operating, gas was released to a retained gas volume fraction of ~1.25 vol% in 22 min, decaying with time. Then the PJMs and spargers were turned off. The gas fraction again increased to a steady state value of 1.5% after 30 minutes. The spargers only were turned on, which did not completely release the gas, allowing a residual holdup of 1%. When the PJMs were turned on, all of the gas was released.

In Figure 2-15, starting at an initial gas fraction (~5 vol% from continuous injection test, Figure 2-10) with the PJMs and spargers operating, gas was released to a retained gas volume fraction of ~0.25 vol% in 22 min. Then the PJMs and spargers were turned off. The gas fraction again increased to a steady state value of 0.5% after 30 minutes. The spargers only were turned on, which completely released the gas.

In comparing Test E-2 and Test E-4 (Figure 2-14 and Figure 2-15, respectively.), it is interesting to note that after the initial release with both the PJMs and spargers on and the holdup attained a residual value, not all the gas was released in Test E-2, but all the gas was released in Test E-4 with the spargers operating only. This is evidently due to a larger air volumetric flow in Test E-4 (5 spargers, 9 scfm) than in Test E-2 (3 spargers, 6 scfm).

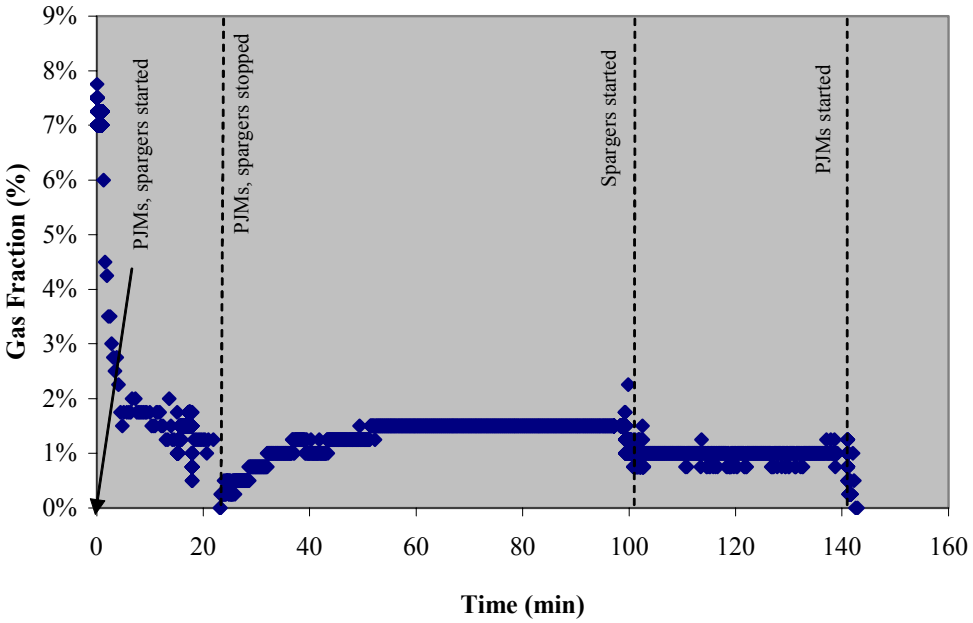

Figure 2-14. Gas Release from Gelled Clay in the CRV Prototype after Gas Holdup in Figure 2-9 (Test E-2, six PJMs + three spargers at 1.9 scfm ea)

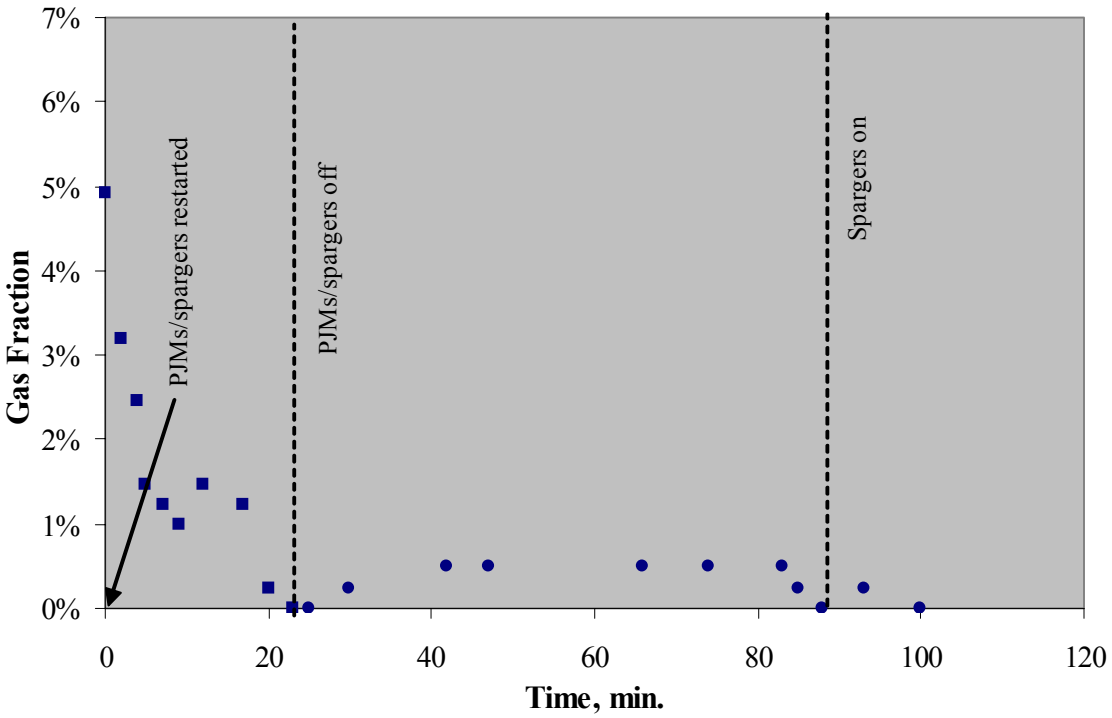

Figure 2-15. Gas Release from Gelled Clay in the CRV Prototype after Gas Holdup in Figure 2-10 (Test E-4, 3 upward and 3 downward 1.5" nozzles + five spargers at 1.8 scfm ea)

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3.0 REFERENCES

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