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Continuous-Flow Stirred-Tank Reactor 20-L Demonstration Test: Final Report

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ACRONYMS

AA	atomic adsorption
ALARA	as low as reasonably achievable
ASTM	American Society for Testing and Materials
CERS	Chemical and Energy Research Section
CFF	cross-flow filter
CP	chrome plated
CSTR	continuous-flow stirred-tank reactor
DF	decontamination factor
DNC	delayed neutron counting
ECC	enhanced catalyst component
EPA	U.S. Environmental Protection Agency
EPDM	ethylene propylene diene monomer
HEPA	high-efficiency particulate air filter
HFIR	High Flux Isotope Reactor
HLW	high-level waste
HPLC	high-performance liquid chromatography
I&C	Instrumentation and Controls
ICP-AES	inductively coupled plasma–atomic emission spectrometry
ICP-MS	inductively coupled plasma–mass spectroscopy
KTPB	potassium tetraphenylborate
LI	level indicator
LIC	level indicator/controller
MST	monosodium titanate
NaTPB	sodium tetraphenylborate
NISS	nitrite interference suppressor solution
NIST	National Institute of Standards and Technology
ORNL	Oak Ridge National Laboratory
P&CT	Process and Control Technology
PCV	pressure control valve
PI	pressure indicator
PIC	pressure indicator/controller
QA	quality assurance
RMAG	Radioactive Materials Analysis Group
RMAL	Radioactive Materials Analytical Laboratory
RWPs	Radiological Work Permits
RWWT	recycle wash water tank
SAMs	statistical analysis methods
SP	sample port
SRL	Savannah River Laboratory
SRS	Savannah River Site
SRTC	Savannah River Technology Center
SS	stainless steel
TMP	transmembrane pressure

TPB	tetraphenylborate
TTP	technical task plan
WAC	waste acceptance criteria
WSRC	Westinghouse Savannah River Company
3PB	triphenylborane
2PB	diphenylborinic acid
1PB	phenylboronic acid

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ABSTRACT

One of the proposed methods of removing the cesium, strontium, and transuranics from the radioactive waste storage tanks at Savannah River is the small-tank tetraphenylborate (TPB) precipitation process. A two-reactor-in-series (15-L working volume each) continuous-flow stirred-tank reactor (CSTR) system was designed, constructed, and installed in a hot cell to test the Savannah River–developed process. The system also includes two cross-flow filtration systems to concentrate and wash the slurry produced in the process, which contains the bulk of radioactivity from the supernatant processed through the system. Installation, operational readiness reviews, and system preparation and testing were completed. The first test using the filtration systems, two CSTRs, and the slurry concentration system was conducted over a 61-h period with design removal of Cs, Sr, and U achieved. With the successful completion of Test 1a, the following tests, 1b and 1c, were not required.

The second test, Test 2, was started and completed on schedule and integrated the TPB slurry washing and wash water recycle to the first CSTR. It was completed after 230 h of continuous operation. All of the CSTR systems performed as designed, and cesium and uranium decontamination factors (DFs) were obtained in both CSTRs and in the concentration filtrate. No foam problems were encountered in the system operation during the treatment of about 300 L of radioactive simulated waste. Approximately 25 L of greater than 7 to 10 wt % concentrated, precipitated washed slurry was collected. Eight liters of unwashed concentrated slurry was also produced by the end of the experiment. More than 435 L of decontaminated filtrate was collected from the concentration filtering system.

The strontium DF was obtained in the feed tank when some of the catalyst components were added which contained hydrous metal oxides, known to bind strontium. The slurry washing step was completed four times on four successive batches of concentrated slurry, and the wash water, containing resolubilized sodium TPB, was fed back into the first CSTR. Test 2 used the enhanced catalyst component catalyst system with enhanced (five times higher) palladium. No degradation of cesium DF was detected, and no measurable increase in the amount of TPB breakdown products was observed in any of the samples from any vessels.

1. INTRODUCTION

1.1 REQUEST FOR WORK

Oak Ridge National Laboratory (ORNL) was requested to demonstrate the continuous small-tank tetraphenylborate (TPB) process at the bench scale in Technical Task Request HLW-SDT-TTR-99-03.0, which is shown in Appendix A.1. The process was designed to effectively remove soluble cesium from the storage tank supernatant salt solutions, providing a means of separating large volumes of solution for disposal in saltstone. The precipitated cesium would be contained in a small volume of solids for disposal in glass. Monosodium titanate (MST) was added to adsorb Sr and actinides (Pu, Np, and U). In 1995, batch processing of the supernatant in Tank 48H started using sodium tetraphenylborate (NaTPB) to precipitate cesium. Production of benzene from TPB occurred during processing and led to the cessation of operations. Subsequent testing suggested that the decomposition was likely catalyzed by copper and palladium in the tank supernatant and sludge (other elements may contribute). Elevated temperature, radiolysis, and the oxidation/reduction potential of the vapor/liquid system in the tank also influence the decomposition rate.

The Savannah River High-Level Waste (HLW) Salt Disposition Program was created from a need to find alternatives to the in-tank precipitation process. Subsequently, the ORNL project was initiated as a response to a request for support for the preconceptual design phase for one of the "Short List" alternatives. No previous demonstration existed for operation of the precipitation reaction with material from the Savannah River Site (SRS) tank farm in a continuous fashion. Savannah River requested that ORNL conduct tests to confirm that the two reactor-in-series configuration of continuous-flow stirred-tank reactors (CSTRs) perform the desired separations using simulated salt waste containing ^{137}Cs and ^{85}Sr as radioactive tracers.

One of the design requirements was that the flowsheet be designed for maximum operational flexibility. To do so, it was necessary to incorporate the capacities to add a third CSTR, to change the residence time in each CSTR, to change the amount of excess TPB added to CSTR 1 in the recycle wash water feed, and to add TPB to the second CSTR if required. The design variables that were considered during the planning for the CSTRs were mixing speeds, mixing direction, agitator types, coiling coils, and chemical concentrations. It also had to be determined if the cooling coils could serve as draft tubes.

Because of potential foaming problems, the CSTRs had to be designed for the feed and product streams to enter below the liquid surface to minimize aeration, which causes foam formation. At the time of the equipment design, it was uncertain if an antifoaming agent would be required to control foaming; however, provisions were made for its addition.

1.2 TECHNICAL TASK PLAN

The Technical Task Plan (TTP; the full document is supplied in Appendix A.1) for the planned testing at ORNL was created to provide an experimental evaluation of the decontamination achievable in the small-tank TPB precipitation process for the removal of Cs, Sr, and U from simulated Savannah River supernatant. It also provided for the possibility of assessing the effects of the catalytic decomposition of TPB during operation.

These tests were also designed to provide needed information for the future scaleup of the small-tank TPB precipitation process. Analyses performed during the operation of the process were used to determine interstage conversion and demonstrate that the treated supernatant met the required levels for waste acceptance criteria (WAC) for Cs (<45 nCi/g, total alpha <18 nCi/g) and for Sr (<40 nCi/g). The desired levels of treatment—Cs <40 nCi/g, alpha <10 nCi/g, and Sr <20 nCi/g —are set at 50% of the WAC to allow for changes in process conditions. In this set of experiments, the actinide behavior was followed by analysis of uranium.

The TTP prescribed that the supernatant solution to be used in the CSTR testing was the "average" simulant developed at SRS. During operation of the CSTR, the feed (6.4 *M* sodium), dilution water with MST slurry, TPB solution, and wash water recycle (1.25 *M* sodium) were added separately to CSTR 1 in order to result in operation at a 4.7 *M* sodium concentration in both CSTR 1 and CSTR 2.

1.3 PLANNING, REPORTING, QA/QC, SAFETY, AND ALARA CONCERNS AND REQUIREMENTS

1.3.1 Analytical Methods

The ORNL Radioactive Materials Analysis Group (RMAG) conducted analyses. The RMAG was responsible for the preparation and maintenance of procedures for analytical samples and techniques used. All documentation supplied by RMAG referenced applicable procedures used for this project. The chain-of-custody procedure of the Sample Management Office was followed for all analytical samples provided.

Routine sample preparation and analytical activities were conducted following controlled statistical analysis methods (SAMs). Controlled SAMs at the Radioactive Materials Analytical Laboratory (RMAL) are generally based upon analytical methods established by the U.S. Environmental Protection Agency (EPA), Office of Solid Waste (SW-846 Manual); American Society for Testing and Materials (ASTM); Department of Energy Methods for Evaluating Environmental and Waste Management Samples (DOE Methods Compendium); or other recognized sources. Nonroutine analysis was documented in logbooks or noted on paperwork included with sample request files. Generally, no deviations from standard methods are allowed without prior notification and approval from the group leader. The RMAL uses a flexibility policy compatible with that of the SW-846. This flexibility is allowed for volumes of reagents and standards prepared; deviation from specified glassware and equipment is acceptable only if the laboratory can demonstrate that the substitution does not affect the analysis. Size dilution or concentration must be maintained. Deviations not covered by this flexibility policy (i.e., changes in chemistry of the method) are required to be documented and approved by the group leader using a variance report form. Variance report forms will be filed with the sample data (QAP-X-96-CASD/RML-001 Rev. 2, Sect. 4.1).

High-performance liquid chromatography (HPLC) analysis was used for NaTPB, triphenylborane (3PB), diphenylborinic acid (2PB), phenylboronic acid (1PB), and phenol. Gamma counting was used for cesium (^{137}Cs) and strontium (^{85}Sr). Before analysis by inductively coupled plasma–mass spectroscopy (ICP-MS), U, Pd, and B samples required a digestion by microwave. Atomic adsorption (AA) spectroscopy or ICP-MS was used for potassium analysis. The ORNL Analytical Services Organization cooperated with SRS personnel on the HPLC methods. Standards and procedures for the boron analysis were supplied by Savannah River Technology Center (SRTC).

1.3.2 Measuring and Test Equipment

Various types of measuring and test equipment were used for this task. Electronic balances, calibrated annually by ORNL Plumbing and Electrical personnel, were used for solids and liquid mass measurements to prepare the simulants. Class A volumetric flasks and graduated cylinders were used for simulant makeup. A gamma-counting system consisting of an ORTEC model GMX-45220-P-S intrinsic germanium detector, an ORTEC model 672 counting-

system amplifier, a Canberra Accuspec-A multichannel analyzer card, and Canberra Genie-2000 spectroscopy software was used for in-house gamma measurements of ^{137}Cs and ^{85}Sr. These measurements were used to determine changes in experimental conditions (e.g., excess NaTPB) during the testing.

Instrumentation used in the CSTR system was calibrated before startup and recalibrated on a routine schedule. Calibration records were maintained in registered logbooks. A list of instruments and corresponding calibration schedules was generated after the process flow diagram and control strategy were reviewed and approved by SRS and Oak Ridge personnel.

1.3.3 Documentation

Requirements and guidance of the *CTD Records Management Plan* (ORNL/CF-97/42) and *ORNL Records Management and Document Control* (ORNL-IO-003) were followed for controlling documents, such as procedures and the management of records generated for the project. The ORNL personnel performing the work recorded all experimental instructions, observations, results, and calculations in a registered notebook. Copies of the notebook pages and analytical reports will be provided to SRS at the conclusion of the work. The results of these experiments are documented in this ORNL report. All records will be managed under the Records Management Plan for the Chemical Technology Division (ORNL/CF-97/42).

1.3.4 Quality Assurance

The quality assurance (QA) activities associated with this task were covered in the QA documentation developed for the testing. The testing and the processes used were described in ORNL/CF-99/5, *Technical Task Plan for Testing Two CSTRs in Series for the Small Tank TPB Precipitation Simulation*, provided in Appendix A.1.

The management of activities and laboratory and facility operations of the Chemical and Energy Research Section (CERS) is described in ORNL/CF-97/27, *Quality Management Plan for the Chemical and Energy Research Section of Chemical Technology Division*. The development of the QA plan specifically for bench-scale reactor studies with TPB was the result of the project evaluation conducted by the principal investigator in accordance with CTD QA-3, *Procedure for Quality Assurance Planning*. This QA plan shall take precedence should any conflict arise with ORNL/CF-97/27.

The systems and procedures described in the CERS Quality Management Plan (ORNL/CF-97/27) were implemented for this project to the extent they were applicable. The applicable ORNL, division-level, and section-level procedures are listed in Appendix A of the QA plan.

In addition to developing a Problem Safety Summary for the CSTR 20-L demonstration, the principal investigator was responsible for initiating procedures (i.e., workaids) for operations and activities that were specific to the project.

1.3.5 ALARA

The ALARA Plan was developed to ensure reasonable efforts to minimize radiological impact to ORNL (as low as reasonably achievable, or ALARA) —both in radiation exposure to the operating personnel and in generation of radioactive waste —by examining the potential hazards associated with all planned activities and minimizing the dose rates and times for all handling of source material, including waste disposal. The ALARA Plan was used in conjunction with Radiological Work Permits (RWPs). Specially designed remote tools and shielding were employed to the extent reasonably possible. Pretest dose estimates were made to facilitate planning.

All operations were reviewed and directed by the appropriate safety and supervisory personnel, and all activities and material transfers were carried out under approved operating procedures. All personnel used the required protective equipment, including company-issued anticontamination clothing, personal monitors, and respirators, where appropriate. The ALARA principle applied to all material handling, and the cumulative radiation dose to each worker was monitored regularly to ensure that no worker approached the allowable limit.

Although this particular process was operated for the first time in this area, all personnel planning and conducting this project had extensive experience in both chemical processing activities and the handling of radioactive materials and were thoroughly familiar with the particular laboratory and hot cell facilities. Furthermore, the levels of radioactivity handled in these tests/facilities were an order of magnitude lower than those handled safely and successfully in previous projects. Consequently, the employment of established operating procedures limited personnel hazards—for both radiation exposure and the spread of contamination—to relatively low levels.

2. DESIGN OF CSTR SYSTEM

2.1 BASIS FOR DESIGN

The basis for the design of the ORNL CSTR sizes was derived from the preconceptual design bases[1] that contained reactors of 15,000-gal working volume. Assuming 20-L bench-scale vessels with 15-L working volume, scales were 1 gal = 1 mL and 1 gal/min = 1 mL/min. For the ORNL bench-scale system, the flow rates of the various feeds were sized accordingly. The following formation was part of the design data used for the bench-scale design for CSTRs, filter systems, and tank and pump sizing.

2.1.1 Simulated Salt Solution

The salt solution simulant used was the "average SRS salt" as defined in Table 1. The salt solution composition in the SRS design bases gave a 5.6 *M* total Na^+ concentration.[2] The small-tank TPB precipitation process is expected to operate with a feed of approximately 6.4 *M* total Na^+ (ref.2). The concentrations in Table 1 have been increased proportionately.

In addition to the soluble salts used in Test 1a, Test 2 simulant also contained 0.5 mg/L of simulated sludge, supplied by the SRTC, and additional catalyst species [enhanced catalyst component (ECC) catalyst] to promote the decomposition of TPB as specified by SRTC.

2.1.2 Tank Volumes and Scale

The preliminary sizing of the CSTRs for the small-tank TPB precipitation process provided a working volume of 15×10^3 gal (15 kgal).[2] Fortuitously, the CSTR size chosen for the ORNL work was 20 L with a working volume of 15 L. Therefore, the experimental scale was 1 mL = 1 gal and the flow rates scale to 1 mL/min = 1 gal/min. The working volumes of the appropriate tanks are shown in Table 2. (See also Fig. 1 for a simplified process flow diagram.)

The 15-kgal CSTRs will provide a holdup time (τ) of 10.6 h. Dilute precipitate product from CSTR 2 was concentrated to 10 wt % in the concentrate tank. Batches of 10 wt % precipitate were accumulated in the concentrate tank and then transferred to the wash tank for batch washing at constant volume. It will take approximately 60–72 h to accumulate a 4- to 5-kgal batch (4–5 L at bench scale) of concentrated precipitate. The unwashed concentrated

precipitate was then washed in the wash tank in a cycle that will take about 24 h. The working volumes of the concentrate and wash tanks were consistent with processing 4- to 5-kgal batches.

Table 1. Average SRS salt composition – 6.4 *M* Na^+

Component	Average (*M*)
Na^+	6.4
Cs^+	0.000160
K^+	0.0171
OH^-	2.182
NO_3^-	2.446
NO_2^-	0.594
AlO_2^-	0.354
CO_3^{2-}	0.183
SO_4^{2-}	0.171
Cl^-	0.0286
F^-	0.0366
PO_4^{3-}	0.0114
$C_20_4^{2-}$	0.00914
SiO_3^{2-}	0.00457
MoO_4^{2-}	0.000229

Table 2. Tank sizes for small-tank TPB precipitation

Tank	Working volume (10^3 gal)
CSTR 1	15
CSTR 2	15
Concentrate tank	10
Wash tank	10
Recycle wash water tank	10

Of some concern was the size of the recycle wash water tank (RWWT). The concentrated precipitate will be batch washed in a cycle that lasts about 24 h out of every 60–72 h. During this period, the [Na^+] in the spent wash water (stream 13) starts out at about 4.7 *M* and rapidly declines to about 0.1 *M* by the end of the wash—averaging about 1.2 *M* over the entire period.

Most, if not all, of the precipitated excess NaTPB will be expected to dissolve during the wash. At the beginning of the wash, very little NaTPB will be dissolved, due to its low solubility at high [Na^+]. However, as the [Na^+] decreases during the washing, the [NaTPB] will increase to a maximum and then decrease to near zero by the time the wash endpoint is reached (0.01 *M* nitrite). The peak concentration and duration of NaTPB in the wash water will depend on the amount of excess NaTPB being used and the dissolution rate of that excess.

Therefore, the composition of the spent wash water varies considerably over the course of the wash cycle. If this stream were fed with little or no holdup in the RWWT (stream 6), it would result in significant composition swings (and upsets) in the first CSTR. MATLAB® was used at Savannah River to perform an undocumented dynamic study of the interaction of the wash cycle with the composition in CSTR 1 (stream 7) as a function of the minimum heel in the RWWT. The [Na^+] was modeled based on a constant-volume wash lasting 24 h. The NaTPB was assumed to dissolve during one-third of the wash cycle, peaking at a concentration about 8 times higher than its average value. The study showed that if the RWWT operates with a minimum level of 8 kgal (8 L at bench scale), the [Na^+] in the first CSTR would vary by only 0.1 *M* total and the [NaTPB] would vary by less than about ±3% of the total NaTPB at 100% excess NaTPB (total NaTPB = stoichiometric + excess).

As much as 25 kgal of wash water would be generated per wash cycle, with about 10,000 gal being fed to CSTR 1 during that cycle. Therefore, about 15 kgal of wash water would accumulate in the RWWT. The size of this tank was therefore increased to 25,000 gal (25 L at bench scale).

2.2 FLOWSHEET AND INITIAL MATERIAL BALANCE

The preconceptual design basis flowsheet is shown in Fig. 1. The overall two-CSTR flowsheet developed for the ORNL 20-L demonstration is shown in Fig. 2. The flowsheets for the concentration loop and the slurry washing loop are shown in Figs. 3 and 4. The calculated material balance for 50% excess TPB is shown in the Table 3. It was the basis for the original estimates and sketches for the processing at ORNL. The design was originally based on the 50% excess TPB material balance and a 10.6-h nominal CSTR residence time. Based on the results of several small-scale tests at Savannah River and the initial water tests and systems evaluations at ORNL, the operating parameters were set at a residence time of 8 h for each CSTR and a 60%

excess TPB feed, with all TPB feed to the first CSTR. The material balance for this case is shown in Table 4. All feed calculations were based on the 8-h residence time. This increased the rates for all of the feeds by about 30% and made some of the earlier choices for control and monitoring equipment exceed the original design specifications. These were modified as needed during the checkout phase before operations began.

2.3 CSTR SYSTEM DESIGN

The small-tank TPB precipitation pilot-scale system used reactors with a 20-L total volume and a 15-L working volume. A sketch of the flow diagram is shown in Fig. 2. The CSTRs were constructed from 316 stainless steel, with bottom drain/flush valves, and had flanged tops with center, flange-mounted agitators. The agitator size was equivalent to one-fourth of the CSTR internal diameter. Each reactor included four baffles that were 1/12 the diameter of the tank and attached to tank walls. The vapor space of the CSTRs was purged continuously with nitrogen gas during operation. The feed streams to the reactors entered via tubes through the top flange and discharged under the surface of the liquid, near the agitator. The transfer lines from the reactors to the downstream vessel exited through the side of the vessel. The supernatant mixture in the CSTRs was internally cooled by stainless steel (SS) cooling coils, which were positioned below the working liquid level in the reactor. The coils were attached to the top flange and were removable to add versatility to the system.

The vessel for concentrating the precipitate was smaller (8-in. ID vs 10-in. ID) but of the same configuration as the CSTRs, including the cooling coils. The working volume of the tank varied as the amount of precipitate increased during the filtration but was usually about 6–8 L. The contents of the tank were pumped through a cross-flow filter (CFF) using a progressive cavity (Moyno) pump with 416 SS chrome-plated (CP) rotor and an ethylene propylene diene monomer (EPDM) stator. The slurry was returned to the vessel while the filtrate was transferred to the product collection tank. The filtered supernatant product was tested for the required decontamination factors (DFs) while the precipitate was concentrated to about 10% by weight solids. After accumulating the desired volume of 10% solids, the slurry was transferred to the precipitate wash vessel. The wash vessel was the same size as the precipitate concentration vessel, with a 7.5-L working volume (one-half of the working volume of the CSTRs). A second

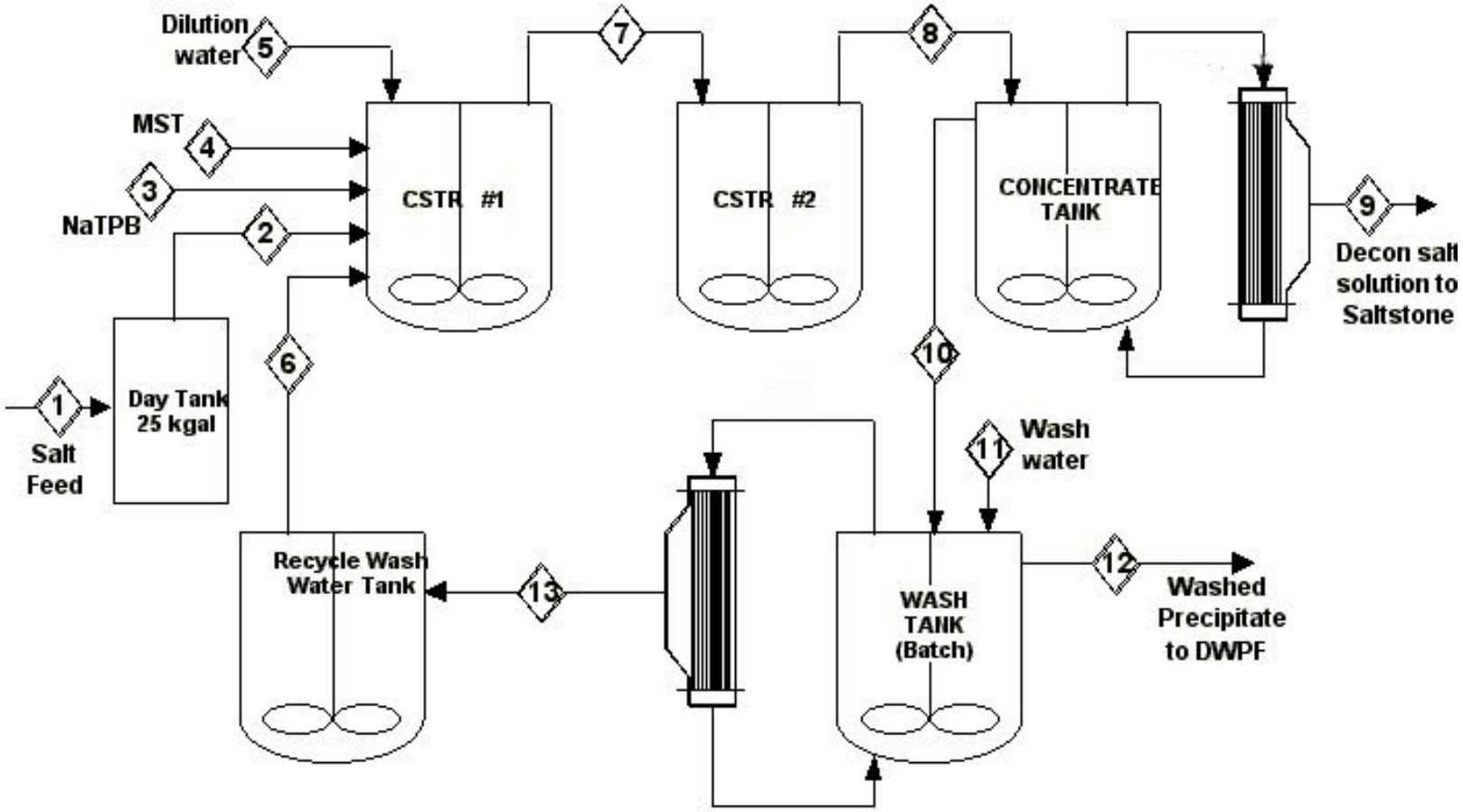


Fig. 1. Simplified flow diagram.

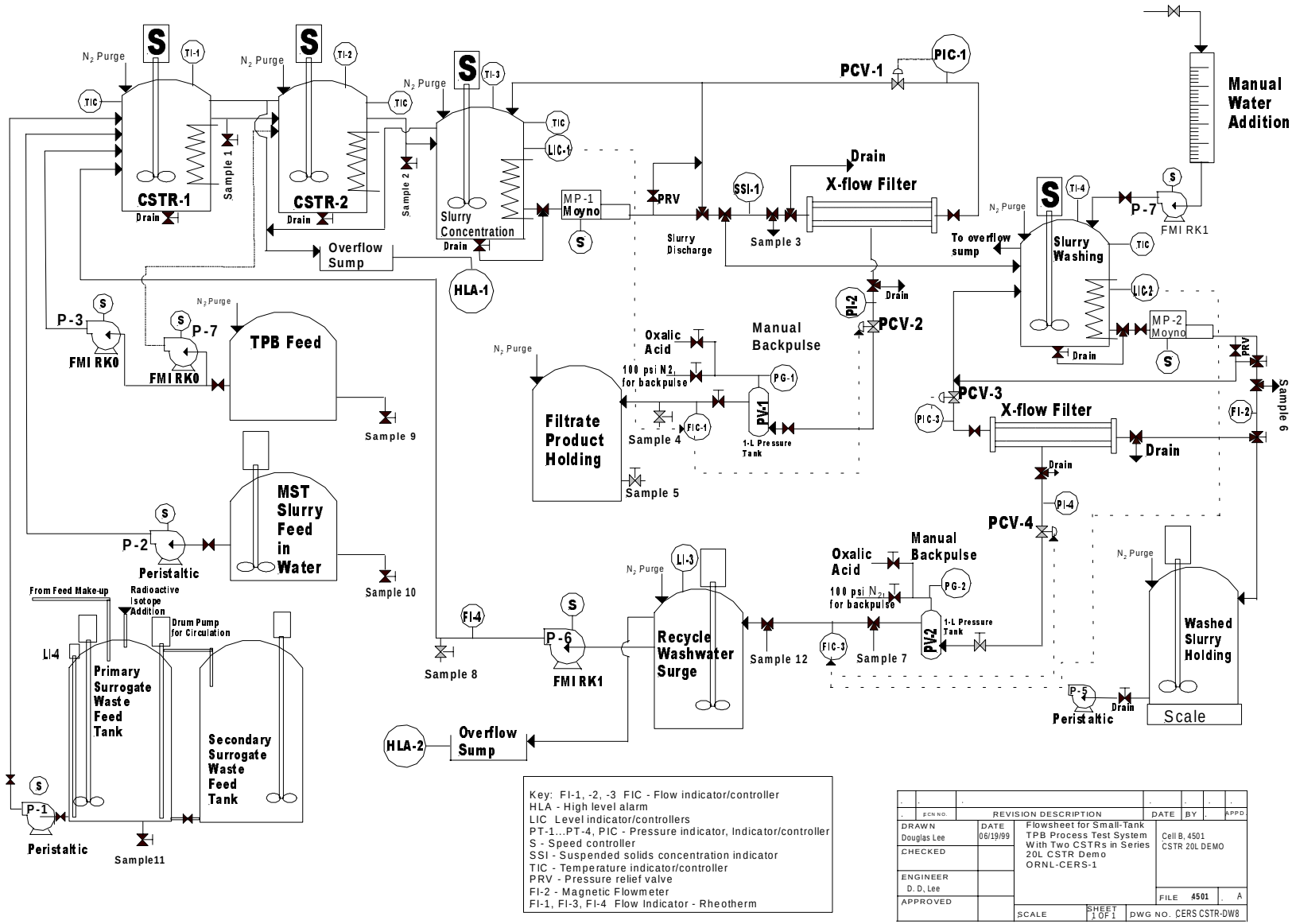


Fig. 2. Flowsheet for small-tank TPB process test system.

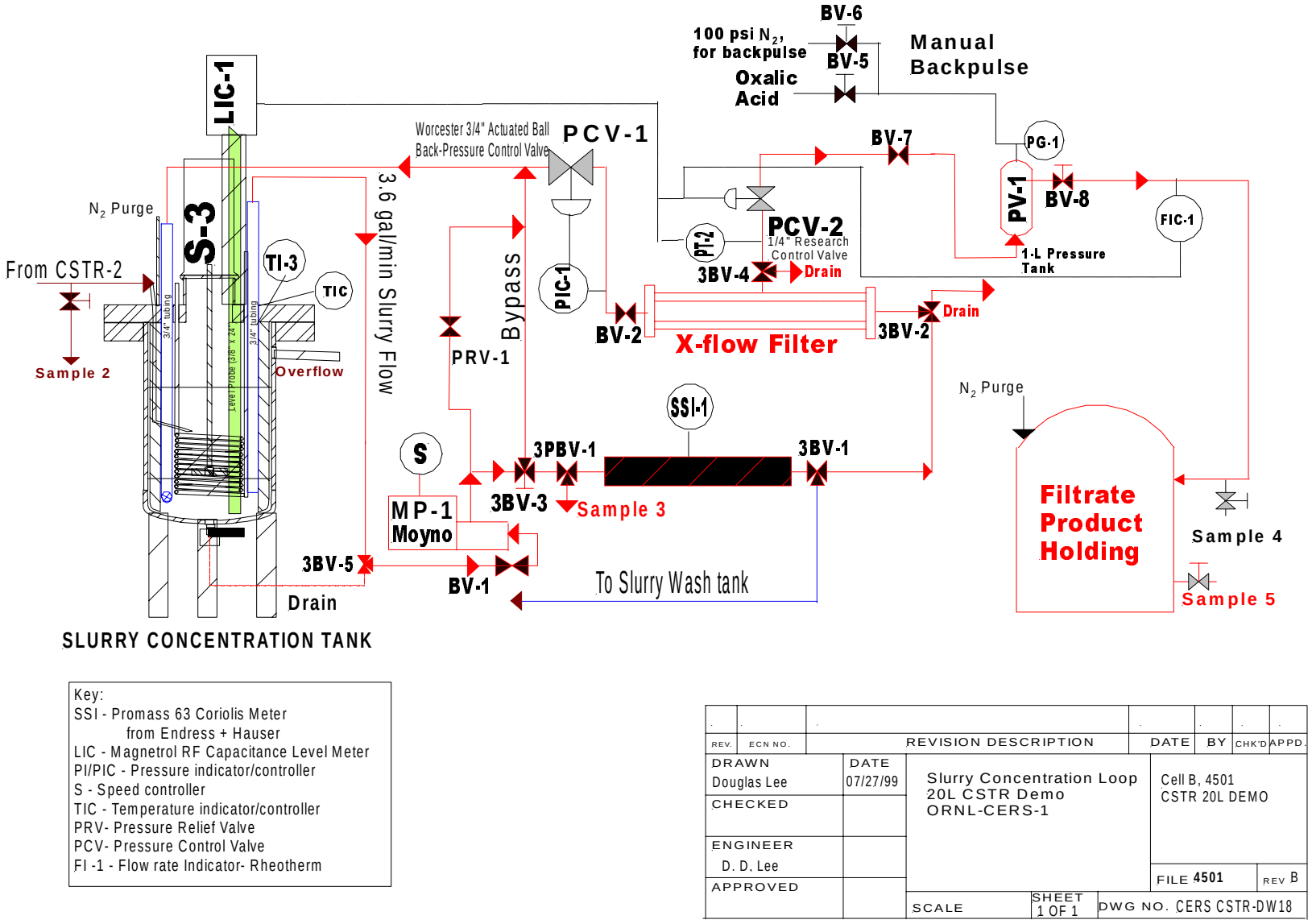


Fig. 3. Slurry concentration loop.

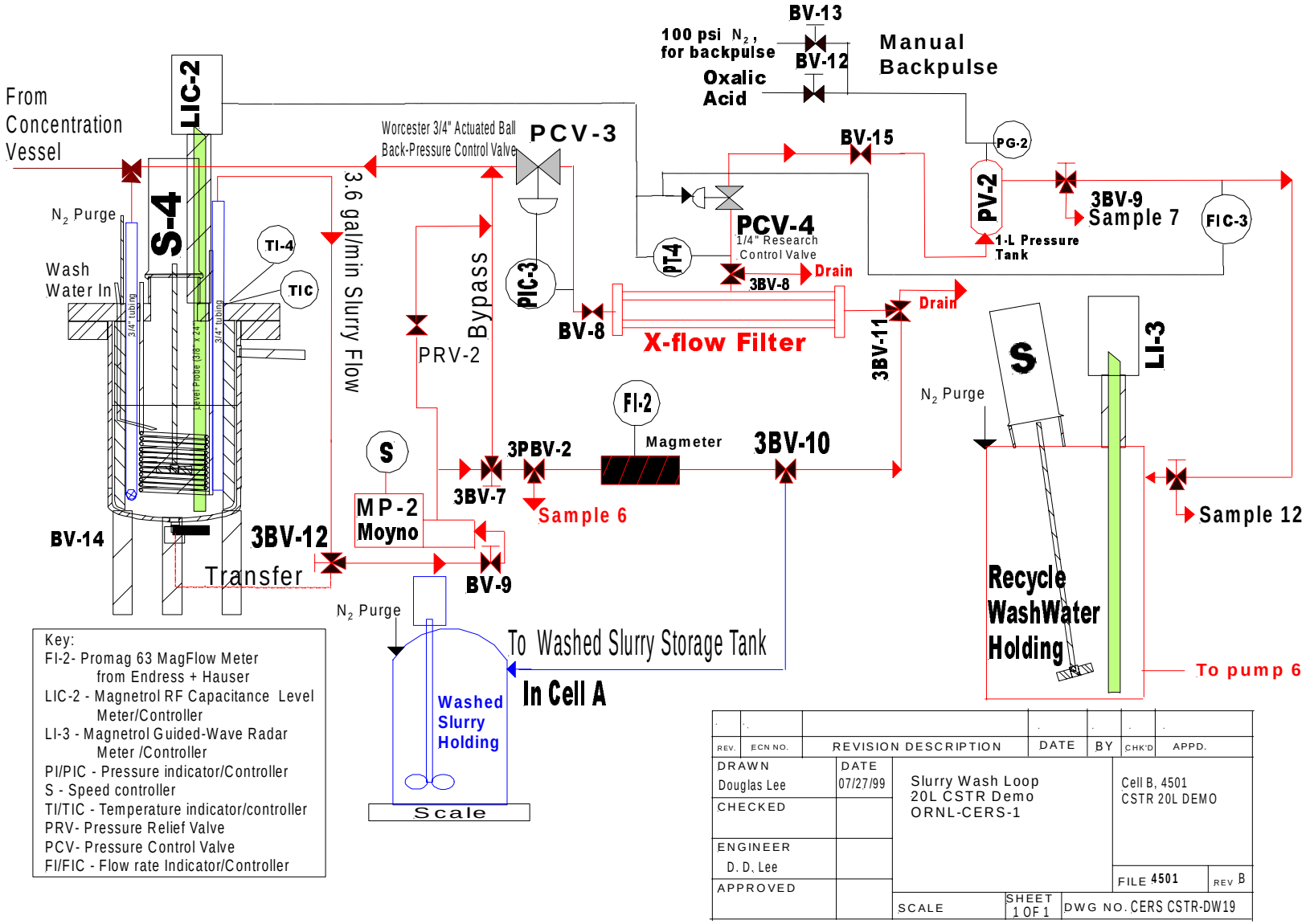


Fig. 4. Slurry washing loop.

Table 3. Material balance for 50% excess NaTPB

Washing endpoint =	0.010	*M* ntirite	Wash ratio =		43.64		Wash water vol =		3.776	gal/gal
Stream Number	1	2	3	4	5	6	7	8	9	10
Stream Name	Salt soln transfer	Salt soln feed	NaTPB	MST	Dilution water	Recycle wash water	CSTR 1 Prod	CSTR 2 Prod	Decon salt soln	Conc'd precip
[B]atch or [C]ontinuous	B	C	C	C	C	C	C	C	C	B
Flow (gal/min)	100	16.5	0.52	0.072	1.76	4.93	23.85	23.85	22.46	--
Flow (lb/hr)	255734	10656	294	39.6	881	2625	14494	14494	13645	--
Aqueous flow (gal/min)		16.5				4.93	23.71	23.71	22.46	--
Cycle time (h)	4.0	--	--	--	--	--	--	--	--	60
Batch size (gal)	23760	--	--	--	--	--	--	--	--	5031
Temp (°C)	amb	amb	25	amb	amb	25	25	25	25	25
Na^+ (*M*)	6.4	6.4	0.65			1.20	4.7	4.70	4.70	4.70
Cs-137 (Ci/gal)	1.38	1.38					0.96	0.96	1.6E-04	18.1
Density aqueous (g/mL)	1.290	1.290	1.12		1.0	1.059	1.214	1.214	1.214	1.214
Density (g/mL)	1.290	1.290		1.10			1.214	1.214		1.213
Insoluble solids (wt %)	0.039	0.039		11.4		0	?	0.59	0	10.0
Flows (lb/hr)										
water	169793	7075	243	35.1	881	2404	10638	10638	10074	33834
Na salts	77615	3234	1.8			177	3413	3413	3232	10856
KOH	190	7.93					?	~0	~0	~0
CsOH	4.76	0.198					?	~0	~0	~0
$NaNO_2$	8131	339				18.6	357	357	338	1137
sludge	98.5	4.1					4.1	4.1	0.0	246
MST				4.5			4.5	4.5	0.0	270
NaTPB (soluble)			49.4			25.0	0.59	0.59	0.56	1.9
NaTPB (insoluble)							?	25.0	0.0	1498
KTPB (insoluble)							?	50.7	0.0	3042
CsTPB (insoluble)							?	0.60	0.0	35.8

Stream Number	10a	11	11a	12	12a	12b	12c	13	13a	13b
Stream Name	Conc'd precip	Wash water	Wash water	Washed precip	Washed precip	Post-wash conc'n precip	Post-wash conc'n precip	Post wash filtrate	Spent wash water	Spent wash water
[B]atch or [C]ontinuous	avg	inst	avg	B	avg	B	avg	avg	avg	B
Flow (gal/min)	1.40	11.9	4.75	--	1.35	--	1.17	0.19	4.93	--
Flow (lb/hr)	849	5938	2375	--	693	--	599	94	2625	--
Aqueous flow (gal/min)	1.26	11.9	4.75	--	1.26	--	1.07	0.19	4.93	--
Cycle time (h)	--	24	--	60	--	60	--	--	--	60
Batch size (gal)	--	17087	--	4875	--	4206	--	--	--	17756
Temp (°C)	25	amb	amb	25	25	25	25	25	25	25
Na^+ (*M*)	4.70			0.02	0.02	0.02	0.02	0.02	1.204	1.20
Cs-137 (Ci/gal)	17.2			18.1	18.1	21.3	21.3	?	?	?
Density aqueous (g/mL)	1.214	1.0	1.0	1.006	1.006	1.006	1.006	1.006	1.059	1.059
Density (g/mL)	1.213			1.022	1.022	1.025	1.025			
Insoluble solids (wt %)	10.0			8.65	8.65	10.0	10.0	0.0	0.0	0.0
Flows (lb/hr)										
water	564	5938	2375	37687	628		535	93	2404	144223
Na salts	181			249	4.15		3.5	0.6	177	10644
KOH	~0			0	0	0	0	0		0
CsOH	~0			0	0	0	0	0		0
$NaNO_2$	19			26	0.43		0.37	0.06	19	1114
sludge	4.1			246	4.1	246	4.1	0		0
MST	4.5			270	4.5	270	4.5	0		0
NaTPB (soluble)	0.03			0	0	0	0	0	25.0	1499
NaTPB (insoluble)	25.0			0	0	0	0	0		0
KTPB (insoluble)	50.7			3042	50.7	3042	50.7	0		0
CsTPB (insoluble)	0.60			35.8	0.60	31.1	0.60	0		0

Table 4. Material balance for 60% excess NaTPB and 8-h residence time

Washing endpoint =	0.010	*M* ntirite	Wash ratio =	43.64			Wash water vol =		3.776	gal/gal
Stream Number	1	2	3	4	5	6	7	8	9	10
Stream Name	Salt soln transfer	Salt soln feed	NaTPB	MST	Dilution water	Recycle wash water	CSTR 1 Prod	CSTR 2 Prod	Decon salt soln	Conc'd precip
[B]atch or [C]ontinuous	B	C	C	C	C	C	C	C	C	B
Flow (gal/min)	100	21.5	0.68	0.094	1.96	6.86	31.18	31.18	29.26	--
Flow (lb/hr)	333229	13885	382	51.6	982	3648	18946	18946	17780	--
Aqueous flow (gal/min)		21.5				6.86	30.99	30.99	29.26	--
Cycle time (h)	5.2	--	--	--	--	--	--	--	--	40
Batch size (gal)	30960	--	--	--	--	--	--	--	--	4609
Temp (°C)	amb	amb	25	amb	amb	25	25	25	25	25
Na^+ (*M*)	6.4	6.4	0.65			1.19	4.70	4.70	4.70	4.70
Cs-137 (Ci/gal)	1.38	1.38					0.96	0.96	<1.6E-04	17.2
Density aqueous (g/mL)	1.290	1.290	1.12		1.0	1.058	1.214	1.214	1.214	1.214
Density (g/mL)	1.290	1.290		1.10			1.214	1.214		1.213
Insoluble solids (wt %)	0.039	0.039		11.4		0	?	0.62	0	10.0
Flows (lb/hr)										
water	221245	9219	316	45.8	982	3340	13901	13901	13127	30983
Na salts	101135	4214	2.3			244	4460	4460	4212	9941
KOH	248	10.33					?	~0	~0	~0
CsOH	6.20	0.258					?	~0	~0	~0
$NaNO_2$	10595	441				25.5	467	467	441	1041
sludge	128.4	5.3					5.3	5.3	0.0	214
MST				5.9			5.9	5.9	0.0	235
NaTPB (soluble)			64.3			38.6	0.59	0.59	0.56	1.3
NaTPB (insoluble)							?	38.5	0.0	1541
KTPB (insoluble)							?	66.1	0.0	2643
CsTPB (insoluble)							?	0.78	0.0	31.1

Stream Number	10a	11	11a	12	12a	12b	12c	13	13a	13b
Stream Name	Conc'd precip	Wash water	Wash water	Washed precip	Washed precip	Post-wash conc'n precip	Post-wash conc'n precip	Post wash filtrate	Spent wash water	Spent wash water
[B]atch or [C]ontinuous	avg	inst	avg	B	avg	B	avg	avg	avg	B
Flow (gal/min)	1.92	10.9	6.52	--	1.85	--	1.52	0.34	6.86	--
Flow (lb/hr)	1166	5437	3262	--	950	--	781	170	3648	--
Aqueous flow (gal/min)	1.73	10.9	6.52	--	1.73	--	1.39	0.34	6.86	--
Cycle time (h)	--	24	--	40	--	40	--	--	--	40
Batch size (gal)	--	15647	--	4448	--	3641	--	--	--	16454
Temp (°C)	25	amb	amb	25	25	25	25	25	25	25
Na^+ (*M*)	4.70			0.11	0.11	0.11	0.11	0.11	1.195	1.19
Cs-137 (Ci/gal)	17.2			17.2	17.2	21.3	21.3	?	?	?
Density aqueous (g/mL)	1.214	1.0	1.0	1.010	1.010	1.010	1.010	1.010	1.058	1.058
Density (g/mL)	1.213			1.025	1.025	1.028	1.028			
Insoluble solids (wt %)	10.0			8.21	8.21	10.0	10.0	0.0	0.0	0.0
Flows (lb/hr)										
water	775	5437	3262	34646	866		697	169	3340	133580
Na salts	249			228	5.69		4.6	1.1	244	9757
KOH	~0			0	0	0	0	0		0
CsOH	~0			0	0	0	0	0		0
$NaNO_2$	26			24	0.60		0.48	0.12	26	1022
sludge	5.3			214	5.3	214	5.3	0		0
MST	5.9			235	5.9	235	5.9	0		0
NaTPB (soluble)	0.03			0	0	0	0	0	38.5	1542
NaTPB (insoluble)	38.5			0	0	0	0	0		0
KTPB (insoluble)	66.1			2643	66.1	2643	66.1	0		0
CsTPB (insoluble)	0.78			31.1	0.78	31.1	0.78	0		0

CFF was used to perform the precipitate washing step. The wash vessel was operated at constant volume with constant wash water feed and filtrate removal until the nitrite concentration was reduced to 0.01 *M* or less. The wash was used to remove excess salt and NaTPB for recycle to the system feed tank. The filtered wash liquid was transferred to the recycle wash water storage tank, and the washed solids were transferred to the solids storage tank. The solids wash cycle required about 24 h.

Liquids and slurries were transferred using small-volume gear and peristaltic pumps. Gear pumps were used for the liquid feed streams to the first CSTR, and the peristaltic pumps were used for intervessel slurry transfers. The 10% solids slurry in the precipitate concentrate tank and the precipitate wash tank was transferred using the Moyno pumps normally used for the CFF feed.

The instrumentation and control strategy included on-line monitoring and data recording of key parameters. Temperatures in both CSTRs were recorded. Parameters on both CFFs that were monitored and recorded included temperatures, pressures, liquid levels, density, pump speed, agitator speed, and flow rates. Feedback control systems were used to control flow, level, and pressure on both CFFs. The complete design package for the system is shown in Appendixes A.2 and A.3.

2.4 FILTRATION SYSTEM DESIGN

2.4.1 Control Strategy for CFF Units

Cross-flow filtration is used to concentrate the product of the TPB and MST treatment process and to separate the wash water during the precipitate washing process. The primary operating parameters that impact the performance of CFFs were axial velocity of the fluid through the CFF element and the transmembrane pressure (TMP) or pressure drop in the direction of filtrate flow. The axial velocity is maintained between 6 and 10 ft/s by adjusting the drive speed of the progressive cavity pump. The TMP is maintained by a control valve in the filtrate piping.

2.4.2 Concentration Filter Loop

The CFF elements were 0.5-µm filters that were 24 in. long with a 0.5-in. ID. A Moyno progressive cavity pump is used to circulate the slurry through the tube side from the supply tank, through the element, and back to the tank. A backpressure valve (Worcester) is used to provide a constant 40-psi pressure on the tube side of the filter at a constant 3.6-gal/min slurry flow through the tube. The pressure across the filter membrane is controlled by a second pressure control valve (research control valve) on the filtrate stream. The valve functions by gradually opening to lower the shell-side backpressure, thus increasing the TMP across the filter.

The slurry concentration tank receives feed at a constant rate from the second CSTR by gravity overflow. The filter loop concentrates slurry continuously during operation from 0.5 wt % solids to about 10 wt % solids, with the filtrate rate out being the slurry rate in minus the accumulation of solids in the tank. The filtrate rate needs to be constant, and the level of the tank slurry can vary slightly as the flow rate of slurry from the CSTRs varies due to hydraulics. The slurry concentration tank is maintained at about 7-L volume, and after 10 wt % solids concentration is achieved, approximately 5 L of the 10% slurry is transferred to the wash tank. Then the level in the concentration tank is allowed to increase back to 7 L with the feed slurry. The slurry concentration recirculation and filtration are then restarted.

If the element becomes fouled, the flux will decrease. Since the expected flow necessary to maintain constant level is 35 mL/min for the concentration filter and 8–15 mL/min for the slurry washing filter, the filter elements were adequately sized to provide the necessary filtrate even if considerable fouling occurs. Backpulsing of the filter element was performed on a routine basis in order to maintain an acceptable filtrate flux. The frequency of backpulsing was usually no greater than once per hour to avoid unnecessarily exposing the filter element surface to small pore-penetrating particles that would otherwise be removed by the thin precoat of filtered solids.

The backpulse systems for each loop consist of a filtrate holding vessel with a nitrogen supply capable of applying a 100-psi sustained pressure on the vessel for the duration necessary for an effective backpulse. The concentration filter loop used a Coriolis meter to monitor the density of the slurry. Additional piping and valves were provided on both loops for discharging concentrated slurry and for providing chemical addition to clean the filter element.

If, despite routine backpulsing, the filtrate flux had dropped below 0.01 $gal \cdot min^{-1} \cdot ft^{-2}$ at 40-psig TMP, a chemical cleaning could have been conducted to restore the flux to an adequate level. If chemical cleaning had been necessary, the TPB processing would have been temporarily stopped (or the slurry concentration vessel effluent diverted to a waste holding tank) to avoid overfilling the slurry concentration vessel. Filter cleaning was not required during or between the test phases.

2.4.3 Slurry Wash Loop

The CFF element and valve system is a duplicate of the concentration loop system, with the exception of the slurry flowmeter, which used a magnetic flowmeter instead of the Coriolis meter. The slurry wash tank receives slurry from the concentration tank that has been concentrated to 10%. About 5 L of slurry is transferred to the wash tank at one time, and a water wash is then initiated. The wash water was maintained at a constant rate of about 5 to 15 mL/min during the entire wash period. The wash loop circulates the slurry through the CFF, and filtered wash water is produced containing soluble TPB and salt solution from the slurry. The sodium content of the wash solution gradually decreased from about 4.7 *M* to about 0.1 *M* during the 24 h of washing. After washing was complete, the washed slurry was transferred to the washed slurry holding tank for disposal.

The washed slurry filter loop washed the slurry continuously during operation, with the product filtrate rate out being equal to the wash water rate in. The filtrate rate setpoint was adjusted to maintain a constant volume of about 5 L in the wash tank. The wash tank slurry level was also varied as the slurry density changed with the washing and dilution.

2.5 INSTRUMENTATION AND CONTROL SYSTEM

The control strategy for the filter systems was devised. Intellution, the man-machine interface used in the test system, was programmed, and its database was created to carry out the strategy. The historical data collection program was configured for all available points in the system. Trend groupings were created to allow data for systems to be followed over time during an experiment, and user displays were generated. All final control elements were tested for operability and were functional. Data were gathered that allowed tuning parameters and settings

for both of the filter system control loops to be generated. The documentation for these settings is located in Appendix D.

2.6 SAMPLING REQUIREMENTS FOR TEST AND ANALYTICAL SAMPLES

During the tests, samples were taken for analysis. The feed solutions for all tests were analyzed after makeup for K, ^{137}Cs, ^{85}Sr, and ^{235}U. The NaTPB feed solution for each test was analyzed by HPLC. During Test 1a, filtrate (output from the precipitate concentrate filter) was sampled every 2 h (a total of 30 samples). Each sample was analyzed for K, ^{137}Cs, ^{85}Sr, and ^{235}U, and every third sample was analyzed by HPLC for NaTPB. Samples were taken between the two CSTRs and after the second CSTR every 4 h, filtered immediately, and analyzed for K, ^{137}Cs, ^{85}Sr, and ^{235}U. Every second sample was analyzed by HPLC for NaTPB

The sampling plan for Test 1a is shown in Table 5, along with all of the sample numbers and space for the person taking the sample to initial that the sample was taken [Initials Of Sampler (IOS)]. The sample numbers were keyed to the sample port, with SP1 located between CSTR 1 and CSTR 2, SP2 located between CSTR 2 and the concentration vessel, and SP4 being the filtrate from the concentration vessel CFF. Tests 1b and 1c, if they had been conducted, would have had similar numbering systems. An example of the SP1 sampling plan in Table 6 shows the samples taken and their disposition. Some were stored on-site, some were sampled for internal counting for ^{137}Cs and ^{85}Sr, and the others were sent to the RMAG for analyses.

One sample of the slurry from each concentrated slurry batch was taken and analyzed by HPLC. During operation, the density (measure of the sodium concentration) of the filtrate from the slurry concentration CFF was checked to confirm that operations were continuing as planned.

During Test 2, the filtrate was sampled every 4 h and analyzed for B, K, ^{137}Cs, ^{85}Sr, and ^{235}U; every other sample was analyzed by HPLC for NaTPB and intermediates. Samples were taken between CSTRs and after the second CSTR every 8 h, filtered immediately, and analyzed for B, K, ^{137}Cs, ^{85}Sr, and ^{235}U. Every other sample was analyzed by HPLC for NaTPB and intermediates. During slurry washing, the wash water was sampled every 2 h and analyzed for B, K, ^{137}Cs, ^{85}Sr, and ^{235}U. Every third sample was analyzed by HPLC for NaTPB. After each batch of concentrated slurry was transferred to the precipitate wash tank and washed, a sample of the solids was obtained before transfer to the slurry waste tank. The samples were analyzed by HPLC.

Table 5. Test 1a sampling schedule

Time (h)	Sample port 1, sample ID	IOS	Sample port 2, sample ID	IOS	Sample port 4, sample ID	IOS
2					T1aSP4-01	
4	T1aSP1-01		T1aSP2-01		T1aSP4-02	
6					T1aSP4-03	
8	T1aSP1-02		T1aSP2-02		T1aSP4-04	
10					T1aSP4-05	
12	T1aSP1-03		T1aSP2-03		T1aSP4-06	
14					T1aSP4-07	
16	T1aSP1-04		T1aSP2-04		T1aSP4-08	
18					T1aSP4-09	
20	T1aSP1-05		T1aSP2-05		T1aSP4-10	
22					T1aSP4-11	
24	T1aSP1-06		T1aSP2-06		T1aSP4-12	
26					T1aSP4-13	
28	T1aSP1-07		T1aSP2-07		T1aSP4-14	
30					T1aSP4-15	
32	T1aSP1-08		T1aSP2-08		T1aSP4-16	
34					T1aSP4-17	
36	T1aSP1-09		T1aSP2-09		T1aSP4-18	
38					T1aSP4-19	
40	T1aSP1-10		T1aSP2-10		T1aSP4-20	
42					T1aSP4-21	
44	T1aSP1-11		T1aSP2-11		T1aSP4-22	
46					T1aSP4-23	
48	T1aSP1-12		T1aSP2-12		T1aSP4-24	
50					T1aSP4-25	
52	T1aSP1-13		T1aSP2-13		T1aSP4-26	
54					T1aSP4-27	
56	T1aSP1-14		T1aSP2-14		T1aSP4-28	
58					T1aSP4-29	
60	T1aSP1-15		T1aSP2-15		T1aSP4-30	

Table 6. Test 1a sampling schedule (port between CSTRs)

Sample seq.	Time (h)	IOS	Sample ID	Type of analyses				
				HPLC[a]	Gamma[b]	ICP[c]	DNC[d]	In-house γ
1	4		T1bSP1-01	Retain	✓	✓		✓
2	8		T1bSP1-02	✓	✓	✓		✓
3	12		T1bSP1-03	Retain	✓	✓		✓
4	16		T1bSP1-04	✓	✓	✓		✓
5	20		T1bSP1-05	Retain	✓	✓		✓
6	24		T1bSP1-06	✓	✓	✓		✓
7	28		T1bSP1-07	Retain	✓	✓		✓
8	32		T1bSP1-08	✓	✓	✓		✓
9	36		T1bSP1-09	Retain	✓	✓		✓
10	40		T1bSP1-10	✓	✓	✓		✓
11	44		T1bSP1-11	Retain	✓	✓		✓
12	48		T1bSP1-12	✓	✓	✓		✓
13	52		T1bSP1-13	Retain	✓	✓		✓
14	56		T1bSP1-14	✓	✓	✓		✓
15	60		T1bSP1-15	Retain	✓	✓		✓
16	64		T1bSP1-16	✓	✓	✓		✓
17	68		T1bSP1-17	Retain	✓	✓		✓
18	72		T1bSP1-18	✓	✓	✓		✓
19	76		T1bSP1-19	Retain	✓	✓		✓
20	80		T1bSP1-20	Retain	✓	✓		✓

[a] HPLC analysis for concentrations of sodium tetraphenylborate (NaTPB).
[b] Gamma analyses for both ^{137}Cs and ^{85}Sr.
[c] ICP-AES or ICP-MS for potassium and uranium.
[d] Delayed neutron counting for uranium (if needed as back up for the ICP analysis for uranium).

3. ANALYTICAL METHODS AND REQUIREMENTS

3.1 RADIOCHEMICAL ANALYSIS

Gamma analysis was used for ^{137}Cs and ^{85}Sr.

3.2 HPLC METHODS

Reverse-phase HPLC is used to monitor the concentrations of NaTPB and its degradation products, triphenylborane (3PB), diphenylborinic acid (2PB), phenylboronic acid (1PB), and phenol in alkaline salt solution. Ion chromatography is used for potassium if the concentration is >1 ppm, while graphite furnace is used for potassium if the concentration is <1 ppm.

In order to ensure that the ORNL HPLC methods were able to determine the NaTPB and its degradation products, an environmental compliance series of comparisons was made between the Savannah River analytical laboratory and the ORNL laboratory. The first comparison gave good agreement for NaTPB, 3PB, and 2PB, as shown in Figs. 5–7. ORNL laboratory results for 1PB and phenol were questionable because of column performance errors. ORNL then purchased and implemented columns identical to those used at SRTC. Both laboratories then completed an additional series on the two compounds and obtained values for 1PB and phenol that were within 5 to 11% and 3 to 5%, respectively, of the known values as shown in Table 7.

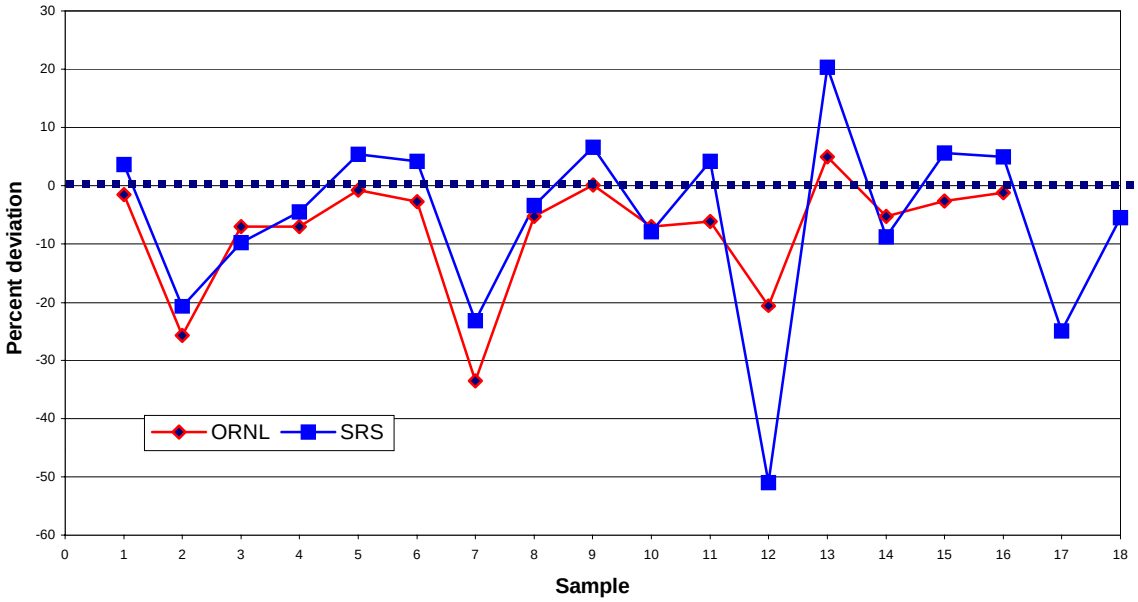


Fig. 5. Percent deviation of TPB results from reference value.

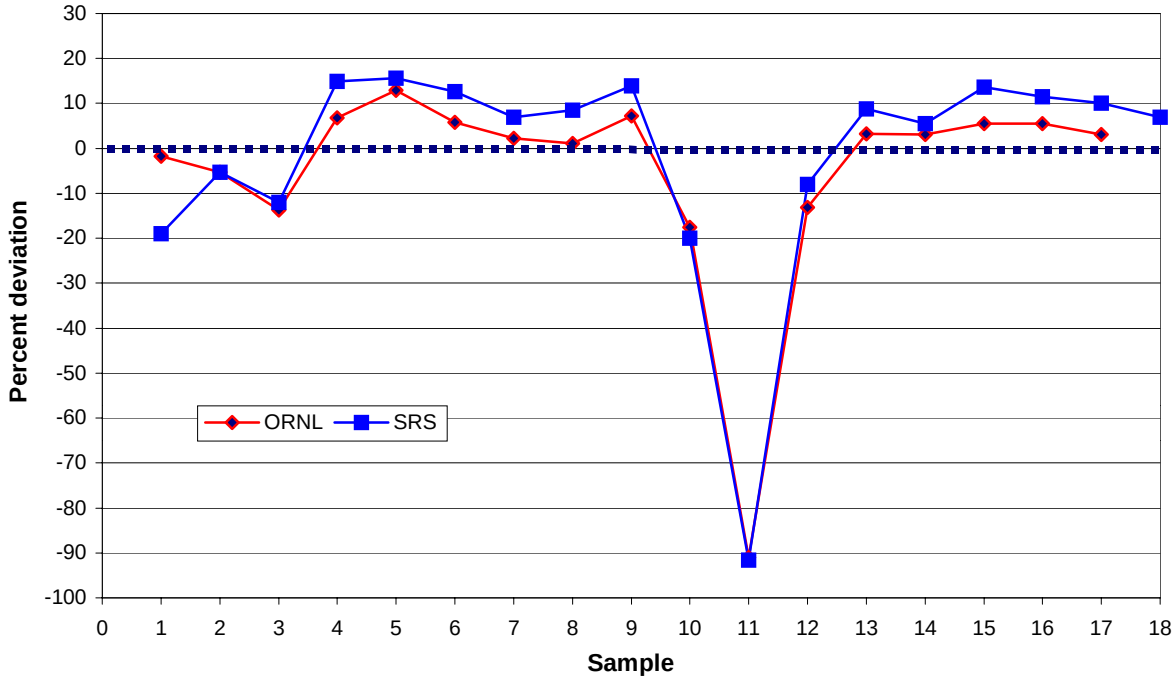


Fig. 6. Percent deviation of 3PB results from reference value.

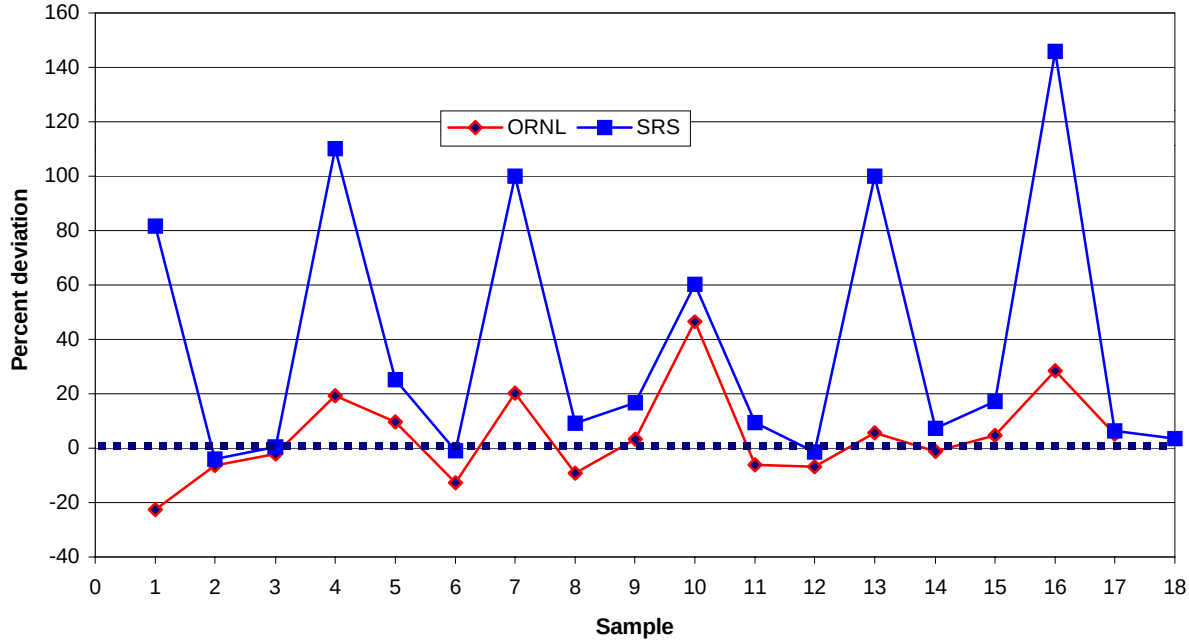


Fig. 7. Percent deviation of 2PB results from reference value.

Table 7. Sample results for phenol and 1PB

Sample	Compound	Instrument reading	Total DF	Recovery efficiency	ORNL "A" results (μg/mL)	SRTC results (μg/mL)	Target value (μg/mL)
1A	Phenol	57.222	6.5	0.78	477	503	500
	1PB	0.000	6.5	0.80	Not detected	Not detected	0
2A	Phenol	62.566	6.5	0.78	521	478	506
	1PB	57.466	6.5	0.80	467	426	500
3A	Phenol	110.828	6.5	0.78	924	1011	998
	1PB	114.841	6.5	0.80	933	894	1000
4A	Phenol	21.395	6.5	0.78	178	182	196
	1PB	175.529	6.5	0.80	1426	1259	1500
5A	Phenol	0.000	6.5	0.78	Not detected	Not detected	0
	1PB	290.938	6.5	0.80	2364	2182	2500

3.3 ICP METHODS

Inductively coupled plasma–atomic emission spectroscopy (ICP-AES) and ICP-MS were used for U (depleted), Cu, Pd, B, Na, and K analysis. Initial sample dilutions were required because of the high salt concentrations. Dilutions of 10 times or more were needed for ICP-AES analyses and 100 to 1000 times for the ICP-MS analyses. The detection limits for the two methods are shown in Table 8. Microwave digestion (HNO_3) was used in sample preparations for boron analyses.

Table 8. Comparison of ICP-AES and ICP-MS detection limits

Element	ICP-AES (ppb)	ICP-MS (ppb)
[Cu]	> 5	< 5
[Pd]	> 100	< 100
[U]	> 200	< 200
[Na]	> 50	
[K]	> 200	
[B]	> 10	

3.4 URANIUM METHODS

The detection limit for uranium using ICP-MS is 1 to 0.1 ppb for normal or depleted uranium and 5 ppt for ^{235}U. However, because of the high salt content of the CSTR test samples, which can cause plugging of the sample delivery system for the ICP-MS, sample dilutions of 100 to 1000 were needed.

The Neutron Activation Analysis Group at the High Flux Isotope Reactor (HFIR) at ORNL conducts delayed neutron counting (DNC) analyses. HFIR, which has a pneumatic tube irradiation system, has the highest thermal neutron flux in the world. DNC depends upon the ^{235}U content in the uranium. Samples were taken and then analyzed by DNC and ICP-MS to check the accuracy of these analyses. Both filtered and unfiltered samples were analyzed.

3.5 NITRITE METHOD

3.5.1 Internal Nitrite Analysis

Measurement of the nitrite concentration in the wash solution was necessary for monitoring slurry washing. A rapid though accurate method was needed, and the use of a nitrite ion–specific electrode was selected: Model 93-46 nitrite electrode from Orion with its corresponding reference electrode Orion Model 90-01and pH meter (Orion model 420A) was used for reading the electrode response. The analysis also required an Orion nitrite interference suppressor solution (NISS), which acted as a pH buffer and removed a variety of interfering anions, including chloride ions, present in samples.

3.5.2 Preparation of the Calibration Curve

A nitrite stock solution was prepared in which the nitrite concentration was 100 ppm. With this solution, standards at 0.5, 1, 10, 40, 70, and 100 ppm of nitrite were prepared. An aliquot of 25 mL of each standard was mixed with 25 mL of the NISS solution. The solution was stirred with a magnetic stirrer. The clean nitrite and reference electrodes were introduced into the solution, and a reading was made after stabilization of the meter. The results were plotted and the parameters of the calibration were calculated. The requirements for these were (1) a correlation coefficient superior to 0.995 and (2) a slope for the electrode between 50 and 60. A calibration curve was prepared before a wash cycle was started.

3.5.3 Analysis of the Wash Samples

For the standard preparation, each sample analyzed was mixed with NISS in a ratio of 25 mL of sample per 25 mL of NISS. The solution was stirred, and the reading was made after stabilization of the meter. The reading was then compared with the calibration curve, and the concentration of nitrite as nitrogen was calculated. Due to the high starting concentration of

nitrite in the samples, a large dilution was performed and only 250 µL of sample was necessary for the analysis.

3.6 INTERNAL SAMPLE ^{137}Cs AND ^{85}Sr ANALYSES FOR PROCESS CONTROL

Samples for internal gamma analysis were filtered, and analyses were performed using an in-house gamma-counting system. Samples were counted for 30 min at various distances from 5 to 30 cm from the detector head, depending on the amount of cesium or strontium in the sample. Typically, 2-mL samples were counted for 30 min at a distance of 30 cm from the detector. A National Institute of Standards and Technology (NIST) gamma standard, SRM-4275 (Sept. 1, 1988), was used for efficiency standardization and energy calibration.

4. CONSTRUCTION, INSTALLATION, AND TESTING OF THE SYSTEMS

4.1 EXPERIMENTAL SITE, CSTRs, SLURRY CONCENTRATION, AND SLURRY WASH TANK CONSTRUCTION

4.1.1 Experiment Location

The equipment was installed in Hot Cells A and B in Building 4501 at ORNL. These are two of four cells in a square, central block, labeled A, B, C, and D. Each cell contains two X-Y-Z master-slave manipulators; two cell-face windows; and access doors for sample, equipment, and convenient personnel entry (6 × 3 ft door). Cell B has very low levels of transferable contamination and has low radiation source levels when not in use for experiments. Routine personnel entry for hands-on maintenance can be carried out with very low radiation exposure incurred. The cell also has low background radiation and allows for minimal cross-contamination from one experiment to the next. Cell B has multiple access ports on two sides and multiple cell-to-cell access ports. Each cell is 8 ft wide, 6 ft deep, and 16 ft high (floor to ceiling). The cell has four electrical panels inside with multiple 120 Vac and 240 Vac outlets connected to panels outside the cell. The panels also contain multiple instrument leads.

The cell has an operating-level table about 4.6 ft above the cell floor, with a containment pan on the table that is about 5.5 ft front to back and 7 ft wide. The area below the table is used for storage and can be used for large feed and waste tanks if an experiment requires them. Each

cell has an air atmosphere supplied by the 3039 stack vacuum system, which pulls air through the cells from the surrounding areas. The high-volume, low-vacuum off-gas from the cell passes through high-efficiency particulate air (HEPA) filters in the Building 4501 basement and then to the 3039 stack. The cells, which are vented to the 3039 stack, contain vessel off-gas connections for low-volume, high-vacuum work. The walls are 3-ft-thick barytes concrete lined with stainless steel. The ceiling is 4-ft-thick barytes concrete. The doors are all 8-in.-thick lead. Each cell has an upper 2 × 2 ft and a lower 16 × 16 in. lead glass window in the center cell face, each 3 ft thick. The windows contain eight 4-in.-thick panes of glass, and the spaces between panes are filled with mineral oil.

4.1.2 CSTRs

The design for the continuous, small-tank TBP bench-scale hot cell system used two CSTR vessels. A spare CSTR was also constructed for possible later use. The materials of construction used in the CSTRs included 316 SS 10-in. schedule 40 pipe with a pipe cap welded to one end and a 150-lb slip-on flange welded to the other. The flange seals were made with Viton gaskets. The vessel was 17 in. from the flange face to vessel bottom. The top flange was machined from a 150-lb blind flange and contained a centered, 4-in. TriClover agitator mount for a Lightnin LabMaster Model G2L05R, 90–1600 rpm, 1/15-hp drive agitator drive with a 3/8-in. shaft and with digital speed display. Impellers available for use included the following:

3-in. A-200 four-blade, 45-deg pitch axial flow (the impeller used for the tests),
3-in. A-100 three-blade marine axial-flow impeller, and
3-in. R-100 six-blade radial-flow impeller.

Each 20-L CSTR had a working volume of 15 L (15.4 L measured) and was equipped with a cooling coil constructed from ¼-in. 316 SS coiled tube, with a 4.5-in. coil ID which served as draft tube (the ratio of the coil ID to that of the agitator was 3:2). The length of the coil assembly from the top to bottom was 5.5 in., and the spacing between each of the 13 coils was 1/8 in. The coil was positioned with one-fourth of the liquid above and below the coil, about 2¾ in. above the bottom of the vessel. Fittings and attachments for CSTR 1 are shown in the design drawings CERS-CSTR-DW1, -DW2, -DW16, -DW17, and -DW20. Fittings and attachments for CSTR 2 and CSTR 3 are shown in design drawings CERS-CSTR-DW1, -DW3, and -DW11.

4.1.3 Slurry Concentration Vessel and Slurry Wash Vessel

The design of the two vessels employed for slurry concentration and washing used 316 SS 8-in. schedule 40 pipe with a pipe cap welded to one end and a 150-lb slip-on flange welded to the other and used Viton flange gaskets. The vessel was 13 in. from the flange face to the tank bottom and had a 150-lb blind flange with a centered 3-in. TriClover agitator mount for the same agitator as was used for the CSTRs. The impellers used in both were 2.5-in. A-200, 4-blade, 45-deg pitch axial-flow impellers. The working volume of each was about 7 L with a total volume of 10.7 L. The concentration vessel had a 4¼-in.-OD cooling coil made from ¼-in. 316 SS coiled tube, with a 4¼-in. coil height, and had ¼-in. spacing between coils. The coil unit was placed $2^{1}/_{8}$ in. off the tank bottom, had about eight coils, and acted as a draft tube. The wash vessel cooling coil was also a ¼-in. 316 SS, 4¼-in.-OD coiled tube, with ¼-in. spacing between coils. The coil was 3.3 in. long and located 1.65 in. from the vessel bottom while acting as draft tube. It had about 6½ coils. Fittings and attachments are shown in the design drawings CERS-CSTR-DW4, -DW5, -DW6, -DW7, -DW12, -DW13, -DW14, -DW15, -DW20, and -DW21.

4.1.4 Wash Water Recycle Hold/Feed Tank

The wash water recycle feed tank had a 25-L maximum working volume and a 10-L minimum volume. It was continuously stirred to maintain a well-mixed volume as the wash water was added during the slurry washing step. This tank also was covered and purged with nitrogen and had about an 8-L freeboard. The tank was 12-in. diam by about 18 in. high constructed from SS tubing. A Lightnin mixer with a 3-in.-diam, angled TriClover fitting, supported the agitator. Fittings and attachments for the tank are shown in the design drawings CERS-CSTR-DW23 and -DW24. A checkvalve was added to the line from the pump to the CSTR on the hot cell side of the line to prevent any chance of backflow from the vessel back through the line.

4.1.5 Level Instrumentation

Several level meters were tried in the concentration and wash vessels. The types that could be used were constrained by the lack of space on the top of the vessel flanges, the materials of construction, the vessel internals, the process fluid/slurry/foam, and the requirements for not introducing foam that causes bubbles. Three level meters were evaluated:

Fig. 8. Photo of CSTRs before installation in the hot cell.

Magnetrol "Eclipse" guided wave radar level transmitter. With 0.1-in. resolution, adjustable 0- to 60-s damping, loop powered, 4- to 20-mA output, 316/416L SS, ¾-in. NPT fitting, 24 in. long, and 0.875-in. OD. These failed in both concentrated slurry–containing tanks due to slurry plugging the annular space between the wave guide and stilling well. This probe did work well in the slurry recycle wash water hold/feed tank.

Magnetrol "Kotron" Series 82 CE, RF capacitance level transmitter. With 0.25% repeatability, 0.1-s response time, loop powered, 4- to 20-mA output, 316/416L SS, ¾-in. NPT fitting, 24 in. long, 0.875-in. OD. These failed in both vessels because of the close proximity of the metal vessel internals along with the slurry bridging between the internals and the probe.

Bindicator "LEVELDATA" Model GAG200006, hydrostatic level transmitter. With ± 1.0% accuracy, 5:1 turndown capability, loop powered, 0–28 in. H_2O, 4- to 20-mA output, 316L SS, ¾-in. NPT process connection, 3/8-in. impulse tube, 7/8-in. reservoir. This probe worked through Test 2 in the slurry wash vessel and gave excellent service.

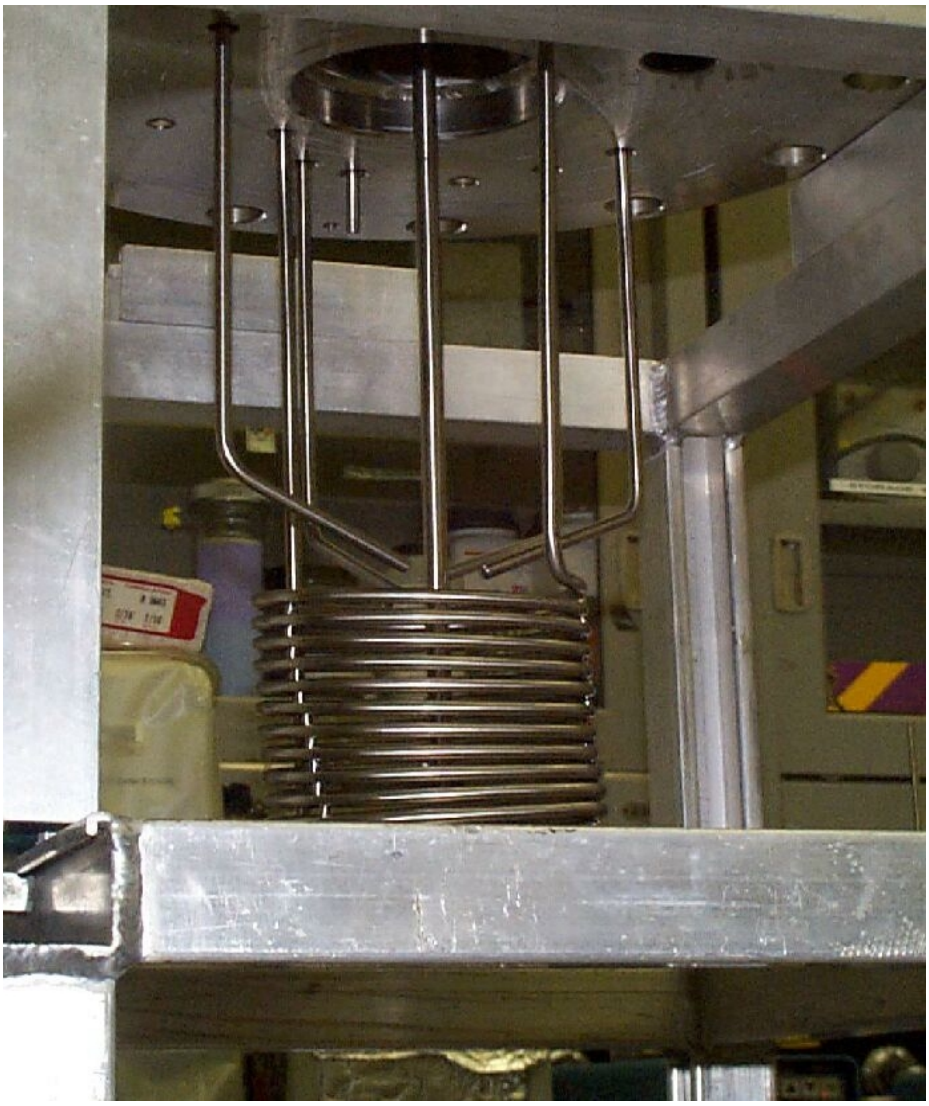


Fig. 9. Photo of CSTR cooling coil internals.

4.2 FILTRATION SYSTEM CONSTRUCTION

Both systems used ¾-in. SS tubing, with valves and instruments readily accessible from the front of the module frame. The slurry sample lines included a small lipped tray below the slurry sample points. A 0- to 100-psig pressure gauge on the backpulse circuit and a 100-psi pressure-relief valve were included on each pump circuit. Each unit was as compact as possible, and one stacked on the other to conserve space inside the hot cell. Each unit contained a 0.5-µm SS sintered metal Mott Filter Corp. filter tube (supplied by Savannah River) that had a length of

24 in. and was 0.5-in. ID and 5/8-in. OD, providing a surface area of 0.26 ft^2. The slurry design flow rate was 6-ft/s axial velocity, which gave a 3.6-gal/min volumetric flow.

Each unit uses a Moyno 500 Series model 33304 pump driven through flexible coupling by a ¾-hp 1750 3/60/460 Vac inverter-duty Baldor motor, mounted on a SS base plate. The pump had a 316 SS body, with a 416 SS CP rotor and EPDM elastomer stator, which had a 4-gal/min capacity at 40 psi. Both systems use tube-side backpressure pressure control valves consisting of Worcester Controls Actuated Ball Control Valve Model no. ¾-in. CPT 4466PMSE-A30 + 10755W actuator (120 Vac), Dataflow Positioner no. 10DFP17 + 4DK75 Feedback Position Module with all wetted parts 316 SS. The shell-side flow/pressure control valves on both systems were research control valves from Badger, no. 1001GCN36SVOEPO3S6, with EVA-1 electric actuator, P-2 trim with stellite, and 416 SS materials for the concentration system, and P-3 trim with stellite and 416 SS materials in the wash system.

The flowmeter for the concentration system was an Endress + Hauser "Promass 63" Coriolis mass flow measurement system, Model 63F-S15AAW00A25A3G, with ½-in. 904S SS tube for 1–22 gal/min at full scale. It had flanged welded process connections and used a DZL 363-551F311 process transmitter/interface with mass flow, density, and temperature outputs. For the slurry wash system, the slurry flow meter was an Endress + Hauser "Promag 33" electromagnetic flow measuring system, Model 33F-T15MD1ED11D21A, with a ½-in. tube for 0.8- to 28-gal/min full-scale operation. It also had flanged welded process connections. Both systems use filtrate flowmeters from Rheotherm, Rheotherm Flow Instrument Model no. 111D-I-TU1/16-4/20 flow sensing transducer, separate electronics, and interconnect cable with flow measurement from 1 to 25 mL/min.

The backpulse system for both filtration systems consists of a 1-L pressure tank with valves and fittings to contain about 0.7 L of filtrate during operation. When a backpulse was needed, upstream and downstream valves were closed, and the nitrogen gas–pressurizing valve was opened. When the pressure was about 100 psi, the pressurizing valve was closed, and the upstream valve to the filter was momentarily opened to force the contained filtrate back through the filter membrane and unplug the filter.

4.3 SHORT-CIRCUIT TESTING OF THE CSTR

To show that there was good hydraulic performance in the CSTR as designed, a test was done using a dye-tracer method with a spectrophotometric detection system. Using this method with an aqueous simulant system, the 15-L CSTR did not exhibit short-circuiting of the simulant solutions fed using any of the three impellers (A-100, A-200, and R-100) at two different stirring rates for the A-100 and the R-100 and one rate for the A-200 impellers. The tests were conducted by S. White of Savannah River, using his on-line visual spectroscopy system with blue dye, which had previously been used at Savannah River for smaller-scale CSTRs. A photo of the test configuration is shown in Fig. 10. Both pretest and posttest calibration checks were done. There was less than 1% error in the detected concentrations. A plot of the test results is shown in Fig. 11.

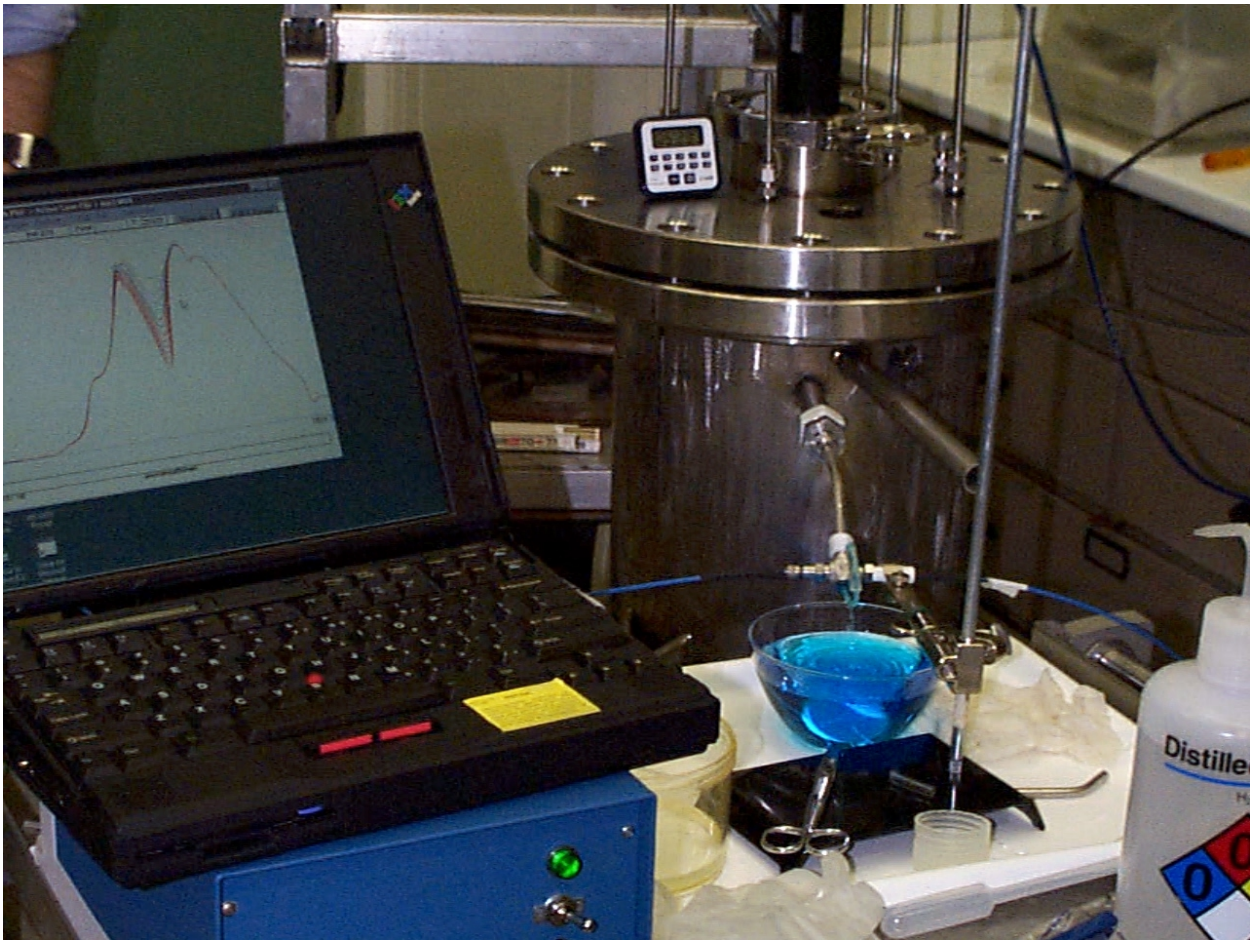


Fig. 10. Photo of spectrophotometric dye test and graph of the configuration for CSTR hydraulic performance.

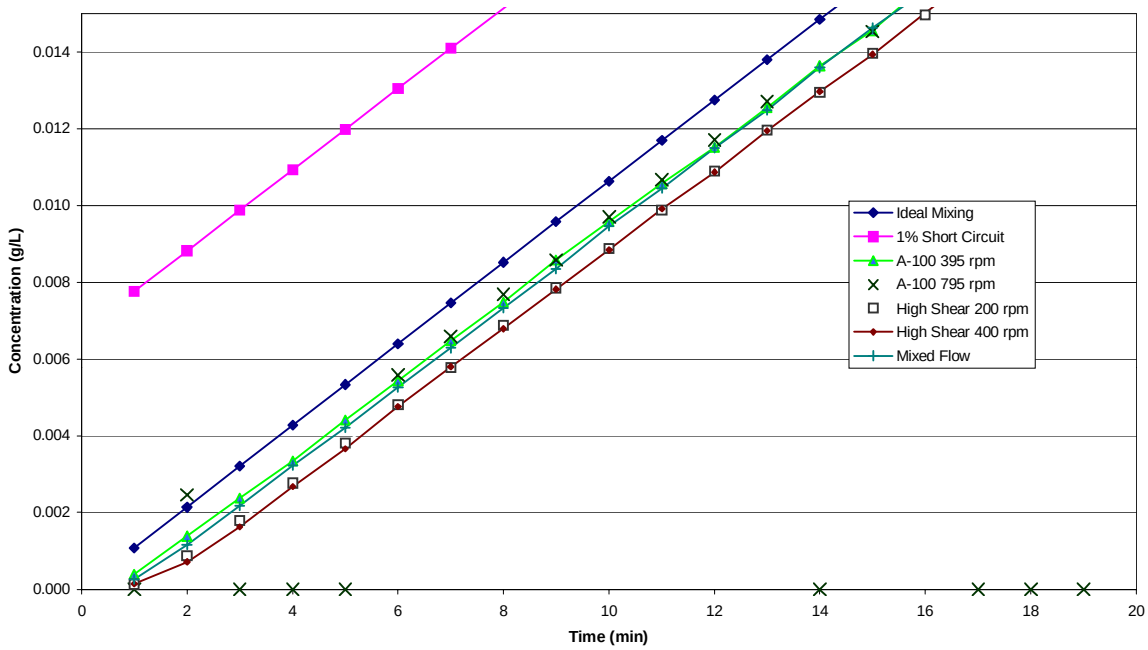


Fig. 11. Short-circuit test for the 20-L demonstration.

4.4 SYSTEMS INTEGRATION

The first series of tests using the CSTR system made use of the two CSTRs and the slurry concentration system. The two CSTRs and the concentration vessel were set up with the concentration filter loop outside the hot cell and operated with a simulated salt–TPB slurry to see how well the slurry would pass from one CSTR to the next and then to the concentration vessel by gravity. The three-way ball valves for slurry sampling were also tested at this time.

Once the flow hydraulics of slurry transfer between vessels was confirmed, the concentration loop control valves, flowmeters, and pressure transducers were connected to the testing system and their operation tested. Some of this information is reported in Sect. 4.6.

4.4.1 CSTR Hydraulic Tests

A set of CSTR experiments for testing the CSTR hydraulics, the behavior of a simulant and TPB/Surfynol® 420 solution, and the slurry concentration system was set up and operated. Two CSTRs in series were followed by the concentration tank, and all were set up for feed into the first CSTR with gravity flow from CSTR 1 to CSTR 2 and then to the concentration vessel. Average Savannah River supernatant simulant that had been prepared by P. A. Taylor (ORNL) to

5.7 *M* sodium and contained some of the Savannah River sludge mix, giving some color to the first CSTR and less to the second, was added to the two CSTRs. The feed was diluted with a NaOH solution containing NaTPB and Surfynol® to yield 500 ppm Surfynol® in each reactor and 0.55 *M* NaTPB in the solutions in each CSTR with the final concentrations shown in Table 9.

Table 9. Average simulated supernatant

Component	Conc. (*M*)	Conc. after dilution (*M*)
Na^+	6.0	4.7
K^+	0.015	0.0126
OH^-	1.9	1.60
NO_3^-	2.14	1.80
NO_2^-	0.52	0.436
AlO_2^-	0.31	0.26
CO_3^{2-}	0.16	0.134
SO_4^{2-}	0.15	0.126
Cl^-	0.25	0.21
F^-	0.032	0.027
PO_4^{3-}	0.01	0.008
$C_2O_4^{2-}$	0.008	0.0067
SiO_3^{2-}	0.004	0.0034
MoO_4^{2-}	0.0002	0.00017

The Surfynol®420 and TPB mixture was prepared as follows: half was prepared with NaTPB and the other half with Surfynol®420. Both were prepared by adding the TPB (half of total) and Surfynol®420 (half of total) to the water first and then adding the caustic to complete the volume.

Bottle A: 1.32 L H_2O
46 mL (50 wt % NaOH)
30.93 g TPB

Bottle B: 1.32 L H_2O
46 mL (50 wt % NaOH)
6.5 g Surfynol®420

The characteristics of the solutions showed that in Bottle A, the TPB was very soluble in the water with no foaming when shaking to dissolve. It became moderately cloudy when caustic was added and mixed; the color was pale amber. In Bottle B, beads of oily Surfynol®420 formed when shaken with water; after caustic was added and mixed vigorously, immiscible white blobs were observed floating on top of the solution. Half of Bottle A was added to Bottle B and mixed vigorously; there was no foaming or additional color. Then the other half of Bottle A was added to Bottle B and mixed vigorously; a whitish, waxy film started forming on the surface, but there

was no foam formation. The remainder of the TPB was then added and mixed; the floating film remained. Then the remainder of the Surfynol®420 was added and mixed; the surface film seemed somewhat heavier. This solution was then added slowly to the CSTR 1 while mixing.

Little foam formation was noticed at the slow stirring rate of 215 rpm. Mild surface movement could be seen as a result of the mixing. The second CSTR was prepared similarly, with the simulant poured in, and a solution of TPB/Surfynol®420/dilute NaOH was then prepared by adding 60.9 g TPB to 2.64 L H_2O and mixing well. To this was added 13 g Surfynol®420, and the solution was again mixed well; then 92 mL of 50 wt % caustic was added with vigorous mixing. There was an oily, immiscible substance floating on the surface in this preparation, which was added slowly to 12.27 L of the simulated supernatant in CSTR 2. An unbroken surface film occurred in CSTR 2, which was not broken up by the mixing of the solution at 215 rpm.

Both CSTRs were filled to 15 L and allowed to stir overnight. Then two pumps fed two streams, one containing the 5.7 *M* simulant and the other containing the previously described diluent with TPB and Surfynol®420, at 23.8 mL/min (simulated supernatant at 19.5 mL/min and TPB/Surfynol®420 solution at 4.3 mL/min) for a continuous flow of 2 h and 40 min. The slurry was allowed to cascade from the first CSTR to the second to the concentration vessel with all tubes that supply liquid terminating below the surface of liquid in the vessel. During the flow, small white clumps were occasionally seen passing through the plastic tubing. Flow was steady and smooth between the CSTRs, and no foam was seen in the vessels. Some samples were taken in graduated cylinders to see how the materials separated. After 2 h and 40 min, the pumps were shut off and the stirrers allowed to continue operation overnight. The next morning, only slight changes were seen in the surface of CSTR 1 solution and no foam formation was seen. However, CSTR 2 contained the blob of congealed Surfynol® 420/TPB, shown in Fig. 12, and the outlet line was plugged where it went from the ¾ -in. to the ¼-in. OD line.

To find the agitation rate at which foam formation began, the stirring speed was sequentially increased in each CSTR. The sequence was 520 rpm (for 10 min), 620 rpm (10 min), 735 rpm (10 min), 860 rpm (10 min), 920 rpm (10 min), and 1040 rpm (20 min), at which point foam formation was evident and cavitation of the impeller occurred. Mixing was returned to low rates, and after an hour, the CSTR liquid surfaces were inspected. Both CSTRs had a slight buildup of waxy particles on the wall at the surface of the solution.

Fig. 12. Photo of the blob of congealed Surfynol® 420/TPB in CSTR 2.

4.4.2 Concentration Filter Loop Tests

Using some of the material made during the previous hydraulic testing, the concentration loop was operated to concentrate slurry and test filtrate production. The cooling unit was attached to the cooling coils and set for operation at 25ºC. The concentration vessel was filled to the operating level of 7 L with the appropriate amount of feed and TPB solution. TPB slurry was fed to the concentration vessel with a peristaltic pump at about 32 mL/min. The loop was run with the Moyno pump operating at the slurry concentration flow rate of 3.6 gal/min for at least 2 h. The unit was operated with a 40-psi slurry backpressure, and filtrate flow was controlled using a hand valve. The system operated without problems and produced several liters of filtrate. A photo of the system is shown in Fig. 13.

Fig. 13. Concentration filter loop system.

4.5 SETUP AND TESTING OF THE CONTROL SYSTEMS FOR THE SLURRY CONCENTRATION AND WASHING LOOPS

An Operational Readiness Review for planned testing at the ORNL facility, relative to the small tank process, was completed on May 18, 1999. The review revealed four areas requiring additional work and preparation, one specific to Instrumentation and Controls (I&C). ORNL responded to the areas of concern with a request to Westinghouse Savannah River Company (WSRC) to provide assistance in correcting I&C issues defined in the recovery plan (HLW-SDT-99-0134).

Mockups for the slurry concentration loop and slurry wash loop equipment were staged in preparation for final installation in the hot cell. The efforts by SRS personnel were divided into three separate activities: Activity 1–checkout of all final control and measuring elements, Activity 2–configuration and checkout of the Intellution workstation and evaluation of the entire system during integrated water runs, and Activity 3–final checkout and tuning of the continuous control loops. The complete results of this activity are given in Appendix D.

Over a 3-week period, a checkout of all equipment associated with the slurry concentration and slurry wash loops was completed. The performance was evaluated and recommendations were made to address unsatisfactory conditions where deficiencies existed. The final control strategy for the test system was implemented as a cascaded control that used

LIC-1, the concentration tank level probe, as the primary (outer) loop that feeds its output to the setpoint of FIC-1, the secondary (inner) loop. During Test 1a, the cascade was broken due to the failure of the level probe, leaving a control scheme based on FIC-1. This was configured and tested in an integrated simulated run and proved very stable. The system was left in this configuration for the remainder of Test. 2. The final control element tuning constants are listed in Table 10. These values include a configuration for controlling filtrate flow rate based on level (LIC-1) or flow (FIC-1).

Table 10. Final control element tuning constants

Loop tag name	PID output low	PID output high	PID setpoint address	PID prop band	PID reset time	PID rate	PID output reverse
PIDPCV-1	0	100	PID1SP.F_CV	657.9	0.0125	0	Yes
PIDPCV-2A	0	40	SETPOINT2LEVEL.F	11.358	94.204	0	Yes
PIDPCV-2B	0	100	PIDPCV-2A.F_CV	2,035.00	0.255	0	No
PIDPCV-3	0	100	PID3SP.F_CV	452.8	0.0125	0	Yes
PIDPCV-4A	0	25	SETPOINT4LEVEL.F	50	20	0	Yes
PIDPCV-4B	0	100	PIDPCV-4A.F_CV	969	0.5618	0	No

4.6 WATER TESTS OF THE INSTALLED SYSTEMS

Once the system was cold tested for operation with the computer control and data acquisition system, all of the signal and control cables were disconnected, and the CSTRs, concentration vessels, and filter systems were installed into Hot Cell B. The feed system, consisting of two 55-gal drums with agitators, a circulation pump, and connecting pipe and valve were installed in adjoining Hot Cell A. After the tank and filter system installation was complete, the control and signal cables were reconnected. The system was retested to confirm acceptable operation of all the required systems. The tests consisted of pumping a feed simulant into the first CSTR and letting it overflow to the second and then to the concentration vessel. During this time, the sampling ports between the CSTRs and after CSTR 2 were tested. The concentration loop and wash loop instruments and control systems were tested and the calibrations for the instruments and valves checked. The salt solution feed tanks were filled from outside the cell. The cesium traced with ^{137}Cs, strontium traced with ^{85}Sr, and ^{235}U were added to the salt feed, mixed well, and then sampled. Photographs of the system are shown in Figs. 14–17.

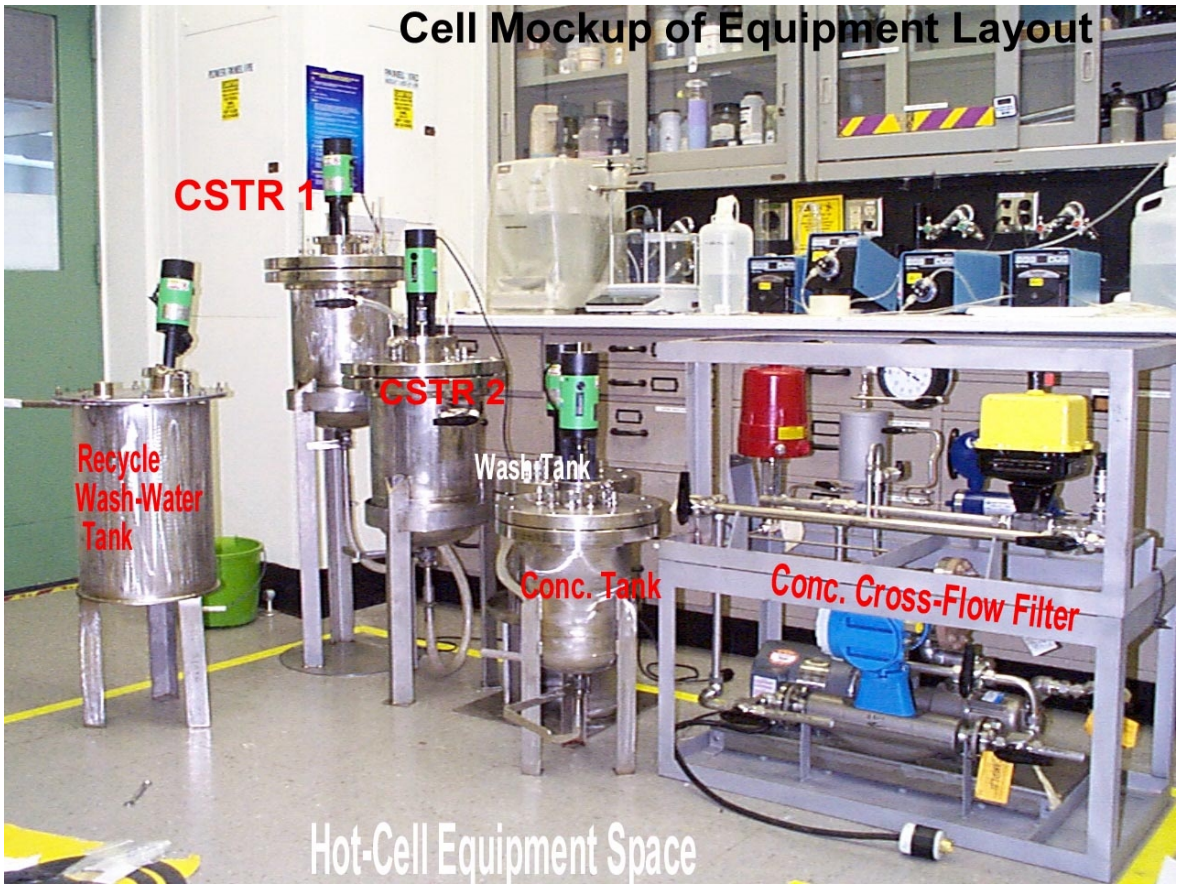


Fig. 14. Cell mockup of equipment layout.

5. CSTR SYSTEM TEST DESIGN

5.1 TEST 1a

Test 1a had the objectives of obtaining the necessary information to continue the experimental program. The information included the ability to attain the required removal of cesium by the TPB and the removal of strontium and uranium by MST during the processing steps. For the test, two CSTRs and the slurry concentration system with the CFF were used with an 8-h residence time per CSTR. During the test, 60% excess NaTPB was added and split between NaTPB feed (100% of the stoichiometric amount) and recycle wash water feed, which was a dilute stream containing the 60% excess NaTPB. The simulated waste salt feed was fed at about 21–21.5 mL/min rate and diluted with the other streams so that the total feed solution rate was about 31.5 mL/min. The experiment lasted about 40 h and provided more than five CSTR turnovers.

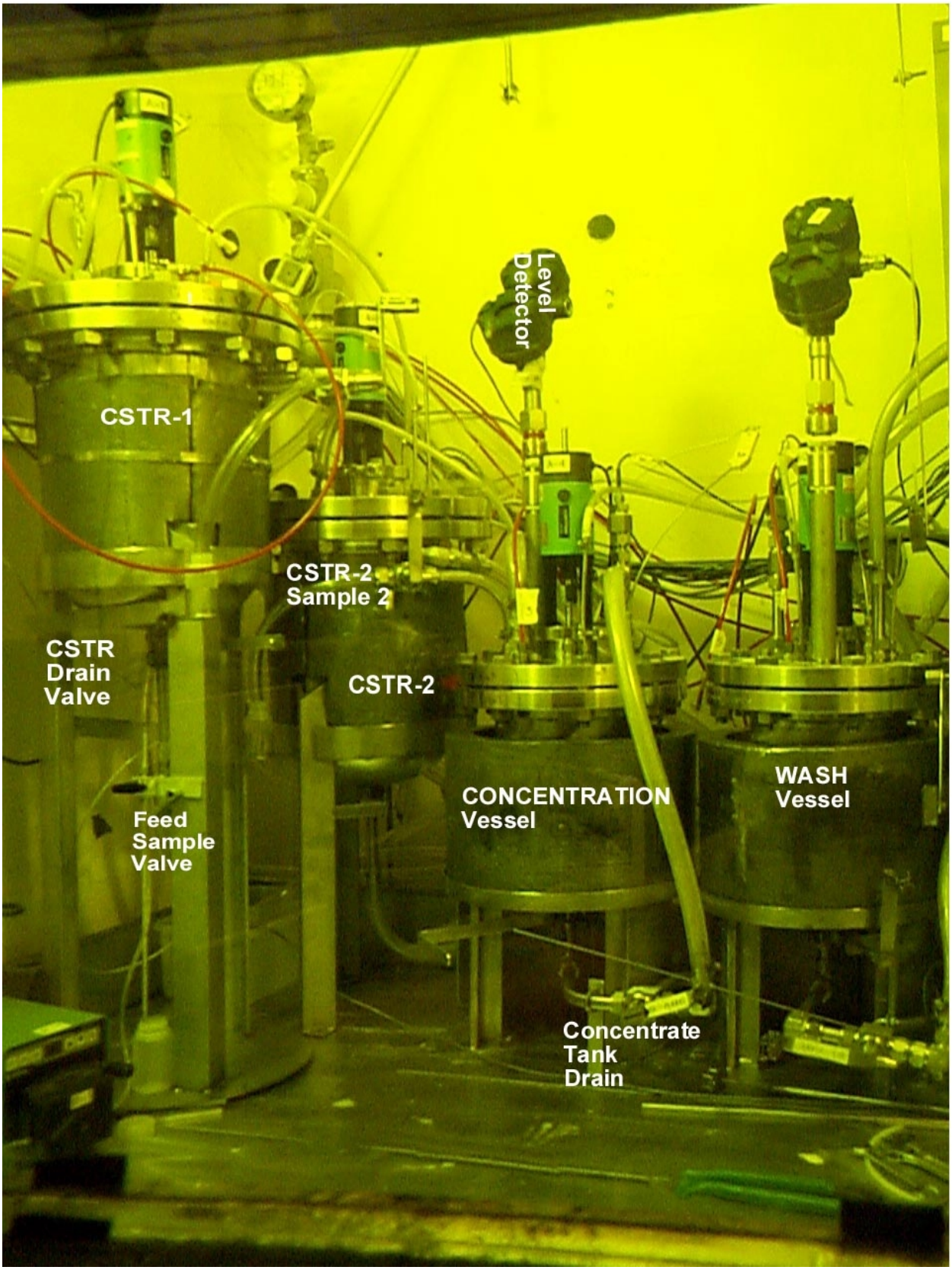


Fig. 15. Equipment setup.

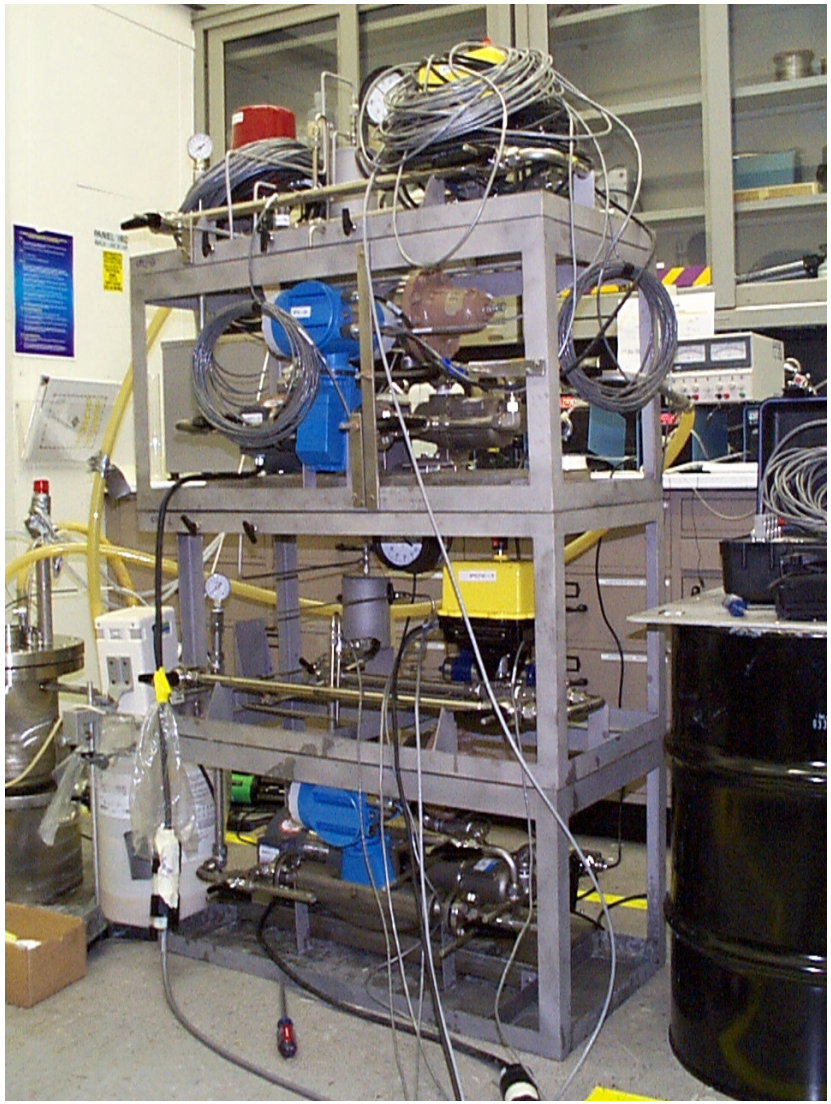


Fig. 16. Slurry wash filter system (top) and slurry concentration system ready for installation into the hot cell.

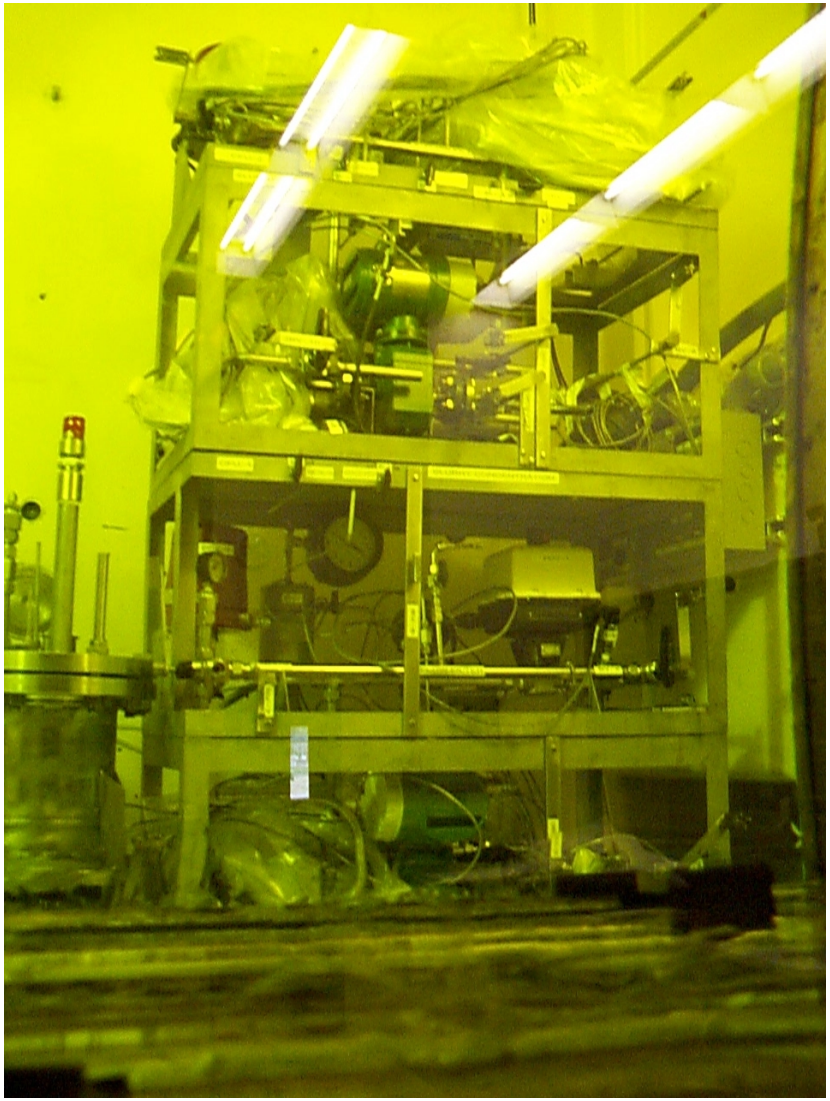


Fig. 17. Slurry wash filter system (top) and slurry concentration system after installation in the hot cell.

No catalysts were added to cause TPB decomposition. Because of the presence of strontium and uranium in the simulated sludge supplied by Savannah River, it was not added to the salt feed in this test. This allowed accurate determination of the strontium and uranium DFs from solution in the CSTRs. When sludge is present, some of the soluble tracer uranium and strontium can exchange with the insoluble strontium and uranium in the sludge and make accurate DF determination difficult or impossible.

The headspaces of all of the vessels that contain TPB were purged with nitrogen gas to prevent a possible explosive mixture of benzene (a decomposition product of TPB) from collecting in the headspace. This was not as important in Test 1a because catalysts were not used.

Cooling coils in the CSTRs, slurry concentration tank, and slurry wash tank were used to remove heat produced by the stirrers and pumps. Chilled water was pumped through each coil from individual refrigerated water baths. Thermocouples in the vessels provide the signal to adjust the temperature of the associated water bath in order to control the temperature in the vessel.

5.2 TEST 2 OPERATING PROCEDURES AND SAMPLING PLAN

Because of the success of Test 1a, the planning for Test 2 specified that two CSTRs in series with an 8-h residence time per CSTR be used. This setup was implemented. Both the slurry concentration CFF system and the slurry wash CFF system to recycle NaTPB were used. The recycle wash water obtained during the slurry washing was recycled to CSTR 1 in a fully integrated, closed-loop process. The goal was also to continue at the level of 60% excess NaTPB split between NaTPB feed, recycle stream feed, and makeup TPB feed, with NaTPB only being fed to CSTR 1. The feed rate was again specified at 21 mL/min for simulated waste, with the other streams diluting this to about 31.5-mL/min total feed. The system was operated continuously for about four system turnovers. Each system turnover required a slurry wash with wash water recycled to the first CSTR. Since each slurry concentration step required about 40–50 h of CSTR feeding, at least 200-h operation was necessary (230-h operation was completed).

In addition to the preceding requirements, the Test 2 plan called for ability to operate the CSTRs continuously, over an extended period of time (11 days). It was also necessary to close

the operation loop by recycling unreacted NaTPB to the first CSTR. In order to determine whether any TPB degradation was occurring due to operating conditions or to the ECC catalysts present, analytical testing for the degradation products of the TPB during processing was required.

Additional goals during the test included showing that the MST/TPB slurry could be concentrated to 10% solids by cross-flow filtration and that the concentrated solids could be washed to remove salt and excess TPB for recycle. It was also important to show that the degradation of TPB could be minimized by controlling the residence time, temperature, and atmosphere during processing, even in the presence of TPB degradation catalysts.

6. CSTR FEED DESIGN AND MAKEUP

6.1 PREPARATION OF SIMULATED WASTE SOLUTIONS FOR CSTR TEST 1a

The preparation of simulated waste solutions for CSTR Tests 1a and 2 at ORNL was based upon the average simulated waste composition as described in the SRS document WSCR-TR-99-00116.[4] The average solution represents a blend of all soluble waste in the SRS tank farms. The average simulated waste formulation has all the major waste components; however, there were a number of hazardous minor components such as chromium that were not included in Test 1a. In Test 2, sludge and catalysts were added to the salt feed that was used. Based on WSCR-TR-99-00116, only high-purity chemicals were purchased for the ORNL CSTR tests. Tables 11 and 12 provide the lot numbers and purities of the chemicals that were used, and Tables 13–15 give the preparation formulas, volumes, and concentrations of the feed used in Test 1a.

6.2 PREPARATION OF SIMULATED WASTE SOLUTIONS FOR CSTR TEST 2

Table 16 provides volumes, densities, and scheduled flow rates for the feed components used in Test 1a. Table 17 gives the concentrations of the radioactive components that were added to the salt feed. The preparation of the salt feed was based upon the formulation given in Table 13. Table 18 gives the composition of some of the primary salt constituents in the salt feed after the sludge and catalytic solution had been added. Those solutions decreased the concentration of the Na^+ and K^+ from 6.4 and 0.0171 *M* to 6.25 and 0.0167 *M*, respectively. The

calculated average simulated supernatant (6.4 *M* Na^+) for 1-L batches is given in Table 13. The compositions of the sludge and catalytic constituents in the salt feed are given in Tables 6, 7, and 8 of Appendix C. A total of 1.75 L (1.909 g sludge solids) of a 50-50 mixture of HM and PUREX sludge (see Tables 6 and 7 of Appendix C) were added to the salt feed. Both HM and PUREX sludge fractions had 15.4 wt % total solids. The total volume of salt feed in the two 55–gal tanks in Hot Cell A at the start of Test 2 was 384 L (101.5 gal).

Table 11. Chemicals used in simulated waste solution preparations

Chemical	Formula	Lot number	Purity (%)
Potassium nitrate	KNO_3	7028KXTG(3), 7028KXDK(4)	99.0
Sodium hydroxide	NaOH	M22635	Purified
Sodium nitrate	$NaNO_3$	M14156	99.8
Sodium nitrite	$NaNO_2$	38253840	97.0
Aluminum nitrate monohydrate	$Al(NO_3)_3 \cdot 9H_2O$	M25146	98–102
Sodium carbonate monohydrate	$Na_2CO_3 \cdot H_2O$	37310823	99.5
Sodium sulfate	Na_2SO_4	38362902, 126754/115904	99.0
Sodium chloride	NaCl	H4794058012C +$N01467C4	99.9
Sodium fluoride	NaF	7636 M42619	99.6
Sodium monohydrogen phosphate heptahydrate	$Na_2HPO_4 \cdot 7H_2O$	7914KXNY	99.9
Sodium oxalate	$Na_2C_2O_4$	38190834	99.5
Sodium metasilicate monohydrate	$Na_2SiO_3 \cdot 9H_2O$	K15161 & M16144	99.9
Sodium molybdate monohydrate	$Na_2MoO_4 \cdot H_2O$	H4793764012F +$M37620F6	99.9

Table 12. Other chemical and radioactive additives

Chemical	Formula	Lot number	Purity (%)
Sodium tetraphenylborate	NaTPB	EU 099009MS	
Cesium chloride	CsCl		99.9
Strontium nitrate	$Sr(NO_3)_2$	626131	99.9
Uranium oxide	UO_2		

The feed preparations for Test 2 were for a 264-h test. However, since the residence time in the CSTRs was shortened from 10.5 to 8.2 h before Test 1, the length of Test 2 was shortened to 230 h, which was enough test time to allow all the objectives of the test to be achieved. The actual averages for the flow rates over Test 2 were 21.17 mL/min for the salt feed, 1.116 mL/min for the NaTPB, 2.15 mL/min for the MST, and 7.24 mL/min for the recycle water.

Table 13. Average simulated supernatant (6.4 *M* Na^+) for 1-L batches

Component	Molarity (mol/L)	Compound	Molecular weight (g/mol)	Mass (g)
K^+	0.0171	KNO3	101.1	1.719
OH^-	3.611	NaOH	40.00	144.46[a]
NO_3^-	1.388[b]	$NaNO_3$	84.99	117.97
NO_2^-	0.594	$NaNO_2$	69.00	40.98
AlO_2^-	0.354	$Al(NO_3)_3 \cdot 9H_2O$	375.14	132.80
CO_3^{2-}	0.183	$Na_2CO_3 \cdot H_2O$	124.01	22.69
SO_4^{2-}	0.171	Na_2SO_4	142.04	24.29
Cl^-	0.0286	NaCl	58.44	1.671
F^-	0.0366	NaF	41.99	1.537
HPO_4^{2-}	0.0114	$Na_2HPO_4 \cdot 7H_2O$	268.09	3.056
$C_2O_4^{2-}$	0.0091	$Na_2C_2O_4$	284.2	1.307
MoO_4^{2-}	0.00023	$Na_2MoO_4 \cdot 2H_2O$	241.95	0.056
H_2O (added)[c]				796.25
Cs^+	0.00016	CsCl	168.45	0.027
UO_2^{2+}	0.0000043	$UO_2(NO_3)_2$		0.0000031
Sr^{2+}	0.00000054	$Sr(NO_3)_2$	211.62	0.000115

[a]The actual mass of NaOH that was required for a 1-L batch of 5.6 *M* Na^+ average simulated salt solution was 126.40 g, which actually yielded a 3.16 *M* solution of NaOH rather than the stated targeted molar concentration of 1.91. Based on the 3.16 *M* solution, a 1-L batch of 6.4 *M* Na^+ average simulated salt solution would require 3.611 mol of NaOH [(6.4/5.6)(3.16)].

[b]The target molarity for NO_3^- was 2.45 *M*. The NO_3^- to be added as $NaNO_3$ per 1-L batch = 2.45 mol – 3(0.354 *M*) = 1.388 *M*.

[c]The density given in the tables on pages 6 and 7 of HLW-SDT-99-0041 for salt solution feed was 1.294 g/mL. The calculated water mass needed for each liter of solution = 1294 g – 493.76 = 796.245 g.

Table 14. Volumes, densities, and flow rates for feed components in Test 1a

Feed component	Volume prepared		Density	Flow rate[a]	
	(L)	(gal)	(g/mL)	(mL/min)	(L/h)
Salt feed	151.4	40.0	1.288	21.5	1.290
MST + water	12.27	3.24	1.004	2.14	0.124
NaTPB	3.73	0.99	1.035	0.68	0.041
Recycle water	32.93	8.70	1.057	6.86	0.412

[a]Total flow rate was 31.18 mL/min (1.871 L/h).

Table 15. Concentration of radioactive components in salt feed in Test 1a

Element	Molarity (mol/L)[a]		Tracer	Activity (μCi/L)	
	In 6.4 *M* Na^+	In 4.7 *M* Na^+		In 6.4 *M* Na^+	In 4.7 *M* Na^+
Cesium	0.000134	0.0000924	^{137}Cs	7500	5172
Strontium	0.00000051	0.00000035	^{85}Sr	66	45.5
Uranium[b]	0.00000394	0.00000271	^{235}U		

[a]Cs (18 mg/L), Sr (0.045 mg/L), and U (0.925mg/L).
[b]Uranium = 93.34 wt % U-235.

Table 16. Feed solutions, volumes, densities, and flow rates for Test 2

Type feed	Volume prepared		Density	Flow rate[a]	
	(L)	(gal)	(g/mL)	(mL/min)	(L/h)
Salt feed	384	101.5	1.281	21.5	1.290
MST + water	12.27	3.24	1.004	2.14	0.124
NaTPB	16.29	4.30	1.034	1.08	0.065
Recycle water			1.057	6.86	0.412

[a]Total flow rate was 31.58 mL/min (1.895 L/h).

Table 17. Concentration of radioactive components in salt feed in Test 2

Element	Molarity (mol/L)[a]		Tracer	Activity (μCi/L)	
	In 6.25 *M* Na^+	In 4.47 *M* Na^+		In 6.25 *M* Na^+	In 4.47 *M* Na^+
Cesium	0.00016	0.00011	^{137}Cs	7917	5390
Strontium	0.000000525	0.000000354	^{85}Sr	84.5	57.6
Uranium[b]	0.0000043	0.0000029	^{235}U		

[a]Cs (21.4 mg/L), Sr (0.046 mg/L), and U (1.01mg/L).
[b]Uranium = 93.34 wt % U-235.

Table 18. Average salt feed with sludge and catalyst added for Test 2

Component	Molarity (mol/L)	Compound
Na^+	6.25	Composite of all Na salts
K^+	0.0167	KNO_3
OH^-	3.526	NaOH
NO_3^-	1.319	$NaNO_3$
NO_2^-	0.566	$NaNO_2$
AlO_2^-	0.337	$AL(NO_3)_3 \bullet 9H_2O$
CO_3^{2-}	0.174	$Na_2CO_3 \bullet H_2O$
SO_4^{2-}	0.163	Na_2SO_4
Cl^-	0.0273	NaCl
F^-	0.0348	NaF
HPO_4^{2-}	0.0108	$Na_2HPO_4 \bullet 7H_2O$
$C_2O_4^{2-}$	0.0086	$Na_2C_2O_4$
SiO_3^{2-}	0.0048	$Na_2SiO_3 \bullet 9H_2O$
MoO_4^{2-}	0.00038	$Na_2MoO_4 \bullet 2H_2$

6.3 PREPARATION AND ADDITION OF CATALYST TO SALT FEED TANKS

Table 19 is a modification of Table 1 of SRT-LWP-99-00099. It gives a description of the components of a modified ECC system, which was prepared at ORNL by WSRC personnel. The modified ECC system should provide the best opportunity for catalytic decomposition of NaTPB in Test 2. In previous work at the SRS, it was determined that the primary active components of the catalyst system in SRS waste were Pd, Cu, TPB decomposition product intermediates, and Hg. In addition to the catalysts, the modified ECC system has a number of other potential catalysts such as nickel and silver. A total of 8.2 L was added to the salt feed tanks in Hot Cell A via a ¾-in. stainless steel attachment with a funnel that was located in front of the cell in the room. Afterwards, the funnel and line were rinsed with a weighed amount of the salt feed. Tables 20 and 21 give a summary of the major constituents before and after dilution of the feed in CSTR 1.

Table 19. Composition of modified enhanced catalyst component system

Stock solution	Compound	Solvent	Stock species target concentration (mg/L)	Volume of stock solution required (mL)[a]	Species concentration in 6.25 *M* Na^+ feed (mg/L)[b]	Species concentration in 4.5 *M* Na^+ slurry (mg/L)[b]
Pd	$Pd(NO_3)_2$	0.1 *M* HNO_3	13010	494	16.7	11.6
Cu	$Cu(SO_4)\cdot 5H_2O$	Water	3710	494	4.8	3.3
2PB/1PB	$(C_6H_5)_2BOH$[c]		62500		161	110
	$(C_6H_5)B(OH)_2$	1.0 *M* NaOH	62500	990	161	110
3PB[c]	$(C_6H_5)_3B$	0.1 *M* NaOH	-	1205 g	161	110
Hg	$Hg(NO_3)_2\cdot H_2O$	0.1 *M* HNO_3	2200	494	2.9	2.0
Diphenylmercury[d]	$(C_6H_5)_2Hg$	Pure	-	74.5 g	194	132
Benzene[e]	C_6H_6	Pure	-	358 g	932	635
Mo/Cr/Si/Se/As	$Na_2MoO_4\cdot 2H_2O$		12010		15.2	10.3
	Na_2CrO_4		60070		77.9	53
	$Na_2SiO_3\cdot 9H_2O$	0.1 *M* NaOH	16020	494	20.9	14.2
	Na_2SeO_4		1000		1.3	0.89
	As_2O_3		41.2		0.048	0.033
Zn/Pb/Fe	$Zn(NO_3)_2\cdot 4H_2O$		8820		11.4	7.8
	$Pb(NO_3)_2$	0.1 *M* HNO_3	1200	494	1.5	1.02
	$Fe(NO_3)_3\cdot 9H_2O$		2600		3.3	2.2
Sn	$SnCl_2$	0.1 *M* HNO_3	2100	494	2.7	1.8
Ca/La/Co	$Ca(NO_3)_2\cdot 4H_2O$		12210		15.8	10.5
	$La(NO_3)_3\cdot 6H_2O$	0.1 *M* HNO_3	50.1	494	0.066	0.045
	$Co(NO_3)_2\cdot 6H_2O$		40.0		0.048	0.037
Cd/Ce	$Cd(NO_3)_2\cdot 4H_2O$		401		0.48	0.33
	$Ce(NO_3)_3\cdot 6H_2O$	0.1 *M* HNO_3	301	494	0.38	0.25
Rh	$Rh(NO_3)_3$	0.1 *M* HNO_3	1400	494	1.8	1.2
Ag	$AgNO_3$	0.1 *M* HNO_3	6810	494	8.8	6.0
Ru	$RuCl_3\cdot xH_2O$	0.1 *M* HNO_3	5410	494	7.0	4.8
Inactive Organics	Isopropanol		50100		64.6	44
	methanol	0.1 *M* NaOH	5010	494	6.7	4.6
Phenol[g]	C_6H_5OH	Pure	-	62.0 g	162	110
Biphenyl[h]	$(C_6H_5)_2$	Pure	-	74.5 g	194	132

[a]Volume of each stock solution required assumed the volume of 6.4 *M* Na^+ feed solution was 365 L (96 gal).

[b]The actual volume of feed solution was 384 L (101.4 gal).

[c]2PB was purchased as diphenylborinic acid ethanolamine complex. No pretreatment was performed on it prior to preparation of the stock solution.

[d]3PB was purchased as a ~6 wt % solution (in 0.1 *M* NaOH) of triphenylborane sodium hydroxide adduct. It was added as purchased (i.e., stock solution not prepared). The quantity required (1205 g) was the total weight of the as-received solution.

[e]Benzene was added as a pure compound (i.e., stock solution not prepared).

[f]Diphenylmercury was added as a pure compound (i.e., stock solution not prepared).

[g]Phenol was added as a pure compound (i.e., stock solution not prepared).

[h]Biphenyl was added as a pure compound (i.e., stock solution not prepared).

Table 20. Salt feed characteristics

Species of interest	Molarity	Concentration (ppm)	Concentration
Na^+	6.25		
K^+	0.0167		
OH^-	3.52		
Cs^+	0.00016	21.4	7.92 mCi ^{137}Cs/L
Sr^{2+}	0.000012	1.08	84.53 μCi ^{85}Sr/L
UO_2^{2+}	0.0000043	1.01	93.34% ^{235}U
Pd	0.000158	16.7	
Cu	0.000076	4.8	
Hg	0.00056	113	

Table 21. Salt feed characteristics as diluted in CSTR 1

Species of interest	Molarity	Concentration (ppm)	Concentration
Na^+	4.54		
K^+	0.01137		
OH^-	2.54		
Cs^+	0.000109	14.57	5.39 mCi ^{137}Cs/L
Sr^{2+}	0.0000082	0.735	57.53 μCi ^{85}Sr/L
UO_2^{2+}	0.0000029	0.688	
Pd	0.000108	11.6	
Cu	0.000052	3.3	
Hg	0.00038	77	

7. RESULTS OF TEST 1 OPERATION

7.1 TEST 1a STARTUP

The CSTR system for Test 1a consisted of two CSTRs in series and the slurry concentration filter loop, which included the feed tank (concentration vessel), filter, and the slurry and filtrate handling systems. The configuration is shown in Figs. 2 and 3. The test was designed to operate with an 8-h CSTR residence time (8 h in each CSTR for a total of 16 h of reaction time) with a simulated waste feed of 6.4 *M* sodium salt solution diluted with 0.55 *M* NaTPB solution; MST/dilution water; and a simulated slurry wash water recycle solution that contained about 1.2 *M* sodium, which gave a final sodium concentration of 4.7 *M*. The NaTPB was fed at about 160% of the stoichiometric amount required to precipitate all of the potassium and cesium present in the feed. The NaTPB was fed in two parts consisting of 100% of the stoichiometric amount as 0.55 *M* NaTPB feed and the 60% excess in the simulated recycle wash water stream (0.0033 *M* NaTPB).

The 15-L CSTR system was prepared for startup on Wednesday, June 23, 1999, by adding the final component—^{137}Cs—to the salt feed mixture. The uranium and ^{85}Sr had been added earlier and allowed to equilibrate for a day. CSTR 1 was emptied and prepared to receive the salt feed mixture. Component checkout testing using CSTR 2, the concentrate vessel, and cross-flow filtration system was completed, and they were then prepared to receive the simulated waste feed and dilution water as it eventually overflowed from CSTR 1. Since 8 h was required to fill CSTR 1, the flow tests on CSTR 2 and the concentration filter loop were completed while CSTR 1 was being filled.

The CSTR salt feed system was set up for the test to pump the feed from the feed tanks in Hot Cell A to CSTR 1 at the designed flow rate, 21.5 mL/min, and pumped at about 21.5 to 22 mL/min for the duration of Test 1a. In preparation for the test, deionized water was used as a substitute for the NaTPB, MST, and recycle feed to produce the desired dilution in the three vessels during the initial filling operation. Dilution water was the only material added with the salt feed during this stage. The dilution water was fed in from outside of Cell B using the recycle wash water feed pump, running at about 9.8–10.3 mL/min to give the desired total feed rate to the CSTRs of about 32 mL/min.

Because of the 32-mL/min flow rate to the CSTRs, the total time to fill the two CSTRs was about 16 h. Another 3 h was required to add about 3 L of the diluted simulant liquid to the concentrate vessel from CSTR 2. This volume was added to allow the CFF circulation loop to be filled prior to starting the actual concentration filtration to avoid the formation of foam from the air in the circulation loop tubing.

When CSTR 2 was about half full, the tubing on the outlet side of the water pump feeding CSTR 1 came off the pump head. Unfortunately, about 100 mL of water and radioactive salt feed from CSTR 1 was siphoned back through the line and spilled onto the floor near the water pump. Once the hose was replaced, the salt feed and dilution water flows were continued until CSTR 2 was filled. Then the feed was shut off, the pumps secured, and the radioactive contamination cleaned up. The cleanup took about 24 h, during which time no operations with the CSTR system took place. The total volume of salt feed and water required was 30.8 L. A total of 9.85 L of water was used, leaving 20.95 L of salt feed added to the two CSTRs. The Na^+ molarity was 4.35.

After decontamination and cleanup had been completed on June 25, the salt feed and dilution water flows to CSTR 1 were resumed. A spare pump was used to replace the contaminated water pump. After about 3 L of diluted salt feed had accumulated in the concentrate vessel, the salt feed and water dilution pumps were turned off to prepare for the actual NaTPB startup. The circulation pump (MP-1) was turned on, and the filter loop was filled. The nitrogen to the three vessels was turned on to provide a flowing inert cover gas. The NaTPB, MST/dilution water, and simulated recycle wash water feed tanks were filled. The feed pumps for these feeds were turned on, and the flows were established to CSTR 1. When all of these streams had reached the CSTR 1, the salt feed was restarted. Officially, the start time for Test 1a was 11:46 p.m., June 25, 1999.

7.2 OPERATION DURING TEST 1a

About 1 h after the test was initiated, a white tint could be seen in the overflow from CSTR 1 to CSTR 2. Shortly afterwards, about 1.25 h into the test, the diluted salt feed from CSTR 2 filled the concentrate vessel to operating level, and the filtration system was started. Filtrate then started to flow through the control valve to the filtrate collection tank. The feed rates for the individual feed streams were adjusted during the next several hours to achieve the desired individual flow rates.

The slurry in the slurry concentration tank was recirculated through the CFF using a Moyno pump. The pressure control valves worked well in adjusting the TMP in the filter unit and maintained a constant filtrate production rate that matched the feed rate to the first CSTR minus the slurry accumulation rate in the concentration step. Alternately, the pressure control valve was used to vary the filtrate production rate to maintain a constant level in the slurry concentration tank (until the level probe failed). In both cases, mass balance was maintained across the system. The filtrate was discharged to a filtrate-product holding tank.

The operation of the filtration loop pressure control valves was controlled from a computer in room 105, in front of the hot cell. The backpressure on the slurry side of the filter was controlled at 40 psi, and the Moyno pump speed was adjusted to obtain a flow rate of about 3.5 gal/min. During most of the test, the speed was 1460 rpm. The filtrate flow control valve was set to give a filtrate flow of 29–30 mL/min and, for most of the test, was controlled by the filtrate flowmeter.

The valve was also controlled for a short time using the level meter in the slurry concentration vessel, but this method had to be discontinued when the slurry got too thick and plugged the level meter tube. The flow control worked well, and only minor problems occurred. One problem was that each time the cell doors were opened to remove samples, the pressure inside the cell increased, causing a rate disturbance in the filtrate flow. The control system quickly brought the disturbance back to the desired flow rate, resulting in little disruption to the system. During one sampling period, however, a measurement of the filtrate flow rate was made during this disturbed flow time. At this point, the operators thought that the rate was too high and the concentration vessel was being emptied. The control system was placed in manual mode for a few hours until it was determined that there were no concerns. There were no other operational problems with the CSTRs or the filtration loop systems, and they all performed as designed, with the exception of the level meter, previously described.

7.3 HYDRAULICS AND THE SLURRY CONCENTRATION SYSTEM

Hydraulics behavior was excellent, and sound overall system operability was accomplished. No foaming problems were noted in CSTRs 1 or 2 or in the slurry concentration loop. The overall process control behavior was exceptional. There were no problems associated with the pressure and flow control valves or pressure transducers in the concentration loop. The

Coriolis flowmeter provided excellent service and provided indications when parts of the system were not in mass balance. Figure 18 is a graph covering the duration of the experiment that shows the behavior of the concentration loop during the 60-h test. The variables shown include the CFF shell- (PIC-1) and tube-side (PI-2) pressures, the temperature of the slurry in the concentration loop (TI-3), the slurry (SSI-1F) and filtrate (FIC-1) flow rates, and the slurry density (SSI-1D). As the slurry was concentrated, the density indication showed a steady decrease, which was expected for solids that were less dense than the salt solution. One of the most obvious points to note in the graph is the increasing TMP (the difference between PIC-1 and PI-2) as the run progressed, indicating that the filter was becoming plugged; however, no backpulsing or chemical cleaning was performed during the run.

One of the operational problems, as mentioned earlier, was the failure of the concentration tank level instrument. The radar probe was a ¼-in. rod inside a ¾-in. stilling well. After the concentration reached about 1.5% solids, the annulus inside the probe became plugged, negating its usefulness as a level-measuring instrument. For the remainder of the test, the filter system was operated with flow control on the filtrate backpressure control valve (FIC-1→ PCV-2). The filtrate was also collected on a balance, giving a secondary check on the flow rate

7.4 ANALYTICAL RESULTS

At the appointed times, samples (55–65 mL, collected over a 2-min duration) were taken between CSTR 1 and CSTR 2 [sample port 1 (SP-1), every 4 h], between CSTR 2 and the concentration vessel [sample port 2 (SP-2), every 4 h] and from the filtrate line before it entered the collection tank [sample port 4 (SP-4), every 2 h]. After 24 h of operation, the radiation level was low enough to pose no radiation dose concerns during the normal sampling procedures, and filtrate flow was routed to the collection bottle in the sampling window.

Samples from SP-1 and SP-2 were prepared for analysis by filtering the slurry with 0.45-µm syringe filters and placing the filtrate in new sample bottles for transfer to the RMAL for gamma counting of the ^{85}Sr and the ^{137}Cs, ICP analysis for the metal ions, and HPLC analysis for TPB. Portions of some of the samples were sent by RMAL to be analyzed for uranium by DNC. A small amount of each sample was also transferred to a 4-mL counting tube for in-house gamma counting of the ^{85}Sr and the ^{137}Cs added to the feed. The filtrate samples from SP-4 were

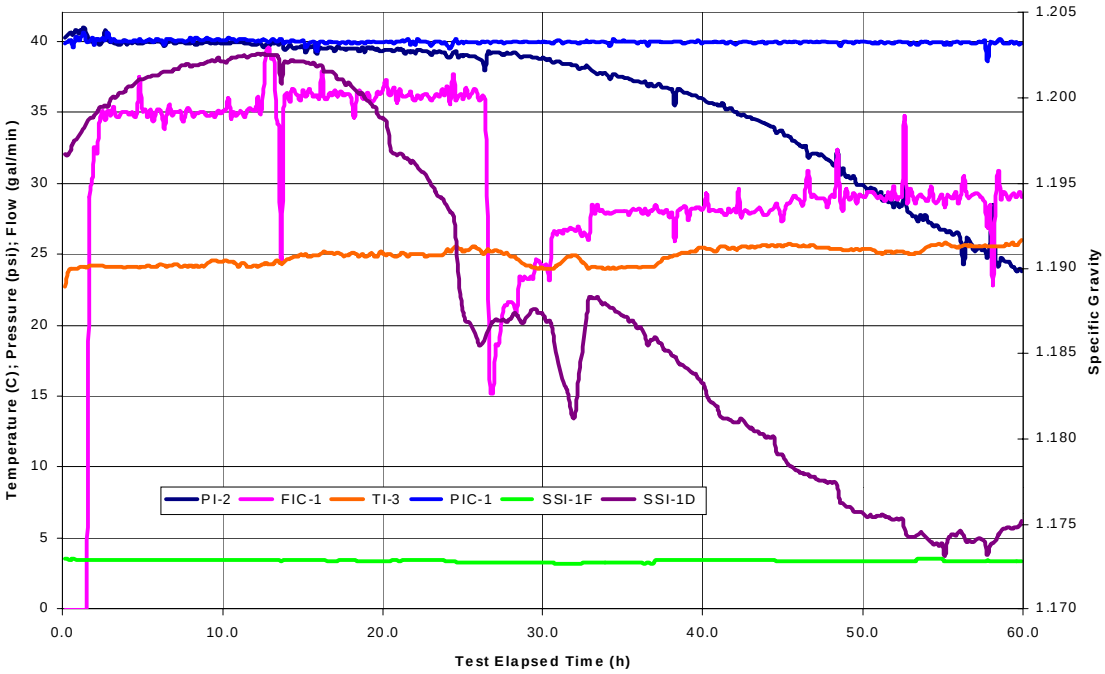


Fig. 18. CSTR Test 1a (start 6/25/99 at 2345).

sent as collected for analysis after a small sample (1–3 mL) of the filtrate was placed in a counting tube for in-house counting. All samples destined for the analytical laboratory were immediately placed in a refrigerator after preparation, where they remained until transport. The analytical results are given in Tables 22–24.

7.5 SALT FEED DECONTAMINATION

The results of the in-house gamma analyses of ^{137}Cs and ^{85}Sr of filtered samples from CSTR 1 and CSTR 2 showed that the desired DFs were achieved quickly. A DF of greater than 10,000 for ^{137}Cs was attained by 20 h of operation, as shown in Fig. 19. The concentration reached a low value and gradually increased to a steady-state value at about 60 h into the test. In CSTR 2, a DF for ^{137}Cs greater than 10,000 occurred after 28 h of operation. The DF reached steady state after about 60 h, as it had for CSTR 1. DFs for the filtrate from the CFF exceeded 10,000 after about 32 h and stayed relatively constant for the rest of the test.

The potassium concentration profile for the CSTR 1 is shown in Fig. 20, giving both the concentration if no TPB was present and the concentration that actually occurred as measured by the sampling after TPB feed started and throughout the test. Potassium behavior is important because the cesium concentration cannot be at the lowest levels until the potassium has all been essentially precipitated as potassium tetraphenyl borate (KTPB).

Additional plots of the cesium concentration with time are shown in Figs. 21–23. They show a comparison between the internal counting and the analytical sample counting at RMAL. The agreement is very good in all cases. Figure 21 is a plot of the filtrate concentration of ^{137}Cs. The required DF was met, and the concentration remained low, with some variation seen in the internal samples. Some of the variation was due to the larger samples, low cesium content, and counting geometry. The results for the post–CSTR 2 samples are shown in Fig. 22. These again show good agreement between internal and RMAL counting. There was also a rising trend from the minimum cesium concentration during the remainder of the run, while the filtrate samples showed a hint of a downward trend. The same tendencies are shown in Fig. 23 for the post–CSTR 1 samples, but the upturn in concentration is more pronounced. It should be noted that all of the samples from CSTR 1 and CSTR 2 are equilibrium samples because the slurry and salt solutions cannot be separated in less than a half hour after removing a sample from the reactors. After removal from the hot cell, which required a survey by a health physics technician, each sample was then taken to a hood for separation using a syringe filter.

While cesium concentration was decreasing, the NaTPB concentration in the supernatant was increasing. As discussed later, the soluble TPB concentration was below detection limits during the first part of the run. The same behavior was observed in batch contact tests using salt solutions, in which NaTPB was added and allowed to mix for various lengths of time.

These results indicate that an 8-h residence time in each CSTR was above the minimum contact time needed for the precipitation reaction to take place and for cesium to be removed from this simulated feed mixture.

The desired strontium DF was also obtained at all three sampling ports, as shown in Figures 24–27. A DF of greater than 50 was obtained at both sample ports 1 and 2 after about 20 h of operation. A DF of greater than 50 was not obtained until near the end of the test (between 56 and 60 h) for the concentrate filtrate from the cross-flow filtration system.

Table 22. Test 1a analytical data for sample port 1

	In-house analysis			External analysis							
Reaction time (h)	CSTR 1 sample ID	Cs-137 (µCi/g)	Sr-85 (µCi/g)	Cs-137 (µCi/mL)	Sr-85 (µCi/mL)	K (mg/L)	Total U (mg/L)	DNC U-235 (mg/L)	NaTPB (mg/L)	B (mg/L)	Na (mg/L)
0	T1aSP1-Feed	3.462E+00	2.824E-02	5.026E+00	4.096E-02	215.81	0.638	0.596	0	0	0
4	T1aSP1-01F	5.830E-01	1.146E-02								
8	T1aSP1-02F	2.361E-01	1.639E-03	3.240E-01	3.780E-03	192.00	0.568	0.530	<.81	<.48	90700
12	T1aSP1-03F	1.229E-01	1.131E-03								
16	T1aSP1-04F	5.205E-02	7.949E-04	5.670E-02	8.370E-04	64.90	0.493	0.460	<.81	<.48	94900
20	T1aSP1-05F	7.528E-05	5.521E-04								
24	T1aSP1-06F	1.640E-04	3.621E-04	2.700E-05	5.400E-04	35.90	0.443	0.413	148	4	96400
28	T1aSP1-07F	1.615E-04	3.008E-04								
32	T1aSP1-08F	1.194E-04	3.550E-04	4.860E-05	4.590E-04	39.40	0.406	0.379	92	2	98
36	T1aSP1-09F	9.095E-05	2.898E-04								
40	T1aSP1-10F	6.403E-06	3.379E-04	5.400E-05	4.590E-04	19.60	0.384	0.358	60	2	123000
44	T1aSP1-11F	7.772E-06	3.146E-04								
48	T1aSP1-12F	1.445E-04	3.384E-04	8.910E-05	5.130E-04	33.70	0.389	0.363	84	2	158000
52	T1aSP1-13F	1.006E-04	4.411E-04								
56	T1aSP1-14F	1.126E-04	4.425E-04	9.720E-05	6.750E-04	29.30	0.403	0.376	72	2	115000
60	T1aSP1-15F	9.931E-05	4.635E-04								

Table 23. Test 1a analytical data for sample port 2

	In-house analysis			External analysis							
Reaction time (h)	CSTR 2 sample ID	Cs-137 (µCi/g)	Sr-85 (µCi/g)	Cs-137 (µCi/mL)	Sr-85 (µCi/mL)	K (mg/L)	Total U (mg/L)	DNC U-235 (mg/L)	NaTPB (mg/L)	B (mg/L)	Na (mg/L)
0	T1aSP2-Feed	3.462E+00	2.824E-02	5.026E+00	4.096E-02	215.81	0.638	0.596	0	0	0
4	T1aSP2-01F	2.407E+00	1.789E-02								
8	T1aSP2-02F	1.451E+00	9.618E-03	1.863E+00	1.404E-02	186.00	0.614	0.573	<.81	<.48	92100
12	T1aSP2-03F	8.123E-01	3.573E-03								
16	T1aSP2-04F	2.981E-01	1.145E-03	4.050E-01	2.133E-03	165.00	0.534	0.499	<.81	<.48	93100
20	T1aSP2-05F	1.073E-01	6.187E-04								
24	T1aSP2-06F	1.719E-02	5.302E-04	2.133E-02	6.750E-04	89.90	0.475	0.444	<.81	<.48	75200
28	T1aSP2-07F	7.453E-05	3.528E-04								
32	T1aSP2-08F	2.933E-05	3.373E-04	3.240E-05	4.860E-04	40.60	0.420	0.392	125	4	95300
36	T1aSP2-09F	5.322E-05	2.928E-04								
40	T1aSP2-10F	3.156E-05	2.406E-04	3.510E-05	3.240E-04	32.70	0.381	0.356	78	2	112000
44	T1aSP2-11F	5.236E-05	2.221E-04								
48	T1aSP2-12F	6.520E-05	2.520E-04	6.480E-05	3.510E-04	29.10	0.353	0.330	98	3	172000
52	T1aSP2-13F	5.581E-05	2.960E-04								
56	T1aSP2-14F	1.228E-05	3.115E-04	7.020E-05	4.050E-04	26.80	0.365	0.341	79	2	116000
60	T1aSP2-15F	1.025E-04	3.264E-04								

Table 24. Test 1a analytical data for sample port 4

		In-house analysis		External analysis					
Reaction time (h)	Conc. tank filtrate sample ID	Cs-137 (µCi/g)	Sr-85 (µCi/g)	Cs-137 (µCi/mL)	Sr-85 (µCi/mL)	K (mg/L)	Total U (mg/L)	DNC U-235 (mg/L)	NaTPB (mg/L)
0	T1aSP4-Feed	3.462E+00	2.824E-02	5.026E+00	4.096E-02	215.81	0.638	0.596	0
2	T1aSP4-01	2.986E+00	0.000E+00						
4	T1aSP4-02	2.949E+00	4.087E-03						
6	T1aSP4-03	2.935E+00	5.227E-03						
8	T1aSP4-04	2.528E+00	6.622E-03	3.510E+00	1.242E-02	365.00	0.548	0.512	<.81
10	T1aSP4-05	2.131E+00	8.130E-03						
12	T1aSP4-06	1.739E+00	6.946E-03						
14	T1aSP4-07	1.284E+00	6.310E-03						
16	T1aSP4-08	9.134E-01	6.699E-03	1.188E+00	9.180E-03	203.00	0.531	0.496	<.81
18	T1aSP4-09	6.087E-01	6.152E-03						
20	T1aSP4-10	3.251E-01	4.777E-03						
22	T1aSP4-11	1.920E-01	4.387E-03						
24	T1aSP4-13	1.015E-01	3.873E-03						
26	T1aSP4-12	9.478E-03	3.641E-03	9.720E-03	4.860E-03	64.40	0.464	0.433	43
28	T1aSP4-14	3.691E-03	3.226E-03						
30	T1aSP4-15	8.144E-04	2.981E-03						
32	T1aSP4-16	1.206E-04	2.572E-03	8.910E-05	3.510E-03	41.60	0.417	0.390	287
34	T1aSP4-17	5.277E-05	2.411E-03						
36	T1aSP4-18	6.711E-05	2.052E-03						
38	T1aSP4-19	1.733E-04	1.842E-03						
40	T1aSP4-20	5.731E-05	1.816E-03	4.590E-05	2.268E-03	30.70	0.365	0.341	222
42	T1aSP4-21	3.902E-05	1.502E-03						
44	T1aSP4-22	4.941E-05	1.348E-03						
46	T1aSP4-23	5.469E-06	1.264E-03						
48	T1aSP4-24	8.158E-05	1.158E-03	4.320E-05	1.593E-03	35.30	0.323	0.302	212
50	T1aSP4-25	7.154E-05	1.167E-03						
52	T1aSP4-26	1.092E-04	1.048E-03						
54	T1aSP4-27	6.087E-05	1.043E-03						
56	T1aSP4-28	6.937E-05	9.133E-04	3.780E-05	1.323E-03	27.20	0.308	0.288	202
58	T1aSP4-29	7.658E-05	8.855E-04						
60	T1aSP4-30	8.239E-05	8.392E-04						
61.7	T1aSP4-31	3.616E-05	7.443E-04						

During the first 30 h of operation, the uranium DF steadily increased with time at the three sampling ports as shown in Fig. 28. Afterwards, it leveled out in the CSTRs but continued to increase in the concentrate filtrate through the end of the run. The maximum uranium DFs obtained were 2.06 for the filtrate, 1.6 for CSTR 1, and 1.75 for CSTR 2.

After the 56-h samples had been analyzed, it was clear that both the cesium and strontium DFs had been obtained in the two CSTRs and that the CSTR operation had almost achieved steady state. The filtrate DF was also beginning to reach steady state. It was then decided to shut the system down after 61-h operation or about 7.5 turnovers (residence times) in CSTR 1. Two hours before shutdown, the control systems for both PCV-1 and PCV-2 were stepped up and down slightly to obtain process parameters under concentrated slurry conditions.

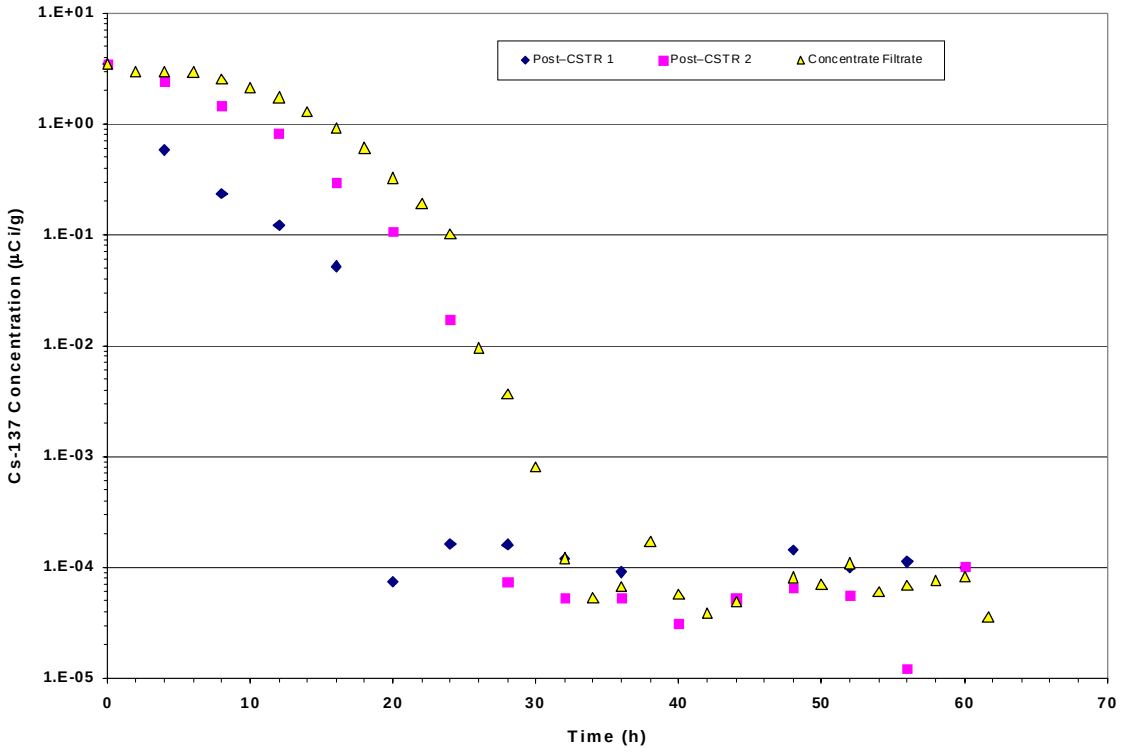


Fig. 19. Cesium concentration vs time for Test 1a (internal samples).

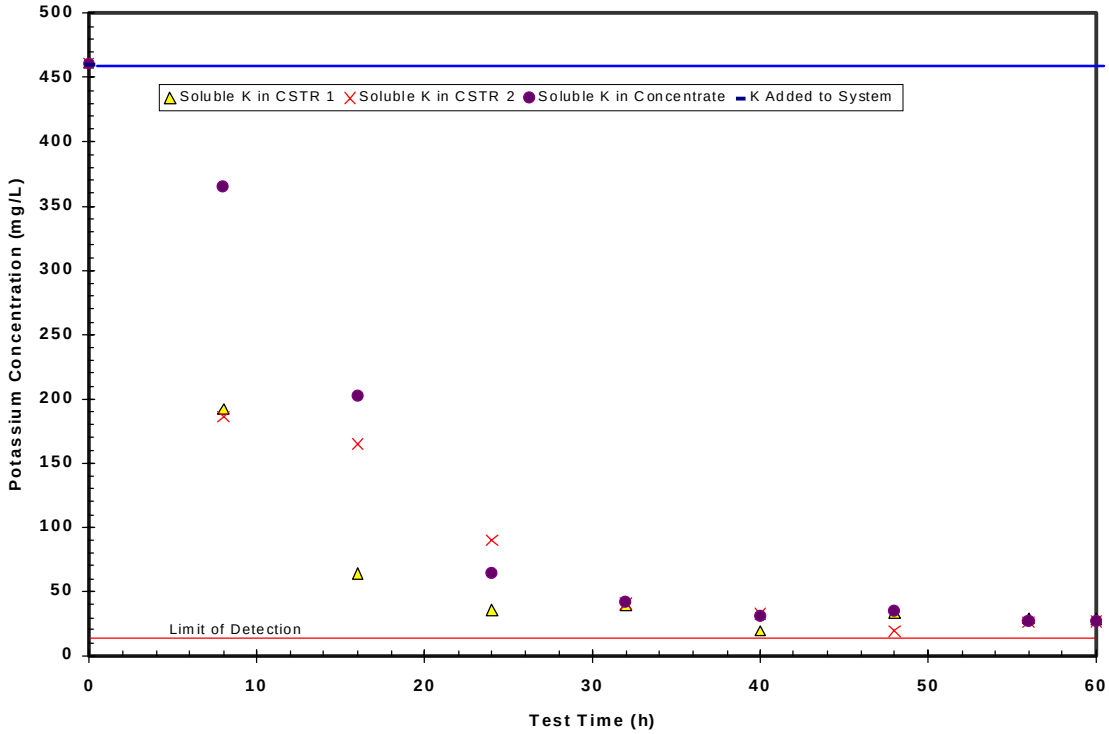


Fig. 20. Potassium concentration vs time for Test 1a.

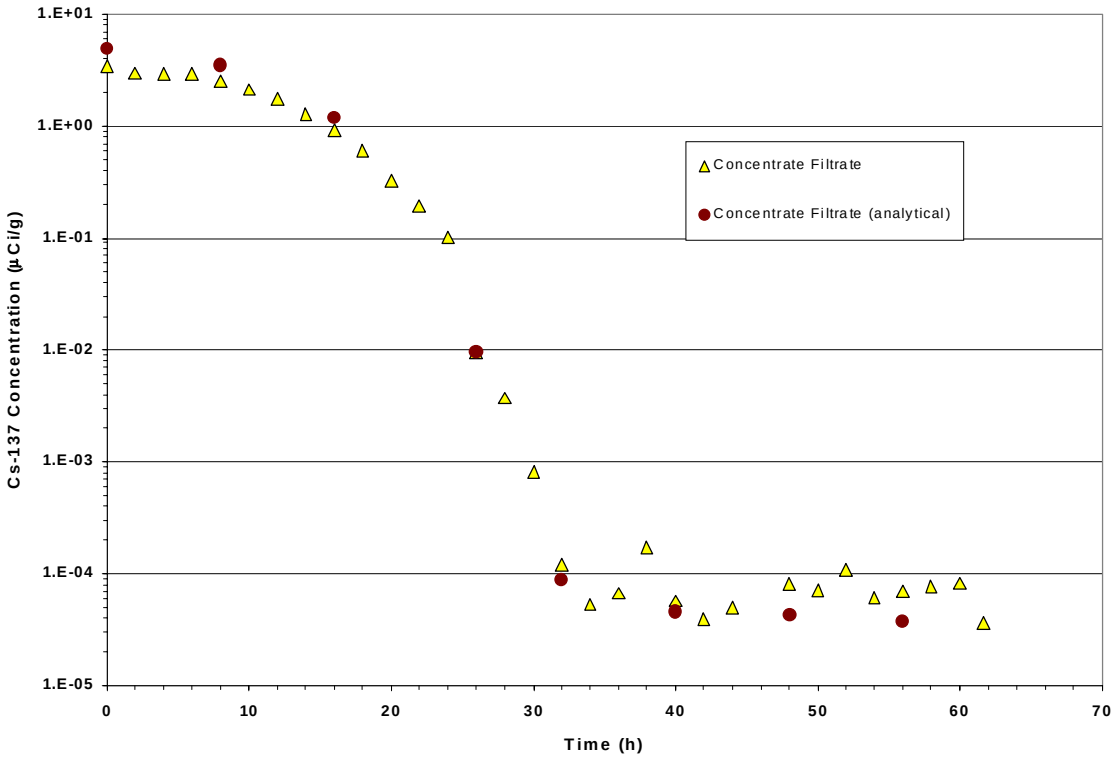


Fig. 21. Cesium concentration vs time for Test 1a concentrate filtrate.

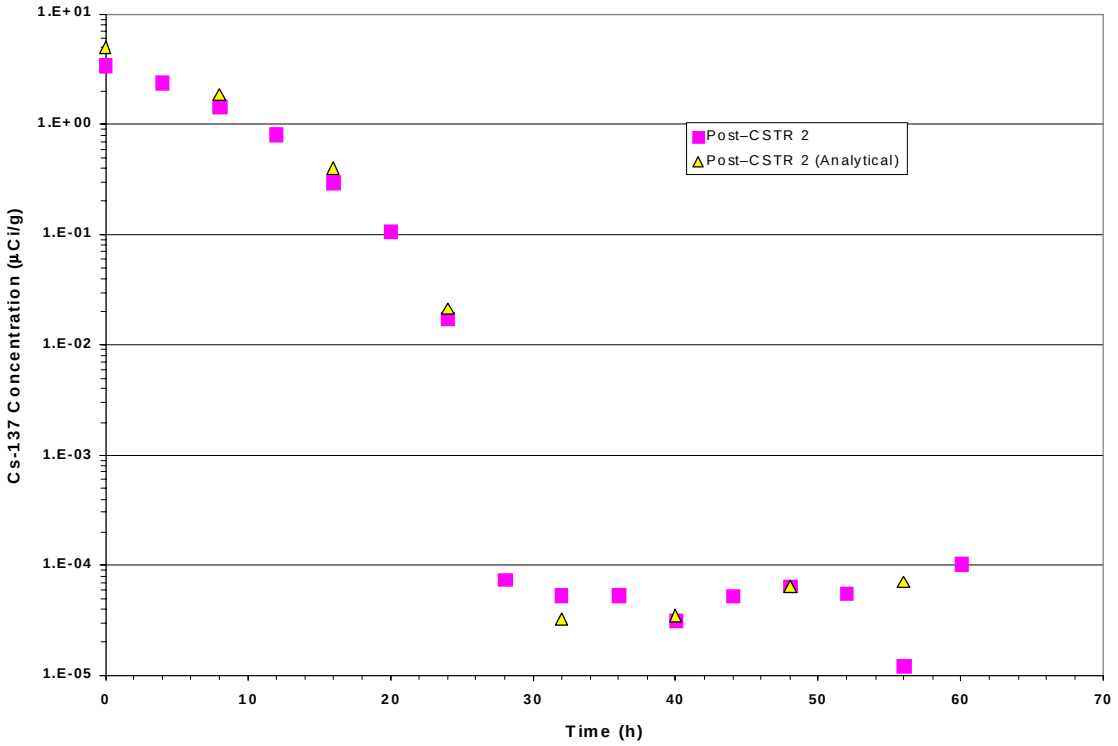


Fig. 22. Cesium concentration vs time for Test 1a for post–CSTR 2.

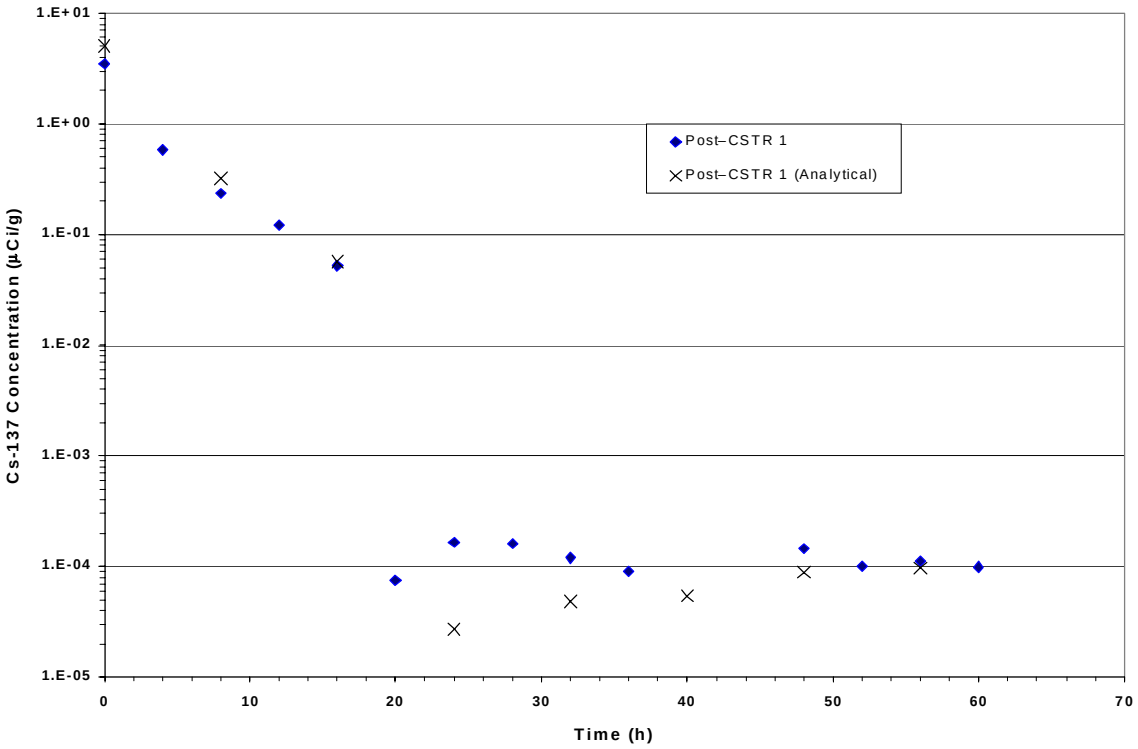


Fig. 23. Cesium concentration vs time for Test 1a for post–CSTR 1.

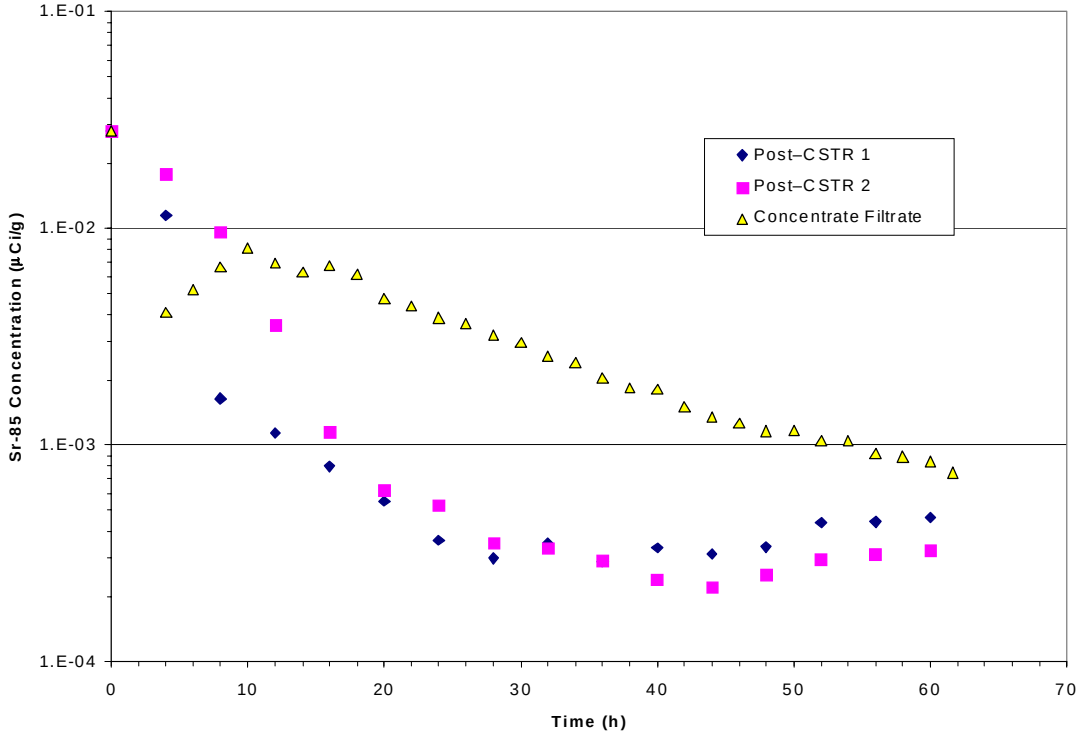


Fig. 24. Strontium concentration vs time.

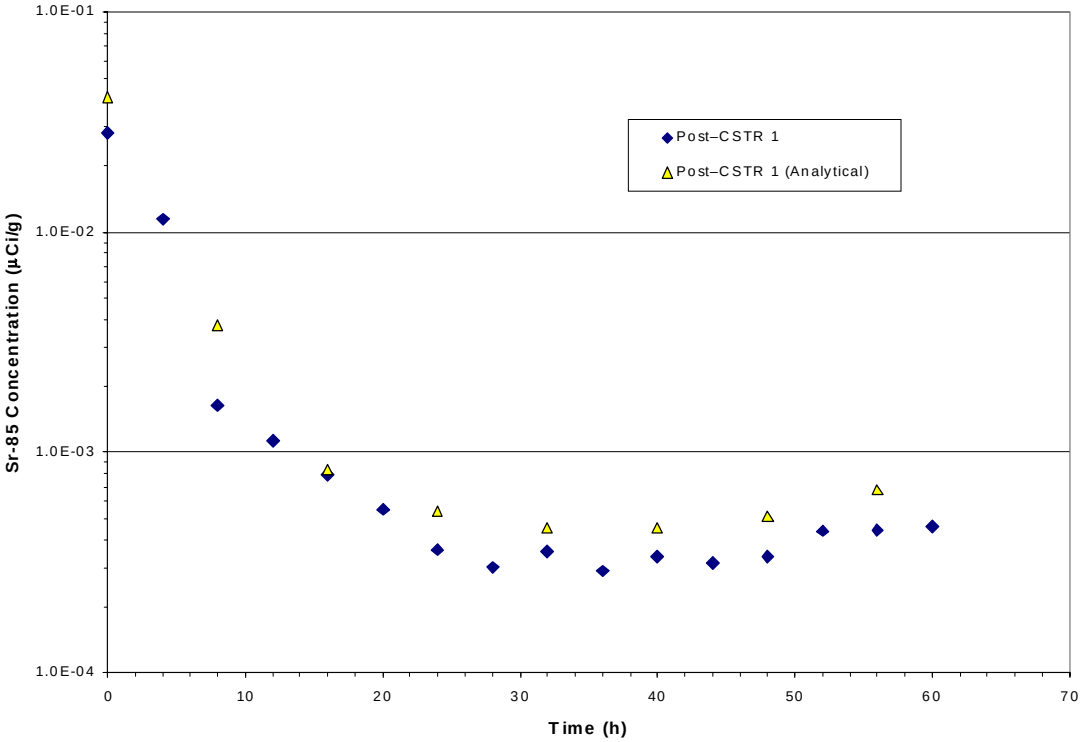


Fig. 25. Strontium concentration vs time for post–CSTR 1.

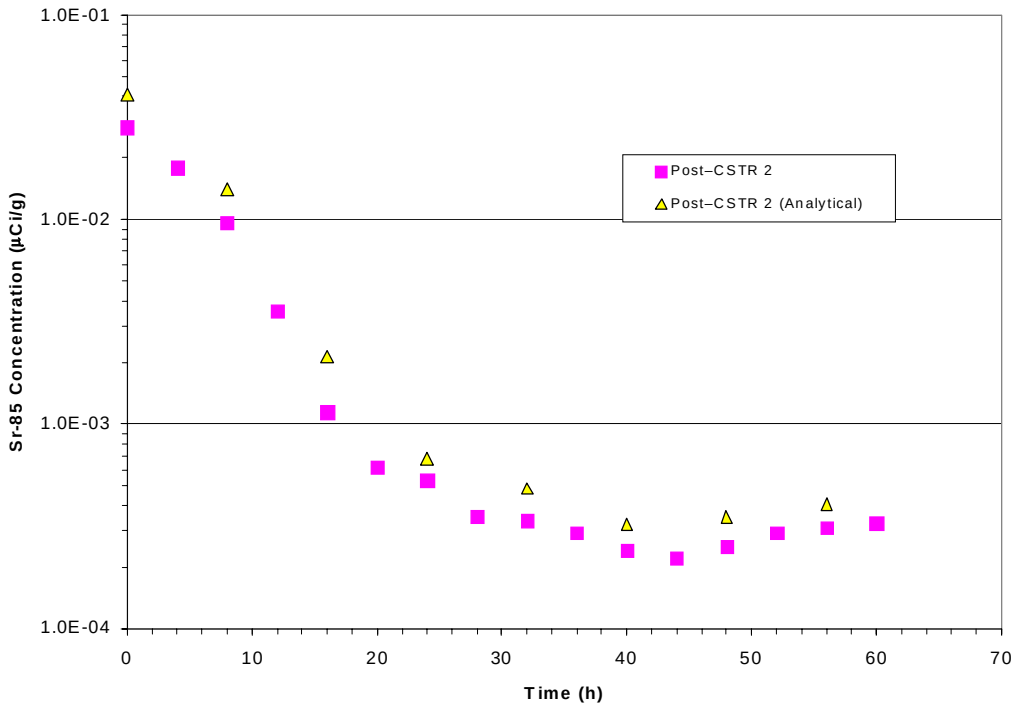


Fig. 26. Strontium concentration vs time for post–CSTR 2.

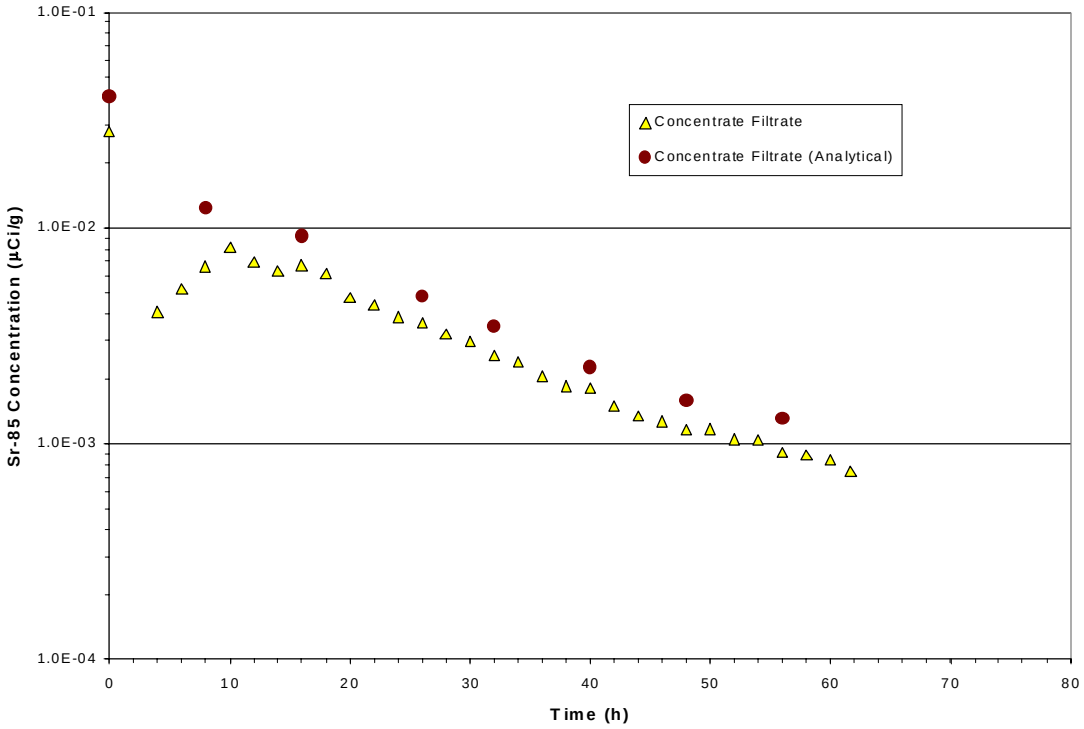


Fig. 27. Strontium concentration vs time for the concentrate filtrate.

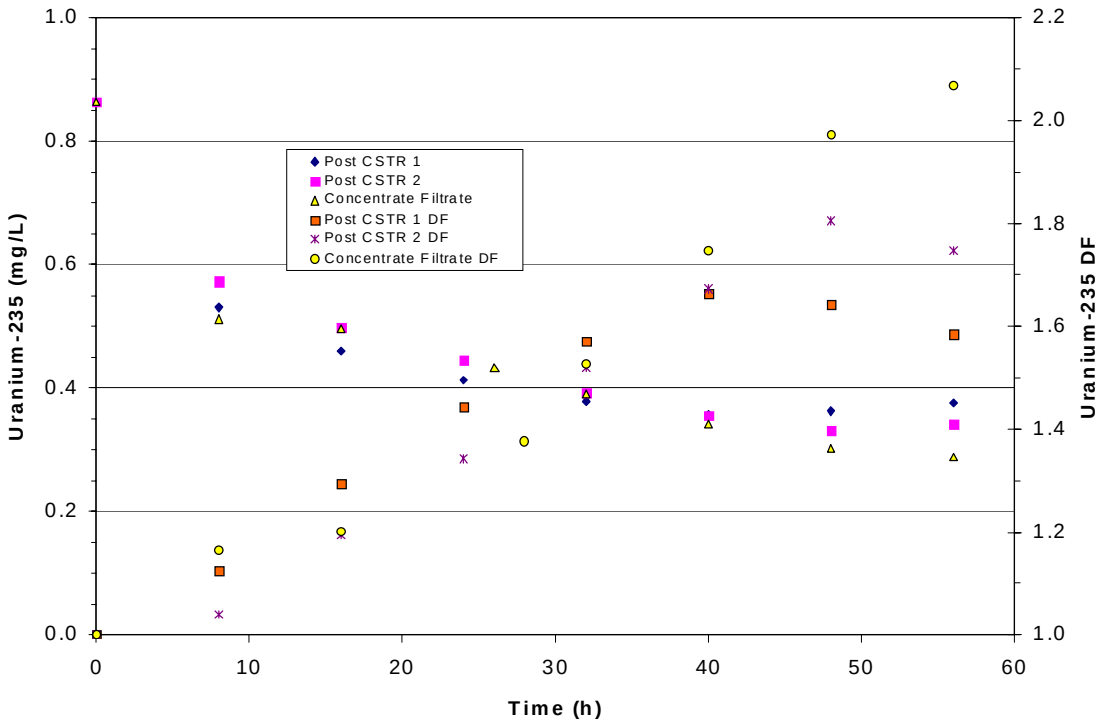


Fig. 28. Uranium-235 concentration vs time for Test 1a.

7.6 MATERIAL BALANCES

The startup sequence resulted in a lower initial concentration of salt in the CSTRs. The sodium and density profiles are shown in Fig. 29. Startup used the supernatant feed and water to attain the desired flow rate into the system to fill it before TPB feed was begun. Recycle wash water could not be used because it contained TPB.

Figures 30 and 31 illustrate the material balances for feed streams into CSTR 1 as measured during the run. An instantaneous flow balance showing the variation in flows at each sample- or data-taking interval is provided in Fig. 30. Figure 31 shows an integrated plot in which the average rates are determined for the feed material from the start of the run until the sample is taken. This plot shows a very smooth flow balance that lines up almost exactly with the desired flow rates. Some of the instantaneous variation can be attributed to the fact that sample time was recorded as occurring on the hour but actually varied somewhat.

7.7 LESSONS LEARNED FOR TEST 2

The most significant issue resulting from the Test 1a operation was the realization that the amount of work required for the completion of all of the tasks during operation required more than two people per shift. Two people were actually needed just to adequately manage the various sampling tasks required during a shift. At the same time, one of the two also had the responsibility of operating the CSTR system. One person from Savannah River was added to each shift crew that worked 12-h shifts for 3 days. The total amount of work to operate the system and sample the process resulted in excessive work hours and necessitated the change to three shifts and three persons per shift to handle system operations and sampling during Test 2 operations. In addition, it was planned that two people from Savannah River would also work on each shift during Test 2 to help take the data, enter it into the data bases, and keep watch on system operations.

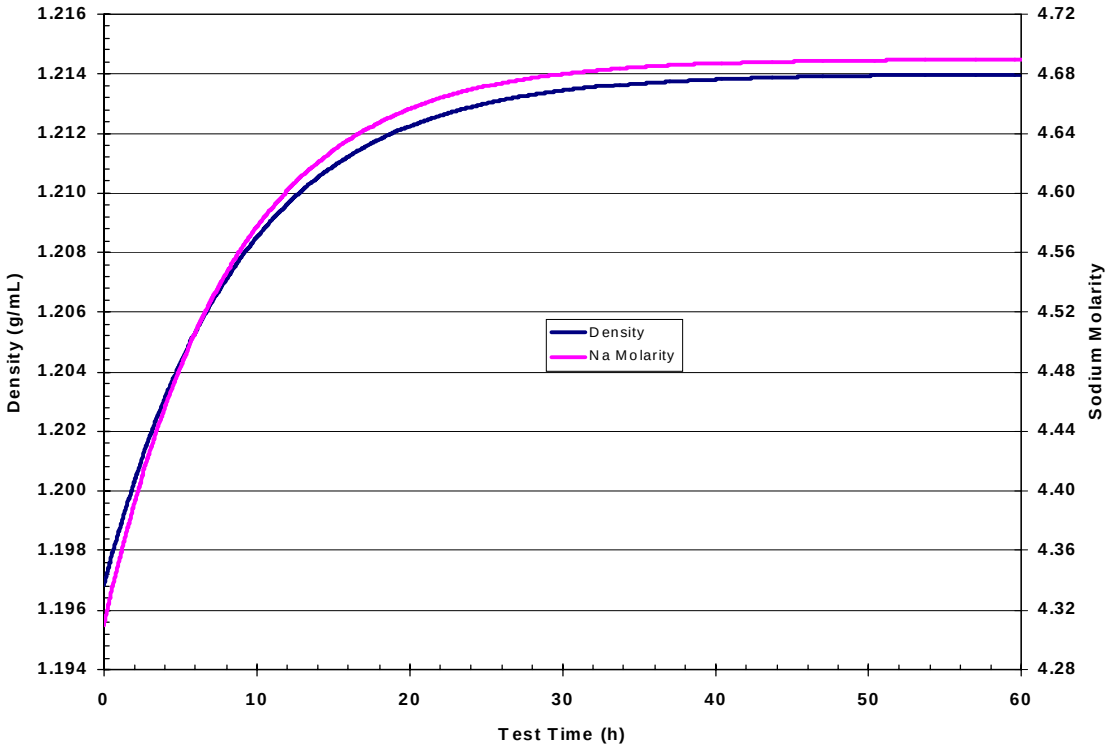


Fig. 29. Density of slurry and sodium molarity in CSTR 1 for Test 1a.

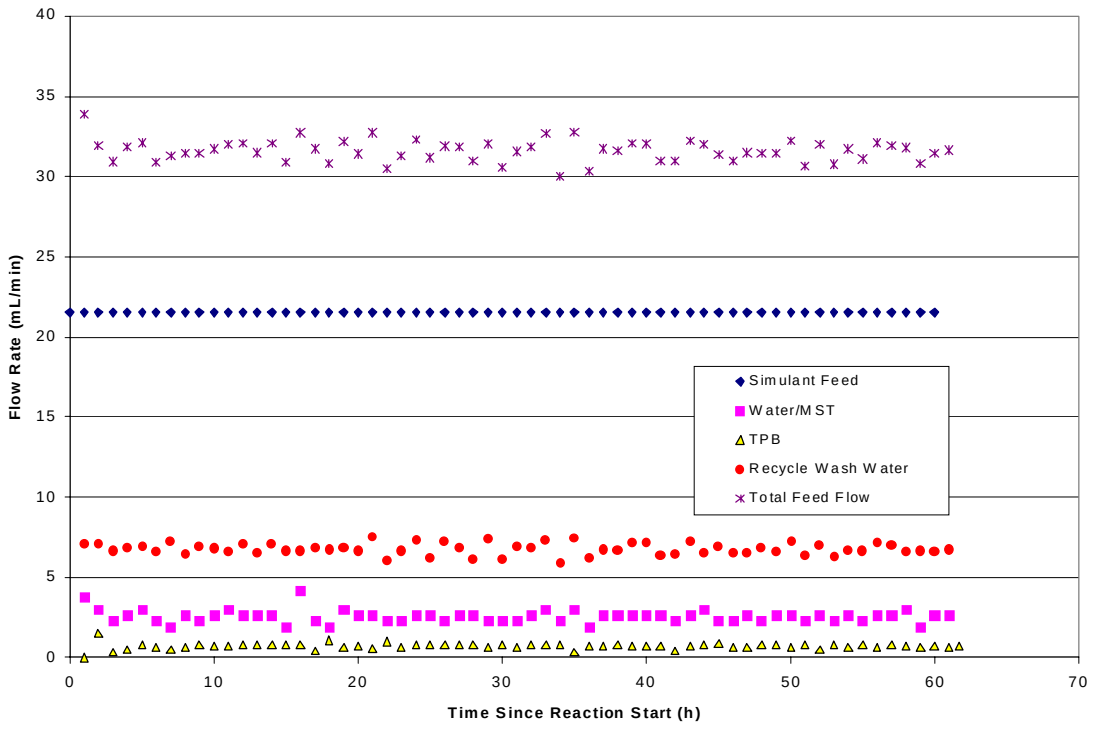


Fig. 30. Instantaneous flow balance for Test 1a.

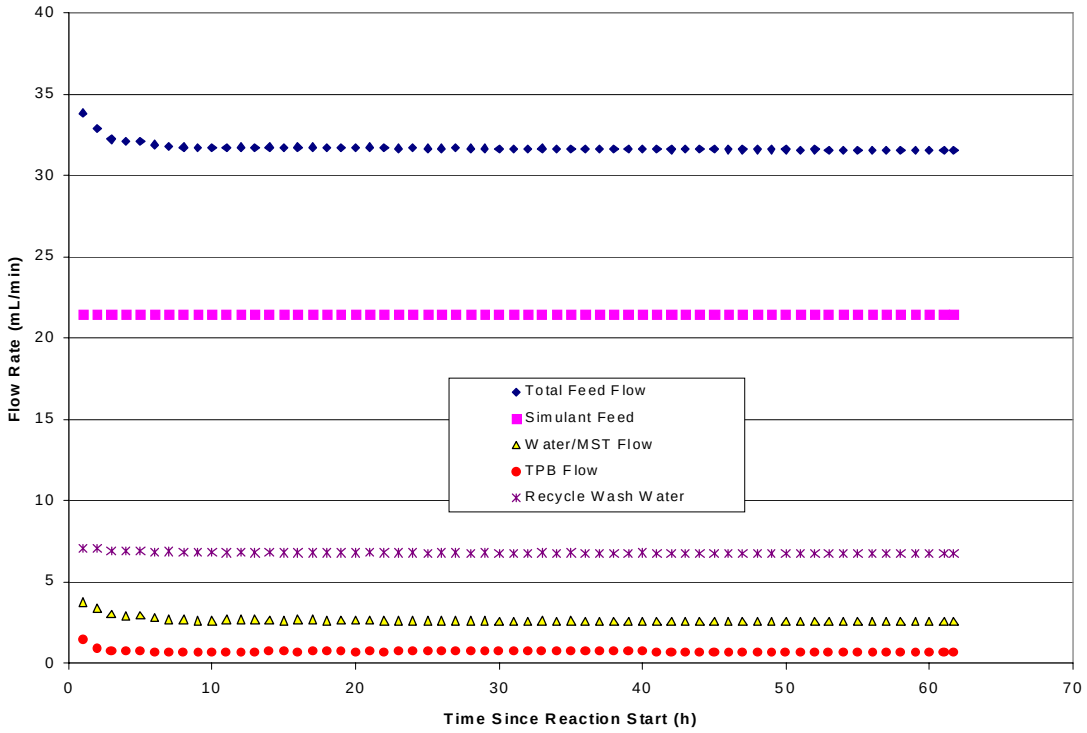


Fig. 31. Overall flow balance for Test 1a.

8. RESULTS OF TEST 2 OPERATION

8.1 TEST 2 STARTUP

The CSTR system for Test 2 again consisted of two CSTRs in series. The CSTRs were followed by the slurry concentration filter loop, which included the concentration vessel, a CFF, and the slurry and filtrate handling systems. The slurry washing system was added, which included the slurry wash tank, a CFF, a wash water filtrate handling system, and the wash water recycle holding tank. As in Test 1a, this test was designed to operate with an 8-h CSTR residence time (8 h in each CSTR for a total of 16 h of reaction time) with a simulated waste feed of 6.4 *M* sodium salt solution diluted to a final sodium concentration of 4.7 *M*.

Test 2 was designed for the salt feed to CSTR 1 to be diluted with 0.55 *M* NaTPB solution, MST/dilution water, and slurry wash water recycle solution contained about 1.2 *M* sodium as it entered the CSTR. The NaTPB was fed at about 172–184% of the stoichiometric amount required to precipitate all of the potassium and cesium present in the feed. The NaTPB was fed in three parts consisting of 100% of the stoichiometric amount from the 0.55 *M* NaTPB feed, 20–30% excess from the recycle wash water stream, and 40% excess from an added makeup 0.55 *M* NaTPB stream which was pumped into a connection and then added to the MST/dilution water feed stream. The recycle wash water stream was supposed to contain the needed recovered excess NaTPB from washing the precipitated slurry (enough to provide a 60% excess NaTPB) but did not. The slurry produced during Test 1a was washed before Test 2 was started, and the wash water was transferred to the recycle wash water holding tank and sampled. The analytical laboratory determined the soluble TPB concentration in the wash water just prior to the start of Test 2. The TPB dissolved from the Test 1a slurry during the washing was only about 20–30% of the excess fed during Test 1a. Therefore, an extra 40% makeup TPB had to be added to compensate for the TPB not washed from the slurry. The extra water needed to represent this extra NaTPB was not added during the initial CSTR filling because the TPB analysis information was not available until actual startup time.

The volume of recycle wash water produced by Test 1a slurry washing was not sufficient to provide wash water until the next batch of slurry concentrate was washed. Therefore, a simulated wash water solution (with the components that were in the same solution for Test 1a) was prepared and added to the recycle wash water tank in the hot cell. The concentration of TPB

in the simulant was made equal to that in the original solution produced by washing the Test 1a slurry. The recycle wash water was pumped to the holding tank through the slurry washing loop. Once the test was started, two 10-L batches of the simulated wash water were added during the first 4 days of operation. Afterwards, enough wash water was available from washing the Test 2 slurry concentrate batches and no additional simulated wash water was used.

The startup proceeded in the same way as for Test 1a. Salt feed was pumped to CSTR 1 at a feed rate of 21.5 mL/min, and deionized water was pumped to CSTR1 at 9.68 mL/min to fill the two CSTRs and to provide about 3 L of diluted salt feed to the concentration tank. After pumping several liters of feed to CSTR 1, it was discovered that the water line had detached where the line connected to one of the transfer valves inside the hot cell. The water had not been pumped into CSTR 1, but instead onto the spill catch pan holding the apparatus. After this discovery, the salt feed flow was stopped and the missing water was pumped to CSTR 1. Subsequently, both flows were restarted and the startup operation was continued.

8.2 HYDRAULICS

The test duration was 230 h, which provided for about 28 CSTR-volume turnovers. As in Test 1a, the hydraulic behavior was again excellent in Test 2. Sound overall system operability was accomplished, with the exception of the level sensor problems in the concentration and wash vessels. Although foam was visually observed in the concentrate tank, no operational issues were noted in CSTR 1 or CSTR 2, in the slurry concentration loop, or in the washing loop for slurry concentrate. The overall process control behavior was excellent. There were no problems associated with the pressure and flow control valves or pressure transducers in the concentration loop or slurry wash loop, but the flow rate measuring devices did not always have the correct calibration. The Coriolis and Mag flowmeters both gave excellent service, and the Coriolis meter again gave helpful indications when parts of the system were not in mass balance.

The figures in Sects. 8.4 and 8.5 and in Appendix E show a series of graphs covering the operating periods for both filtration loops. The variables shown include the CFF shell- and tube-side pressures, the loop slurry temperature, the slurry and filtrate flow rates, and the slurry density. One of the most obvious points in the graphs of the slurry concentration loop was the TMP (the difference between PIC-1and PI-2) increasing as the run progressed, which indicated that the filter was becoming plugged. To keep filtrate flow at the desired rates for the last part of

the test, the modified backpulse technique, which was developed during the test, was frequently used (about once an hour). No chemical cleaning was required during the run. There were no problems with the slurry wash filter, and no backpulsing or chemical cleaning was necessary.

8.3 SALT FEED DECONTAMINATION

The necessary cesium decontamination (DF > 10,000) was again obtained in all vessels, and DFs greater than 30,000 were obtained in all three vessels. A DF of 10,000 was obtained in the concentrate filtrate after 32 h. Decontamination was also maintained during and after short-term reduced supplemental NaTPB flow incidents. Decontamination returned quickly (within 8 h) after the concentration vessel contamination incident described in the following paragraph. Most of the strontium decontamination occurred in the feed tanks when the ECC catalyst and sludge were added. The hydrous metal oxides in these components are sorbents for strontium and apparently sorbed the strontium. This was apparent when the strontium contents in the filtered and unfiltered feed samples were compared. In addition, uranium decontamination was obtained at a similar level to that in Test 1a. An average DF of 1.76 over the course of the 230 h was obtained, which was based on the ^{235}U analysis by DNC.

The concentrate tank contamination incident occurred when slurry that had been spilled out of the concentration tank during an attempted pressure backpulse with nitrogen and filtrate was returned to the concentrate tank. During an attempt to backpulse the concentration tank filter with nitrogen, the nitrogen valve was left open too long and gas broke through the filter, causing some of the slurry in the concentration tank to overflow into the catch sump. That slurry was pumped back into the concentration tank with a peristaltic pump. Although the sump was dry, it had previously contained some salt feed from a small CSTR overflow. The residue left in the sump contained enough cesium to briefly raise the cesium concentration in the slurry tank filtrate by an order of magnitude.

Figures 32–35 show ^{137}Cs decontamination from the salt feed. The cesium removal during Test 1a is also shown in Figs. 32–34 for comparison. In the samples from CSTR 1, the desired DF was obtained slightly faster in Test 2 than in Test 1a. The same held true for CSTR 2 samples and the concentrate filtrate. Note that in Test 2, a higher concentration of NaTPB was used (72 to 84% excess in Test 2 compared with 60% excess in Test 1a). Another difference was the Na^+ molarity, which was 4.47 in Test 2 and 4.7 in Test 1a. The cesium concentrations in both

CSTRs also showed a cyclic response of about 30 h. Both the internal and the RMAL analyses indicated this response, and it continued for about five cycles in both CSTR 1 and CSTR 2. The response in the concentrate filtrate samples was hardly visible, and the cesium concentration gradually decreased over the course of the last 120 h of operation. The CSTR 1 and CSTR 2 responses are also slightly offset from each other. As a comparison, Fig. 34b shows the filtrate samples for the calculated concentration of ^{137}Cs that would be present if the total cesium used in Test 2 was of the composition of actual average Savannah River salt waste. Assuming the same salt feed decontamination results that were obtained in Test 2 could be obtained with actual SRS average salt feed, this plot clearly shows that the needed ^{137}Cs decontamination to provide a WAC value of ≤45 nCi/g would have been rapidly reached and greatly exceeded. For the last 60 h of the test, the ^{137}Cs concentration in the filtrate would have been less that 10 nCi/g providing DF values ≥40,000.

The strontium decontamination of the simulant salt feed is shown in Figs. 36–39. The initial amount of strontium added in Test 2 was almost the same as in Test 1a. The difference between Test 1a and Test 2 was the addition of the Savannah River sludge and the ECC catalyst ingredients to the salt feed for Test 2. These ECC ingredients included several hydrous metal oxides that are known to sorb strontium and removed the strontium from solution. This was confirmed by analyzing feed samples both before and after filtering. The amount of strontium added to the salt feed was found when analyzing the unfiltered samples, but less than one-tenth was found in the filtered samples. The concentration of strontium remained almost constant during the whole test at all three sample ports at a DF of between 30 and 40.

The performance of the MST in removing uranium was measured both by ICP-MS and by DNC on the concentrate filtrate samples. Both methods gave DF results between 1.6 and 2.4 for all filtrate samples, but the ICP-MS values were consistently about 15–20% higher than the DNC values. Based upon the known amount of ^{235}U added to the salt feed at the start of the test, DNC proved to be the more accurate of the two analytical methods for uranium. The DF gradually decreased during the course of the test and ended at about 1.5 for the three samples ports. The loss of MST/dilution water flow for 2 to 3 h twice late in the test due to solids buildup in the pump suction line did not appear to affect the uranium DF at any of the sample ports. This DF is comparable to the Test 1a uranium DF. The samples of the feed taken before introduction

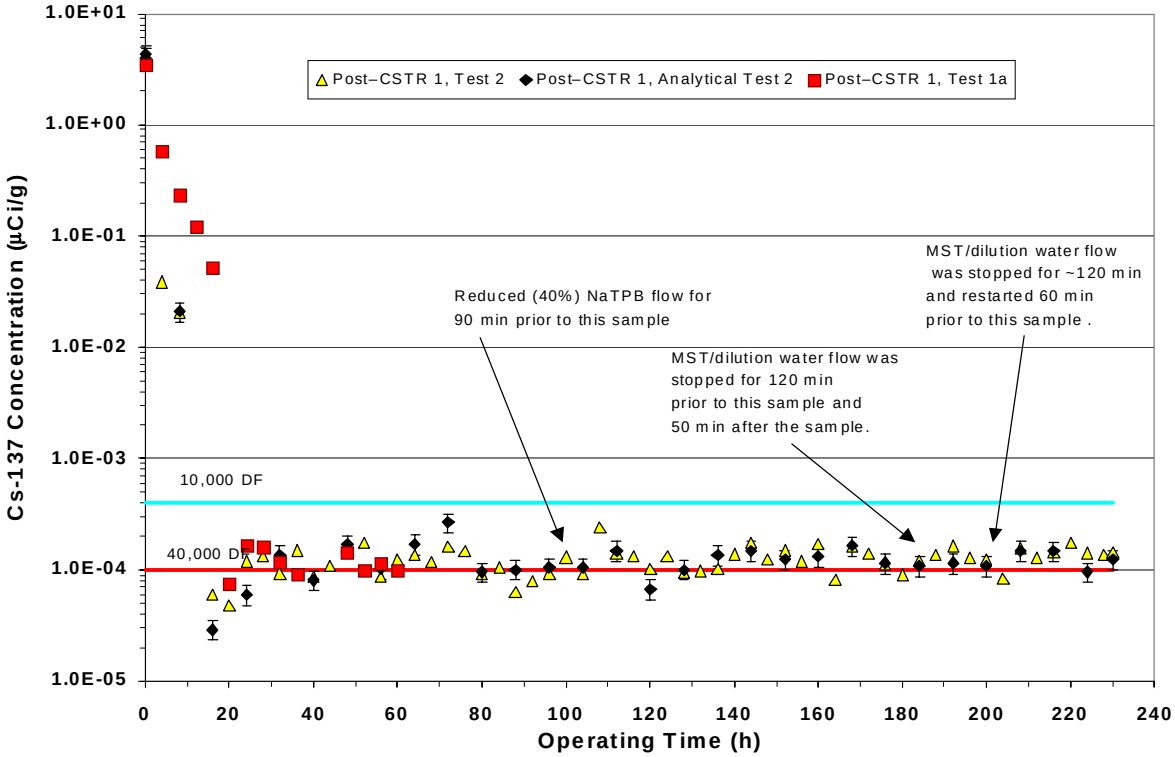


Fig. 32. SP1 cesium concentration vs time for Test 2.

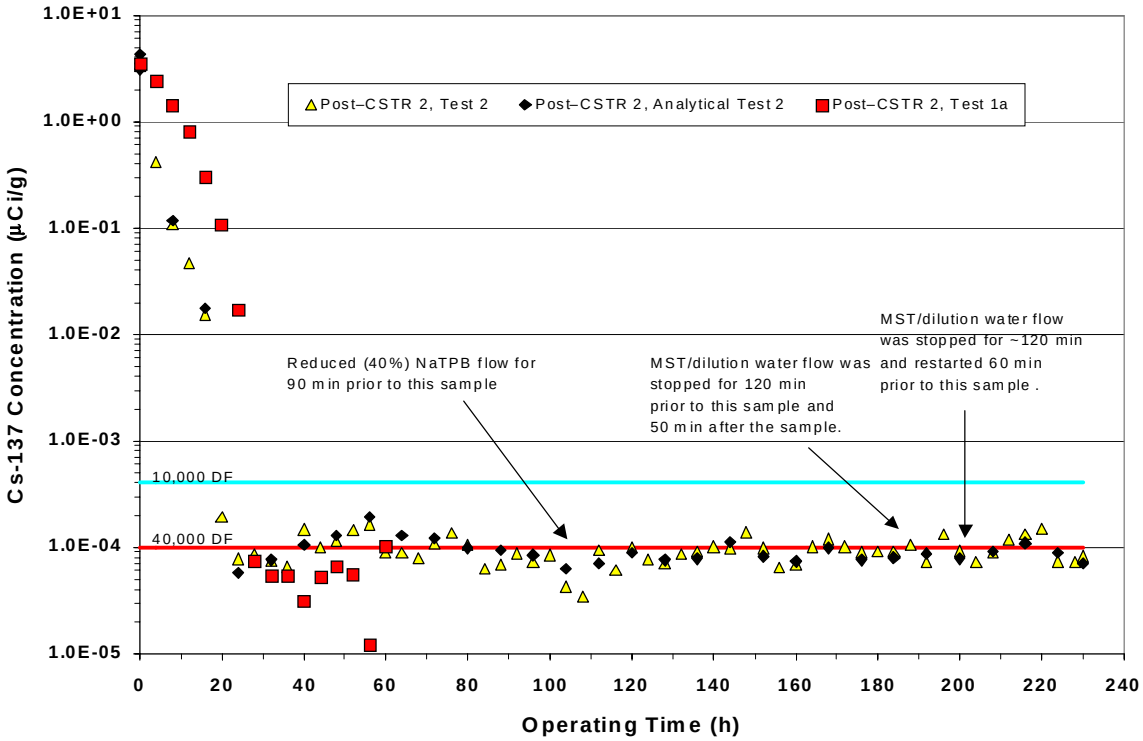


Fig. 33. SP2 cesium concentration vs time.

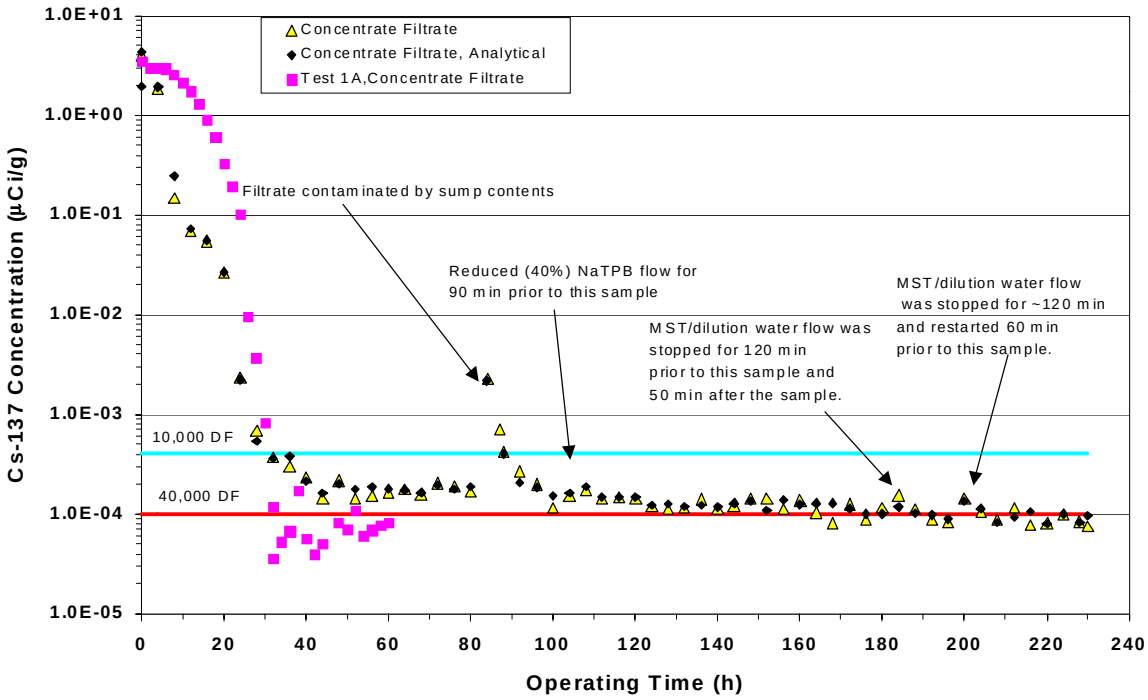


Fig. 34a. SP4 cesium concentration vs time.

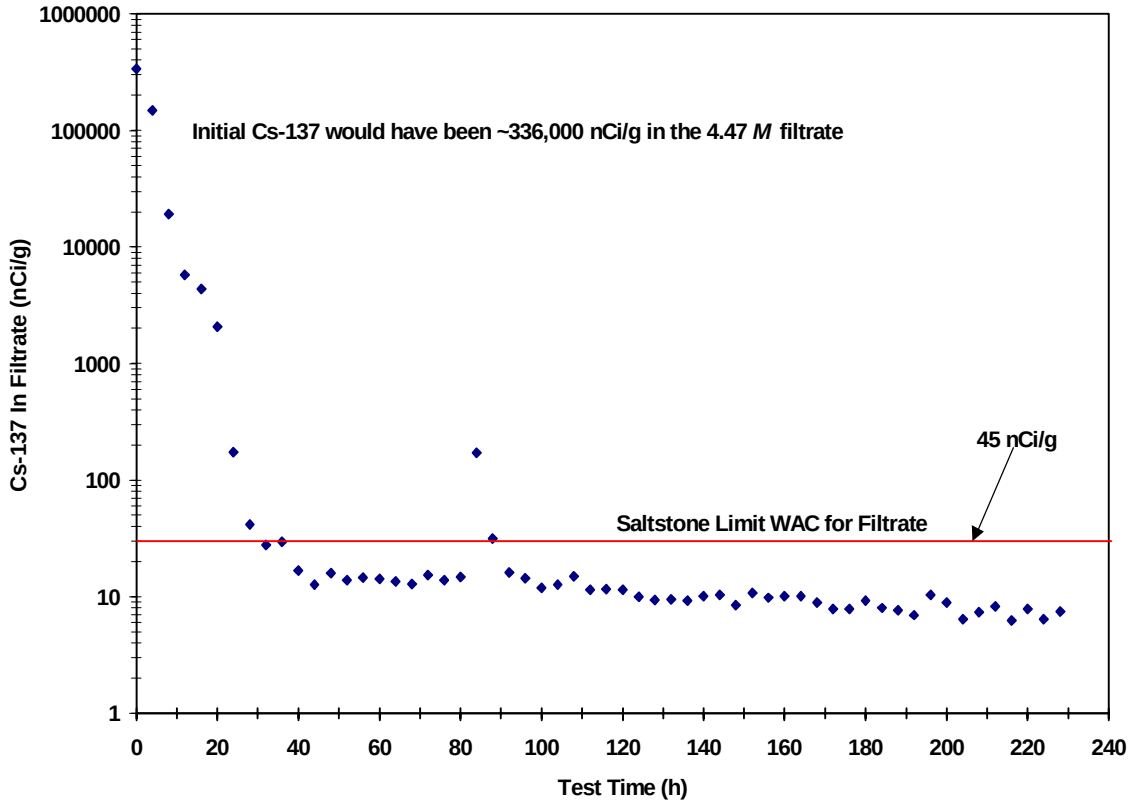


Fig. 34b. SP4 ^{137}Cs filtrate equivalent if Test 2 had been conducted with real SRS average salt feed.

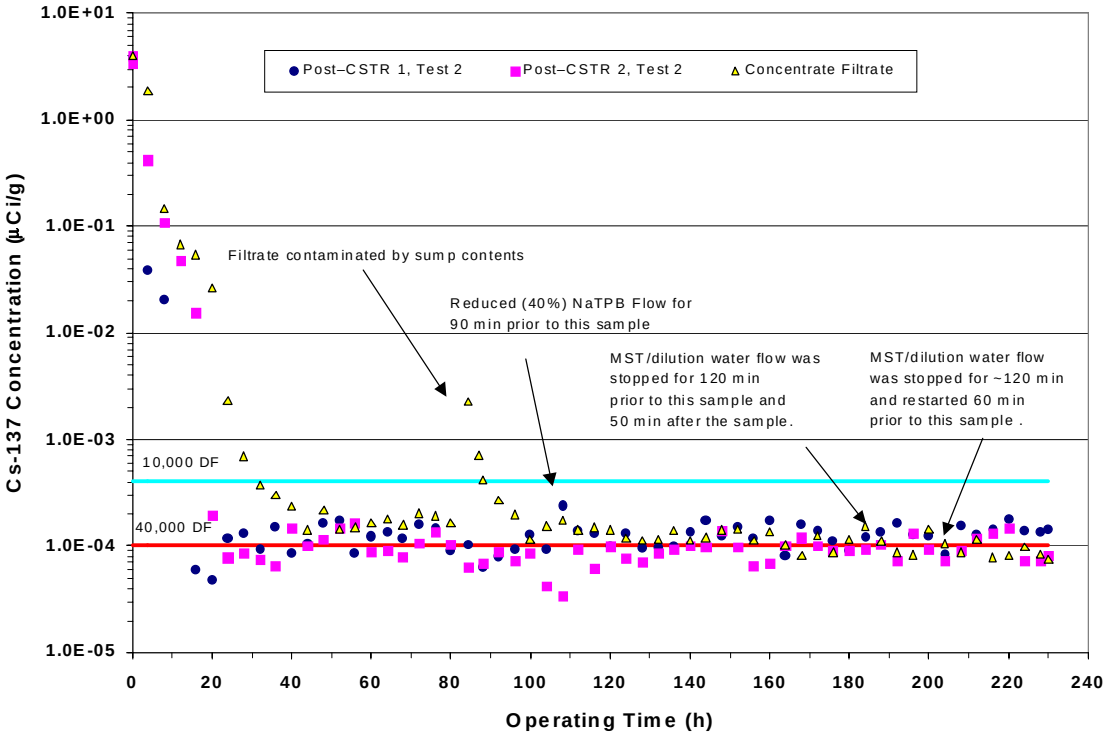


Fig. 35. Cesium concentration vs time, SP1, SP2, and SP4.

of MST indicated that the ECC catalyst system also was active in removing some of the uranium, although not nearly to the extent that strontium was removed.

The analytical data for this experiment is also shown in Tables 25–28. Shown in the tables are the internal counting data for Cs and Sr and the analytical results for the Cs, Sr, and U; ICP-MS results for B, Cu, and Pd; and the HPLC results for the TPB and its decomposition products.

8.4 SLURRY CONCENTRATION AND FILTRATION SYSTEM

The slurry concentration loop was operated throughout the 230 h of Test 2. Five batches of slurry were concentrated. Four batches of concentrate were transferred to the slurry wash tank for washing after being concentrated. The last batch of slurry concentrate produced was not washed during the test but was washed later. The operation of the slurry wash system can be seen in Figs. 41–44. The figures show the Intellution® history data for the filtration loop. Figures 41 and 42 each show about one-half of the test, with the CFF tube (inside) and shell

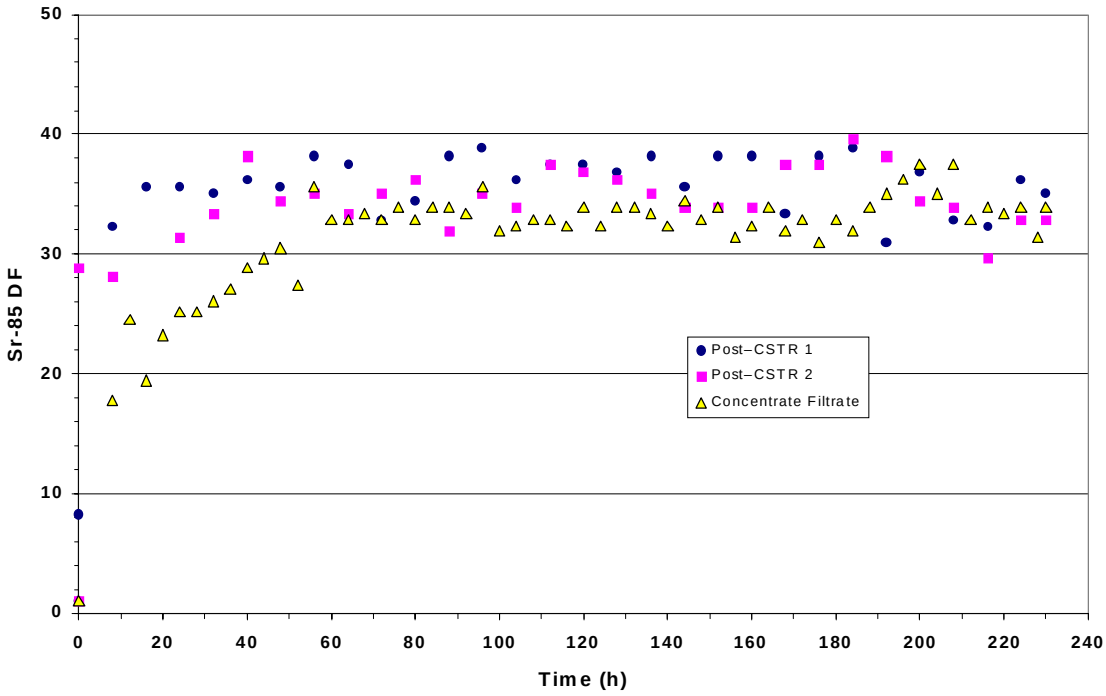


Fig. 36. Strontium DF vs time.

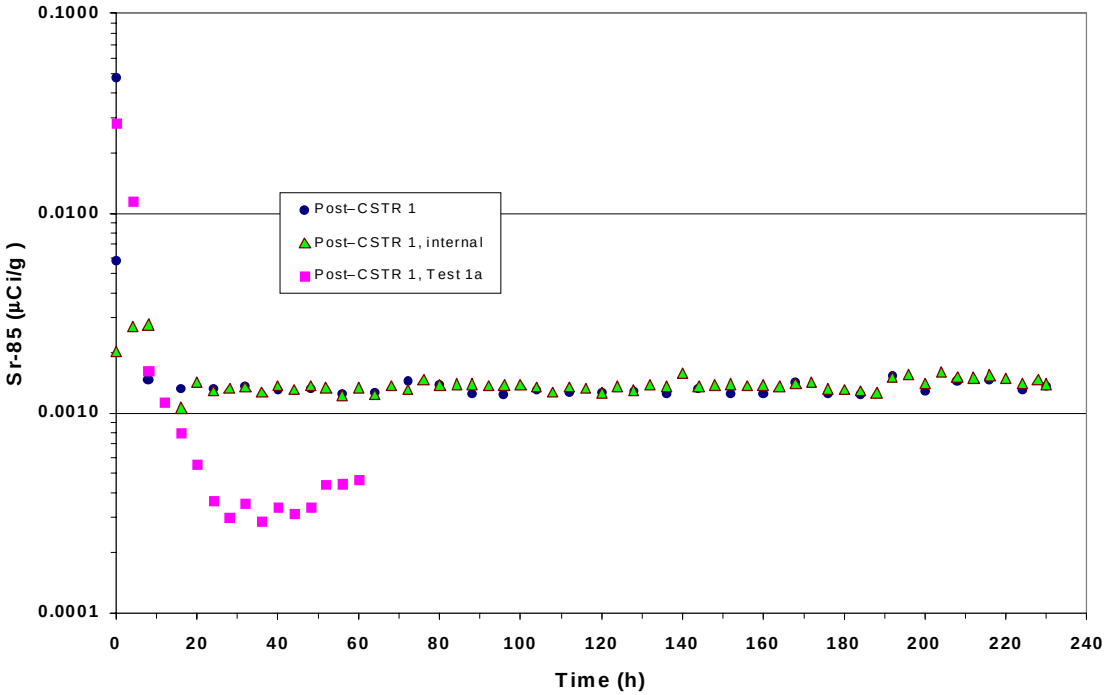


Fig. 37. Strontium concentration after CSTR 1 vs time.

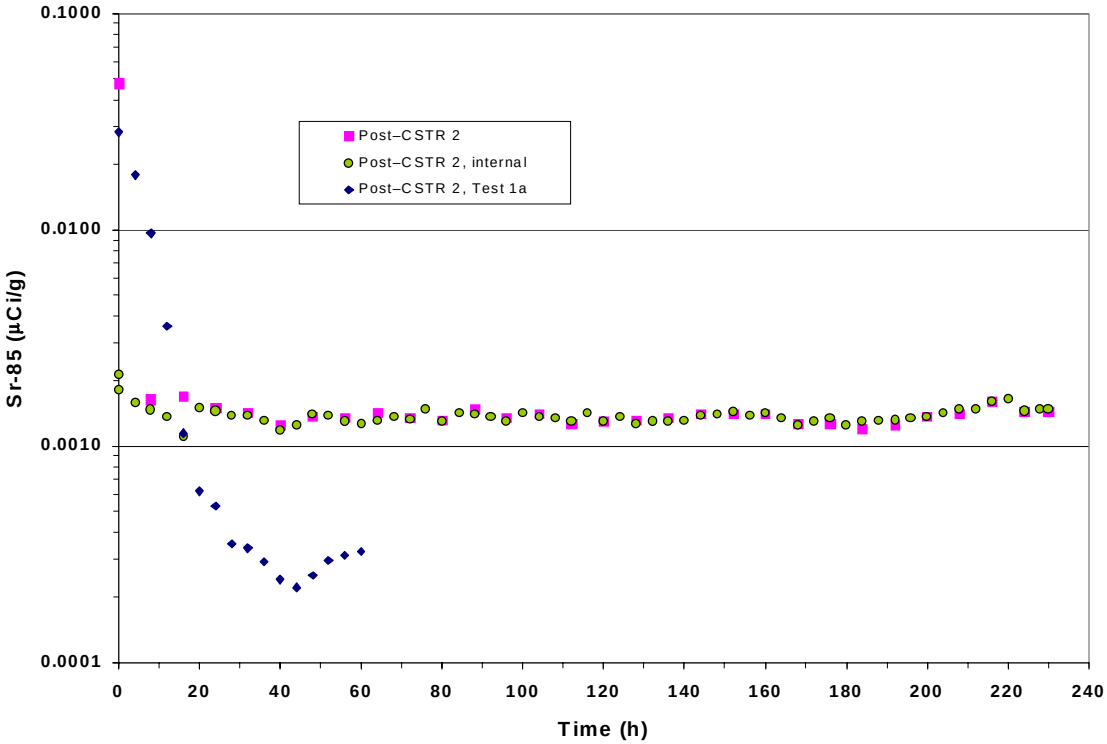


Fig. 38. Strontium concentration after CSTR 2 vs time.

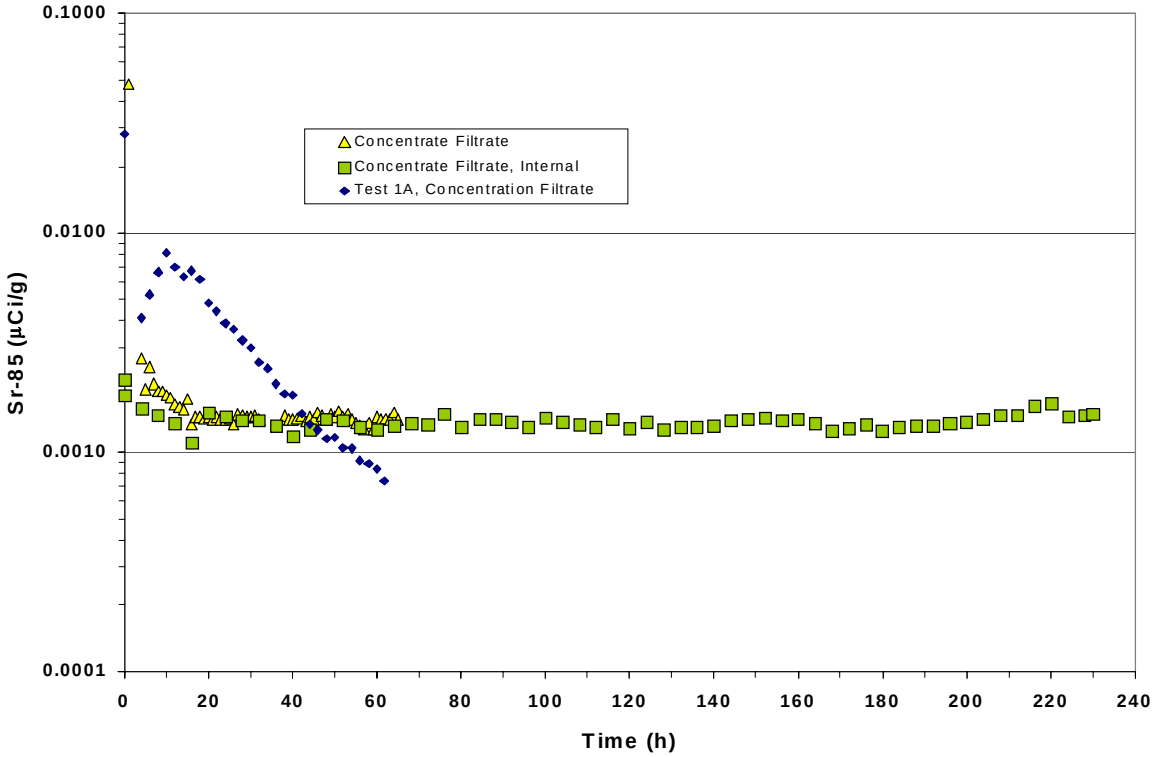


Fig. 39a. Strontium concentration in slurry concentration filtrate vs time.

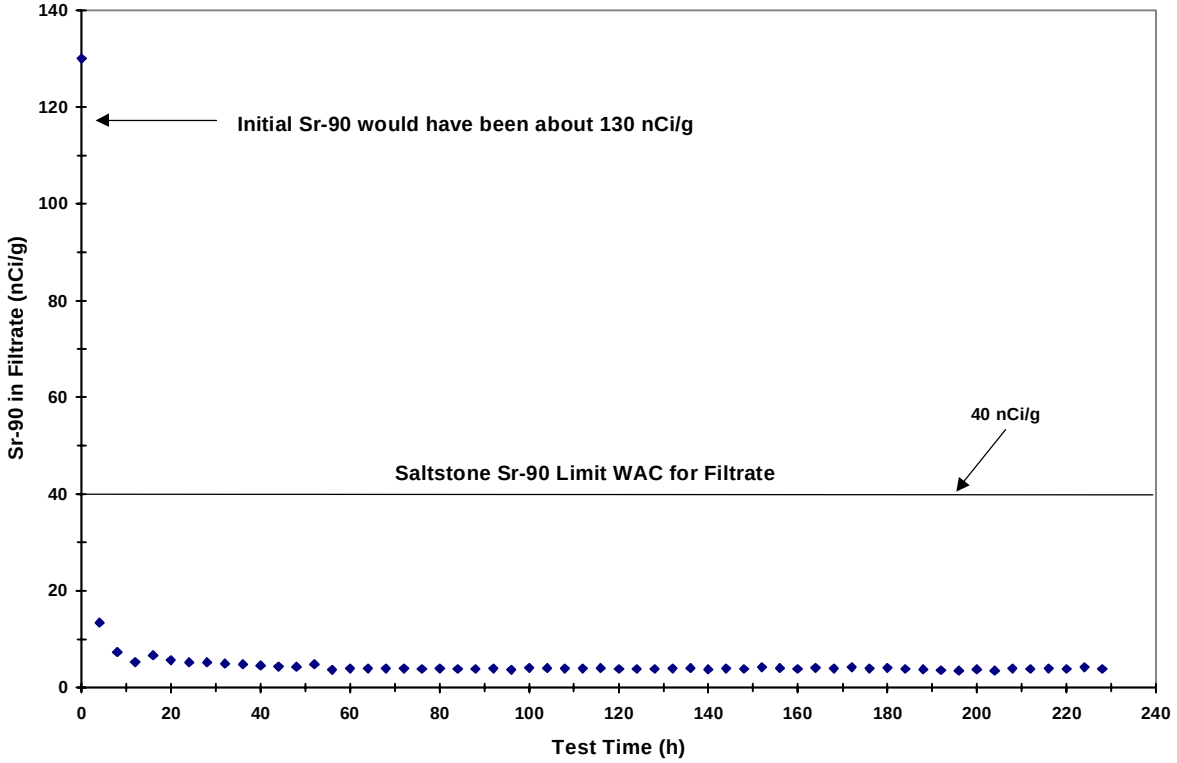


Fig. 39b. Strontium-90 filtrate equivalent if Test 2 had been conducted with real SRS average salt feed.

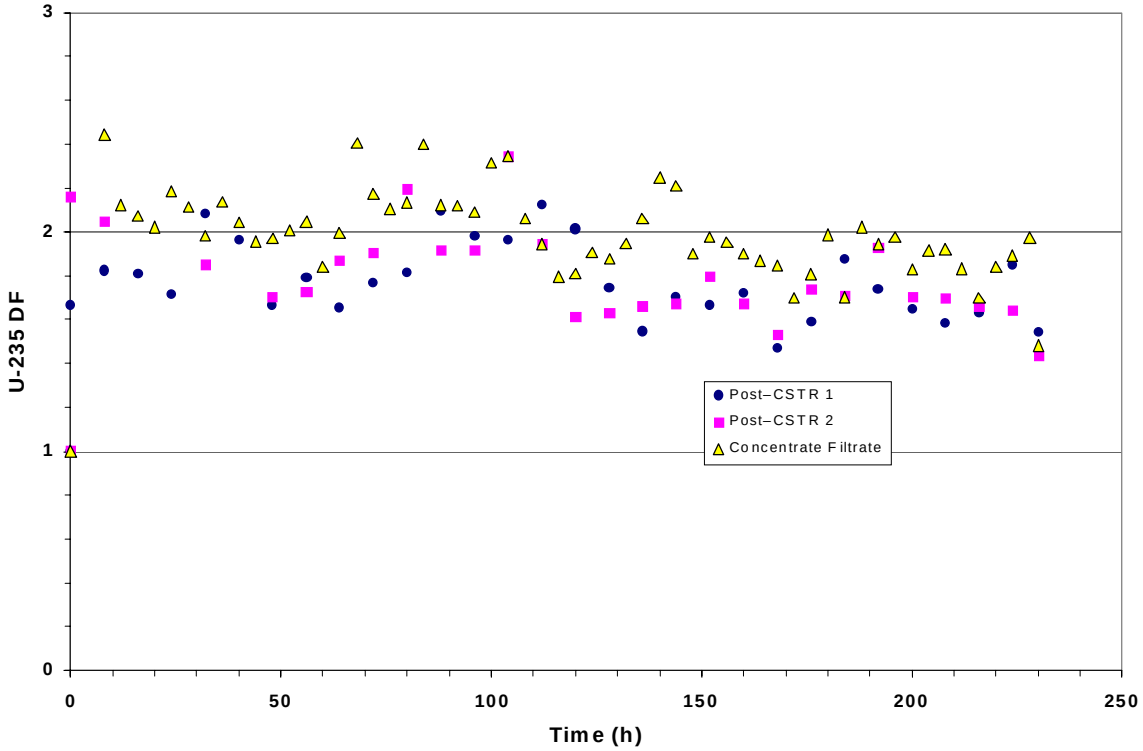


Fig. 40. Uranium-235 DF vs time.

Table 25. Uranium-235 analysis by delayed neutron counting and comparison with that by CP-MS

Sample	^{235}U (ng)	Sample[a] mass (g)	^{235}U by DNC (ng/g)	Error[b] (ng/g)	Filtrate densities[c] (g/mL)	Corrected[d] ^{235}U by ICP-MS (ng/mL)	DF[e]	^{235}U by ICP-MS[f] (ng/mL)	Deviation[g] (%)
T2SP4-02F	59.0	0.2341	252.0	1.9	1.2090	304.7	2.1	366.9	20
T2SP4-04F	66.4	0.2325	285.5	2.1	1.2090	345.2	1.9	431.8	25
T2SP4-06F	70.9	0.2293	309.1	2.2	1.2090	373.7	1.7	410.3	10
T2SP4-08F	71.4	0.2341	304.9	2.1	1.2090	368.6	1.7	452.2	23
T2SP4-10F	69.6	0.2374	293.2	2.1	1.2090	354.5	1.8	438.7	24
T2SP4-12	72.0	0.2456	293.1	2.0	1.2090	354.3	1.8	454.7	28
T2SP4-14	63.4	0.2315	273.9	2.0	1.2090	331.1	1.9	438.1	32
T2SP4-16	67.9	0.2342	289.8	2.1	1.2090	350.3	1.8	449.1	28
T2SP4-18	68.7	0.2320	296.0	2.1	1.2090	357.9	1.8	412.3	15
T2SP4-20	67.8	0.2318	292.4	2.1	1.2080	353.2	1.8	419.7	19
T2SP4-22	70.2	0.2423	289.7	2.0	1.2080	349.9	1.8	422.0	21
T2SP4-24	47.0	0.1489	315.9	2.7	1.2076	381.5	1.7	429.0	12
T2SP4-26	73.0	0.2391	305.4	2.1	1.2076	368.8	1.7	381.9	4
T2SP4-28	69.7	0.2341	297.9	2.1	1.2076	359.7	1.8	461.2	28
T2SP4-00	32.2	0.2229	144.6	1.5	1.2046	174.2		195.5	12
T2SP4-30	70.5	0.2343	300.8	2.1	1.2046	362.4	1.8	495.0	37
T2SP4-32	69.3	0.2324	298.4	2.1	1.2036	359.1	1.8	476.8	33
T2SP4-34	70.5	0.2353	299.7	2.1	1.2042	360.9	1.8	435.4	21
T2SP4-36	71.5	0.2410	296.9	2.1	1.2042	357.5	1.8	406.4	14
T2SP4-38	76.3	0.2395	318.7	2.2	1.2041	383.8	1.7	453.4	18
T2SP4-40	75.8	0.2413	314.2	2.1	1.2041	378.3	1.7	472.1	25
T2SP4-42	73.5	0.2340	314.3	2.2	1.2046	378.6	1.7	485.5	28
T2SP4-44	71.3	0.2271	314.1	2.2	1.2046	378.4	1.7	496.5	31
T2SP4-46	73.2	0.2351	311.4	2.2	1.2043	375.0	1.7	527.0	41
T2SP4-48	69.9	0.2346	298.1	2.1	1.2055	359.4	1.8	460.7	28
T2SP4-50	77.7	0.2364	328.6	2.2	1.2054	396.1	1.6	489.9	24
T2SP4-52	73.7	0.2324	317.1	2.2	1.2064	382.6	1.7	466.8	22
T2SP4-54	77.2	0.2324	332.1	2.2	1.2110	402.2	1.6	527.7	31
T2SP4-56	76.2	0.2309	330.0	2.2	1.2110	399.6	1.6	474.1	19
T2SP4-FINAL	75.6	0.2307	327.8	2.2	1.2110	396.9	1.6	604.9	52

[a] Aliquots (200 µL) were dried under a heat lamp on Eichrom TRU-Spec resin. Aliquots were weighed. Pipette calibration suggests that aliquot size is 0.193 mL with 0.5% precision. Mass-per-milliliter numbers are calculated using 0.193-mL pipetting volume.

[b] Uncertainty calculated from counting statistics at the 2-sigma level of confidence.

[c] Taken from the measured densities of filtrate during Test 2.

[d] Original ICP-MS numbers corrected based on the sample mass and the filtrate densities.

[e] Calculated based on the amount of ^{235}U added to the feed and then diluted so that the diluted feed = 642 ng ^{235}U/mL.

[f] Calculated based on the sampling procedure described in note *a*.

[g] Percent difference between the original ICP-MS numbers and the corrected ones.

Table 26. T2SP1 normalized data for CSTR 1 samples

Reaction time (h)	CSTR 1 sample ID	Cs-137 (µCi/g)	Sr-85 (µCi/g)	K (mg/L)	Cu (mg/L)	Pd (mg/L)	B (mg/L)	U-235 (mg/L)	NaTPB (mg/L)	3PB (mg/L)	2PB (mg/L)	1PB (mg/L)	Phenol (mg/L)
0	T2SP1-Feed	4.30	4.76E-02	2137.45	4.69	13.86	22.3	0.90		56.2	57.6	88.9	119.3
0	T2SP1-00	4.01	5.79E-03	640.00	2.40	<1.85	15.9	0.54	<16.3	<15.7	60.8	0.0	113.0
8	T2SP1-01	2.09E-02	1.47E-03	187.00	2.25	<1.28	14.8	0.49					
16	T2SP1-02	2.90E-05	1.34E-03	40.30	2.15	<1.28	19.5	0.50	109.0	30.4	69.4	145.0	
24	T2SP1-03	6.01E-05	1.34E-03	21.50	2.12	<1.28	20.0	0.52					
32	T2SP1-04	1.36E-04	1.36E-03	30.80	2.11	<1.28	17.3	0.43	38.5	22.8	88.8	97.0	113.0
40	T2SP1-05	8.02E-05	1.31E-03	31.10	2.25	<1.28	20.5	0.46					
48	T2SP1-06	1.69E-04	1.34E-03	21.00	2.25	<1.28	23.0	0.54	123.0	62.7	66.5	112.0	142.0
56	T2SP1-07	1.02E-04	1.25E-03	26.70	2.23	<1.28	20.2	0.50					
64	T2SP1-08	1.72E-04	1.27E-03	20.80	2.37	<1.28	21.6	0.54	72.9	48.1	46.3	86.5	138.0
72	T2SP1-09	2.67E-04	1.45E-03	<17	2.37	<1.28	22.1	0.51					
80	T2SP1-10	9.58E-05	1.38E-03	<1.5	2.16	1.50	18.5	0.49	46.7	32.2	28.7	135.0	140.0
88	T2SP1-11	1.00E-04	1.25E-03	<8.57	2.17	<1.28	22.4	0.43					
96	T2SP1-12	1.05E-04	1.23E-03	<6.67	2.40	<1.28	22.8	0.45	175.0	81.3	36.8	108.0	123.0
104	T2SP1-13	1.05E-04	1.31E-03	<12	2.34	<1.28	18.6	0.46					
112	T2SP1-14	1.49E-04	1.27E-03	<3.06	2.15	<2.42	20.0	0.42	86.9	55.3	33.5	0.0	126.0
120	T2SP1-15	6.68E-05	1.27E-03	<11.7	2.36	<1.28	22.3	0.45					
128	T2SP1-16	1.00E-04	1.29E-03	<12.5	2.49	<1.28	19.6	0.51	63.8	39.3	<16.5	80.8	0.0
136	T2SP1-17	1.36E-04	1.25E-03	<1.5	2.47	2.59	21.4	0.58					
144	T2SP1-18	1.49E-04	1.34E-03	<3.98	2.40	<1.28	22.3	0.53	146.0	78.2	<16.5	115.0	132.0
152	T2SP1-19	1.25E-04	1.25E-03	<8.56	2.31	<1.28	19.8	0.54					
160	T2SP1-20	1.31E-04	1.25E-03	<10.6	2.34	<1.28	19.8	0.52	94.3	55.5	<16.5	121.0	123.0
168	T2SP1-21	1.65E-04	1.43E-03	<6.6	2.34	<1.28	21.8	0.61					
176	T2SP1-22	1.16E-04	1.25E-03	<11.6	2.47	<1.28	18.8	0.56	39.5	26.7	<16.5	83.5	144.0
184	T2SP1-23	1.09E-04	1.23E-03	<7.67	2.40	<1.28	21.3	0.48					
192	T2SP1-24	1.16E-04	1.54E-03	<5.28	2.35	<1.28	22.2	0.52	85.0	79.4	<16.5	79.0	127.0
200	T2SP1-25	1.09E-04	1.29E-03	<10.1	2.46	<1.28	18.7	0.54					
208	T2SP1-26	1.49E-04	1.45E-03	<6.06	2.45	<1.28	21.7	0.57	91.1	68.4	<16.5	78.0	129.0
216	T2SP1-27	1.47E-04	1.47E-03	<2.49	2.37	<1.28	22.0	0.55					
224	T2SP1-28	9.58E-05	1.31E-03	<6.88	2.41	<1.28	18.8	0.48	33.4	23.4	<16.5	81.0	130.0
230	T2SP1-FINAL	1.25E-04	1.36E-03	<4.46	2.14	<1.28	18.4	0.58	20.0	17.0	<16.5	74.8	128.0

Table 27. T2SP2 normalized data for CSTR 2 samples

Reaction time (h)	CSTR 2 sample ID	Cs-137 (µCi/g)	Sr-85 (µCi/g)	K (mg/L)	Cu (mg/L)	Pd (mg/L)	B (mg/L)	U-235 (mg/L)	NaTPB (mg/L)	3PB (mg/L)	2PB (mg/L)	1PB (mg/L)	Phenol (mg/L)
0	T2SP2-Feed	4.30	4.76E-02	458.52	4.69	13.86	22.27	0.90		56.19	57.64	88.95	119.28
0	T2SP2-00	3.12	<0.01	580.00	1.93	<1.28	15	0.38	<16.3	28	53	0	98
8	T2SP2-01	1.18E-01	1.65E-03	437.00	1.98	<1.28	14	0.42					
16	T2SP2-02	1.76E-02	1.69E-03	116.00	2.08	<1.28	14	0.44	<16.3	<15.7	67	149	132
24	T2SP2-03	5.79E-05	1.51E-03	25.70	2.02	<1.28	18						
32	T2SP2-04	7.57E-05	1.43E-03	31.60	2.15	<1.28	18	0.48	52	25	92	100	113
40	T2SP2-05	1.05E-04	1.25E-03	29.90	2.14	<1.28	20						
48	T2SP2-06	1.29E-04	1.38E-03	23.90	2.16	<1.28	22	0.53	91	53	64	88	220
56	T2SP2-07	1.94E-04	1.36E-03	26.80	2.31	<1.28	18	0.52					
64	T2SP2-08	1.29E-04	1.43E-03	22.60	2.37	<1.28	21	0.48	74	47	49	91	137
72	T2SP2-09	1.23E-04	1.36E-03	<21.5	2.30	<1.49	22	0.47					
80	T2SP2-10	9.80E-05	1.31E-03	<7.99	2.26	<1.28	19	0.41	55	42	34	142	138
88	T2SP2-11	9.36E-05	1.49E-03	<12.1	2.10	<1.28	20	0.47					
96	T2SP2-12	8.47E-05	1.36E-03	<8.99	2.39	<1.28	23	0.47	184	84	39	129	117
104	T2SP2-13	6.24E-05	1.40E-03	<12.6	2.32	<1.28	18	0.38					
112	T2SP2-14	7.13E-05	1.27E-03	<5.99	2.34	<1.28	22	0.46	127	65	35	118	124
120	T2SP2-15	8.91E-05	1.29E-03	<12.5	2.39	<1.28	22	0.56					
128	T2SP2-16	7.57E-05	1.31E-03	<12.5	2.44	<1.28	20	0.55	63	38	<16.5	83	166
136	T2SP2-17	7.80E-05	1.36E-03	<4.77	2.43	1.54	22	0.54					
144	T2SP2-18	1.11E-04	1.40E-03	<5.83	2.38	<1.28	22	0.54	165	82	<16.5	117	140
152	T2SP2-19	8.24E-05	1.40E-03	<9.09	2.34	<1.28	19	0.50					
160	T2SP2-20	7.35E-05	1.40E-03	<5.24	2.17	<1.28	20	0.54	113	63	<16.5	112	131
168	T2SP2-21	9.80E-05	1.27E-03	<10.0	2.53	<1.28	24	0.59					
176	T2SP2-22	7.57E-05	1.27E-03	<12.8	2.55	<1.28	20	0.52	37	26	<16.5	88	148
184	T2SP2-23	8.02E-05	1.20E-03	<10.2	2.46	<1.28	23	0.52					
192	T2SP2-24	8.69E-05	1.25E-03	<7.32	2.34	<1.28	23	0.46	150	80	<16.5	78	122
200	T2SP2-25	7.80E-05	1.38E-03	<11	2.43	<1.28	18	0.53					
208	T2SP2-26	9.13E-05	1.40E-03	<9.3	2.46	<1.28	22	0.53	96	61	<16.5	79	127
216	T2SP2-27	1.09E-04	1.60E-03	<3.98	2.43	<1.28	23	0.54					
224	T2SP2-28	8.91E-05	1.45E-03	<5.36	2.37	<1.28	18	0.55	26	23	<16.5	83	134
230	T2SP2-FINAL	7.13E-05	1.45E-03	<8.13	2.25	<1.28	19	0.63	22	17	<16.5	77	130

Table 28. T2SP4 normalized data for concentrate tank filtrate samples

Reaction time (h)	Conc. tank filtrate sample ID	Cs-137 (µCi/g)	Sr-85 (µCi/g)	K (mg/L)	Cu (mg/L)	Pd (mg/L)	B (mg/L)	DNC U-235 (ng/mL)	ICP-MS U-235 (mg/L)	NaTPB (mg/L)	3PB (mg/L)	2PB (mg/L)	1PB (mg/L)	Phenol (mg/L)
0	T2SP4-Feed	4.30	0.05	458.52	4.69	13.86	22.27	642	0.90		56.19	57.64	88.95	119.28
0	T2SP4-00	1.94E+00	<210	392.00	1.26	<1.28	15	174	0.20	31	64	39	77	78
4	T2SP4-01	1.92E+00	<220	495.00	1.62	<1.28	16		0.26					
8	T2SP4-02	2.45E-01	2.67E-03	490.00	1.80	<1.28	17	305	0.37	<16.3	68	70	106	119
12	T2SP4-03	7.35E-02	1.94E-03	365.00	1.89	<1.28	16		0.42					
16	T2SP4-04	5.57E-02	2.45E-03	238.00	2.04	<1.28	17	345	0.43	<16.3	44	69	158	129
20	T2SP4-05	2.67E-02	2.05E-03	69.30	2.09	<1.28	18		0.44					
24	T2SP4-06	2.23E-03	1.89E-03	28.80	2.02	<1.28	22	374	0.41	118	74	98	107	121
28	T2SP4-07	5.35E-04	1.89E-03	30.20	2.09	<1.28	23		0.42					
32	T2SP4-08	3.56E-04	1.83E-03	18.10	2.07	<1.28	23	369	0.45	192	73	92	98	122
36	T2SP4-09	3.79E-04	1.76E-03	26.10	2.05	<1.28	24		0.42					
40	T2SP4-10	2.14E-04	1.65E-03	29.50	2.12	<1.28	25	354	0.44	210	77	91	88	117
44	T2SP4-11	1.63E-04	1.60E-03	9.36	2.02	<1.55	25		0.46					
48	T2SP4-12	2.03E-04	1.56E-03	25.40	2.12	<1.28	27	354	0.46	189	81	56	90	311
52	T2SP4-13	1.78E-04	1.74E-03	26.60	2.14	<1.28	27		0.45					
56	T2SP4-14	1.87E-04	1.34E-03	12.20	2.19	<1.28	27	331	0.44	196	83	54	79	0
60	T2SP4-15	1.80E-04	1.45E-03	20.20	2.34	<1.28	27		0.49					
64	T2SP4-16	1.74E-04	1.45E-03	22.30	2.26	<1.28	27	350	0.45	203	84	51	100	140
68	T2SP4-17	1.65E-04	1.43E-03	<14.7	2.32	<1.28	26		0.37					
72	T2SP4-18	1.96E-04	1.45E-03	<24.4	2.38	<1.28	26	358	0.41	211	94	39	121	143
76	T2SP4-19	1.76E-04	1.40E-03	<15.9	2.43	<1.28	27		0.43					
80	T2SP4-20	1.89E-04	1.45E-03	<11.2	2.17	<1.28	25	353	0.42	200	96	36	125	138
84	T2SP4-21	2.18E-03	1.40E-03	<5.49	2.03	<1.28	25		0.37					
88	T2SP4-22	4.01E-04	1.40E-03	<13.2	2.08	<1.28	25	350	0.42	295	<15.7	<16.5	140	141
92	T2SP4-23	2.07E-04	1.43E-03	<2.78	2.28	<1.28	26		0.42					
96	T2SP4-24	1.85E-04	1.34E-03	<10.2	2.10	<1.28	24	382	0.43	276	103	39	121	118
100	T2SP4-25	1.51E-04	1.49E-03	<7.06	2.27	<1.28	25		0.39					
104	T2SP4-26	1.63E-04	1.47E-03	<14.5	2.33	<1.28	26	369	0.38	290	101	37	120	117
108	T2SP4-27	1.92E-04	1.45E-03	<13.9	2.21	<1.28	26		0.44					
112	T2SP4-28	1.47E-04	1.45E-03	<9.61	2.27	<1.28	26	360	0.46	286	100	34	117	121
116	T2SP4-29	1.49E-04	1.47E-03	<9.96	2.25	<1.28	27		0.50					

Table 28 (continued)

Reaction time (h)	Conc. tank filtrate sample ID	Cs-137 (µCi/g)	Sr-85 (µCi/g)	K (mg/L)	Cu (mg/L)	Pd (mg/L)	B (mg/L)	DNC U-235 (ng/mL)	ICP-MS U-235 (mg/L)	NaTPB (mg/L)	3PB (mg/L)	2PB (mg/L)	1PB (mg/L)	Phenol (mg/L)
120	T2SP4-30	1.47E-04	1.40E-03	<7.99	2.28	<1.28	26	362	0.50	232	93	80	81	255
124	T2SP4-31	1.23E-04	1.47E-03	<10.2	2.24	<1.28	26		0.47					
128	T2SP4-32	1.27E-04	1.40E-03	<12.3	2.30	<1.28	27	359	0.48	231	92	33	82	0
132	T2SP4-33	1.20E-04	1.40E-03	<12.4	2.23	<1.28	26		0.46					
136	T2SP4-34	1.23E-04	1.43E-03	<6.67	2.28	1.62	28	361	0.44	233	90	<16.5	41	0
140	T2SP4-35	1.18E-04	1.47E-03	<1.76	2.38	<1.84	28		0.40					
144	T2SP4-36	1.29E-04	1.38E-03	<6.35	2.18	<1.28	26	358	0.41	281	106	<16.5	123	0
148	T2SP4-37	1.34E-04	1.45E-03	<8.75	2.27	<1.28	26		0.47					
152	T2SP4-38	1.09E-04	1.40E-03	<9.75	2.27	<1.28	26	384	0.45	260	88	<41.1	129	0
156	T2SP4-39	1.38E-04	1.51E-03	<10.4	2.22	<1.28	27		0.46					
160	T2SP4-40	1.25E-04	1.47E-03	<8.42	2.15	<1.28	25	378	0.47	291	106	<16.5	91	0
164	T2SP4-41	1.29E-04	1.40E-03	<2.63	2.21	<1.72	26		0.48					
168	T2SP4-42	1.29E-04	1.49E-03	<11.0	2.37	<1.28	28	379	0.49	241	96	<16.5	78	146
172	T2SP4-43	1.14E-04	1.45E-03	<12.2	2.45	<1.28	28		0.53					
176	T2SP4-44	1.00E-04	1.54E-03	<11.9	2.34	<1.28	28	378	0.50	214	93	<16.5	91	145
180	T2SP4-45	1.00E-04	1.45E-03	<11.7	2.32	<1.28	27		0.45					
184	T2SP4-46	1.18E-04	1.49E-03	<10.2	2.31	<1.28	28	375	0.53	223	95	<16.5	92	146
188	T2SP4-47	1.02E-04	1.40E-03	<3.67	2.27	<1.88	28		0.44					
192	T2SP4-48	9.80E-05	1.36E-03	<8.29	2.21	<1.28	27	359	0.46	277	109	<16.5	79	123
196	T2SP4-49	8.91E-05	1.31E-03	<9.02	2.21	<1.28	27		0.45					
200	T2SP4-50	1.34E-04	1.27E-03	<10.8	2.31	<1.28	28	396	0.49	271	107	<16.5	78	124
204	T2SP4-51	1.14E-04	1.36E-03	<10.8	2.31	<1.28	28		0.47					
208	T2SP4-52	8.24E-05	1.27E-03	<10.2	2.33	<1.28	27	383	0.47	94	61	<16.5	78	123
212	T2SP4-53	9.36E-05	1.45E-03	<1.5	2.31	<1.66	27		0.49					
216	T2SP4-54	1.05E-04	1.40E-03	<5.78	2.36	<1.28	27	402	0.53	202	97	<16.5	88	132
220	T2SP4-55	8.02E-05	1.43E-03	<5.58	2.31	<1.28	27		0.49					
224	T2SP4-56	1.00E-04	1.40E-03	<6.72	2.29	<1.28	27	400	0.47	197	96	<16.5	83	133
228	T2SP4-57	8.24E-05	1.51E-03	<7.12	2.32	<1.28	28		0.45					
230	T2SP4-FINAL	9.58E-05	1.40E-03	<8.52	2.14	<1.28	27	397	0.61	200	97	<16.5	78	130

(outside) pressures, the difference being the TMP for the filter. Initially, several hours of filtration was required before the pressure difference became too great to achieve the desired filtrate flow. As the test continued, the time between backpulses shortened. By the last day of operation, backpulsing was required every 15–30 min.

Although the constant backpulsing required a lot of attention, the test was completed without any CSTR downtime. One attempt at a complete backpulse was made about 90 h into the test. The concentrate tank suffered a loss of slurry when the nitrogen gas used to pressurize the filter shell through the filter pressure vessel entered the tank after the filtrate that was used for backpulsing was gone. This surge of gas caused a level increase that made the slurry concentrate overflow into the catch sump. It appeared from visual observation through the hot cell window that about 1–1.5 L was in the sump. It was then pumped back into the concentrate tank. Although this event caused a contamination upset, very little total slurry was lost during the incident. Unfortunately, the full backpulse did not improve the filter operation.

Several modified backpulse methods were developed during operation that allowed the filtrate flow to be maintained. They all used the pressure on the shell side of the filter when the tube-side pressure control valve, PCV-1, was fully opened and the shell-side valve, PCV-2, was closed. This reversed the normal pressure and helped to remove some of the slurry solids from the filter. This process reduced the TMP and increased the filtrate flow rate for a short time. In addition to backpulsing, the tube-side pressure was gradually increased toward the end of the test. This provided a higher TMP and allowed the run to continue. As the pressure was increased, the Moyno pump speed was also increased to try to keep the axial flow through the filter tube near the design specifications. The graphs show these efforts, which increased the filtrate flow. At the end of the test, the tube-side pressure was operated at 60–65 psi vs the normal 40 psi. The TMP also increased to about 40–45 psi compared with 1–3 psi in Test 1a.

Figures 43 and 44 are expanded graphs showing data for daily operations. The rest of the 1-day plots are given in Appendix E. The day 3 plot in Fig. 43 is typical for the first part of the test, while Figure 44 shows the day 10 plot and the short time between backpulsing, which was required toward the end of the test. Figures 43 and 44 also include the slurry flow rate and slurry density plots as measured by the Coriolis meter. The PCV-2 valve position is also shown and indicates that the valve had to be held wide open in order to obtain enough filtrate flow to keep the concentrate tank in mass balance. During the first few days, some filtrate flow control was

possible, which worked well to maintain a constant flow rate; however, during the last 3 days, the valve had to be left wide open except during backpulsing.

After the test was completed, the concentrated slurry in the concentrate tank was transferred to the wash tank and the concentration loop was rinsed for several hours with 0.5 *M* NaOH solution. The solution was circulated through the loop on two different days at a flow rate of about 3 gal/min. Afterwards, the slurry concentration system was drained and chemically cleaned using 8 L of a 2 wt % solution of oxalic acid, which was were added to the system and circulated for about 3 h at 3 gal/min. Several backpulses were conducted near the end of the chemical cleaning. Subsequently, the cleaning solution was drained from the system and the system was refilled with a 0.25 *M* NaOH solution. The dilute caustic was circulated and the system pressurized to test the filtration rate. It appeared that the filter had been substantially cleaned because the flow rate attainable was greater than what could be measured by the flowmeter (>40 mL/min). Furthermore, it was attained at a TMP of less than 2 psi with the control valve partially closed (<95% open). The flow rate and pressure drop were similar to those obtained in the simulant testing before the start of Test 1a.

To lessen the possibility of TPB decomposition, which is accelerated at temperatures above 30°C, it was important to control the temperature of the slurry in the concentration loop during the test. During Test 1a, temperature control was not a problem, but during Test 2, with the pressure requirements much higher and with the Moyno pump forced to operate at higher speeds to maintain axial velocity, more heat was added to the slurry. The concentration tank temperature, TI-3, is shown in Fig. 43. Between hours 48 and 56 of the test, the temperature control was poor. To correct the problem, a larger cooling unit was installed, which maintained control of the temperature until it failed several days later between hours 165 and 185. Another cooling unit was then installed, which performed adequately for the duration of the test.

One of the main concerns in the concentration loop was the performance of the level-indication instrumentation. Before the start of Test 2, an alternate level indicator was sought. A Magnetrol RF capacitance level transmitter was tried, but it did not work. It failed because of the close proximity of the metal vessel internals along with the slurry bridging between the internals and the probe. A Bindicator hydrostatic level transmitter was tried next, but the range was insufficient and the pressure transducer failed. This led to using a wooden dowel as a level gauge. A ¾-in. wooden dowel, 3 ft long, was placed into the opening in the top of the tank, which had been used for the other level instruments that were tried, and lowered to the bottom of

the tank. It was then carefully removed from the tank, and the depth of the liquid on the stick was measured. This worked well and allowed the mass balance to be maintained, which minimized the possibility of the slurry becoming overconcentrated or the tank becoming too full and spilling over into the overflow sump. Slurry level reading were taken at intervals of 1 to 2 h and compared with the previous reading to provide information for filtrate flow rate control. A blue chalk was used to coat the dowel stick to make it easier to see the exact level of slurry on the stick. The measurement was made with a ruler that could be observed through the hot cell window.

8.5 SLURRY CONCENTRATE WASHING AND FILTRATION SYSTEM

Washing of the slurry concentrate was the next step in the operation of the CSTR system. Concentrated slurry (about 4 or 5 L) was transferred from the concentration tank to the wash tank. Deionized wash water flow was then started at a rate of 10 mL/min while the slurry was circulated through the slurry wash CFF. The filtrate was pumped to the recycle wash water holding tank. The filtrate from the CFF was sampled at 2-h intervals to determine the nitrite concentration. The determination of nitrite removal by dilution with water was a measure of the approach to ideal mixing. Figures 45–48 show the concentrations of some of the components in the slurry measured during washing. Figure 45 shows results obtained from washing slurry concentrate at an addition rate of 10 mL/min for about 19 h. As expected, the nitrite concentration decreased steadily during washing. Figures 46–48 show the results for washing rates of 15, 8, and 5 mL/min, respectively.

As the salt content of the slurry concentrate solution became less and less concentrated, more NaTPB dissolved. The cesium concentration in the filtrate also increased slightly. The boron content decreased slightly during all the washes except for the 5-mL/min wash, in which it increased during washing. The washes were continued until the nitrite concentration dropped below about 800 ppm, with the target concentration being about 500–600 ppm. Figures 49 and 50 compare the nitrite concentration in the filtrate for the four wash rates vs time and wash water volume, respectively. The nitrate concentrations vs time plots indicate that the wash water rate greatly affected the efficiency of washing; however, when the wash volumes were considered, the efficiency of nitrite removal was almost constant.

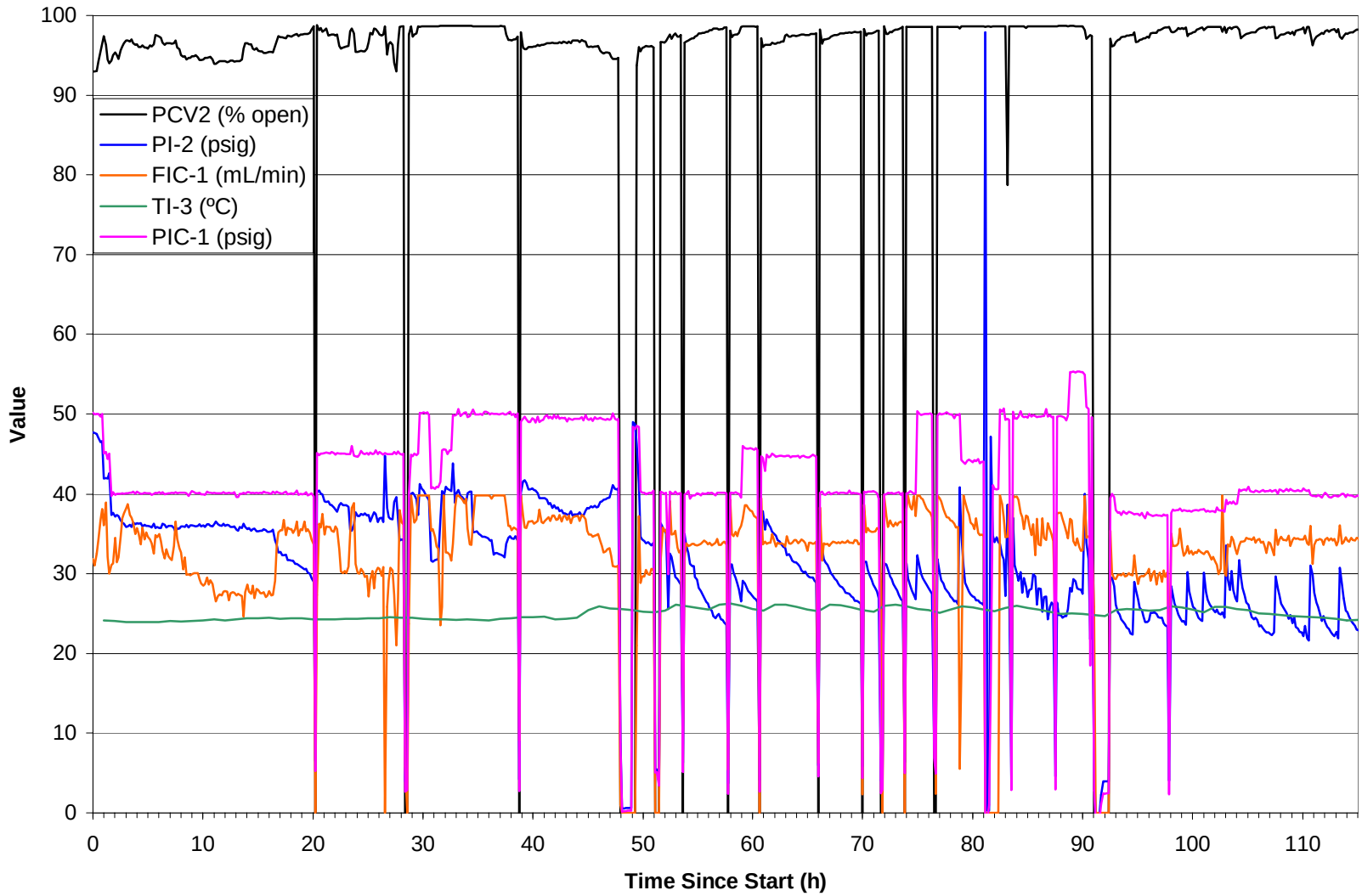


Fig. 41. Concentration loop, first half of Test 2.

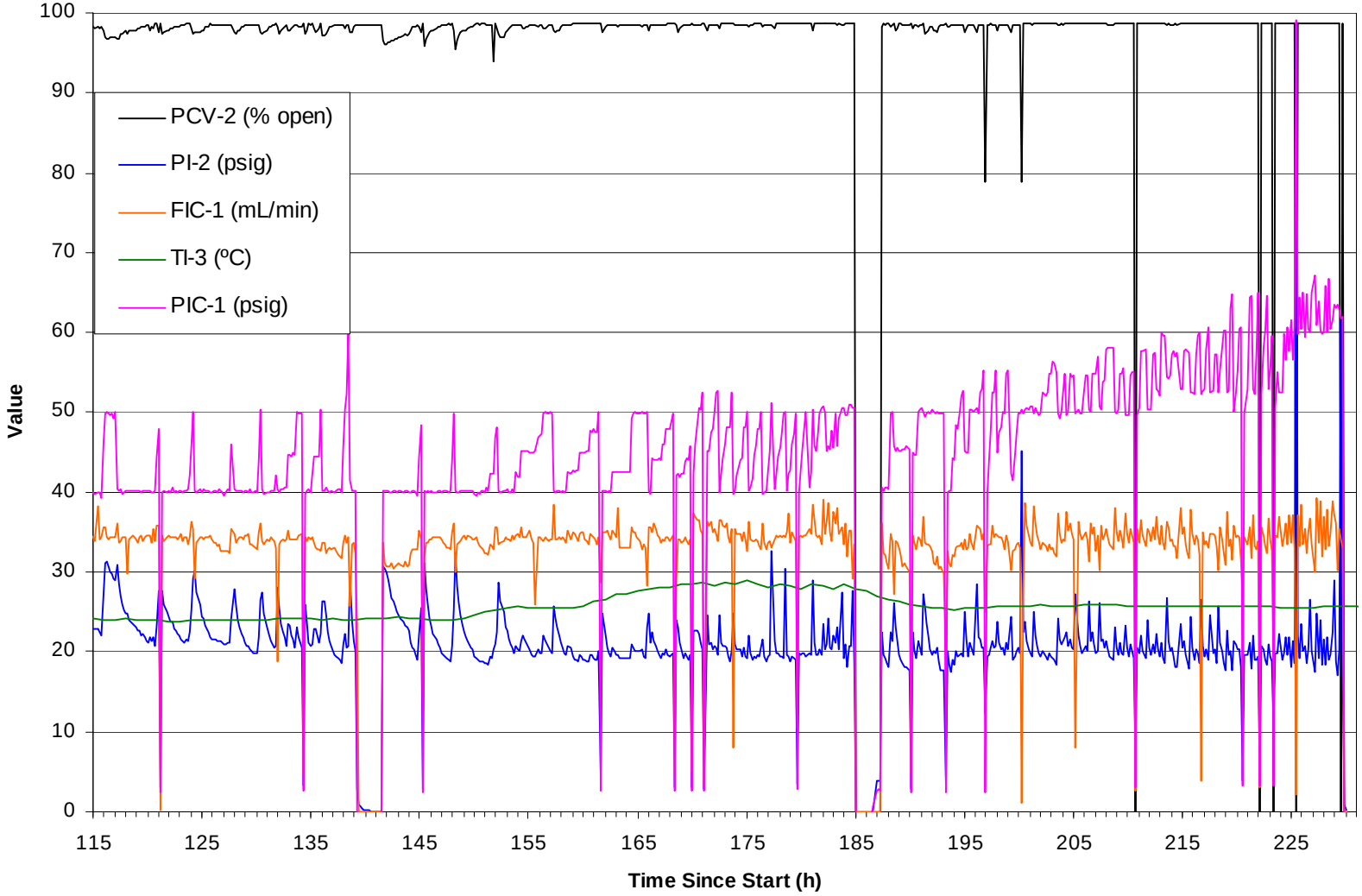


Fig. 42. Concentration loop, second half of Test 2.

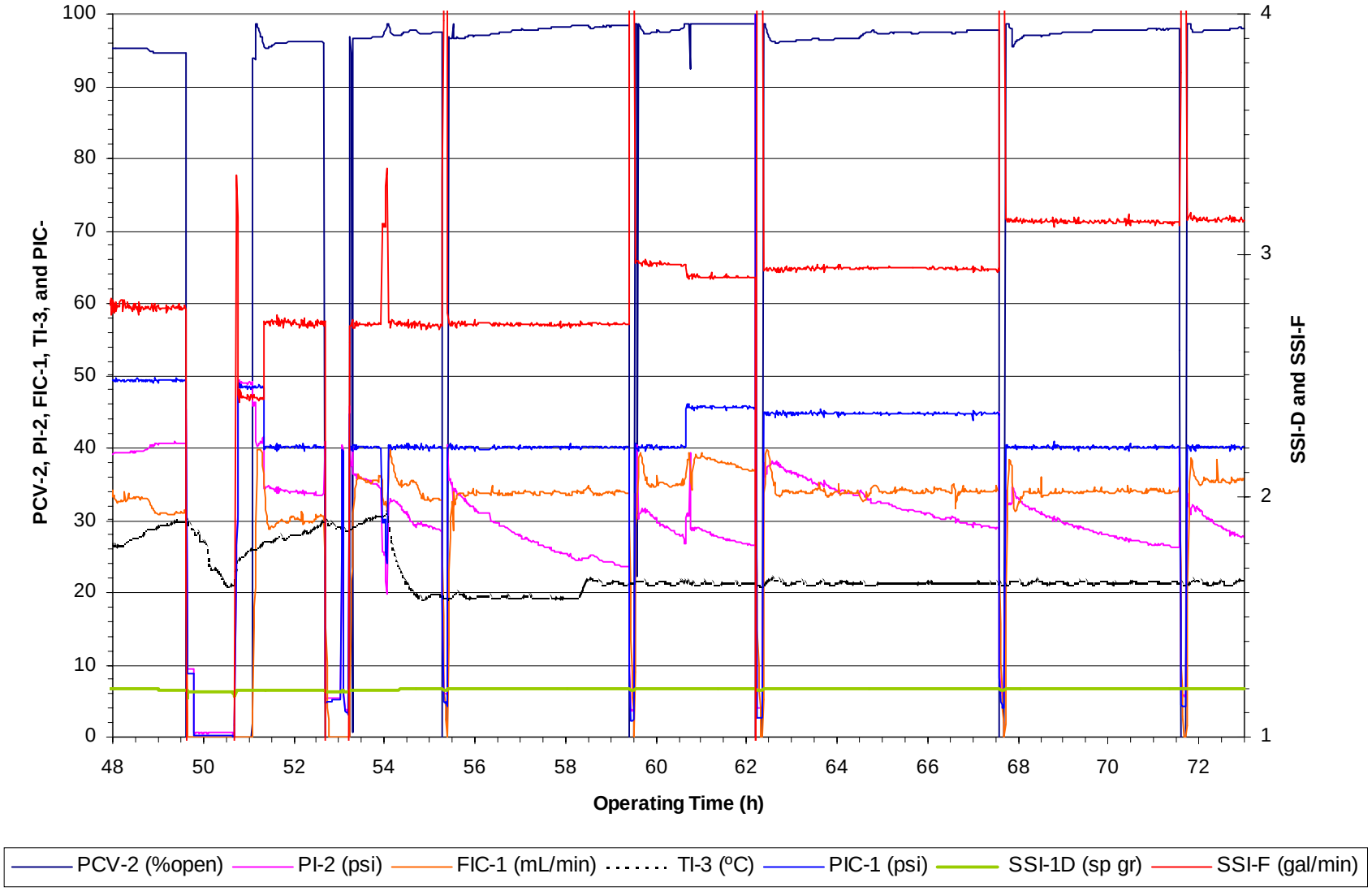


Fig. 43. Slurry concentration loop – day 3.

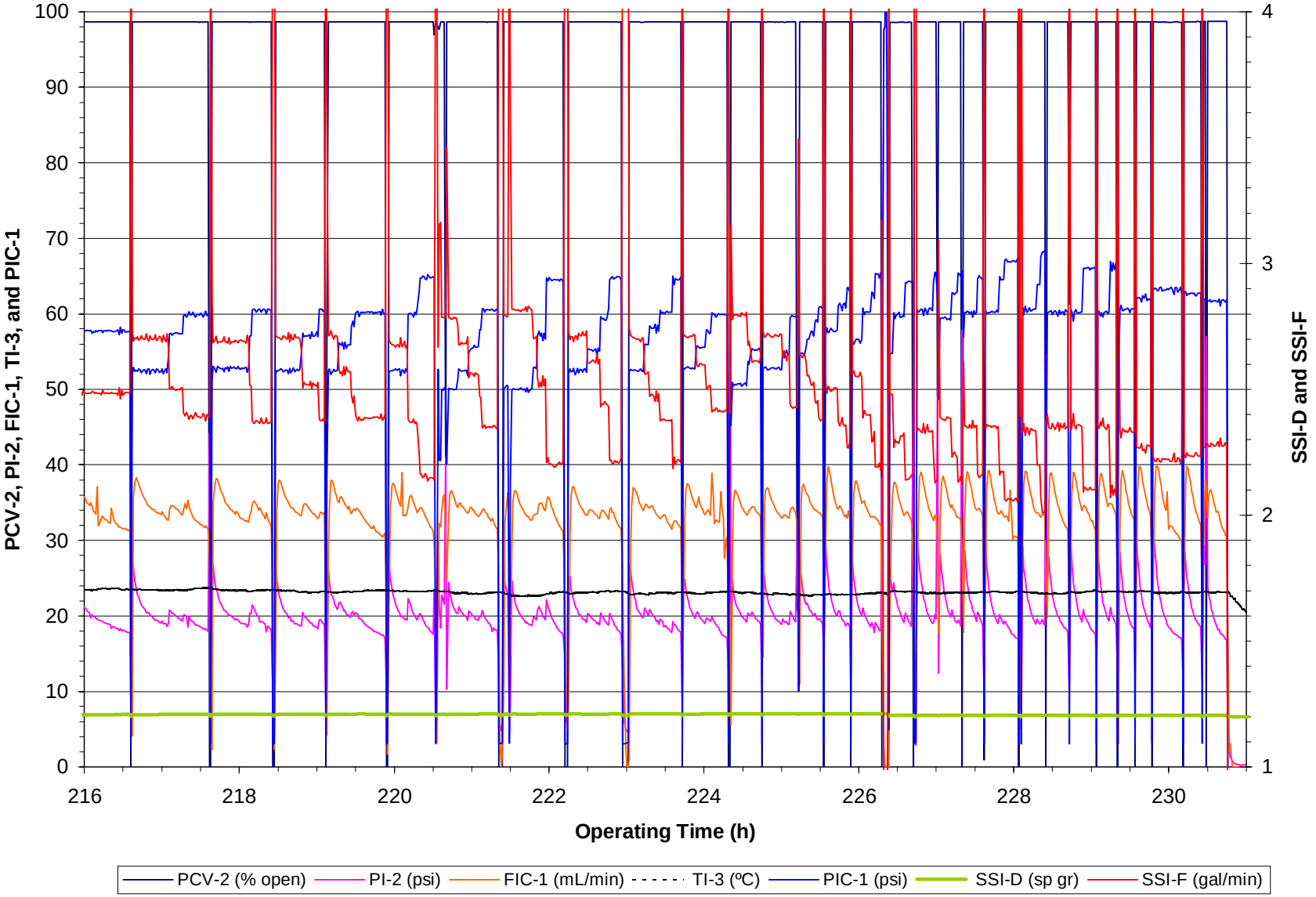


Fig. 44. Slurry concentration loop – day 10.

Figures 51–54 show the nitrite and the radionuclide concentrations for Cs, Sr, and U during the wash step. Strontium and uranium appeared to follow the dilution curve similar to nitrite in all cases, while cesium concentration increases during washing. Apparently, the increased cesium content was caused by the cesium TPB being slightly soluble. The increase in cesium was small, and it was recycled to the first CSTR in the recycle wash water.

The operation of the slurry washing loop during the four slurry washes is shown Figs. 55–58. These graphs show similar results to those given earlier for the slurry concentration loop. In this case, the level instrument, the Bindicator hydrostatic level transmitter, worked well and the loop was run under level control to keep the filtrate rate equal to the wash water feed rate. Due to the control constants used in the control algorithms, considerable time was required for the system to reach steady state. This was evident in the large cycles of the control valve and the tank level; the larger cycles occurred at lower flow rates.

As indicated earlier, the TMP in the wash filter stayed in an acceptable range throughout the wash sequence. However, as the figures show, the pressure increased slightly during each cycle and also increased from one cycle to the next. By the end of the fourth washing, when the wash water flow rate was at 5 mL/min, the pressure increased to about 3 psi. After the test was completed, as part of the preparations for disposal of the slurry, the fifth batch of slurry concentrate was washed at a rate of about 8 mL/min for a few hours per day over several days. The results were similar, with the pressure increasing slightly, but with no operability problems. No backpulsing was required. The rinse water produced in the last washing was added to the rinse water that was already in the holding tank and will be available for use in the startup of any future experiments.

The only problem that occurred during the slurry washing was associated with the flow monitor, FIC-3, on the filtrate going to the recycle wash water hold tank. The value of the flow rate sent by the monitor was about twice that of the measured value. Although the unit was calibrated prior to use, the calibration was not accurate. Nevertheless, the results were consistent and useful for relative flow rate monitoring. Temperature control was never a problem in the slurry wash loop.

When the slurry concentrate washing was completed, the contents of the tank and loop were transferred to a plastic carboy, which were prepared for about 5–10 L of slurry. A sample

of the washed slurry was taken for analysis from the circulation loop just before the transfer. The carboy was placed on a balance, the lid with attached transfer tubing was attached, and the balance tared. When the concentrated slurry was transferred, the drop in the wash tank level and the increase in the mass of slurry on the balance could be followed. Once weighed, the original cap was placed loosely back on the carboy and stored in the same hot cell to await removal and disposal. The four batches of washed slurry contained 19.2, 1.2 , 64.8, and 78.3 µCi/mL of ^{137}Cs, respectively, for batches 1–4. This would make batch 4, which contained about 8 L of slurry, have about 0.63 Ci of ^{137}Cs and result in a radiation field of >10 R at contact with the bottle. Batch 2, containing only 1.2 µCi/mL, contained a very low slurry concentration due to a short slurry collection time and a dilute washed slurry. The carboy containing this slurry had a substantial volume (>80%) of water after washing, compared with the other batches.

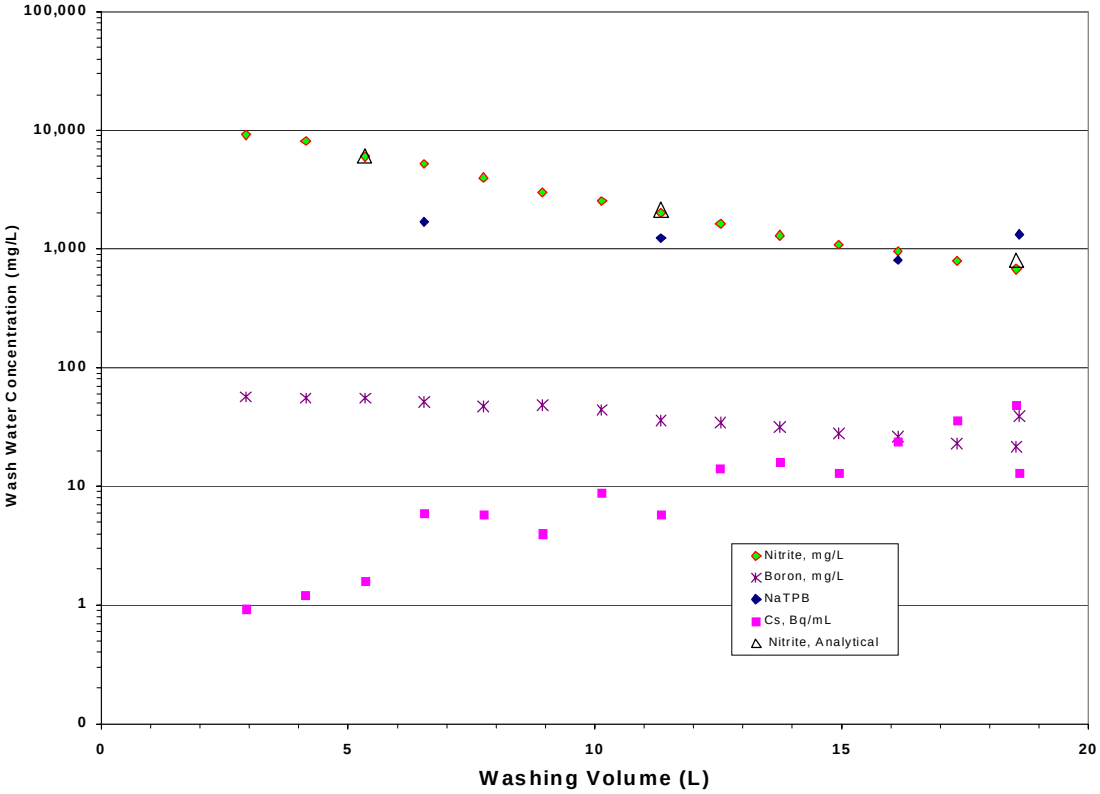


Fig. 45. Nitrite concentration vs wash volume for first slurry washing, 10-mL/min wash rate.

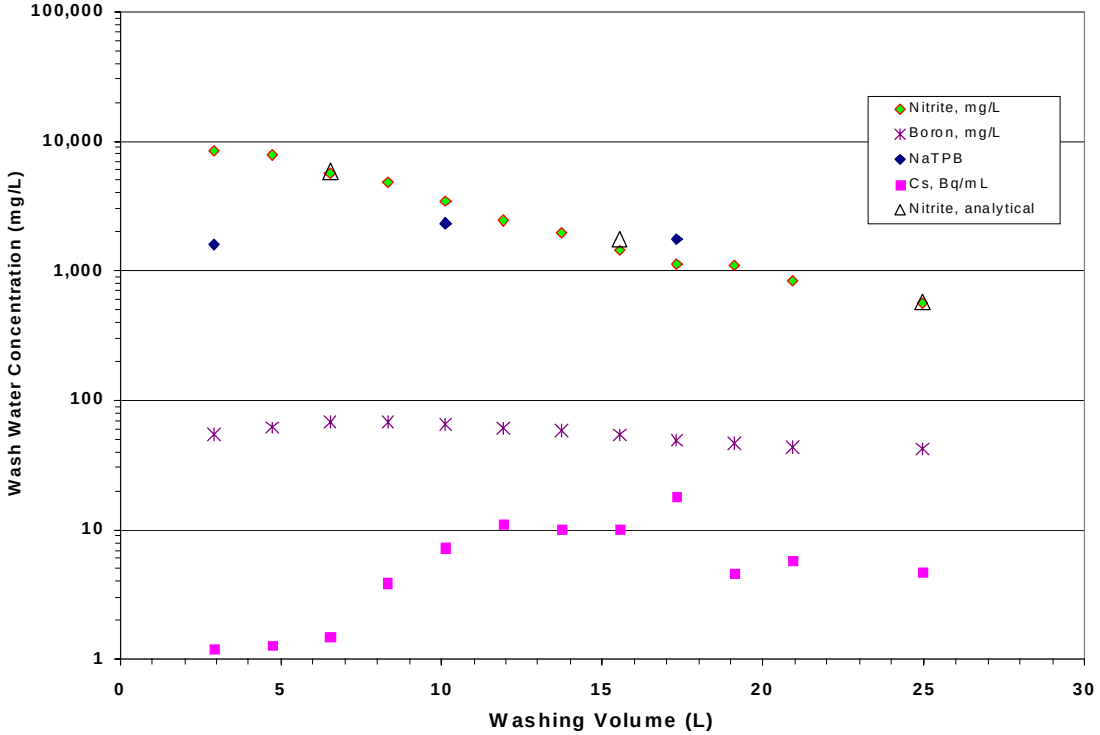


Fig. 46. Nitrite concentration vs wash volume for second slurry washing, 15-mL/min wash rate.

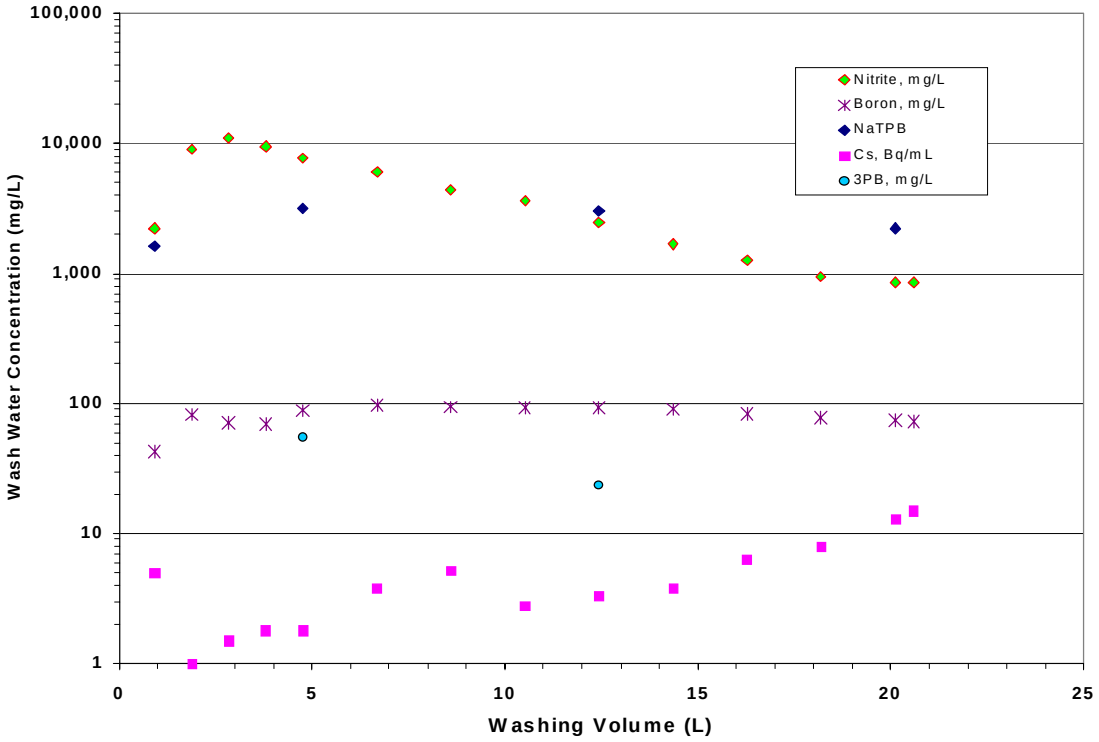


Fig. 47. Nitrite concentration vs wash volume for third slurry washing, 8-mL/min wash rate.

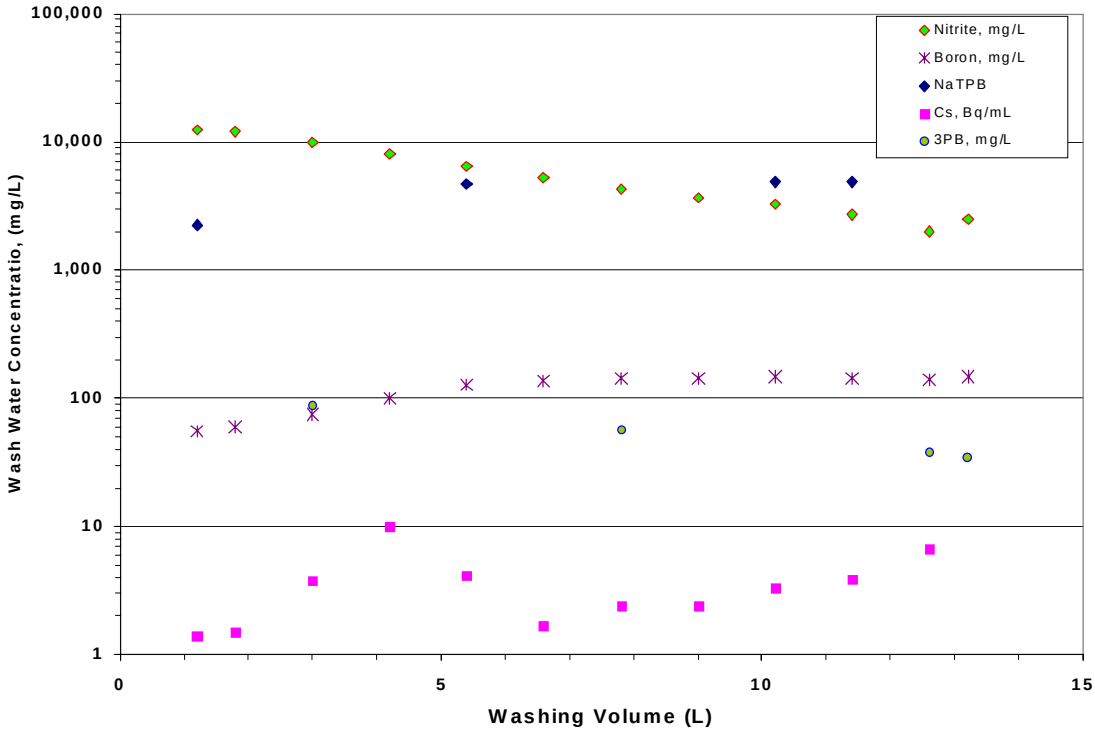


Fig. 48. Nitrite concentration vs wash volume for fourth slurry washing, 5-mL/min wash rate.

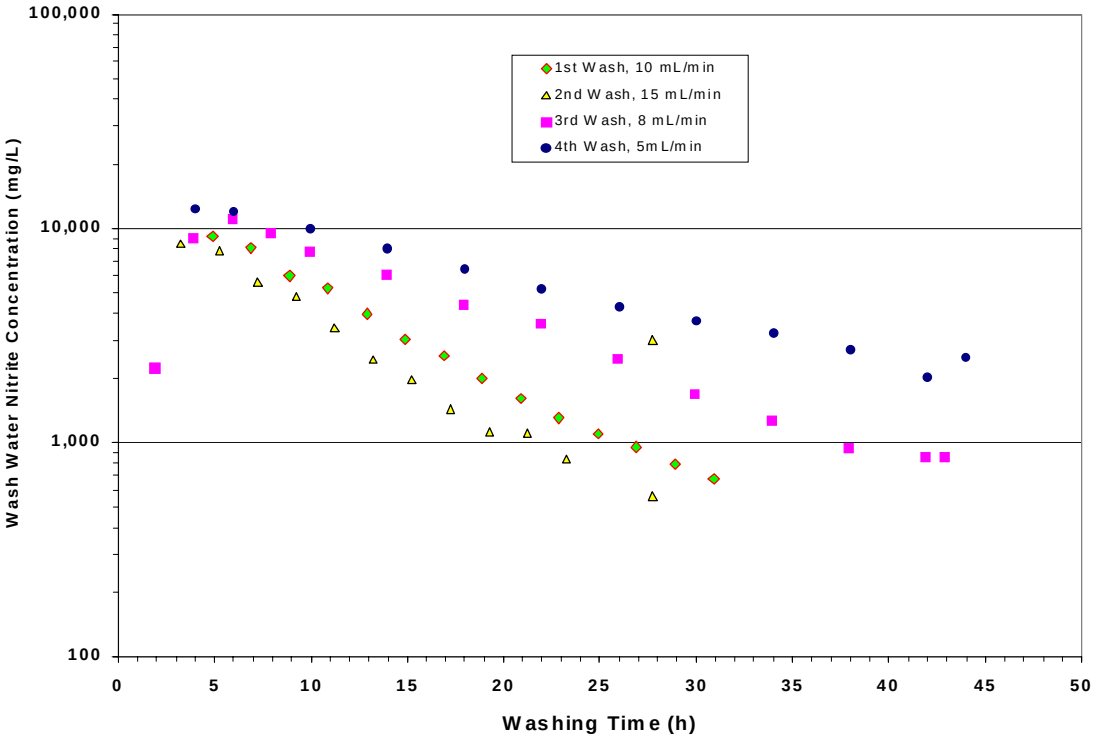


Fig. 49. Nitrite concentrations vs wash times for slurry washing at four wash rates.

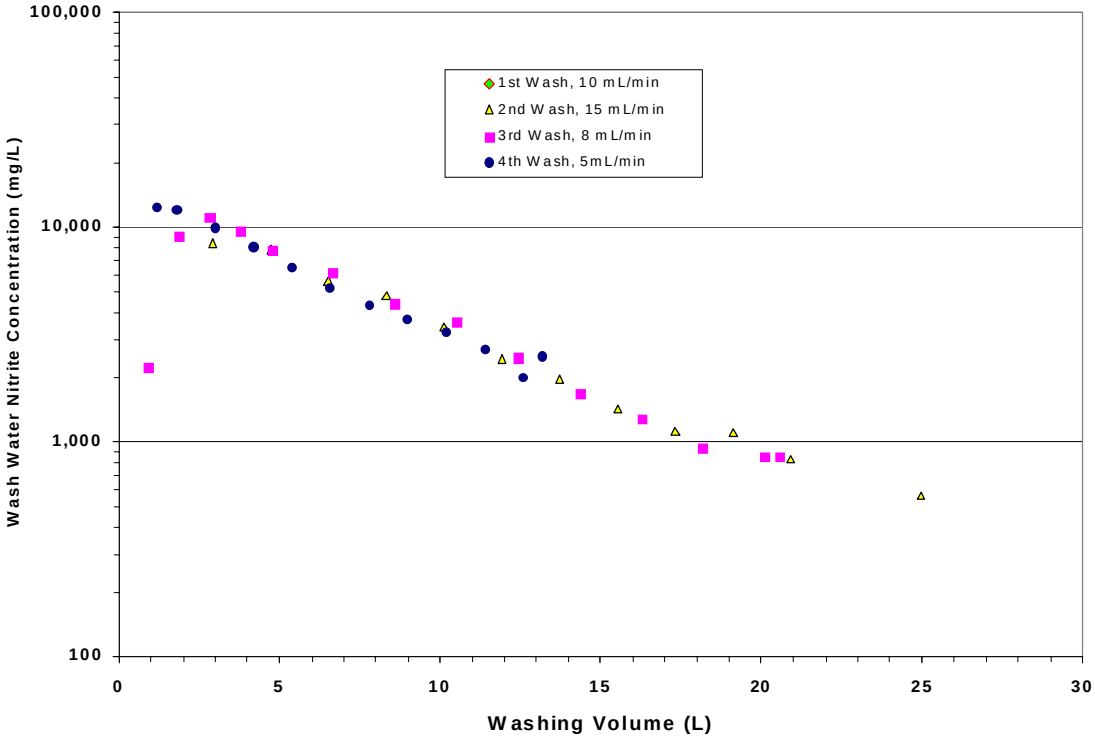


Fig. 50. Nitrite concentrations vs wash volume for slurry washing at four wash rates.

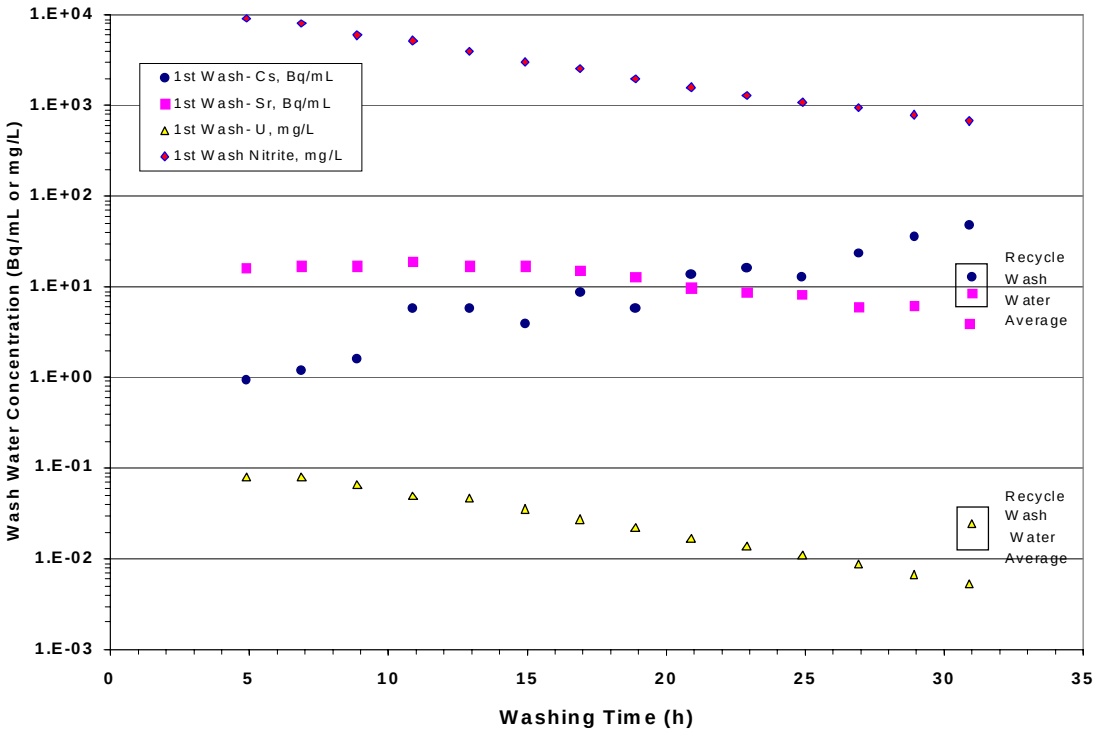


Fig. 51. Cesium, strontium, and uranium concentrations vs time for SP7-wash 1(10 mL water/min).

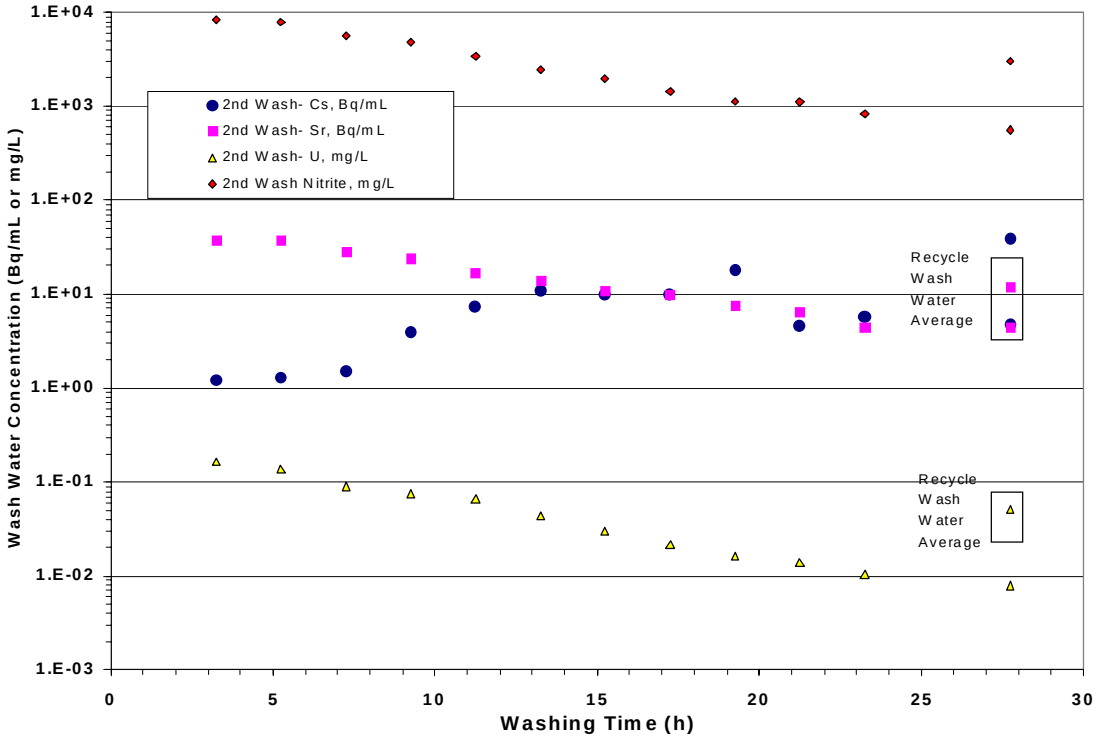


Fig. 52. Cesium, strontium, and uranium concentrations vs time for SP7-wash 2 (15 mL water/min).

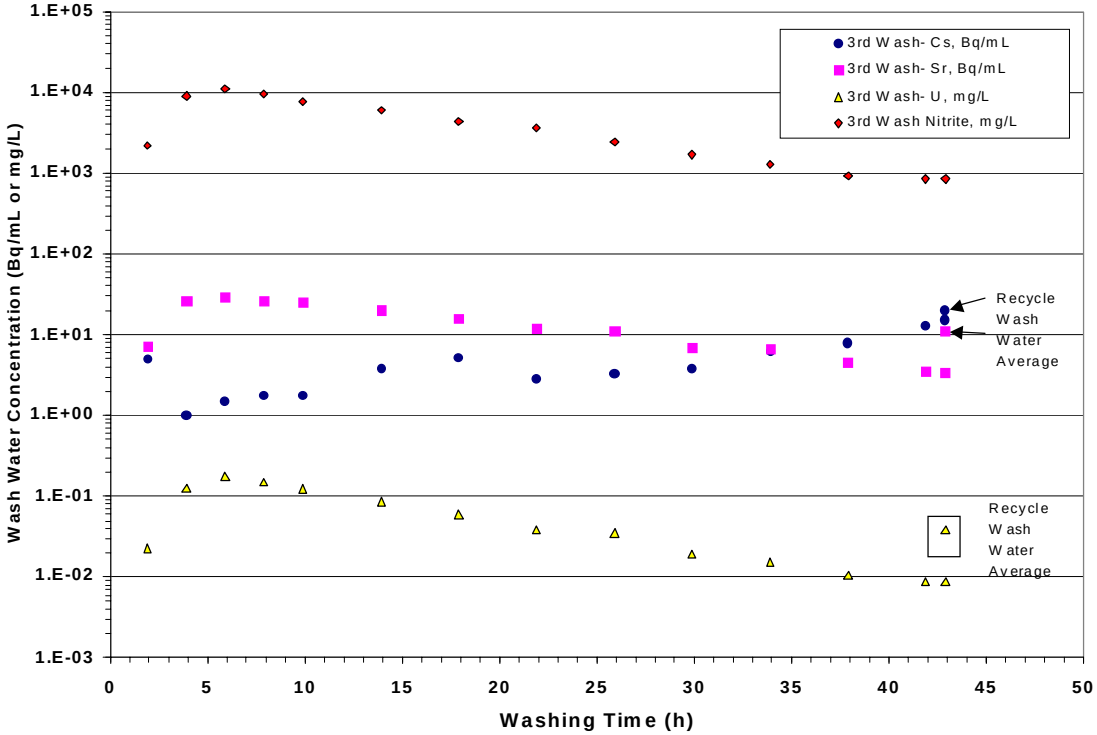


Fig. 53. Cesium, strontium, and uranium concentrations vs time for SP7-wash 3 (8 mL water/min).

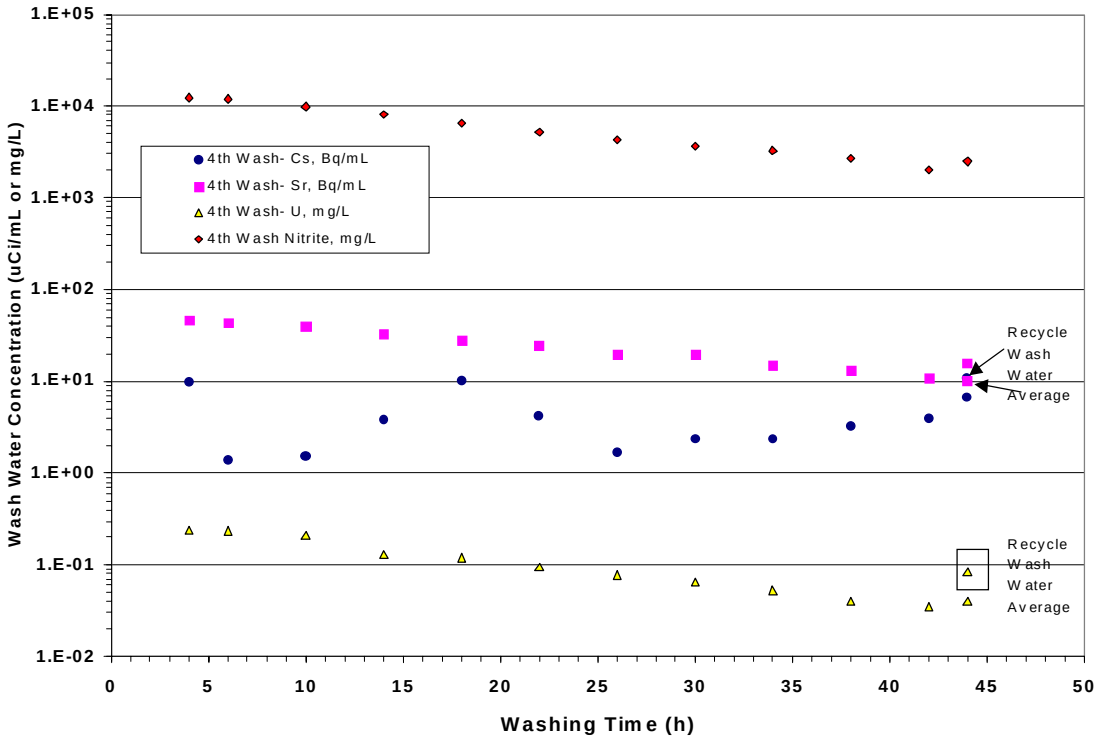


Fig. 54. Cesium, strontium, and uranium concentrations vs time for SP7-wash 4 (5 mL water/min).

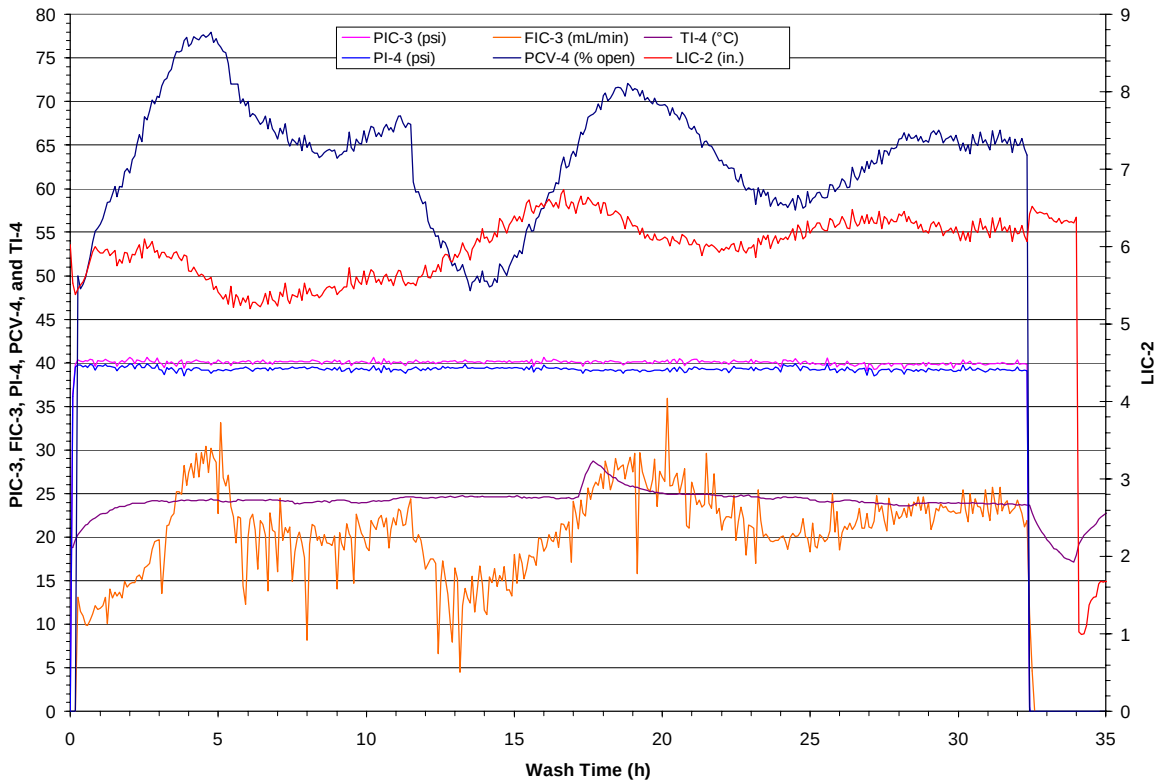


Fig. 55. First slurry wash.

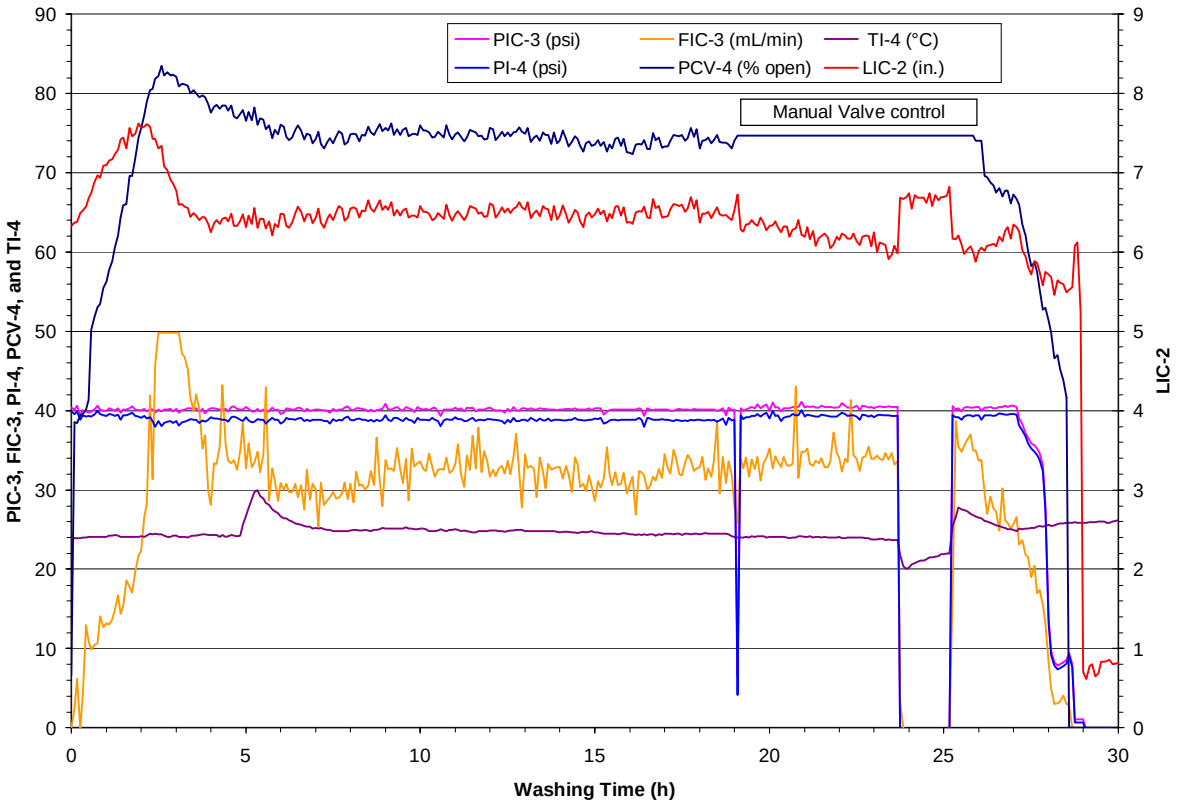


Fig. 56. Second slurry wash.

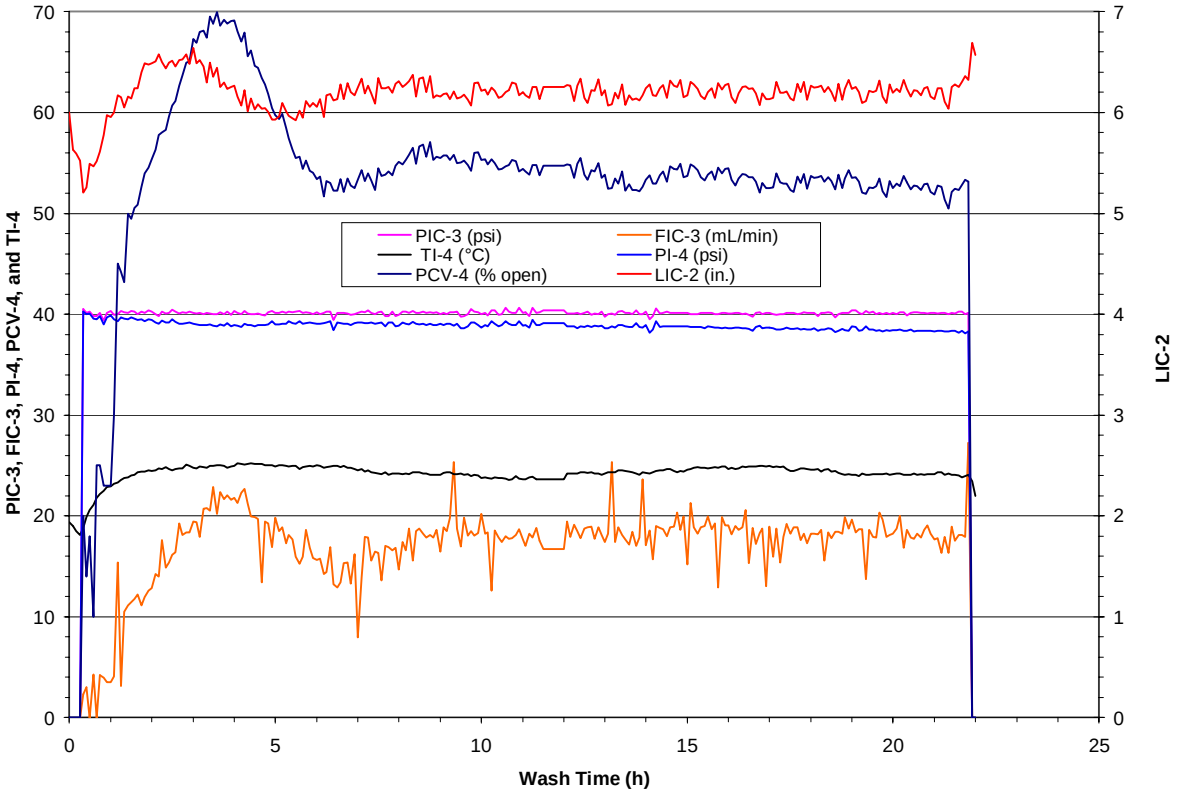


Fig. 57. Third slurry wash.

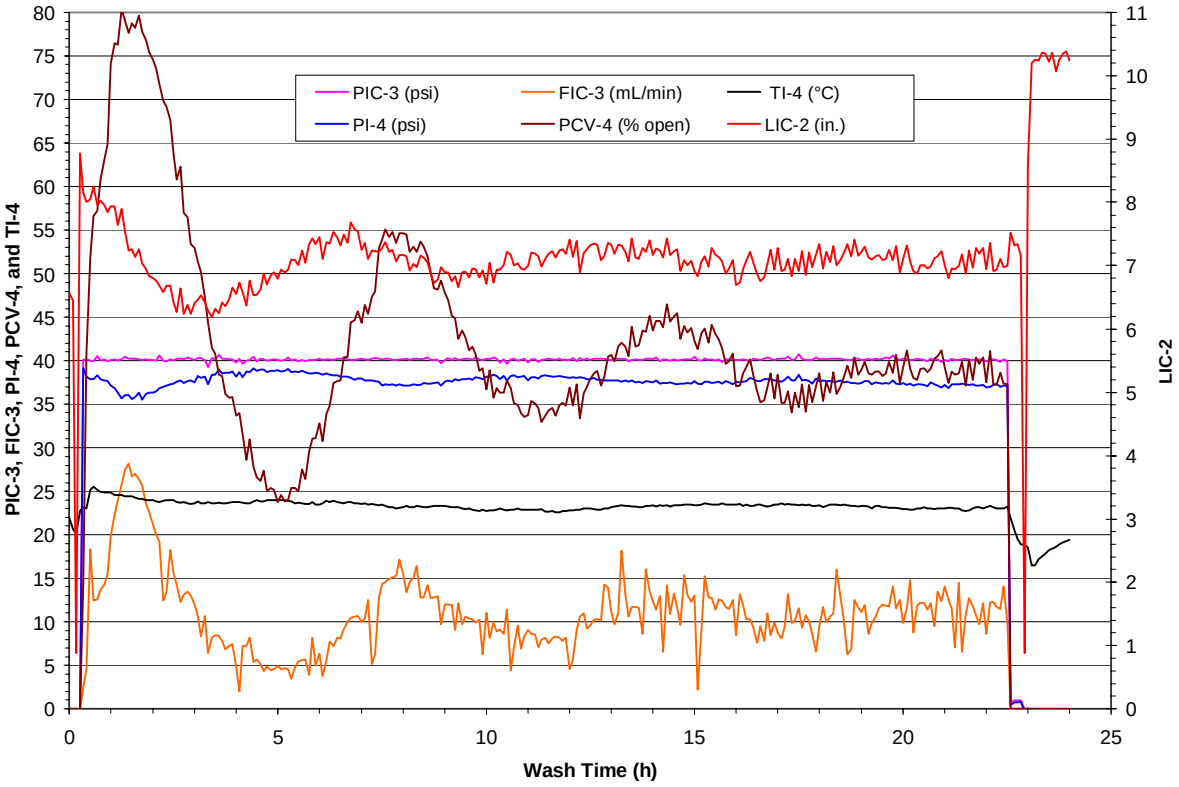


Fig. 58. Fourth slurry wash.

8.6 WASH WATER RECYCLING

Flow balances for the wash water recycle system are shown in Figures 59–62. The figures show how consistent the rate of feed was to the wash system and how close the rate was to the planned wash rate. Some of the flow rate values that appear erratic were caused by incorrectly recording values on the hour when they were actually taken as much as 15 minutes before or after the hour. For example, when the change in mass was measured with a balance holding the water feed bottle, those small differences in the time gave quite different flow rates, especially in cases when a measurement was taken before the hour and the next after the hour.

The data for the samples of wash water filtrate are shown in Table 29. The results show the sample time after the washing was started and the analytical results for the Cs, Sr, K, Cu, Pd, B, nitrate, U, NaTPB, 3PB, 2PB, 1PB, and phenol present in the filtrate. Internal and analytical nitrite analyses agreed very well. The boron levels in the filtrate increased from about 40 mg/L in the first wash to 50 mg/L in the second, 75 mg/L in the third, and about 140 mg/L in the fourth. In addition, more 3PB was present in the filtrate of the fourth wash than in the third or second washes. It was below the detection limit for most of the first wash. No 2PB was detected in any of the filtrate samples, but some 1PB and phenol were detected in both the third and fourth washes, providing some evidence, along with the slight increase in boron concentration, of minor decomposition of the TPB. The amount of TPB present in the filtrate seemed to be a function of the wash water feed rate, with the greatest amount of TPB present in the fourth wash, which was a 5-mL/min wash.

The flow results for the recycle wash water section of the system are shown in Figs. 63 and 64. Figure 63 shows the startup and then the first two washes. The plots show the recycle wash water flow out of the filter (FIC-3), the wash water flow to CSTR 1 (FI-4), and the wash water tank level. The plots also show when the two 10-L batches of simulated wash water were added during the initial 60 h of operation. Since insufficient wash water was produced during the Test 1a slurry washing, simulated wash water had to be added to make up the difference that was needed. Enough had to be available to allow time for the initial batch of concentrated slurry to be made in Test 2 and then washed.

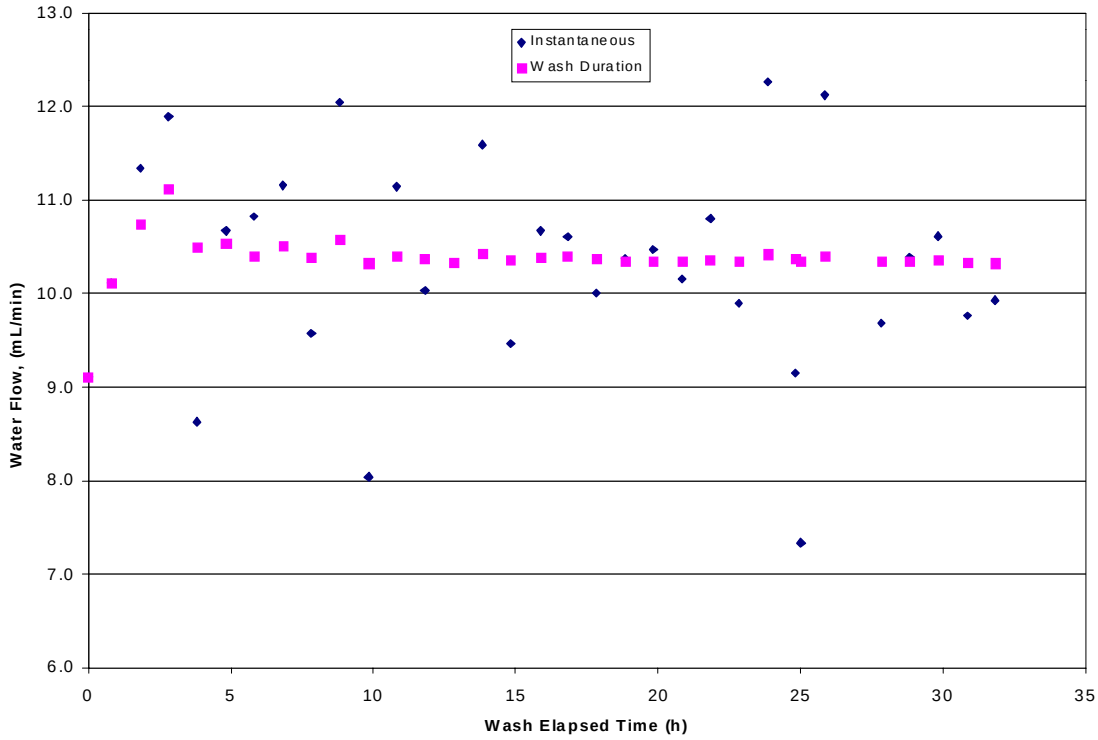


Fig. 59. First slurry wash–-wash water feed.

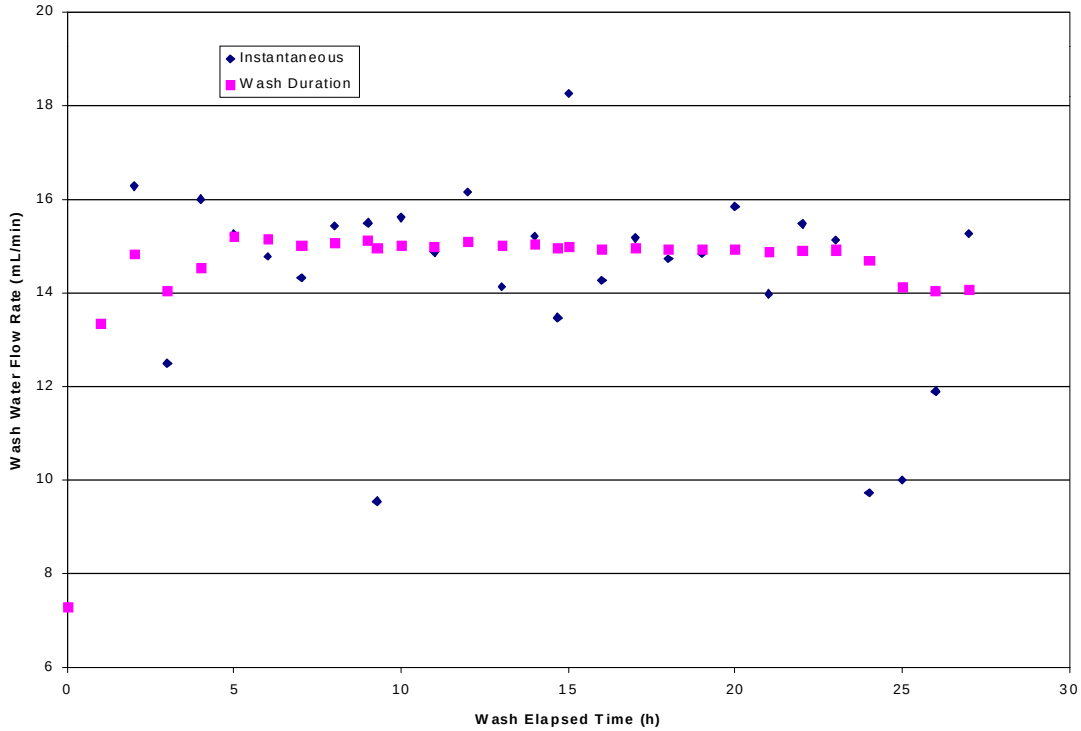


Fig. 60. Second slurry wash—wash water feed.

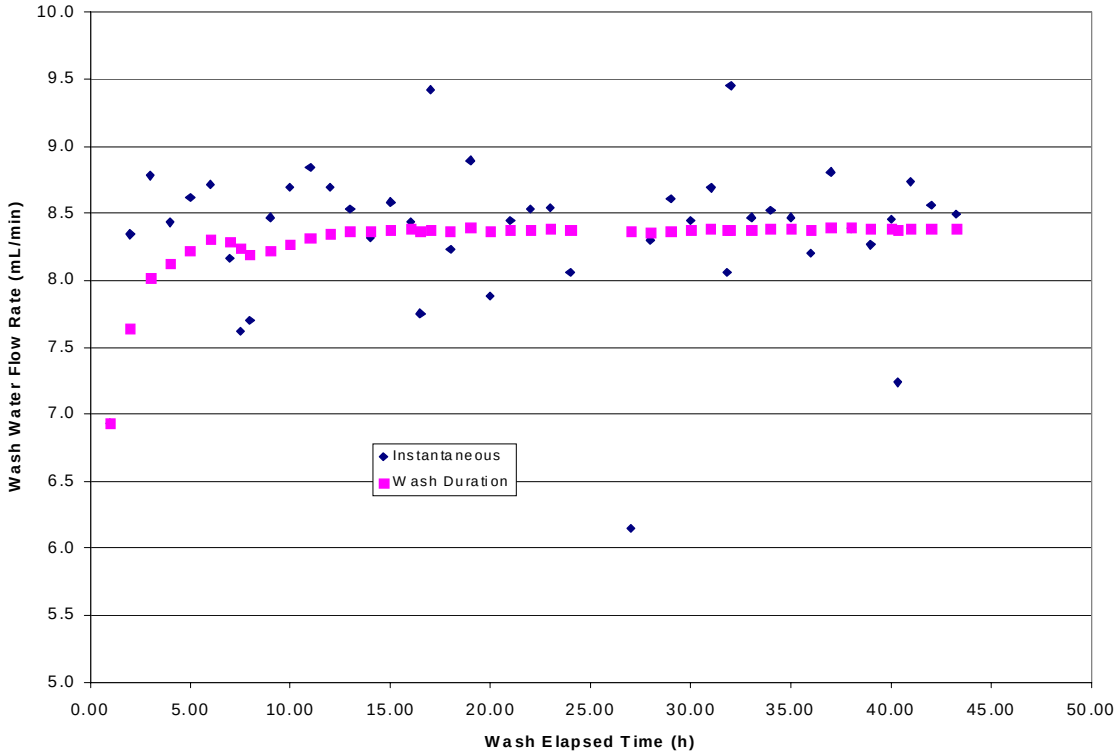


Fig. 61. Third slurry wash–-wash water feed.

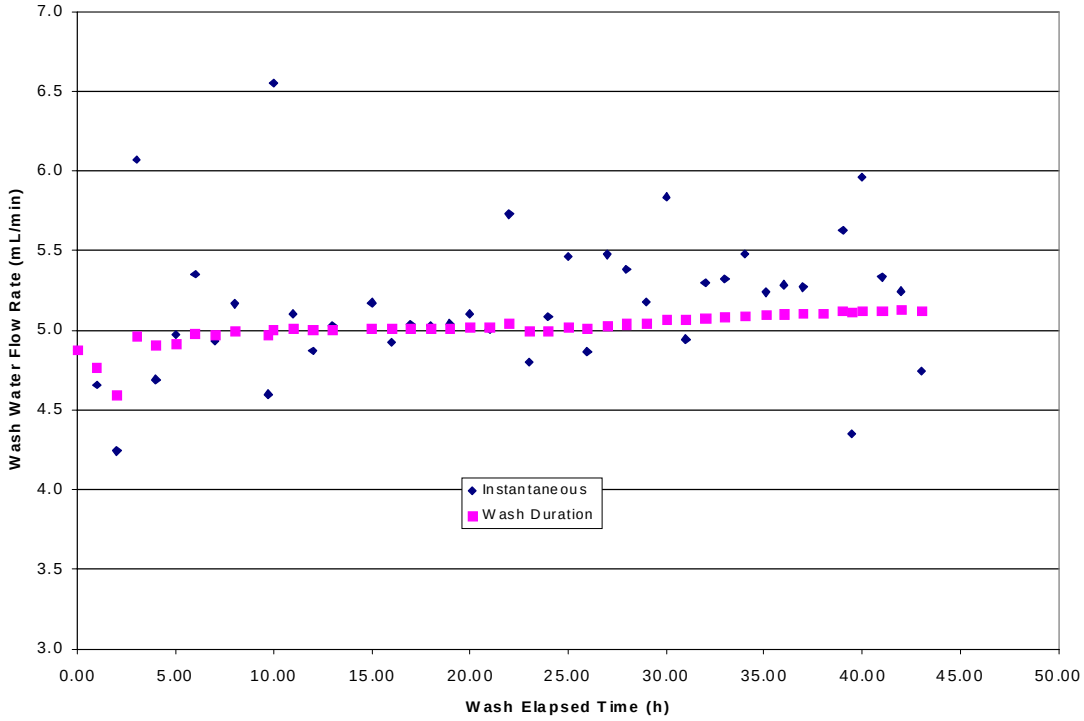


Fig. 62. Fourth slurry wash–-wash water feed.

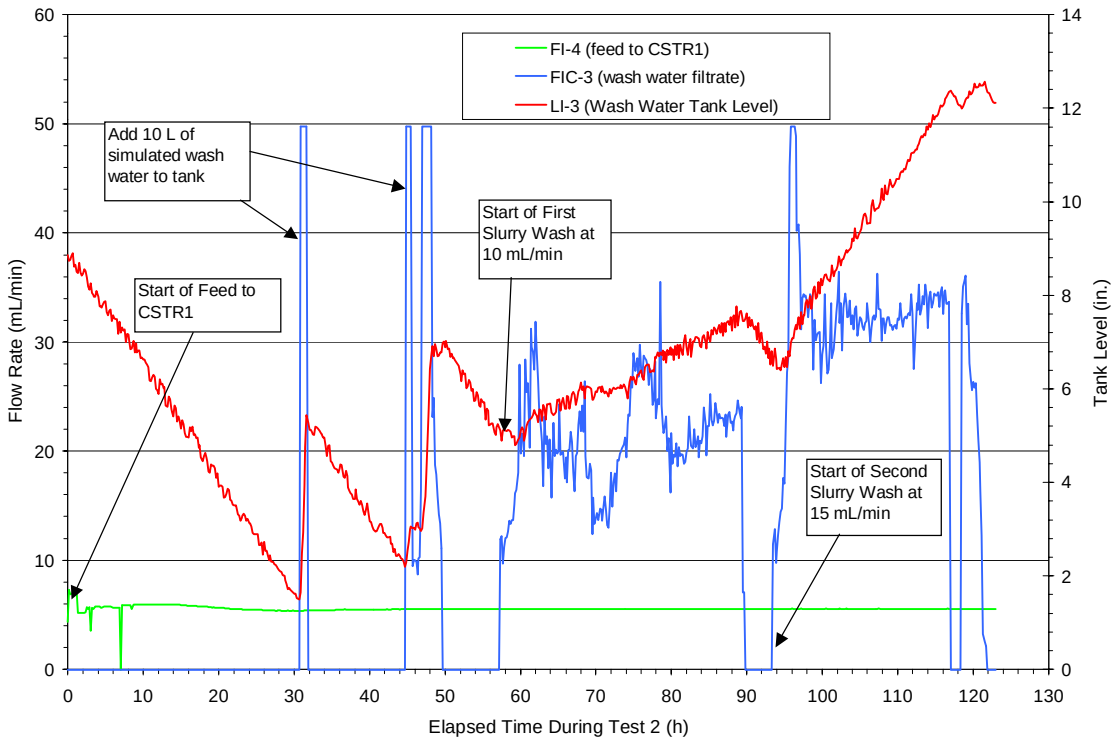


Fig. 63. Recycle wash water tank system through two slurry washes.

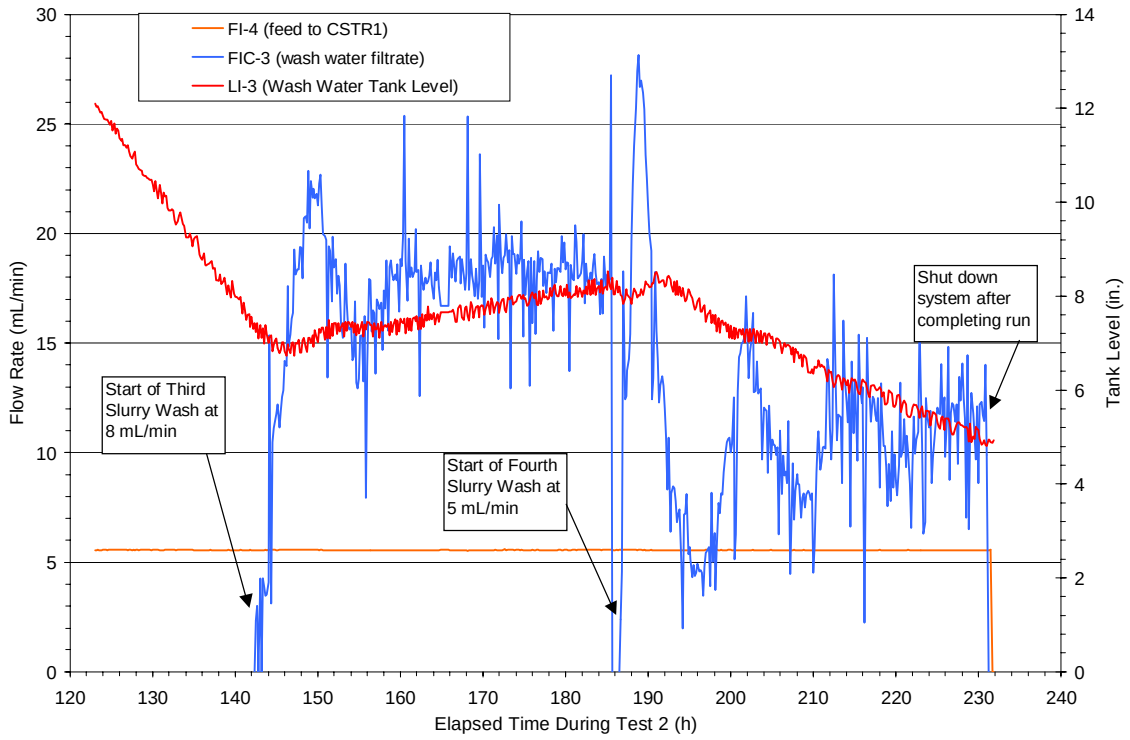


Fig. 64. Recycle wash water tank system–third and fourth slurry washes.

Table 29. Analytical data for filtrate samples for Test 2

Washing time (h)	Filtrate sample ID	Cs-137 (mCi/mL)	Sr-85 (mCi/mL)	K (mg/L)	Cu (mg/L)	Pd (mg/L)	B (mg/L)	NO_2 (mg/L)	U-235 (mg/L)	NaTPB (mg/L)	3PB (mg/L)	2PB (mg/L)	1PB (mg/L)	Phenol (mg/L)
10 mL/min wash														
4.90	T2SP7-01	2.51E-05	4.32E-04	<2.5	0.52	<1.28	56.8	9165	0.08					
6.90	T2SP7-02	3.24E-05	4.59E-04	<2.96	0.38	<1.28	55.6	8111	0.08					
8.90	T2SP7-03	4.32E-05	4.59E-04	<1.99	0.28	<1.28	55.5	5990	0.07					
10.90	T2SP7-04	1.59E-04	5.13E-04	5.77	0.31	<1.28	51.5	5253	0.05	1700	22.60	<16.5	<16.3	<16.7
12.90	T2SP7-05	1.57E-04	4.59E-04	<3.57	0.25	<1.28	47.5	3986	0.05					
14.90	T2SP7-06	1.08E-04	4.59E-04	<3.98	0.26	<1.28	48.1	3025	0.04					
16.90	T2SP7-07	2.40E-04	4.05E-04	<1.8	0.17	<1.28	44.2	2547	0.03					
18.90	T2SP7-08	1.57E-04	3.51E-04	<1.5	0.13	<1.28	35.9	2004	0.02	1230	<15.7	<16.5	<16.3	<16.7
20.90	T2SP7-09	3.78E-04	2.67E-04	<1.5	0.13	<1.28	34.6	1613	0.02					
22.90	T2SP7-10	4.32E-04	2.40E-04	<1.5	0.10	<1.28	31.7	1304	0.01					
24.90	T2SP7-11	3.51E-04	2.21E-04	<1.5	<0.08	<1.28	28.0	1093	0.01					
26.90	T2SP7-12	6.48E-04	1.65E-04	<1.5	0.10	<1.28	26.1	954	0.01	810	<15.7	<16.5	<16.3	<16.7
28.90	T2SP7-13	9.72E-04	1.70E-04	<1.5	0.23	<1.28	23.1	793	0.01					
30.90	T2SP7-14	1.30E-03	1.08E-04	<1.5	0.20	<1.28	21.8	677	0.01					
31.00	T2SP7-15	3.51E-04	2.32E-04	<3.1	0.18	<1.28	39.0		0.02	1330	<15.7	<16.5	<16.3	<16.7
31.00	T2SP5-01	1.92E+01	1.05E-01	1580.00	2.07	<1.28	526.0		0.48					
15 mL/min wash														
3.25	T2SP7-16	3.24E-05	9.99E-04	<5.19	0.62	<1.28	54.1	8448	0.17	1600	56.80	<16.5	<16.3	<16.7
5.25	T2SP7-17	3.51E-05	9.99E-04	<4.23	0.44	<1.28	61.6	7858	0.14					
7.25	T2SP7-18	4.05E-05	7.56E-04	<3.04	0.30	<1.28	68.5	5622	0.09					
9.25	T2SP7-19	1.05E-04	6.48E-04	<2.55	0.24	<1.28	68.0	4799	0.07					
11.25	T2SP7-20	1.97E-04	4.59E-04	<1.68	0.19	<1.28	65.6	3434	0.07	2320	24.90	<16.5	<16.3	<16.7
13.25	T2SP7-21	2.97E-04	3.78E-04	<1.5	0.13	<1.28	61.5	2435	0.04					
15.25	T2SP7-22	2.70E-04	2.97E-04	<1.5	0.13	<1.28	58.0	1968	0.03					
17.25	T2SP7-23	2.70E-04	2.65E-04	<1.5	0.10	<1.28	53.7	1434	0.02					
19.25	T2SP7-24	4.86E-04	2.05E-04	<1.5	0.12	<1.28	49.1	1123	0.02	1750	<15.7	<16.5	<16.3	<16.7
21.25	T2SP7-25	1.24E-04	1.73E-04	<1.5	0.14	<1.28	46.4	1105	0.01					
23.25	T2SP7-26	1.54E-04	1.22E-04	<1.5	0.11	<1.28	43.4	836	0.01					
27.75	T2SP7-27	1.27E-04	1.19E-04	<1.5	0.12	<1.28	42.2	562	0.01					
27.75	T2SP8-02	1.05E-03	3.24E-04	<2.77	0.17	<1.28	49.3	3002	0.05	1580	17.00	<16.5	16.00	<17
27.75	T2SP5-02	1.19E+00	5.94E-02	61.20	0.57	<1.28	34.9		0.36	1100	<15.7	<16.5	<16	<17

Table 29 (continued)

Washing time (h)	Filtrate sample ID	Cs-137 (mCi/mL)	Sr-85 (mCi/mL)	K (mg/L)	Cu (mg/L)	Pd (mg/L)	B (mg/L)	NO_2 (mg/L)	U-235 (mg/L)	NaTPB (mg/L)	3PB (mg/L)	2PB (mg/L)	1PB (mg/L)	Phenol (mg/L)
						8 mL/min wash								
1.90	T2SP7-28	1.35E-04	1.97E-04	<1.5	0.10	<1.28	42.6	2230	0.02	1630	<15.7	<16.5	<16.3	<16.7
3.90	T2SP7-29	2.70E-05	7.02E-04	<2.49	0.42	<1.28	82.0	9033	0.12					
5.90	T2SP7-30	4.05E-05	7.83E-04	<2.83	0.51	<1.28	71.2	11030	0.18					
7.90	T2SP7-31	4.86E-05	7.02E-04	<2.78	0.52	<1.28	69.3	9546	0.15					
9.90	T2SP7-32	4.86E-05	6.75E-04	<1.59	0.21	<1.28	88.0	7751	0.12	3150	54.90	<16.5	18.10	16.70
13.90	T2SP7-33	1.03E-04	5.40E-04	<1.51	0.15	<1.28	97.2	6083	0.09					
17.90	T2SP7-34	1.40E-04	4.32E-04	<1.5	<.08	<1.28	93.6	4402	0.06					
21.90	T2SP7-35	7.56E-05	3.24E-04	<3.25	<.08	<1.28	93.1	3622	0.04					
25.90	T2SP7-36	8.91E-05	2.97E-04	<2.05	<.07	<1.28	92.7	2470	0.04	3010	23.90	<16.5	<16	17.00
29.90	T2SP7-37	1.03E-04	1.86E-04	<1.91	0.07	<1.28	90.2	1678	0.02					
33.90	T2SP7-38	1.70E-04	1.81E-04	<1.53	<.06	<1.28	82.6	1278	0.02					
37.90	T2SP7-39	2.13E-04	1.24E-04	<1.5	<.06	<1.28	78.1	941	0.01					
41.90	T2SP7-40	3.51E-04	9.45E-05	<1.5	0.10	<1.28	74.6	854	0.01	2220	<16.0	<16.5	<16.0	<17.0
42.90	T2SP7-41	4.05E-04	9.18E-05	<1.5	<0.08	<1.28	72.5	854	0.01					
42.90	T2SP8-03	5.40E-04	2.97E-04	<3.07	0.14	<1.28	78.9		0.04	2460	24.50	<16.5	<16.3	21.00
42.90	T2SP5-03	6.48E+01	3.78E-01	5780.00	7.42	<2.65	2230.0		1.70	2280	<15.7	<16.5	<16.0	<17
						5 mL/min wash								
4.00	T2SP7-42	2.62E-04	1.27E-03	<6.9	1.05	<1.28	55.4	12400	0.24					
6.00	T2SP7-43	3.78E-05	1.19E-03	<5.79	0.92	<1.28	60.2	12046	0.24					
10.00	T2SP7-44	4.05E-05	1.08E-03	<5.6	0.77	<1.28	75.2	9880	0.21	2260	86.70	<16.5	48.70	74.00
14.00	T2SP7-45	1.03E-04	8.91E-04	4.18	0.51	<1.28	99.6	8070	0.13					
18.00	T2SP7-46	2.70E-04	7.56E-04	<3.37	0.29	<1.28	128.0	6457	0.12					
22.00	T2SP7-47	1.13E-04	6.75E-04	3.04	0.22	<1.28	137.0	5231	0.09					
26.00	T2SP7-48	4.59E-05	5.40E-04	<1.5	0.13	<1.28	143.0	4290	0.08	4710	56.70	<16.5	27.00	42.30
30.00	T2SP7-49	6.48E-05	5.40E-04	<1.5	<.06	<1.28	144.0	3697	0.06					
34.00	T2SP7-50	6.48E-05	4.05E-04	<1.5	<.06	<1.28	149.0	3253	0.05					
38.00	T2SP7-51	8.91E-05	3.51E-04	<1.5	<.06	<1.28	143.0	2701	0.04					
42.00	T2SP7-52	1.05E-04	2.97E-04	<1.5	<.06	<1.28	140.0	2006	0.04	4880	37.70	<16.5	<16.3	18.60
44.00	T2SP7-final	1.81E-04	2.70E-04	<2.94	<.06	<1.28	148.0	2497	0.04	4880	35.00	21.10	<16.3	17.40
44.00	T2SP8-Final	2.97E-04	4.32E-04	<1.5	<.06	<1.28	103.0		0.08	3580	41.50	<16.5	21.30	32.10
44.00	T2SP6-Final	7.83E+01	4.32E-01	607.00	8.66	<2.56	252.0		2.74	5230	36.50	<16.5	<16.3	<16.7

8.7 MATERIAL BALANCES

The potassium concentration profile during Test 2 for CSTR 1 is shown in Fig. 65, giving both the calculated concentration of potassium, which assumed no KTPB precipitation and the actual concentration of potassium that actually occurred, as measured by the sampling. The graph shows the potassium concentrations in CSTRs 1 and 2, and the filtrate concentrations during the test. Figure 66 shows the sodium concentration in Test 2 and in Test 1a for comparison.

The NaTPB excess during the test varied with the amount of TPB in the recycle wash water. The initial NaTPB in the recycle wash water was produced during the wash of the Test 1a slurry. The simulated recycle wash water used to supply extra solution while waiting to wash more slurry was made up with the concentration of TPB measured in the initial recycle wash water from Test 1a. Following the initial period, the recycle wash water contained different amounts of excess TPB, depending on the efficiency of the water wash of the slurry. The values are as follows:

Test 1A	6386 + 352 = 6738 mg NaTPB/L slurry (73.2% excess)
Test 2, Batch 1	6386 + 289 = 6675 mg NaTPB/L slurry (71.5% excess)
Test 2, Batch 2	6386 + 343 = 6729 mg NaTPB/L slurry (72.9% excess)
Test 2, Batch 3	6386 + 535 = 6921 mg NaTPB/L slurry (77.9% excess)
Test 2, Batch 4	6386 + 778 = 7164 mg NaTPB/L slurry (84.1% excess)
K^+ (in slurry)	*0.01137 mol/L*

Stoichiometric amount: (0.01137 mol/L)(342.23 g NaTPB/mol) = 3.891 g NaTPB/L

During the test, the concentrating of the TPB slurry in each batch was continued until it was estimated that the solids content had reached about 8–10 wt %. Each estimate was made by calculating the amount of slurry that would be formed in the system if all the potassium and the excess TPB were precipitated. There was no TPB in the CSTRs at the beginning of the test, and by knowing the concentrations of TPB in the feed streams to CSTR 1, it was rather easy to track the TPB. The curved plots are caused by the initial TPB buildup during startup (shown in Fig. 67). The plots are straight in Fig. 68 because the TPB is already present. About four to five residence times were required to reach the equilibrium TPB concentration. The accumulation of TPB solids could then be estimated with time and with the volume of circulating slurry in the

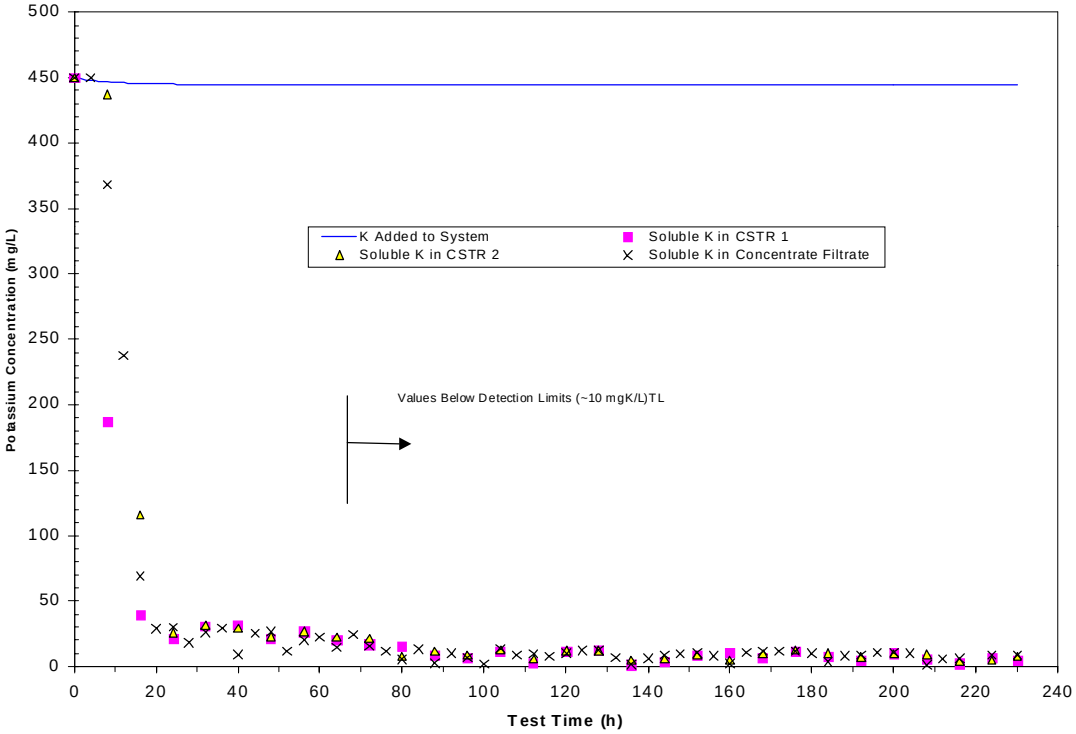


Fig. 65. Potassium concentration vs test time.

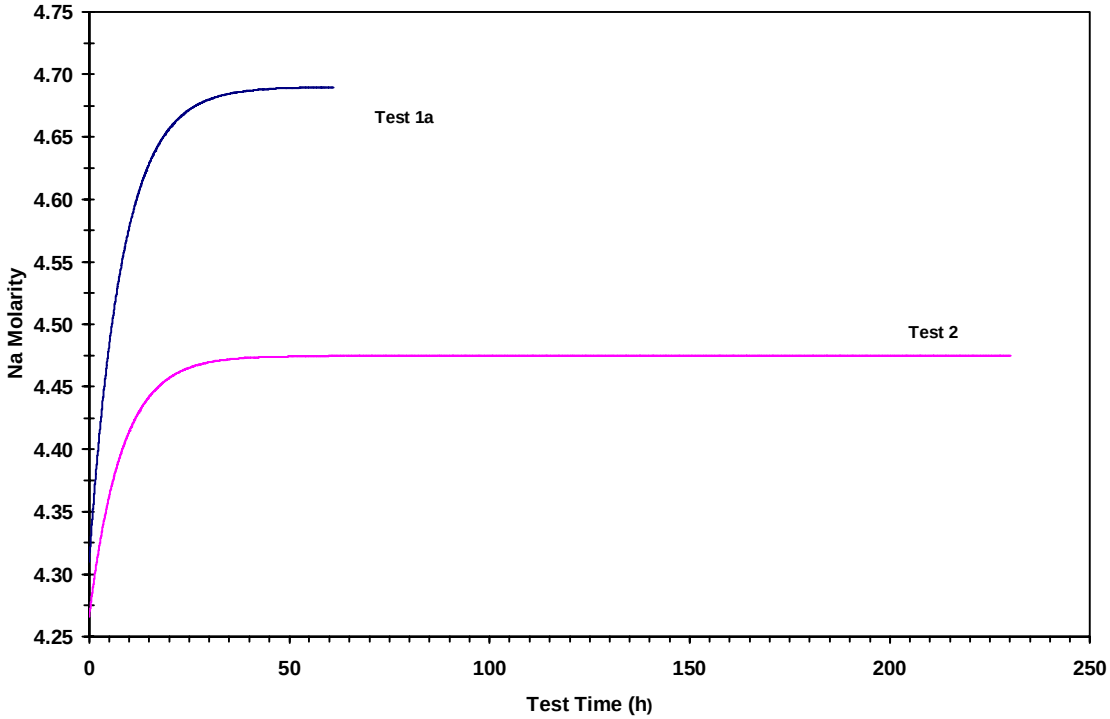


Fig. 66. Sodium concentration vs test time.

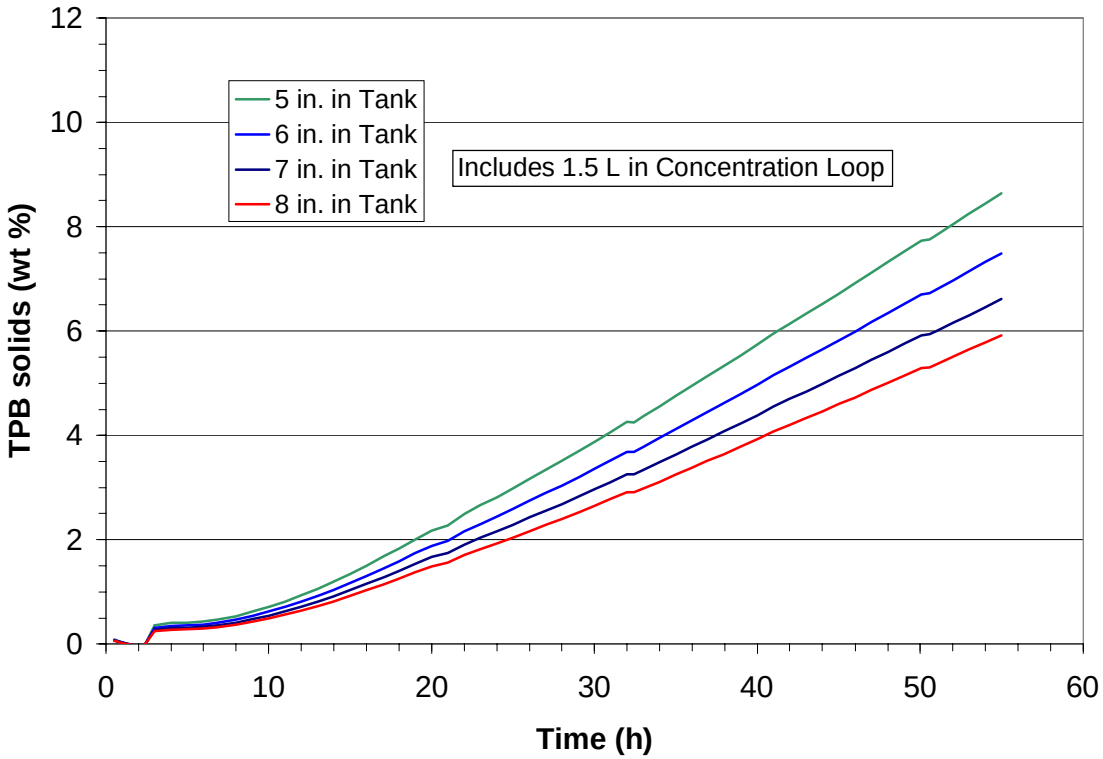


Fig. 67. Weight percent TPB solids vs time—concentration loop, first concentration.

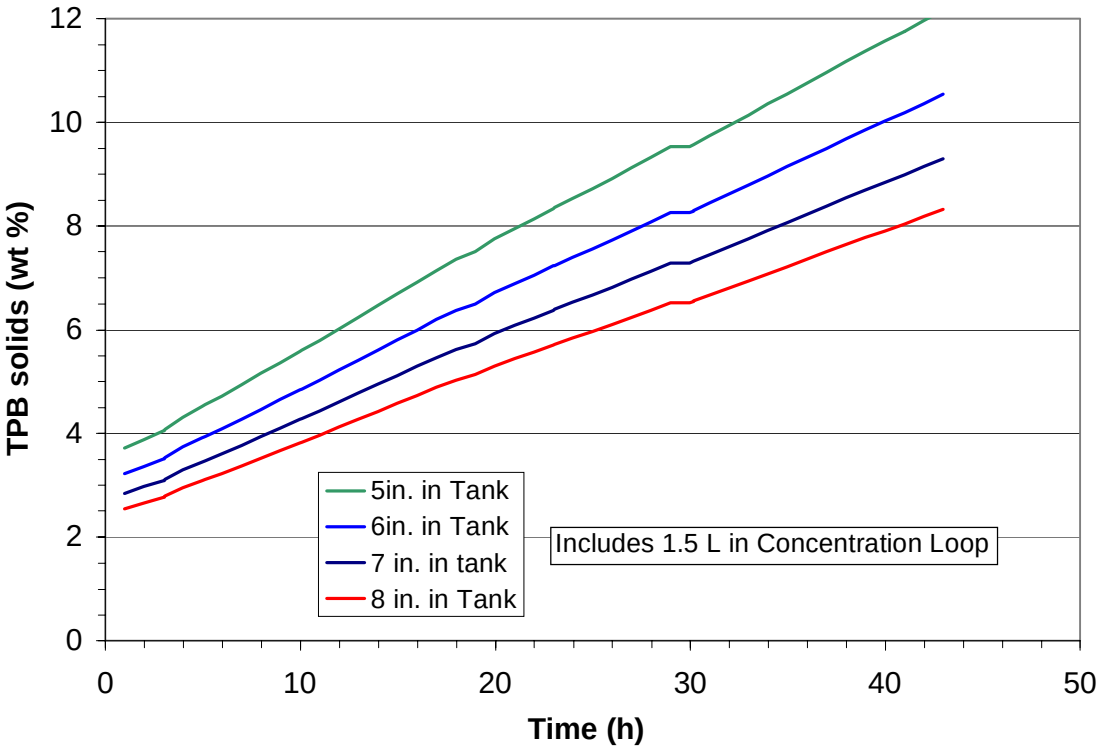


Fig. 68. Weight percent TPB solids vs time—concentration loop, second concentration.

slurry concentration loop. Plots of the expected accumulation are shown in Figs. 67 and 68 for several different levels (slurry volumes) in the concentration tank. Once the solids concentration reached the desired level, most of the slurry was transferred to the slurry wash tank. This left about 2 in. (equal to 1.6 L) of slurry in the concentration tank. Another 1.5 L remained in the circulation loop, so the subsequent batches started at about 2.5–3 % solids, as indicated in Fig. 68.

The total material balance on the CSTR system is shown in Figs. 69 and 70. These plots show the hourly feed rates and concentrate filtrate rates (Fig. 69) and the integrated rates over the whole run. Variations in the filtrate rate, which resulted in the points on the graph looking like random shotgun holes, were attributed to the changes in flow rate caused by the lack of level control in the concentrate tank and the filter pressure drop problems in the filter loop. The many backpulse attempts resulted in some high flow rates following a backpulse and lower rates near the end of the period before the next backpulse was required. Since the system was maintained in mass balance, the wide fluctuations were tolerable. The integrated rates looked much better and resulted in final rates that very close to what we had desired for the run. The filtrate plot in Fig. 70 shows the dips where slurry was transferred. The filtrate flow was off for the time required to refill the slurry concentration tank.

Figure 69 also shows the variations in the total feed rate caused by the variations in recycle wash water, salt feed, and MST/dilution water feed rates. Some of the variations were caused when feed pumps were adjusted to give rates closer to the planned rates, and others by variations in the times the balance readings were recorded. The feed rate for the salt feed was estimated based on a grab sample taken at least once per shift, in which a graduated cylinder was used to collect a 3-min sample. The flow rate was calculated based on the volume of feed collected in the 3 min. A similar flow check was completed on the recycle wash water feed to CSTR 1. The flow rates for the feeds (NaTPB, MST/dilution water) that were plumbed from outside the hot cell were checked at least once per shift by taking a timed difference in the mass readings on the balance. The overall flow balance was very good.

At the end of the test, about 63 L of salt feed was removed from the feed tanks in Hot Cell A. Considering that 384 L of salt feed was prepared for this test and that 23 L of salt feed was pumped to the CSTRs and concentration tank before the test was initiated (when TPB addition started), a volume of 298 L of salt feed was actually used in the 230-h test. This provided an average flow rate of 21.6 mL/min. This value was very close to the operational flow rate goal of 21.5 mL/min.

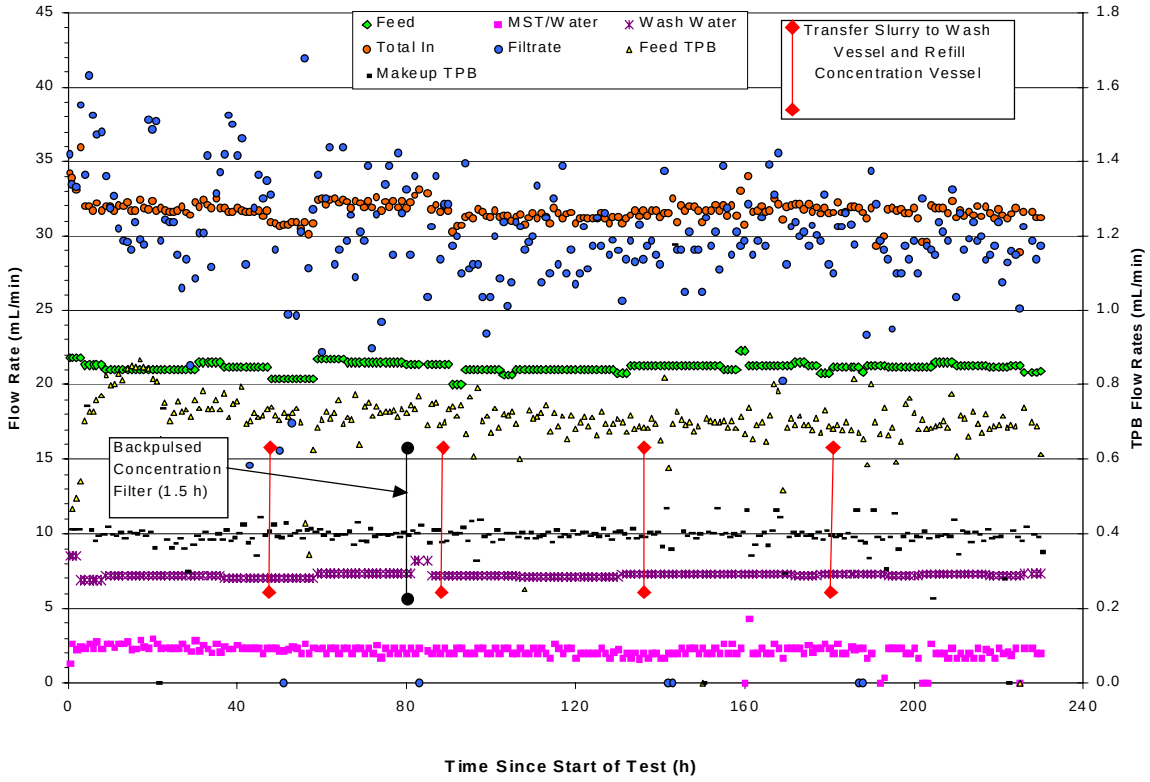


Fig. 69. Hourly flow balances, Test 2.

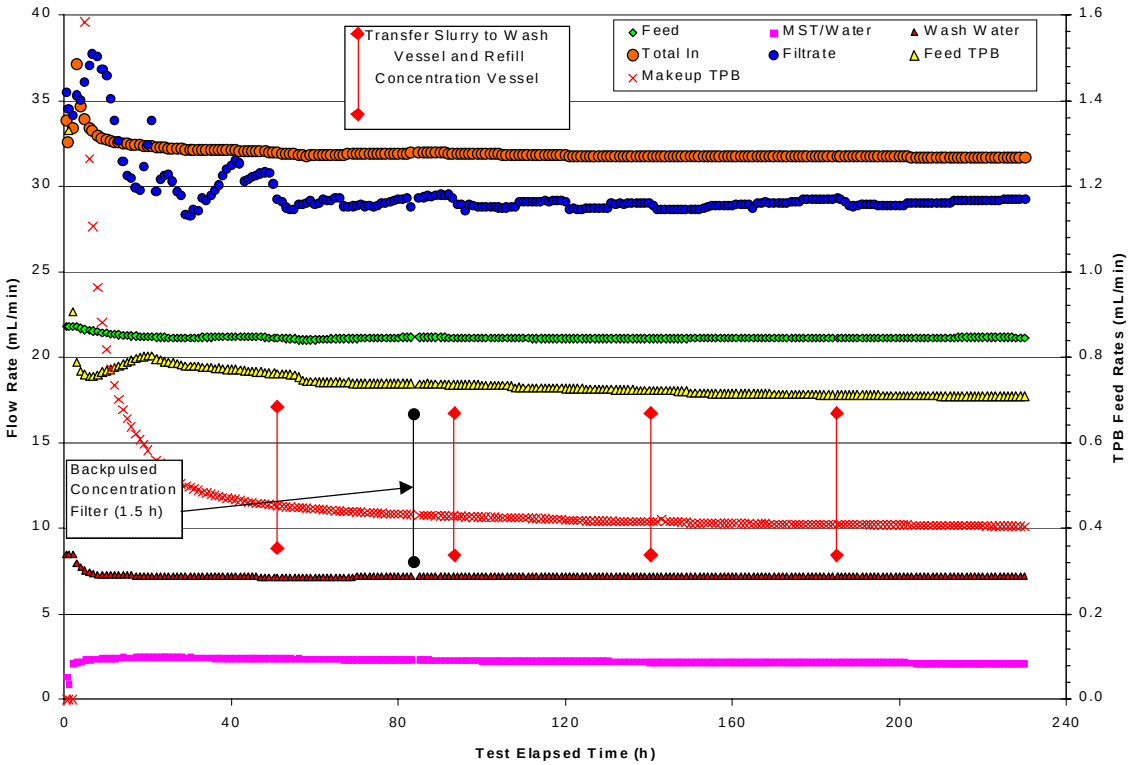


Fig. 70. Test average flow balances, Test 2.

8.8 ANALYTICAL RESULTS

The samples that were taken from the CSTRs and the concentrate filtrate were submitted to RMAL for the many analyses required. The results of these analyses were used to determine whether the many aspects of Test 2 were successful. The analytical results are displayed in Figs. 71–76 and Tables 30–32. Figure 71 shows a plot of the soluble NaTPB content in the effluent from CSTR 1 and CSTR 2 and in the concentrate filtrate. The analysis error bars (generally ± 20% for the HPLC analysis) are shown with the data points. The plot also contains the cesium concentrations so that changes in cesium content can be correlated with changes in TPB content. The quantities of soluble TPB in CSTR 1 and CSTR 2 filtrate are at similar levels at most sample times. As expected, TPB appears in the CSTR 1 filtrate a few hours before it appears in CSTR 2. Levels of TPB in the filtrate from the concentration loop are generally about 200 mg/L, compared with about 75–100 mg/L for TPB in CSTR 1 and CSTR 2 filtrate.

The analyses plots for the 3PB decomposition product are shown in Fig. 72 for the three sample points, along with the analysis error bars. The initial concentration of 3PB in the feed was about 110 mg/L, and the filtrate concentration gradual stabilized at 100 ppm. The amount of 3PB in the CSTRs varied around 50 mg/L, from 20 mg/L up to 80 mg/L. There was no apparent trend in the amount of 3PB in either CSTR 1 or CSTR 2. It did not appear to be increasing during the test, nor did it disappear from the system during operation. The amount of 3PB in the salt feed was unchanged from the start, within the experimental error.

The filtrate analysis for 2PB in solution at the three sample points is shown in Fig. 73. The initial amount of 2PB added to the feed was 110 mg/L. The three sample points have comparable amounts of 2PB until it disappears from the analyses after about 130 h of operation. During the period from 20 to 120 h, the amount of 2PB in solution appears to decrease steadily and, during the last 80 h of operation, was below detection limits. The copper added to the feed as part of the ECC catalyst system is known to catalyze the decomposition of 2PB. The copper-catalyzed decomposition of 2PB is apparently the fastest of the TPB decomposition reactions. A sample of the feed at 150 h into the test showed that the 2PB had dropped to about 44 mg/L after starting at 110 mg/L at the time of preparation. At the end of the 230-h test, another feed sample showed that the 2PB had dropped to below the detection limit of about 16 mg/L.

The concentration of 1PB in the system is shown in Fig. 74. The values for all three sample ports are shown and stay reasonably close to the value of 110 mg/L present in the diluted

salt feed. It appears that there is an increase in the 1PB concentration at all sample points at the same time that the 2PB concentration is decreasing, and while this is possible, the results are not definitive. The concentration remains fairly stable after the 2PB has disappeared at about 80 mg/L for the remainder of the test.

The product of each step in the breakdown of TPB is benzene or phenol depending on the oxidation potential of the solution. The general yield is about 90% benzene/10% phenol with trace amounts of biphenyl and terphenyl. Increases in oxygen favor a higher phenol yield but under most conditions, benzene is favored. The plot of the phenol concentration in the filtered samples at each sample port is shown in Fig. 75. Phenol was added to the salt feed at a diluted feed concentration of about 110 mg/L. Over the course of the test, the concentration of phenol in the samples remained near the salt feed levels, indicating that there was no large input to the phenol concentration from TPB or its decomposition products. In addition, since 3PB did not increase, any TPB decomposition was minor at most.

The final figure in this series (Fig. 76) plots the decomposition products in the concentration filtrate. If there were patterns in the TPB breakdown, they might be evident here. Other than the disappearance of the 2PB, the other components remain near the levels in the salt feed. When TPB is present in the filtrate, its concentration is fairly constant between 200 and 300 mg/L. Because higher temperatures can increase the rate of TPB decomposition, the operating temperature of the concentration vessel is also plotted in the graph. Since the temperature stayed between 20 and 25ºC for most of the test, no discernable effects were expected.

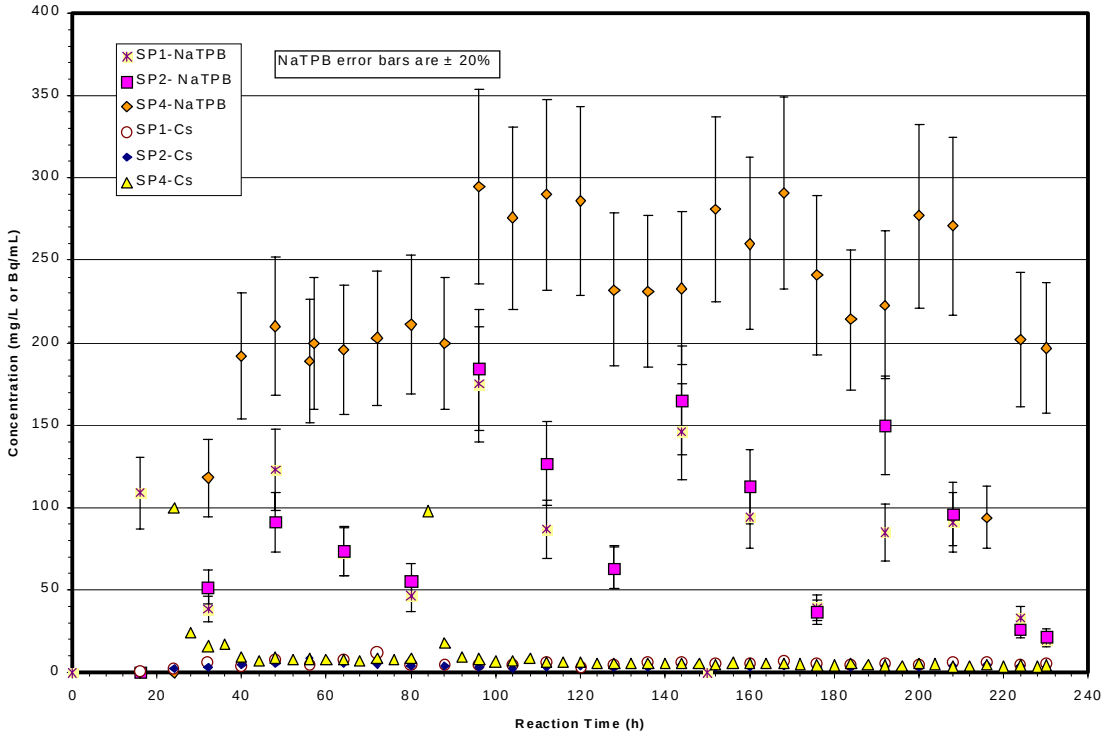


Fig. 71. Comparison of NaTPB from CSTR 1, CSTR 2, and filtrate in Test 2.

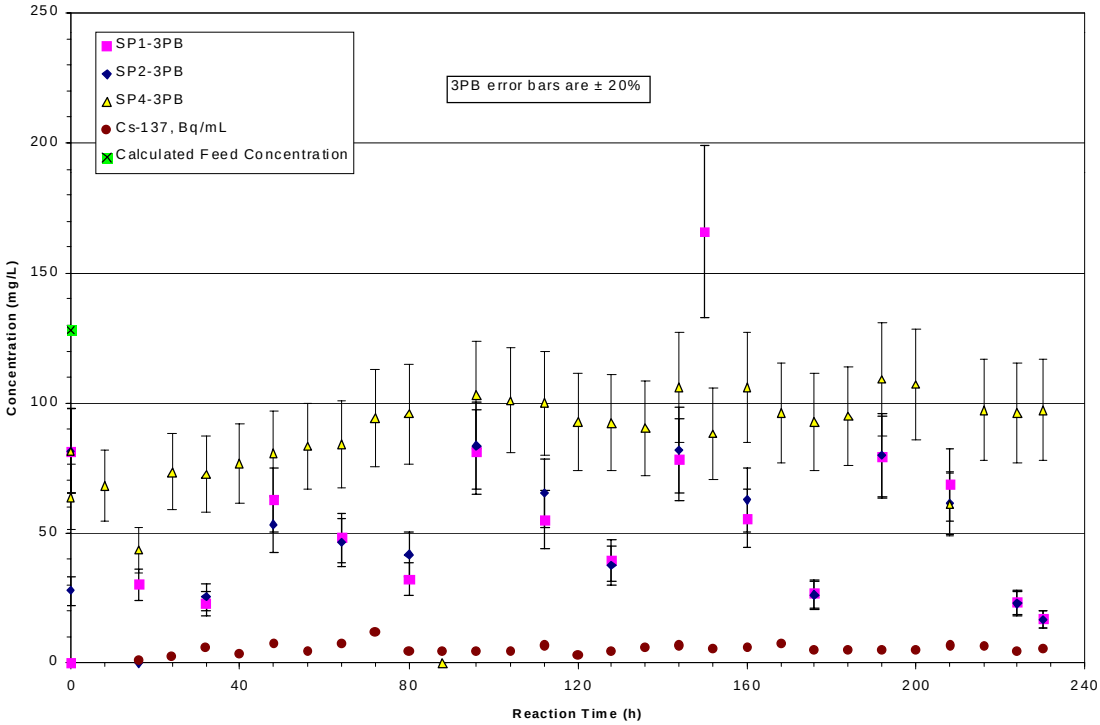


Fig. 72. Concentrations of 3PB in the Test 2 CSTR system.

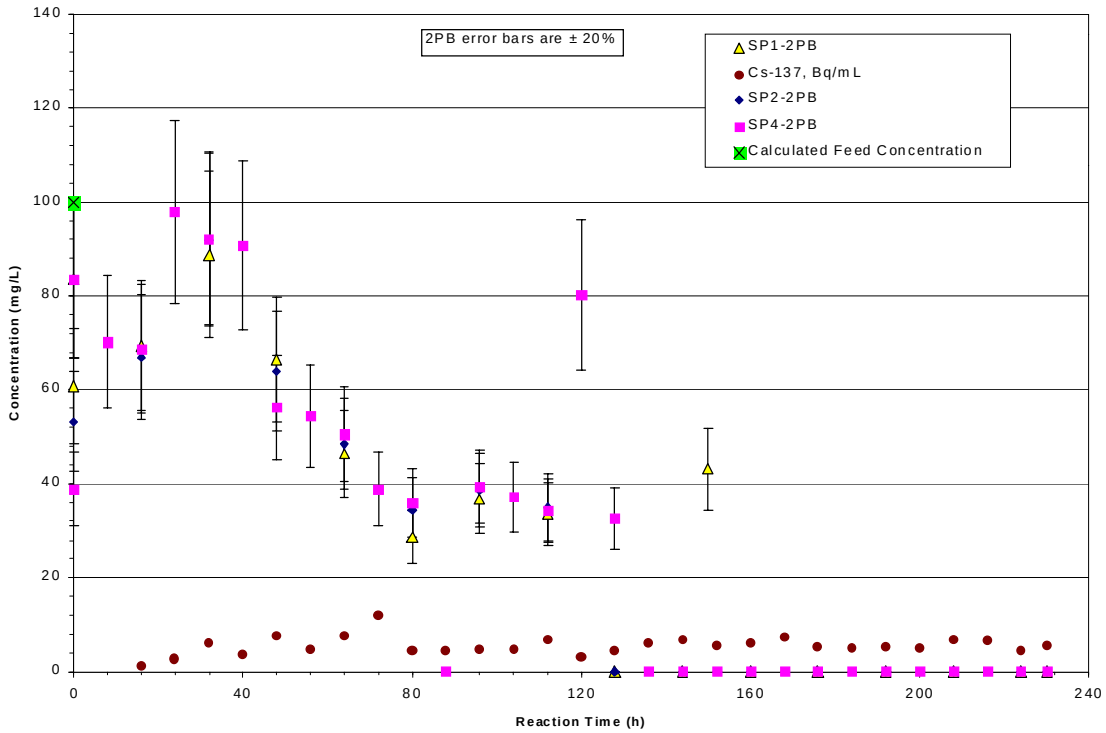


Fig. 73. Concentration of 2PB in the Test 2 CSTR system.

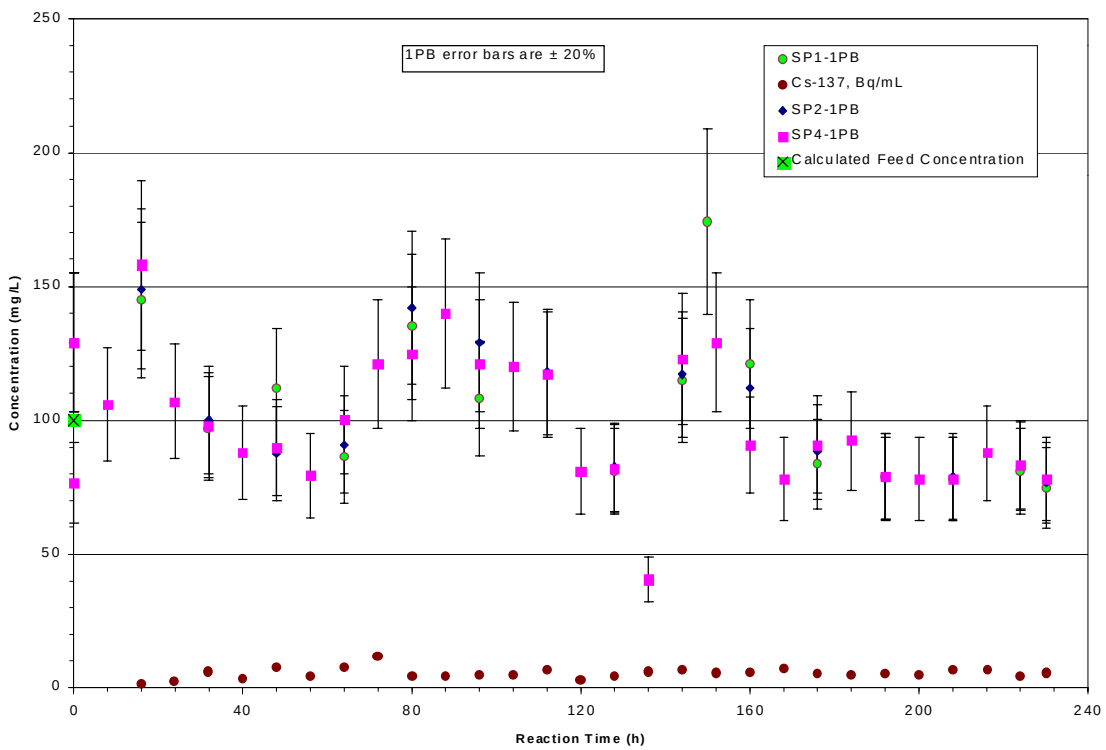


Fig. 74. Concentration of 1PB in the Test 2 CSTR system.

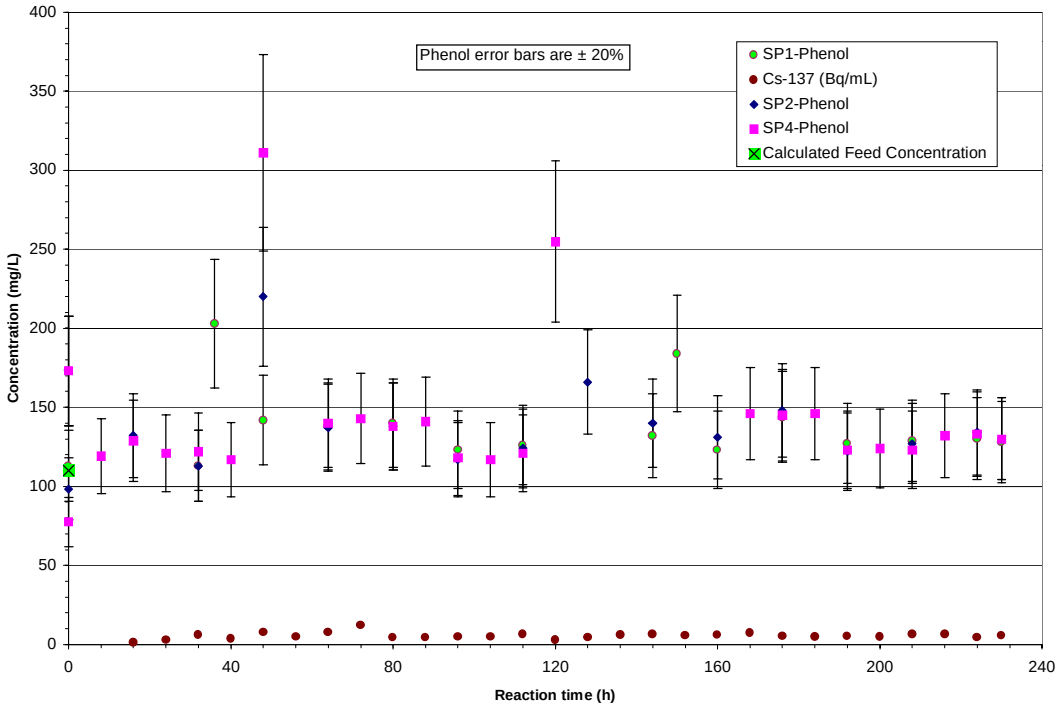


Fig. 75. Concentrations of phenol in the Test 2 CSTR system.

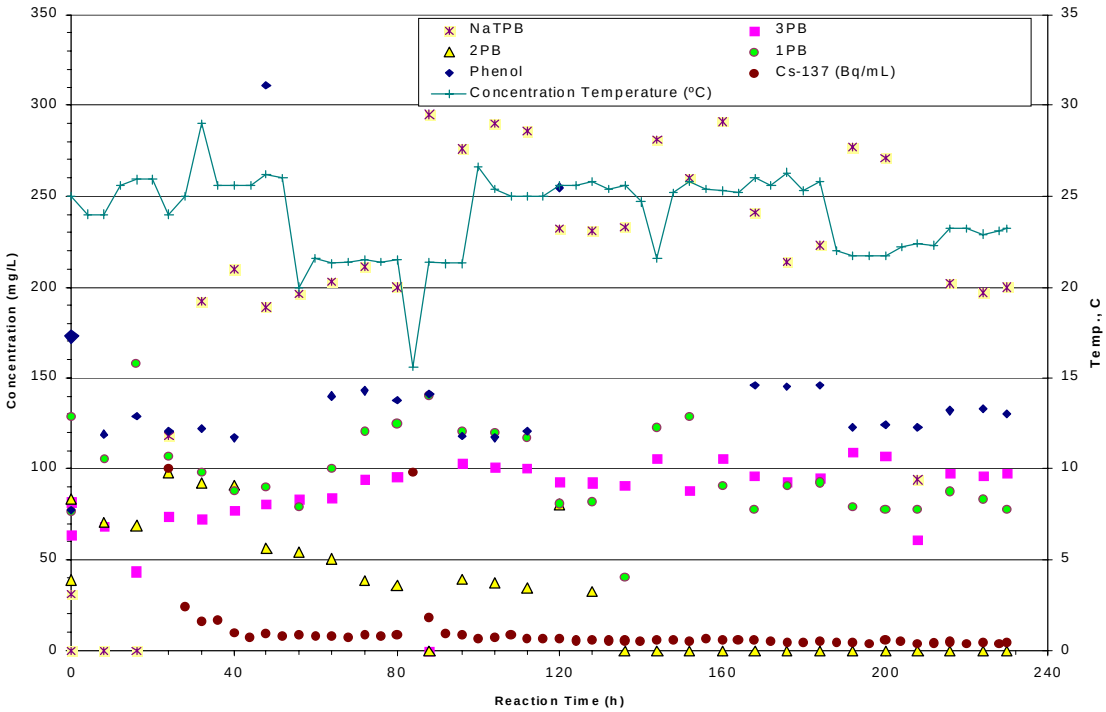


Fig. 76. Decomposition of products from concentration filtrate, Test 2.

Table 30. Analytical data for CSTR 1 samples from Test 2

Reaction time (h)	CSTR 1 sample ID	Cs-137 (µCi/g)	Sr-85 (µCi/g)	K (mg/L)	Cu (mg/L)	Pd (mg/L)	B (mg/L)	U-235 (mg/L)	NaTPB (mg/L)	3PB (mg/L)	2PB (mg/L)	1PB (mg/L)	Phenol (mg/L)
0	T2SP1-Feed	4.30	4.76E-02	2137.45	4.69	13.86	22.3	0.90		56.2	57.6	88.9	119.3
0	T2SP1-00	4.01	5.79E-03	640.00	2.40	<1.85	15.9	0.54	<16.3	<15.7	60.8	0.0	113.0
8	T2SP1-01	2.09E-02	1.47E-03	187.00	2.25	<1.28	14.8	0.49					
16	T2SP1-02	2.90E-05	1.34E-03	40.30	2.15	<1.28	19.5	0.50	109.0	30.4	69.4	145.0	
24	T2SP1-03	6.01E-05	1.34E-03	21.50	2.12	<1.28	20.0	0.52					
32	T2SP1-04	1.36E-04	1.36E-03	30.80	2.11	<1.28	17.3	0.43	38.5	22.8	88.8	97.0	113.0
40	T2SP1-05	8.02E-05	1.31E-03	31.10	2.25	<1.28	20.5	0.46					
48	T2SP1-06	1.69E-04	1.34E-03	21.00	2.25	<1.28	23.0	0.54	123.0	62.7	66.5	112.0	142.0
56	T2SP1-07	1.02E-04	1.25E-03	26.70	2.23	<1.28	20.2	0.50					
64	T2SP1-08	1.72E-04	1.27E-03	20.80	2.37	<1.28	21.6	0.54	72.9	48.1	46.3	86.5	138.0
72	T2SP1-09	2.67E-04	1.45E-03	<17	2.37	<1.28	22.1	0.51					
80	T2SP1-10	9.58E-05	1.38E-03	<1.5	2.16	1.50	18.5	0.49	46.7	32.2	28.7	135.0	140.0
88	T2SP1-11	1.00E-04	1.25E-03	<8.57	2.17	<1.28	22.4	0.43					
96	T2SP1-12	1.05E-04	1.23E-03	<6.67	2.40	<1.28	22.8	0.45	175.0	81.3	36.8	108.0	123.0
104	T2SP1-13	1.05E-04	1.31E-03	<12	2.34	<1.28	18.6	0.46					
112	T2SP1-14	1.49E-04	1.27E-03	<3.06	2.15	<2.42	20.0	0.42	86.9	55.3	33.5	0.0	126.0
120	T2SP1-15	6.68E-05	1.27E-03	<11.7	2.36	<1.28	22.3	0.45					
128	T2SP1-16	1.00E-04	1.29E-03	<12.5	2.49	<1.28	19.6	0.51	63.8	39.3	<16.5	80.8	0.0
136	T2SP1-17	1.36E-04	1.25E-03	<1.5	2.47	2.59	21.4	0.58					
144	T2SP1-18	1.49E-04	1.34E-03	<3.98	2.40	<1.28	22.3	0.53	146.0	78.2	<16.5	115.0	132.0
152	T2SP1-19	1.25E-04	1.25E-03	<8.56	2.31	<1.28	19.8	0.54					
160	T2SP1-20	1.31E-04	1.25E-03	<10.6	2.34	<1.28	19.8	0.52	94.3	55.5	<16.5	121.0	123.0
168	T2SP1-21	1.65E-04	1.43E-03	<6.6	2.34	<1.28	21.8	0.61					
176	T2SP1-22	1.16E-04	1.25E-03	<11.6	2.47	<1.28	18.8	0.56	39.5	26.7	<16.5	83.5	144.0
184	T2SP1-23	1.09E-04	1.23E-03	<7.67	2.40	<1.28	21.3	0.48					
192	T2SP1-24	1.16E-04	1.54E-03	<5.28	2.35	<1.28	22.2	0.52	85.0	79.4	<16.5	79.0	127.0
200	T2SP1-25	1.09E-04	1.29E-03	<10.1	2.46	<1.28	18.7	0.54					
208	T2SP1-26	1.49E-04	1.45E-03	<6.06	2.45	<1.28	21.7	0.57	91.1	68.4	<16.5	78.0	129.0
216	T2SP1-27	1.47E-04	1.47E-03	<2.49	2.37	<1.28	22.0	0.55					
224	T2SP1-28	9.58E-05	1.31E-03	<6.88	2.41	<1.28	18.8	0.48	33.4	23.4	<16.5	81.0	130.0
230	T2SP1-FINAL	1.25E-04	1.36E-03	<4.46	2.14	<1.28	18.4	0.58	20.0	17.0	<16.5	74.8	128.0

Table 31. Analytical data for CSTR 2 samples from Test 2

Reaction time (h)	CSTR 2 sample ID	Cs-137 (µCi/g)	Sr-85 (µCi/g)	K (mg/L)	Cu (mg/L)	Pd (mg/L)	B (mg/L)	U-235 (mg/L)	NaTPB (mg/L)	3PB (mg/L)	2PB (mg/L)	1PB (mg/L)	Phenol (mg/L)
0	T2SP2-Feed	4.30	4.76E-02	458.52	4.69	13.86	22.27	0.90		56.19	57.64	88.95	119.28
0	T2SP2-00	3.12	<270	580.00	1.93	<1.28	15	0.38	<16.3	28	53	0	98
8	T2SP2-01	1.18E-01	1.65E-03	437.00	1.98	<1.28	14	0.42					
16	T2SP2-02	1.76E-02	1.69E-03	116.00	2.08	<1.28	14	0.44	<16.3	<15.7	67	149	132
24	T2SP2-03	5.79E-05	1.51E-03	25.70	2.02	<1.28	18						
32	T2SP2-04	7.57E-05	1.43E-03	31.60	2.15	<1.28	18	0.48	52	25	92	100	113
40	T2SP2-05	1.05E-04	1.25E-03	29.90	2.14	<1.28	20						
48	T2SP2-06	1.29E-04	1.38E-03	23.90	2.16	<1.28	22	0.53	91	53	64	88	220
56	T2SP2-07	1.94E-04	1.36E-03	26.80	2.31	<1.28	18	0.52					
64	T2SP2-08	1.29E-04	1.43E-03	22.60	2.37	<1.28	21	0.48	74	47	49	91	137
72	T2SP2-09	1.23E-04	1.36E-03	<21.5	2.30	<1.49	22	0.47					
80	T2SP2-10	9.80E-05	1.31E-03	<7.99	2.26	<1.28	19	0.41	55	42	34	142	138
88	T2SP2-11	9.36E-05	1.49E-03	<12.1	2.10	<1.28	20	0.47					
96	T2SP2-12	8.47E-05	1.36E-03	<8.99	2.39	<1.28	23	0.47	184	84	39	129	117
104	T2SP2-13	6.24E-05	1.40E-03	<12.6	2.32	<1.28	18	0.38					
112	T2SP2-14	7.13E-05	1.27E-03	<5.99	2.34	<1.28	22	0.46	127	65	35	118	124
120	T2SP2-15	8.91E-05	1.29E-03	<12.5	2.39	<1.28	22	0.56					
128	T2SP2-16	7.57E-05	1.31E-03	<12.5	2.44	<1.28	20	0.55	63	38	<16.5	83	166
136	T2SP2-17	7.80E-05	1.36E-03	<4.77	2.43	1.54	22	0.54					
144	T2SP2-18	1.11E-04	1.40E-03	<5.83	2.38	<1.28	22	0.54	165	82	<16.5	117	140
152	T2SP2-19	8.24E-05	1.40E-03	<9.09	2.34	<1.28	19	0.50					
160	T2SP2-20	7.35E-05	1.40E-03	<5.24	2.17	<1.28	20	0.54	113	63	<16.5	112	131
168	T2SP2-21	9.80E-05	1.27E-03	<10.0	2.53	<1.28	24	0.59					
176	T2SP2-22	7.57E-05	1.27E-03	<12.8	2.55	<1.28	20	0.52	37	26	<16.5	88	148
184	T2SP2-23	8.02E-05	1.20E-03	<10.2	2.46	<1.28	23	0.52					
192	T2SP2-24	8.69E-05	1.25E-03	<7.32	2.34	<1.28	23	0.46	150	80	<16.5	78	122
200	T2SP2-25	7.80E-05	1.38E-03	<11	2.43	<1.28	18	0.53					
208	T2SP2-26	9.13E-05	1.40E-03	<9.3	2.46	<1.28	22	0.53	96	61	<16.5	79	127
216	T2SP2-27	1.09E-04	1.60E-03	<3.98	2.43	<1.28	23	0.54					
224	T2SP2-28	8.91E-05	1.45E-03	<5.36	2.37	<1.28	18	0.55	26	23	<16.5	83	134
230	2SP2-FINAL	7.13E-05	1.45E-03	<8.13	2.25	<1.28	19	0.63	22	17	<16.5	77	130

Table 32. Analytical data for concentration tank filtrate samples from Test 2

Reaction time (h)	Conc. tank filtrate sample ID	Cs-137 (µCi/g)	Sr-85 (µCi/g)	K (mg/L)	Cu (mg/L)	Pd (mg/L)	B (mg/L)	DNC U-235 (ng/mL)	ICP-MS U-235 (mg/L)	NaTPB (mg/L)	3PB (mg/L)	2PB (mg/L)	1PB (mg/L)	Phenol (mg/L)
0	T2SP4-Feed	4.30	0.05	458.52	4.69	13.86	22.27	642	0.90		56.19	57.64	88.95	119.28
0	T2SP4-00	1.94E+00	<210	392.00	1.26	<1.28	15	174	0.20	31	64	39	77	78
4	T2SP4-01	1.92E+00	<220	495.00	1.62	<1.28	16		0.26					
8	T2SP4-02	2.45E-01	2.67E-03	490.00	1.80	<1.28	17	305	0.37	<16.3	68	70	106	119
12	T2SP4-03	7.35E-02	1.94E-03	365.00	1.89	<1.28	16		0.42					
16	T2SP4-04	5.57E-02	2.45E-03	238.00	2.04	<1.28	17	345	0.43	<16.3	44	69	158	129
20	T2SP4-05	2.67E-02	2.05E-03	69.30	2.09	<1.28	18		0.44					
24	T2SP4-06	2.23E-03	1.89E-03	28.80	2.02	<1.28	22	374	0.41	118	74	98	107	121
28	T2SP4-07	5.35E-04	1.89E-03	30.20	2.09	<1.28	23		0.42					
32	T2SP4-08	3.56E-04	1.83E-03	18.10	2.07	<1.28	23	369	0.45	192	73	92	98	122
36	T2SP4-09	3.79E-04	1.76E-03	26.10	2.05	<1.28	24		0.42					
40	T2SP4-10	2.14E-04	1.65E-03	29.50	2.12	<1.28	25	354	0.44	210	77	91	88	117
44	T2SP4-11	1.63E-04	1.60E-03	9.36	2.02	<1.55	25		0.46					
48	T2SP4-12	2.03E-04	1.56E-03	25.40	2.12	<1.28	27	354	0.46	189	81	56	90	311
52	T2SP4-13	1.78E-04	1.74E-03	26.60	2.14	<1.28	27		0.45					
56	T2SP4-14	1.87E-04	1.34E-03	12.20	2.19	<1.28	27	331	0.44	196	83	54	79	0
60	T2SP4-15	1.80E-04	1.45E-03	20.20	2.34	<1.28	27		0.49					
64	T2SP4-16	1.74E-04	1.45E-03	22.30	2.26	<1.28	27	350	0.45	203	84	51	100	140
68	T2SP4-17	1.65E-04	1.43E-03	<14.7	2.32	<1.28	26		0.37					
72	T2SP4-18	1.96E-04	1.45E-03	<24.4	2.38	<1.28	26	358	0.41	211	94	39	121	143
76	T2SP4-19	1.76E-04	1.40E-03	<15.9	2.43	<1.28	27		0.43					
80	T2SP4-20	1.89E-04	1.45E-03	<11.2	2.17	<1.28	25	353	0.42	200	96	36	125	138
84	T2SP4-21	2.18E-03	1.40E-03	<5.49	2.03	<1.28	25		0.37					
88	T2SP4-22	4.01E-04	1.40E-03	<13.2	2.08	<1.28	25	350	0.42	295	<15.7	<16.5	140	141
92	T2SP4-23	2.07E-04	1.43E-03	<2.78	2.28	<1.28	26		0.42					
96	T2SP4-24	1.85E-04	1.34E-03	<10.2	2.10	<1.28	24	382	0.43	276	103	39	121	118
100	T2SP4-25	1.51E-04	1.49E-03	<7.06	2.27	<1.28	25		0.39					
104	T2SP4-26	1.63E-04	1.47E-03	<14.5	2.33	<1.28	26	369	0.38	290	101	37	120	117
108	T2SP4-27	1.92E-04	1.45E-03	<13.9	2.21	<1.28	26		0.44					
112	T2SP4-28	1.47E-04	1.45E-03	<9.61	2.27	<1.28	26	360	0.46	286	100	34	117	121
116	T2SP4-29	1.49E-04	1.47E-03	<9.96	2.25	<1.28	27		0.50					

Table 32 (continued)

Reaction time (h)	Conc. tank filtrate sample ID	Cs-137 (µCi/g)	Sr-85 (µCi/g)	K (mg/L)	Cu (mg/L)	Pd (mg/L)	B (mg/L)	DNC U-235 (ng/mL)	ICP-MS U-235 (mg/L)	NaTPB (mg/L)	3PB (mg/L)	2PB (mg/L)	1PB (mg/L)	Phenol (mg/L)
120	T2SP4-30	1.47E-04	1.40E-03	<7.99	2.28	<1.28	26	362	0.50	232	93	80	81	255
124	T2SP4-31	1.23E-04	1.47E-03	<10.2	2.24	<1.28	26		0.47					
128	T2SP4-32	1.27E-04	1.40E-03	<12.3	2.30	<1.28	27	359	0.48	231	92	33	82	0
132	T2SP4-33	1.20E-04	1.40E-03	<12.4	2.23	<1.28	26		0.46					
136	T2SP4-34	1.23E-04	1.43E-03	<6.67	2.28	1.62	28	361	0.44	233	90	<16.5	41	0
140	T2SP4-35	1.18E-04	1.47E-03	<1.76	2.38	<1.84	28		0.40					
144	T2SP4-36	1.29E-04	1.38E-03	<6.35	2.18	<1.28	26	358	0.41	281	106	<16.5	123	0
148	T2SP4-37	1.34E-04	1.45E-03	<8.75	2.27	<1.28	26		0.47					
152	T2SP4-38	1.09E-04	1.40E-03	<9.75	2.27	<1.28	26	384	0.45	260	88	<41.1	129	0
156	T2SP4-39	1.38E-04	1.51E-03	<10.4	2.22	<1.28	27		0.46					
160	T2SP4-40	1.25E-04	1.47E-03	<8.42	2.15	<1.28	25	378	0.47	291	106	<16.5	91	0
164	T2SP4-41	1.29E-04	1.40E-03	<2.63	2.21	<1.72	26		0.48					
168	T2SP4-42	1.29E-04	1.49E-03	<11.0	2.37	<1.28	28	379	0.49	241	96	<16.5	78	146
172	T2SP4-43	1.14E-04	1.45E-03	<12.2	2.45	<1.28	28		0.53					
176	T2SP4-44	1.00E-04	1.54E-03	<11.9	2.34	<1.28	28	378	0.50	214	93	<16.5	91	145
180	T2SP4-45	1.00E-04	1.45E-03	<11.7	2.32	<1.28	27		0.45					
184	T2SP4-46	1.18E-04	1.49E-03	<10.2	2.31	<1.28	28	375	0.53	223	95	<16.5	92	146
188	T2SP4-47	1.02E-04	1.40E-03	<3.67	2.27	<1.88	28		0.44					
192	T2SP4-48	9.80E-05	1.36E-03	<8.29	2.21	<1.28	27	359	0.46	277	109	<16.5	79	123
196	T2SP4-49	8.91E-05	1.31E-03	<9.02	2.21	<1.28	27		0.45					
200	T2SP4-50	1.34E-04	1.27E-03	<10.8	2.31	<1.28	28	396	0.49	271	107	<16.5	78	124
204	T2SP4-51	1.14E-04	1.36E-03	<10.8	2.31	<1.28	28		0.47					
208	T2SP4-52	8.24E-05	1.27E-03	<10.2	2.33	<1.28	27	383	0.47	94	61	<16.5	78	123
212	T2SP4-53	9.36E-05	1.45E-03	<1.5	2.31	<1.66	27		0.49					
216	T2SP4-54	1.05E-04	1.40E-03	<5.78	2.36	<1.28	27	402	0.53	202	97	<16.5	88	132
220	T2SP4-55	8.02E-05	1.43E-03	<5.58	2.31	<1.28	27		0.49					
224	T2SP4-56	1.00E-04	1.40E-03	<6.72	2.29	<1.28	27	400	0.47	197	96	<16.5	83	133
228	T2SP4-57	8.24E-05	1.51E-03	<7.12	2.32	<1.28	28		0.45					
230	T2SP4-FINAL	9.58E-05	1.40E-03	<8.52	2.14	<1.28	27	397	0.61	200	97	<16.5	78	130

8.9 LESSONS LEARNED

During the course of the two experiments, much was learned about the CSTR system for precipitating cesium from solution using NaTPB. The ORNL system operated well in providing the necessary foundation for determining whether or not the process would work as designed in engineering-scale continuous equipment. The experiment was labor intensive, primarily due to the large number of samples and the handling of the samples after removing them from the hot cells. This effort required at least two dedicated workers on each of the three shifts per 24-h day. Since this was a new process and a new system running the process, additional data were taken during both experiments to help in determining operability, to help define operating parameters, and to generate enough data to analyze trends and troubleshoot problems. The failure of the level-indicating device in the slurry concentration vessel also created extra needs for manpower to constantly watch the concentration loop operation so as to prevent tank overflow or too much slurry concentration. The large volume of feed required for a 10- to 11-day continuous experiment was intensive in terms of time, material, and manpower. The several additional feed streams also added time and manpower requirements.

The Intellution computer control system with the database and historical record capabilities was invaluable in allowing troubleshooting of problems and trending of performance. The setup of the control system by Rocci Norcia of the Control Company and Jim Firth and Tony Tipton of Savannah River made the operation of the two filtration systems almost effortless. If the problems with the level meter in the concentration vessel had been resolved before the run, the extra time and effort required to gauge the tank with a dip stick would not have been required—as is evident from the smooth operation of the slurry wash filtration system. This preparation and automation were essential for the scale and complexity of the equipment in this test.

8.10 NEEDS FOR FURTHER WORK

These tests established that the chemistry and engineering of the two CSTRs in series process are feasible. Several questions remain, including the efficiency of slurry washing, the effects of long-term operation on the CSTR and filtration systems, the ability of the system to recover from an upset, the process conditions that can initiate catalytic decomposition of TPB, and the effects of the decomposition on the control of the system.

9. REFERENCES

1. Scot Beck et al., *Bases, Assumptions, and Results for the Flowsheet Calculations for the Short List Salt Disposition Alternatives*, WSRC-RP-98-00168, Rev. 1, Westinghouse Savannah River Company, Aiken, S.C., Oct. 22, 1998.

2. *Small Tank TPB Precipitation Facility Pre-Conceptual Design Package*, G-CDP-H-00003, Rev. 0, Westinghouse Savannah River Company, Aiken, S.C.

3. R. A. Jacobs, *Partial Design Input for Bench Scale Reactor Studies*, HLW-SDT-99-0041, Rev. 0, Westinghouse Savannah River Company, Aiken, S.C. , March 17, 1999.

4. D. D. Walker, *Preparation of Simulated Waste Solutions*, WSRC-TR-00116, Rev. 0, Westinghouse Savannah River Company, Savannah River Technology Center, Aiken, S.C., April 15, 1999.

Appendix A.1

TECHNICAL TASK REQUEST AND TECHNICAL TASK PLAN FOR TESTING TWO CSTRs IN SERIES FOR THE SMALL TANK TPB PRECIPITATION SIMULATION

OSR 19-2558(Rev 4-6-97)

Technical Task Request

Funding Source	Plant Modification Traveler No.	Technical Task Request No.		Revision
EM-50 (ORNL Financial Plan)	N/A	HLW-SDT-TTR-99-03.0		0
Task Initiation (Signature/Date) R A Jacobs 3/8/99	Name (Print) Roy A. Jacobs	Phone 557-4513	Site Address 704-196N	Date 1/25/99
Performing Organization Oak Ridge National Lab	Approval (Signature)			Date 3/8/99

Task Description Due Date 9/24/99

☐ All activities are to be performed and documented in accordance with Manual E7. Specific procedures are referenced with the associated tasks.

☒ Task Specific QA Plan Reference ORNL eq. to SRS 1Q Manual QAP 2-3

Definition of Scope of Plant Modification

☐ Provided Reference _____ ☐ Not Applicable to this Request

☒ To be developed as part of this request. Specific activities are

- ☐ Scoping Studies
- ☐ Feasibility Studies
- ☐ Technology Assessment
- ☒ Other Specify Technology Development

Functional Requirements and Basis

☐ Provided Reference _____ ☒ Not Applicable to this Request

☐ To be developed as part of this request. Specific activities are

- ☐ Develop Functional Performance Requirements to be included as Part of The PMT (Procedure 2.05) or Task Requirements and Criteria (Procedure 2.13)

Hazard Classification

☐ High Hazard ☒ Moderate Hazard ☐ Low Hazard ☐ General Use

☐ To be developed as part of this request (Manual 11Q)

Functional Design Criteria

☐ Provided Reference _____ ☒ Not Applicable to this Request

To be developed as part of this request. Specific activities are

- ☐ Alternative Studies
- ☐ Develop FDC Document (Procedure 2.11)
- ☐ Develop Functional Design Criteria to be included as Part of the PMT (Procedure 2.05) or Task Requirements and Criteria (Procedure 2.13)

Functional Classification

- ☐ Safety Class
- ☐ Safety Significant
- ☒ Production Support
- ☐ General Service
- ☐ To be developed as part of This request (Procedure 2.25)

Criteria Technical Review

☒ Not Applicable to This Request ☐ To be performed as part of this request (Procedure 2.80)

Design and Analysis/Technical Baseline Development

☐ Provided Reference _____ ☐ Not Applicable to this Request

☒ To be developed as part of this request. Specific activities are

- ☐ Calculations (Procedure 2.31)
- ☐ Drawings (Procedure 2.30)
- ☐ Specifications (Manual 3E)
- ☐ SAR (Manual 11Q)
- ☐ FDD (Procedure 2.12)
- ☐ SDD (Procedure 2.12)
- ☐ Design Process Hazard Review (Manual 11Q)
- ☐ Quality Assessments (Procedure 2.35)
- ☐ Quality Inspection Plans (Procedure 2.36)
- ☐ Functional Acceptance Criteria (Procedure 2.26)
- ☐ Technical Specifications
- ☒ Other Specify ORNL eq. to SRTC L1.7.10

Back of
DSR 19-[illegible] (Rev 4-25-97)

TTR No. HLW-SDT-TTR-99-03.0

Rev. 0

Technical Task Request (Continuation)

Design and Analysis/Technical Baseline Document Technical Review

☐ Not Applicable to This Request ☒ To Be Performed as Part of This Request (Procedure 2.60) 3.60 & 2.41)

Acceptance Testing

☒ Acceptance Testing is Not Part of This Request
☐ Test Procedures Provided Reference _____
☐ Test Procedures to be Developed as Part of this Request (Manual 6E)
☐ Test Results Provided Reference _____
☐ Test Results Evaluation Not Part of this Request
☐ Test Acceptance Report to be Provided as Part of this Request (Manual 6E)

Other Tasks or Clarification

Consultants and Bench Scale Reactor Studies

Background: Continuous TPB precipitation of Cs and alpha/Sr removal by MST have not been demonstrated. Consulation with experts on mixing and scaleup and on precipitation/crystallization is needed to guide experimental and bench scale design. Small scale (0.5 L) single reactor CSTR work will be performed at SRTC. Integrated bench scale studies (~20 L) will be conducted at ORNL.

Specific Tasks:

3.1 Consultants: (1) Obtain services from a mixing expert for guidance in design of small scale kinetics experiments, design and scale of bench scale reactor studies, and scale-up to full scale facilities. (2) Obtain services from an expert in precipitation and crystallization for guidance in experimental design and for guidance in CSTR design and operation.

3.2 Bench scale reactor studies: (1) Design and build an integrated bench scale facility with two CSTRs sized at ~20 L (~ 15 L working vol.). Facilities to include feed tanks, product tanks, two CSTRs in series, concentrate tank w/ filter, wash tank w/ filter, recycle wash water tank. See attached drawing for guidance. (2) Conduct one or more continuous open loop tests (CSTR #1 thru concentrated precipitate) using simulant without noble metals to determine operating conditions for adequate DFs. Feeds will be spiked with tracers to determine Cs, U, and Sr DFs. Initial operating conditions (simulant composition, feed rates, excess NaTPB, mixing) will be determined by prior testing at SRTC. Sample schedule to be specified by SRTC. Duration of each run will be approximately 60 hours at nominal feed rates. Concentrated precipitate to be batch washed when satisfactory operating conditions have been determined. (3) Conduct a continuous closed loop test (close wash water recycle loop) producing four batches of concentrated precipitate (~ 240 hours at nominal feed rate) with complete simulant spiked with tracers (includes noble metals/catalyst) to demonstrate DF in the presence of catalyst for TPB decomposition. Sample schedule per SRTC.

Other Reviews/Reports Required?

☒ Yes ☐ No Specify Described in Closure/Deliverables Provided

Technical Agency	Name (Print)	Site Address	Phone No.
Oak Ridge National Lab	C. P. McGinnis	PO Box 2009, ORTN 37831	423 576-6865
Acceptance of Task (Signature) C. Phil McGinnis			Date 3/9/99

Closure/Deliverables Provided

3.1 Consultants:

Initial consultations:	3-Mar-99
Final reports:	TBD

3.2 Bench scale reactor studies:

Approved Task/QA Plan:	9-Mar-99
Complete preliminary bench scale design:	19-Mar-99
Complete facility fabrication:	14-May-99
Progress reports:	as required
Complete experiments:	19-Aug-99
Approved technical report:	17-Sep-99

Technical report to be prepared and approved according to SRS E7 procedures 3.60 and 2.41.

Task Initiator (Signature)	Date
Approvals (Signature)	Date

ORNL/CF–99/5

Rev. 0, 3/9/99

TECHNICAL TASK PLAN FOR TESTING TWO CSTRs IN SERIES FOR THE SMALL TANK TPB PRECIPITATION SIMULATION

D. D. Lee

1. INTRODUCTION

This document describes proposed testing at Oak Ridge National Laboratory (ORNL) to evaluate the decontamination achievable in the small-tank TPB precipitation process for the removal of cesium, strontium, and uranium from simulated Savannah River supernatant. The proposed technical task plan is in response to a request for support for the conceptual design phase for one of the selected alternatives to the High-Level Waste (HLW) Salt Deposition project. As part of these tests, a continuous stirred-tank reactor (CSTR) system will be constructed containing two CSTRs in series, each with a 15 L working volume. A precipitate concentration system (including a cross-flow filter), a precipitate wash system (with cross-flow filter), and various tanks and pumps to integrate the operations are included. Simulant supernatant, dilution water, recycle wash solution, sodium tetraphenylborate (NaTPB), and monosodium titanate (MST) will be fed to the first CSTR. Overflow from this CSTR will be passed into a second CSTR, which will then overflow to the third vessel in which the slurry of precipitated potassium-TPB, cesium-TPB, excess NaTPB, sludge, and MST are concentrated. The filtrate from the slurry concentration step is the decontaminated product. The concentrated slurry will then be washed in a fourth vessel and the washed solids will be stored as the HLW waste product. The wash solution will be recycled to the feed tank.

Needed information for the scale-up of the Small Tank TPB Precipitation process will be provided by these tests. Analyses to be performed during the operation of the process include high performance liquid chromatography (HPLC), K, Cs, Cu, B, Pd, Sr, and U to determine interstage conversion and demonstrate that the treated supernatant meets the required levels for waste acceptance criteria (WAC) of Cs <70 nCi/g, total alpha <18 nCi/g, and Sr <40 nCi/g. The desired levels of treatment are Cs <40 nCi/g, alpha <10 nCi/g, and Sr <20 nCi/g, which are set at 50% of the WAC to allow for changes in process conditions. In this set of experiments, the alpha will be followed by analyzing uranium. The apparatus will be constructed of stainless steel with the CSTR vessels having a minimum of four inlet tubes. The CSTRs and other process vessels will be purged with nitrogen gas as required and the CSTRs and filter feed vessels will contain a cooling coil to maintain +/- 2°C temperature control. The first test will be performed **without** the addition of the ECC, copper, and palladium catalysts that have been shown to cause the decomposition of the tetraphenylborate (TPB), but sludge surrogate supplied by SRL will be added to the feed mixture. The precipitate washing system will not be used during the first test series so it will be performed with a simulated recycle stream. Test 1 will be divided into three phases (A, B, and C) with phases B and C to be

conducted only if prior phases do not achieve the required decontamination factor (DF). Phase A will use the excess TPB and residence time defined by prior Savannah River Technology Center (SRTC) tests and provided by the Savannah River Site (SRS). If the DF goals are not met, the amount of excess TPB will be increased (Phase B), and if the DFs are still not obtained, then the residence time in Phase C will be increased to achieve the goal.

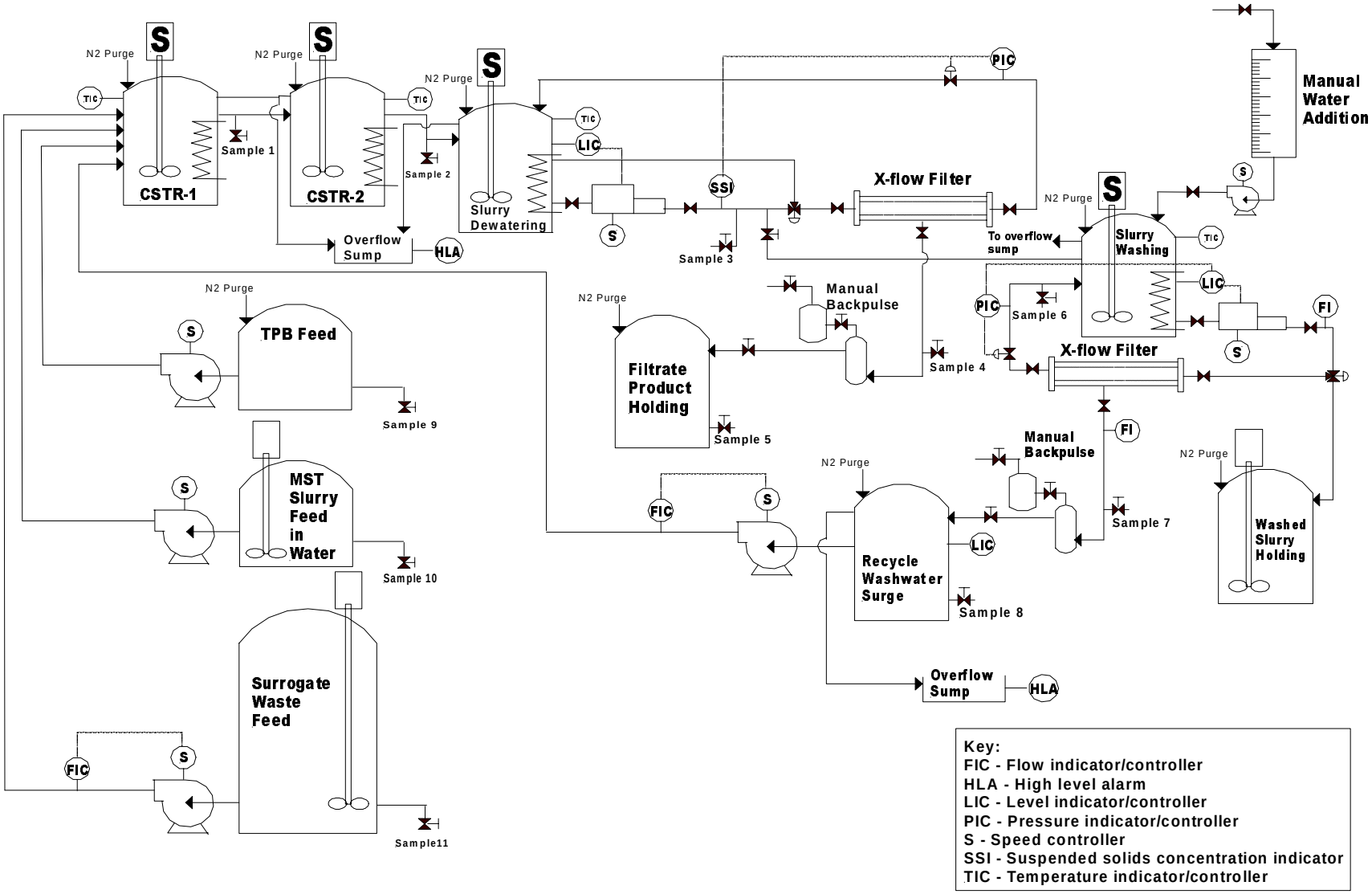
Test 2 will examine the fully integrated flow sheet which includes measurements of several parameters including interstage conversions of Cs, Sr, and U, and evaluation of the catalytic decomposition of TPB by copper and palladium catalysts. A minimum of 4 system turnovers (defined by 4 turnovers of the wash and concentrate tanks) will be performed for the test. The required residence times for CSTRs will be based on data from the test 1.

2. TASK DESCRIPTION

2.1 Equipment

The Small Tank TPB Precipitation pilot scale system will use reactors with a 20 L total volume and a 15 L working volume. A preliminary sketch of the flow diagram is shown in Fig. 1. The CSTRs will be constructed from 316 stainless steel, with bottom drain/flush valves, and have flanged tops with center, flange-mounted agitators. The agitator size will be equivalent to one fourth of the CSTR internal diameter. Each reactor will also include 4 baffles that are 1/12 the diameter of the tank and attached to the top flange. The vapor space of the CSTRs will be purged continuously with nitrogen gas during operation. The feed streams to the reactors will enter through the top flange and discharge under the surface of the liquid in the CSTR, near the agitator. The transfer lines from the reactors to the downstream vessel will also open at the liquid surface and exit through the side of vessel. The supernatant mixture in the CSTRs will be internally cooled or heated by stainless steel cooling coils which will be positioned below the working liquid level in the reactor. The coils will be attached to the top flange and will be removable to add versatility to the system.

The vessel for concentrating the precipitate will be the same size and configuration as the CSTRs, including the cooling coils. The working volume of the tank will vary as the amount of precipitate increases during the filtration. The contents of the tank will be pumped through a cross-flow filter using a progressive cavity (Moyno) pump with 416 stainless steel CP (chrome plated) rotor and an EPDM stator. The slurry will be returned to the vessel while the filtrate is being sent to the product collection tank. The supernatant filtered product will be tested for the required DFs while the precipitate is being concentrated to about 10% by weight solids. After accumulating the desired volume of 10 % solids, the slurry will be transferred to the precipitate wash vessel. The wash vessel is similar to the precipitate concentration vessel, but has a 7.5 L working volume (one-half of the working volume of the CSTRs). A second cross-flow filter is used to perform the precipitate washing step. The wash vessel is operated at constant volume with constant wash water feed and filtrate removal until nitrite concentration is reduced to 0.01 $\underline{M}$ or less. The wash is used to remove excess salt and NaTPB for recycle to the system feed tank. The filtered wash liquid will be sent to the recycle storage tank and the washed solids will be transferred to the solids storage tank. The solids wash cycle requires about 24 h.



1. **Flow diagram for the ITP Test System.**

Liquids and slurries will be transferred using small-volume gear and peristaltic pumps. Gear pumps will be used for the liquid feed streams to the first CSTR, and the peristaltic pumps will be used for inter vessel slurry transfers. The 10% solids slurries in the precipitate concentrate tank and the precipitate wash tank will be transferred using the Moyno pumps normally used for the cross-flow filter feed.

The instrumentation and control strategy is currently being developed for the CSTR test system. Parameters that will be monitored and recorded include temperature, pressure, liquid level, density, pump speed, agitator speed, and flow rate. Feedback control systems will be used to control flow, level, and pressure. The control strategy for the process will be reviewed and approved by SRS and Oak Ridge.

2.2 Installation

The equipment will be installed in Hot Cell B in Building 4501 at ORNL. This cell is one of four cells in a square, central block, labeled A, B, C, and D. Cell B contains two X-Y-Z master-slave manipulators, two cell face windows, and access doors for sample, equipment, and convenient personnel entry (6 ft x 3 ft door). Cell B has very low levels of transferable contamination, and has low radiation source levels when not in use for experiments. Routine personnel entry for hands-on maintenance can be carried out with very low radiation exposure incurred. The cell also has low background radiation and allows for minimal cross contamination from one experiment to the next. Cell B has multiple access ports on two sides, and multiple cell-to-cell access ports. Each cell is 8 ft wide, 6 ft deep, and 16 ft floor to ceiling. The cell has 4 electrical panels inside with multiple 120 VAC and 240 VAC outlets connected to panels outside the cell. The panels also contain multiple instrument leads.

The cell has an operating level table about 4.6 ft above the cell floor with a containment pan on the table that is about 5.5 ft front to back and 7 ft wide. The area below the table is used for storage and can be used for large feed and waste tanks if an experiment requires them. Each cell has an air atmosphere supplied by the 3039 stack vacuum system which pulls air through the cells from the areas surrounding the cells. The high-volume, low-vacuum off-gas from the cell passes through HEPA filters in the Building 4501 basement and then to the 3039 stack. The cells also contain vessel off-gas connections for low volume, high vacuum work and it also goes to the 3039 stack. The walls are 3 ft thick barytes concrete lined with stainless steel. Ceiling is 4 ft thick barytes concrete. The doors are all 8 in. thick lead. Each cell has an upper 2 x 2 ft and a lower 16 x 16 in. lead glass window in the center cell face, each 3 ft thick. The windows contain 8 four in. thick panes of glass and the spaces between panes are filled with mineral oil.

2.3 Materials

The MST, NaTPB, and sludge used in these tests will be supplied by SRS, and the supernatant simulants will be prepared at ORNL using a recipe provided by SRS. Normal cesium and strontium traced with ^{137}Cs and ^{85}Sr will be used to obtain the needed concentration of cesium and strontium, in the simulated supernatant. In order to assure the level of strontium added, the simulant will be treated with MST and then filtered to remove strontium impurities

present in some of the chemicals used to make up the feed. The amounts of tracers added will be dictated by the counting accuracy required after the TPB precipitation.

2.4 Test Description

Continuous stirred-tank reactor sizes from the Phase III document[1] are 15,000 gallons working volume. Assuming 20 L bench scale vessels with 15 L working volume, scales are 1 gal = 1 mL and 1 gal/min = 1 mL/min. For bench scale system, the flow rates of the various feeds include:

Salt feed — 16.5 mL/min at 6.4 *M* Na+ and 0.017 *M* K+

Dilution water plus MST (@ 0.4 g MST/L salt solution) — 2.8 mL/min

Makeup NaTPB — 0.5 mL/min at 0.55 *M* TPB – (stoichiometric – assumes no TPB decomposition)

Recycle wash water — 3.8 mL/min at 1.25 *M* Na + and 0.03 *M* TPB – average (TPB – is 50% excess)

Total flow exit the CSTRs ~23.6 mL/min of 0.5 wt % slurry; residence time in each CSTR = 10.6 h.

The operating process will make about 1.2 mL/min of 10 wt % unwashed slurry and 22.5 mL/min decontaminated salt solution with about 4.7 *M* Na. Experimental residence time for the CSTR for the first case is τ = 10.6 h. For the first test series, the tests are expected to last for 60 h (>five residence times or the time required for the production of one batch of precipitated concentrated solids) and make approximately 4 L of unwashed 10 wt % precipitate. Each part of the first test is expected to require the following volumes of feed and produce the following volumes of waste.

Salt solution	60 L
Dilution water	10 L
Makeup NaTPB	1.8 L
MST slurry	0.2 L
Recycle wash water simulant (1.25 *M* Na)	13.7 L
Decontaminated salt solution	81 L
10 wt% unwashed precipitate	4.3 L

The closed loop run will last about 264 h or about 11 days and makes three batches of washed precipitate. The approximate volumes in liters for a 10.6 h residence time in the CSTR are:

Salt solution feed	264 L
Dilution water	44 L
Makeup NaTPB	8 L
MST slurry	0.9 L
Wash water/recycle wash produced	10.25 x 4 L = 41 L
Decontaminated salt solution	356 L
10 wt% unwashed precipitate	4.75 L x 4 = 19 L
8.7 wt% washed precipitate	4.83 L x 3 = 14.5 L*

*Only three batches of precipitate will be washed

The Savannah River Site has determined that the supernatant solution that will be used in the CSTR testing will be the average simulant with the composition shown below. During operation of the CSTR, the feed (6.4 *M* Na), dilution water with MST slurry, TPB solution, and wash water recycle (1.25 *M* Na) are added separately to CSTR 1 to operate at a 4.7 *M* sodium concentration in both CSTR 1 and CSTR 2.

SRS Waste Simulant (SR "Average") Composition for CSTR Tests

Component	(*M*)		NaX for 60 L Feed
Na^+	6.4		
Cs^+	0.00016	(CsCl)	1.62 g
K^+	0.017	(KNO_3)	103.89 g
OH^-	2.18		5.236 kg
NO^-_2	2.45		12.473 kg
NO^-_3	0.59		2.459 kg
$AlO^=$	0.35		1.743 kg
$CO_3^=$	0.18		1.163 kg
$SO_4^=$	0.17		1.461 kg
Cl^-	0.029		100.29 g
F^-	0.036		92.16 g
PO_4^{3-}	0.011		112.43 g
$C_2O_4^=$	0.0091		73.51 g
$SiO_3^=$	0.0046		33.49 g
$Sr^=$	0.0000025	($Sr(NO_3)_2$)	0.0326g
U			0.0114 g
$MoO_4^=$	0.00023		2.82 g
Sludge		(target = 0.571 wt%, 0.5wt% after dilution)	431 g

This composition may have the tendency for the aluminate to be either supersaturated or precipitated at 25°C. Therefore, the simulant feed tank will be agitated to make sure a uniform feed material is transferred to the CSTR.

Test and Analytical Samples

During the tests, a large number of samples will be taken for analysis. Due to the high total cost of the analytical portion of the experiment, some of the analyses may be deferred and only performed if required. It is planned that the following analytical tests will be done, but the circumstances during operation may change the needs for particular results. The feed solutions for all tests will be analyzed after make-up for K, ^{137}Cs, ^{85}Sr, and alpha. The NaTPB feed solution for each test will be analyzed by HPLC for a total of 4 HPLC samples. During each of Tests 1a, 1b and 1c, filtrate (output from the precipitate concentrate filter) will be sampled every two h (total of 30 samples for each test). Each sample will analyzed for K, ^{137}Cs, ^{85}Sr, and alpha and every third sample analyzed by HPLC for NaTPB every 6 h for a total of 10 HPLC samples for each test. Samples will be taken between CSTRs

every 4 h (15 samples), filtered immediately, and analyzed for K, ^{137}Cs, ^{85}Sr, and alpha and analyzed by HPLC for NaTPB every 8 h for a total of 8 HPLC samples for each test. Samples will also be taken after the second CSTR every 4 h (15 samples), filtered immediately, and analyzed for K, ^{137}Cs and ^{85}Sr, alpha and analyzed by HPLC for NaTPB every 8 h for a total of 8 HPLC samples for each test. These HPLC analyses do not look for all of the TPB intermediates. One sample of the slurry from the concentrated slurry batch will be taken after each of the first three tests and filtered, then washed with water, and the water analyzed by HPLC. The washed solids should then be dried, weighed and analyzed for potassium and boron. The blanks and standards that are recommended for analysis as part of each instrument run include 2 HPLC standards with all 4 components at about 100 and 1000 mg/L each, and each batch of feed solution will be analyzed for K, ^{137}Cs and ^{85}Sr, and alpha. During operation, the density (measure of the sodium concentration) of the filtrate from the slurry concentration cross-flow filter will be checked to confirm that operations are continuing as planned.

During Test 2, the filtrate is to be sampled every 4 h (total of 66 samples) and analyzed for B, K, ^{137}Cs and ^{85}Sr, alpha and analyzed by HPLC for NaTPB for every 8 h for a total of 33 HPLC samples. Samples are to be taken between CSTRs every 8 h (33 samples), filtered immediately, and analyzed for B, K, ^{137}Cs and ^{85}Sr, alpha and analyzed by HPLC for NaTPB and intermediates every 16 h for a total of 17 HPLC samples. Samples are to be taken after the second CSTR every 8 h (33 samples), filtered immediately, and analyzed for B, K, ^{137}Cs and ^{85}Sr, alpha and analyzed by HPLC for NaTPB and intermediates every 16 h for a total of 17 HPLC samples. During slurry washing, the wash water is to be sampled every 2 h (~45 samples) and analyzed for B, K, ^{137}Cs and ^{85}Sr, alpha and analyzed by HPLC for NaTPB every 6 h for a total of 15 HPLC samples. After each batch of concentrated slurry is transferred to the precipitate wash tank, a sample of the solids before washing will be obtained. The samples will be filtered, then washed with water, and the water analyzed by HPLC. The washed solids should then be dried, weighed and analyzed for potassium and boron. After washing in the precipitate washing step, a sample of the solids from each batch will be obtained and then dried, weighed and analyzed for potassium and boron. During the precipitate washing step, three wash water samples will be obtained from the beginning, middle and end of the wash and analyzed by HPLC, and for anions, nitrate and nitrite.

Start-up of the CSTRs will begin by filling both CSTRs with the simulant feed solution, NaTPB, and MST calculated to be present at steady state. The mix will be allowed to stand overnight or a period specified by SRTC to allow for an induction period, to decrease the operating time to reach steady state, since the residence time in each CSTR is about 10.4 h. This start-up procedure will also allow the output of product from the precipitate concentration filter system within several hours of start-up.

Other considerations during operation include the amount of antifoam (Surfynol 420, to be supplied by SRTC) and the operating conditions needed to suppress the formation of foam during the agitation and pumping operations, both in the CSTRs and in the two filter systems. Foaming is a known problem and the design of the CSTRs takes this

into account by not allowing any of the feeds or product streams that empty into a vessel to enter above the surface of the liquid in the vessel. To help effectively prevent the foaming, the Surfynol 420 will be added at amounts of about 150 mg/L of simulated feed and the amount increased if necessary. The baffles and agitator also operate to prevent the entrainment of gas during the agitation.

The waste streams that are produced during all of the tests can be disposed of as low-level radioactive mixed waste and transferred to Bechtel Jacobs Company LLC for disposition. As an alternative, the liquids can be neutralized and transferred as Low-Level Liquid Waste (LLLW) to the ORNL Low-Level liquid waste treatment

3. MEASUREMENTS, DOCUMENTATION, and QA

3.1 Analytical Methods

HPLC will be used for NaTPB, triphenyl, diphenyl, and monophenyl borate and phenol. Gamma counting will be used for cesium (^{137}Cs) and strontium (^{85}Sr). ICP-MS will be used for uranium, and palladium and boron, which requires a digestion prior to analysis. AA or ICP-MS will be used for potassium. These will be done by Analytical Services Organization in cooperation with SRS personnel on the HPLC methods. Standards and procedures for the boron analysis will be supplied by SRL.

3.2 Measuring and Test Equipment

The following M&TE will be used for this task. Electronic balances, which are calibrated annually by ORNL P&E personnel, will be used for solids and liquid mass measurements to make up the simulants. Class A volumetric flasks and graduated cylinders will be used for simulant make-up. A gamma counting system consisting of an ORTEC model GMX-45220-P-S intrinsic germanium detector, an ORTEC model 672 counting system amplifier, a Canberra Accuspec-A MCA card and Canberra Genie-2000 spectroscopy software will be used for in-house gamma measurements for the trace levels of ^{137}Cs and ^{85}Sr, to determine the course of the experiment.

Instrumentation used in the CSTR system will be calibrated before startup and recalibrated on a routine schedule. Calibration records will be maintained in registered logbooks or in a controlled project file. A list of instruments and corresponding calibration schedule will be generated once the process flow diagram and control strategy has been reviewed and approved by SRS and Oak Ridge.

3.3 Documentation

The ORNL personnel performing the work will record all experimental instructions, observations, results and calculations in a registered notebook. Copies of the notebook pages and analytical reports will be provided to SRS at the conclusion of the work. The results of these experiments will be documented in a numbered ORNL report (ORNL-TM-xxxx). Drafts of the final report will be reviewed by at least two independent ORNL researchers and

by selected SRS personnel. The final report will be published after the author addresses all comments. All records will be managed under the Records Management Plan for the Chemical Technology Division, ORNL/CF-97/42.

3.4 QA

The work performed under this task plan will be conducted under the Quality Management Plan for the Chemical and Energy Research Section of the Chemical Technology Division, ORNL/CF-97/27. It is governed by the Division Quality Program Description, ORNL/DF-98/31, and follows the CTD Procedure QA-3, R-2, 10/5/98.

4. SCHEDULE

Issue Approved Task/QA Plan	3/9/99
Complete bench scale design verification	3/19/99
Begin Equipment assembly	4/19/99
Complete equipment assembly in hot cell	5/14/99
Initiate Test 1a	6/7/99
Initiate Test 2	7/12/99
Complete Testing	8/19/99
Issue Draft Report	8/30/99
Issue approved technical report	9/17/99

5. BUDGET

Small Tank TPB Precipitation Facility Equipment Costs

1.	Four - 20 L CSTRs@$5K/reactor	$20K
2.	Four agitators (Lightnin or equiv. @ $2K)	$ 8K
3.	One - 10 L Stirred Tank with agitator	$ 5K
4.	Eight Feed and Transfer Pumps (0.5-30 mL/min)	$15.7K
5.	Sample refrigerator	$0.7K
6.	Recirculating Cooling Bath (2 @$3K)	$ 6K
7.	Cross-Flow Filters	$50-70K
8.	Moyno pumps for Cross flow filters	$10K
9.	Feed and Product Tanks (30 or 50 gal drums)-	$ 2K
10.	Agitators for feed tanks Four @$750	$ 3K
11.	Meters – Level (ultrasonic) (6@$1K), Flow (2 mag@$2.6K and 3 Coriolis @ $5K), Temperature, Pressure (10@$0.3K), RPM, flow (rotameters for gas (8@ $0.2)), and pressure controllers (3@$0.7)	$33K
12.	Valves (remote electric and manual) and Fittings	$10K
13.	Computer and Control Program (including programming)	$15K
	Total for Equipment	**$179-199K**
	+ 20% Contingency	$35K
	Total for Equipment	**$214-234K**

Small Tank TPB Precipitation Installation Costs

1.	Prepare Hot Cell (Remove previous experiment)	$20K
2.	Run new services (Nitrogen purge, electrical power, instrument, etc.)	$20K
3.	Build equipment rack and install reactor system in hot cell	$40K
4.	Prepare Bulk Liquid Radioactive Waste Handling Facilities	$8K
5.	Prepare Problem Safety Summary, Documentation, QA material, Operating Manual, etc.	$30K
	Total Installation Costs	**$118K**

SMALL TANK TPB PRECIPITATION FACILITY ANALYTICAL COSTS

Test 1a, 1b, and 1c

1.	Initial setup of HPLC methods —	$10K
2.	HPLC — 28 samples @3h ($150/h) or (4h/sample) each for 1a, b, and c ($12.6K ea.)	$37.8-50.4K
3.	Gamma for Cs & Sr — 61 samples @0.5h each for 1a, b, and c ($4.6K ea.)	$13.7K
4.	ICP-MS for U & B — 61 samples @0.5h or (0.75h) each for 1a, b, and c ($4.6-6.9K ea.)	$13.7-20.6K
5.	AA for K — 61 samples @0.75h each for 1a, b, and c ($6.9K ea.)	$20.6K
6.	HPLC Standards — 2 samples with 4 components @4h	$1.2K
7.	Three Feed solutions (AA, Gamma, and ICP-MS) @3h	$0.9K
	Analytical Totals for Test 1	$97.9 – 117.4K
	+ *Contingency (15% = $16.1K)*	*$16.1K*
	Totals	**$114 – 133.5K**

Test 2

1.	HPLC — 68 samples @3h ($150/h) or (4h/sample)	$41-54.6K
2.	Gamma for Cs & Sr — 136 samples @0.5h	$13.3K
3.	ICP-MS for U & B — 145 samples @0.5h or (0.75h)	$14 - 21K
4.	AA for K — 145 samples @0.75h	$21K
5.	HPLC Standards — 2 samples with 4 components @4h	$1.2K
6.	Feed solutions (AA, Gamma, and ICP-MS) @3h	$0.6K
	Analytical Totals for Test 2	$90.9 – 111.5K
	***Con**tingency (15% = $15.2K)*	*$15.2K*
	Totals	**$106.1 – 126.7K**
	Total Analytical for Both Tests	**$220.1K – $260.2K**

SMALL TANK TPB PRECIPITATION FACILITY OPERATIONAL COSTS

1. Chemicals (excluding TPB and MST) — NaOH, $NaNO_3$, $NaNO_2$, $NaAlO_2$, Na_2CO_3, Na_2SO_4, NaCl, NaF, Na_3PO_4, Strontium-85

	Test 1	$20K	
	Test 1	$28K	$48K

2. Technicians for operation - 2.5 for 3 months at $9.7K/m/tech

(2 months)	Test 1	$50K	
(1 month)	Test 2	$25K	$75K

3. Technical Staff - 2 for 2 months at $12.7K/m

(1.25 months)	Test 1	$32K	
(0.75 months)	Test 2	$19K	
			$51K

4. Support (P&E, I&C)

	Test 1	$10K	
	Test 2	$2K	$12K

5. Analytical

	Test 1	$114 – 133.5K	
	Test 2	$106.1 – 126.7K	$220.1K – $260.2K

6. Health Physics

	Test 1	$15K	
	Test 2	$5K	$20K

7. Waste Handling

	Test 1	$10K	
	Test 2	$5K	$15K

Total Operations Cost for Test 1	***$251 – 270.5K***
Total Operations Cost for Test 2	***$190.1 – 210.7K***
Total Operations Cost	***$441.1 –481.2K***

SMALL TANK TPB PRECIPITATION FACILITY TOTAL COSTS SUMMARY

1.	Equipment Costs		**$214 - 234K**
2.	Installation Costs		**$118K**
3.	Operational Costs	***Total Test 1***	**$251 – 270.5K**
		Total Test 2	**$190.1 – 210.7K**

Total Operational Costs

Construction, Installation and operations for Test 1		**$583 - 602.5K**
	Add 20% Contingency	**$115K**
	Total Test 1	**$698K - $717.5K**
	Test 2	**$190.1 – 210.7K**
	Add 20% Contingency	**$40K**
	Total Test 2	**$230.1K - $250.7K**
	Total For Both Tests	***$928.1K - $968.2K***

6. REFERENCES

1. Roy Jacobs, WSRC-RP-98-00168, Revision 1, *Bases, Assumptions, and Results of the Flowsheet Calculations for the Short List Salt Disposition Alternatives,* 1998.

2. *Small Tank TPB Precipitation Facility Pre-Conceptual Design Package* (U), G-CDP-H-00003, Revision 0.

7. APPROVALS

_____

D. D. Lee, Task Leader Date

_____

R. T. Jubin, CERS Section Head Date

_____

W. L. Tamosaitis, Manager, SRS-WPTS Date

_____

J. T. Carter, Salt Disposition Flow-Sheet Team Leader Date

Appendix A.2

DESIGN OF CSTR 20-L VESSELS AND ASSOCIATED EQUIPMENT WORKAID

Design of CSTR 20-L Vessels and Associated Equipment Workaid

Title: Design of CSTR 20-L Vessels and Associated Equipment Workaid

Purpose: To provide design specifications for the 20-L CSTR System

Scope: Design package for construction of the CSTR system

Acronyms:

3BV	Three-way ball valve
3PBV	Three-way proportional-flow ball valve
BV	Ball valve
CSTR	Continuous-flow stirred tank reactor
FI	Flow indicator
LI/LIC	Level indicator/controller
MP	Moyno Pump
PCV	Pressure control valve
PI	Principal investigator
PG	Pressure Gauge
PT	Pressure transmitter
PRV	Pressure relief valve
PV	Pressure Vessel
SSI	Suspended solids indicator - Coriolis meter
TI/TIC	Temperature indicator/controller

Action Steps:

Documentation:

The design drawings presented in this document will also be issued separately after they have received final approval.

Design of CSTR 20-L Vessels and Associated Equipment

These are the specifications and drawings that were given to the millwrights, boilermakers, and pipefitters to work from. This includes placement of the overflow and slurry transfer openings in the vessel walls and the openings, sizes, and placements of openings in the vessel top flanges. The cooling coil drawings are also included.

Design Specifications

1. **CSTRs** (Process needs 2 with 1 extra if needed later and for spare)
   Materials of Construction: 316 SS
   Gaskets: Viton
   Size: 10" schedule 40 pipe with pipe cap end, 17" from flange face to bottom with a standard mill finish, 150 lb. flanges, and 4" TriClover agitator mount.
   Agitator: Lightnin LabMaster Model G2L05R, 90-1600 RPM, 1/15 HP drive with 3/8" shaft and with digital speed display, and 4" TriClover vessel mount.. Impellers available for use include:
   3" A-200 4-blade, 45-degree pitch axial flow,
   3” A-100 3-blade marine axial-flow impeller, and
   3” R-100 6-blade radial-flow impeller,
   Working Volume: 15 Liters, (15.4 Liters measured)
   Total Volume: 20 Liters
   Cooling Coil: 1/4" 316SS coiled tube, 4.5" coil ID acting as draft tube (coil is 3/2 ID ratio to the agitator), with length of 5.5” from the bottom to the top of the coil, and 1/8” between the approximately 13 coils. The coil is positioned with ¼ of the liquid above and below the coil, about 2 ¾” above the bottom of the vessel.
   Fittings and attachments for CSTR1 are shown in the design drawings CERS-CSTR-DW1, -DW2, DW16, DW17, and DW20. Fittings and attachments for CSTR2 and 3 are shown in design drawings CERS-CSTR-DW1, -DW3, and -DW11.

2. **Slurry Concentration Vessel** (1)
   Materials of Construction: 316 SS
   Gaskets: Viton
   Size: 8" schedule 40 pipe with pipe cap end, 13" from flange face to bottom with standard mill finish, 150 lb. flanges, and 3" TriClover agitator mount.
   Agitator: Lightnin LabMaster Model G2L05R, 90-1600 RPM, 1/15 HP drive with 3/8" shaft and 2.5"A-200, 4-blade, 45 degree pitch axial flow impeller, with a digital speed display, and 3" TriClover mount.
   Working Volume: 7 Liters
   Total Volume: 10.7 Liters

Cooling Coil: 1/4" 316SS coiled tube, 4.25" coil ID acting as draft tube with coils spaced ¼" between coils. The coil is 4 1/4" from the bottom of the coil to the top and 2 1/8" off the bottom. It has about 8 coils.

Level Meter: Magnetrol "Kotron" Series 82 CE, RF Capacitance Level transmitter, With 0.25% repeatability, .1 second response time, Loop powered, 4-20 mA output, 316/416L SS, 3/4" NPT fitting, 24 " long, .875" OD

Fittings and attachments are shown in the design drawings CERS-CSTR-DW4, -DW6, DW14, DW15, and DW20.

3. **Slurry wash Vessel** (1)

Materials of Construction: 316 SS

Gaskets: Viton

Size: 8" schedule 40 pipe with pipe cap end, 13" from flange face to bottom with standard mill finish, 150 lb flanges, and 3" TriClover agitator mount.

Agitator: Lightnin LabMaster Model G2L05R, 90-1600 RPM, 1/15 HP drive with 3/8" shaft and 2.5"A-200, 4-blade, 45 degree pitch axial flow impeller, with digital speed display, and 3" TriClover mount. Spare A-100 and R-100 Agitators are to be available.

Working Volume: 7 Liters

Total Volume: 10.7 Liters

Cooling Coil: 1/4" 316SS coiled tube, 3 ¾" coil ID acting as draft tube spaced ¼" between coils. The coil is 3.3" from the bottom of the coil to the top and 1.65" off the bottom. It has about 6 ½ coils and sits about 1 5/8" above the bottom of the vessel.

Level Meter: Bindicator "LEVELDATA" Model GAG200006, Hydrostatic Level transmitter, With ± 1.0% Accuracy, 5:1 Turndown Capability, Loop powered, 0 to 28" H2O, 4-20 mA output, 316L SS, 3/4" NPT Process Connection, 3/8" Impulse Tube, 7/8" Reservoir

Fittings and attachments are shown in the design drawings CERS-CSTR-DW5, -DW7, DW12, DW13, and DW21.

4. Slurry Concentration Filter Loop (1)

System: Lines 3/4" SS tubing, valves and instruments readily accessible from the front of the module, sample lines include a small lipped tray below sample point, with 0-120 psig pressure gauge, 100 psi pressure relief valve with total unit as compact as possible.

Filter Tube: 0.5 micron SS sintered metal elements made by Mott Filter Corp. length is 24" with 0.5"ID, 5/8" OD and surface area of 0.26 ft^2, supplied by SR.

Slurry Flowrate: Design flow of 6 ft/s axial velocity gives 3.6 gpm volumetric flow

Pump: Moyno 500 Series model 33304 driven through flexible coupling by 3/4 HP 1750 3/60/460 VAC inverter duty Baldor motor, mounted on SS base plate. Pump is 316 SS body, 416 SS CP rotor and EPDM (ethylene-propylene-diene monomer) Elastomer stator, with 4 gpm capacity at 40 psi.

Piping: 3/4" 316 SS tubing, valves and fittings.

Flow Meter: Endress + Hauser "Promass 63" coriolis Mass Flow measurement system Model 63F-S15AAW00A25A3G with ½" 904S SS Tube for 1-22 GPM full scale, with flanged welded process connections, and DZL 363-551F311 Process transmitter/interface with Mass flow, density, and temperature outputs.

Pressure Control Valve: (Tube side back-pressure) Worcester Controls Actuated Ball Control Valve Model # 3/4" CPT 4466PMSE-A30 + 10755W actuator (120VAC), Dataflow Positioner #10DFP17 + 4DK75 Feedback Position Module with all wetted parts 316SS.

Pressure Control Valve: (Shell side back-pressure for Concentration Tank Level control) Research control valve from Badger #1001GCN36SVOEPO3S6 with EVA-1 Electric Actuator, P-3 Trim with stellite and 416 SS materials:

Filtrate Flow Meter: Rheotherm Flow Instrument Model#111D-I-TU1/16-4/20 flow sensing transducer, separate electronics and interconnect cable

Backpulse system: 1-L pressure tank with valves and fittings to contain about 0.7 L of filtrate during operation. When backpulse is needed, upstream and downstream valves are closed, and the nitrogen gas pressurizing valve is opened. When the pressure is about 100 psi, the pressurizing valve is closed, and the upstream valve to the filter is momentarily opened to force the contained filtrate back through the filter membrane and unplug the filter.

5. **Slurry Wash Filter Loop**

System: Lines 3/4" SS tubing, valves and instruments readily accessible from the front of the module, sample lines include a small lipped tray below sample point, with 0-120 psig pressure gauge, 100 psi pressure relief valve with total unit as compact as possible.

Filter Tube: 0.5 micron SS sintered metal elements made by Mott Filter Corp. length is 24" with 0.5"ID, 5/8" OD and surface area of 0.26 ft^2, supplied by SR.

Slurry Flowrate: Design flow of 6 ft/s axial velocity gives 3.6 gpm volumetric flow

Pump: Moyno 500 Series model 33304 driven through flexible coupling by 3/4 HP 1750 3/60/460 VAC inverter duty Baldor motor, mounted on SS base plate. Pump is 316 SS body, 416 SS CP rotor and EPDM Elastomer stator, with 4-gpm capacity at 40 psi.

Piping: 3/4" 316 SS tubing, valves and fittings.

Backpulse system: 1-L pressure tank with valves and fittings to contain about 0.7 L of filtrate during operation. When backpulse is needed, upstream and downstream valves are closed, and the nitrogen gas pressurizing valve is opened. When the pressure is about 100 psi, the pressurizing valve is closed, and the upstream valve to the filter is momentarily opened to force the contained filtrate back through the filter membrane and unplug the filter.

Flow Meter: Endress + Hauser "Promag 33" Electromagnetic Flow Measuring System Model

33F-T15MD1ED11D21A, with ½" Tube for 0.8-28 GPM full scale, with flanged welded process connections.

Pressure Control Valve: Worcester Controls Actuated Ball Control Valve Model # 3/4" CPT 4466PMSE-A30 + 10755W actuator (120VAC), Dataflow Positioner #10DFP17 + 4DK75 Feedback Position Module with all wetted parts 316SS.

Pressure Control Valve: (Shell side back-pressure for Slurry Wash Tank Level control) Research control valve from Badger #1001GCN36SVOEPO3S6 with EVA-1 Electric Actuator, P-2 Trim with stellite and 416 SS materials

Filtrate Flow Meter: Rheotherm Flow Instrument Model#111D-I-TU1/16-4/20 flow sensing transducer, separate electronics and interconnect cable

7. **Cooling Systems**

Refrigerated Circulating cooling baths: (3) VWR 13270-615, 190 watts cooling, 1-20 L/min coolant recirculation rate, external temperature control.

Refrigerated Circulating cooling baths: (1) VWR 13270-540, 650 watts cooling, 1.7-20 L/min coolant recirculation rate, and external temperature control.

8. **Process Pumps**

Peristaltic pumps (4): Accu CP-10 VWR 54845-686, 0.4-685 mL/min
Piston pumps (4): Accu FM-40 VWR 54845-690, 0.02-45 mL/min

9. **Pressure Transmitters**

Pressure Transducer (6): 0-30 psig Validyne P2-30-I
0-100 psig Validyne P2-100-I
0-200 psig Validyne P2-200-I

10. **Other Process Vessels**

Supernatant Salt/Sludge Feed Tank: Since at least 107 L of feed is required for each part of Test 1, using an eight hour residence time and a total of ten residence times operation, a 55-gal stainless-steel drum, an agitator for keeping the sludge in suspension, and a cover, would be all that is needed. However, for Test 2, a total of at least 281.2 L of feed is required and this is about 75 gal. The feed needs constant agitation to keep the sludge in suspension and to allow the trace cesium and strontium to be in equilibrium with their non-radioactive counterparts. Therefore, a system of two drums will be used so that all feed for test two can be made at one time. To keep flexibility, we will use a 55-gal drum for Test 1, and then add a 55-gal drum and connect the two together for Test 2, with a circulation pump between them. Both will be agitated and placed in Cell A for ALARA purposes. Connections between the tanks and the pump contained in Cell B to feed

the CSTR will be through the cell wall using SS tubing. We will use drum agitators for the agitation and a drum pump for circulation between the drums. Both drums will be covered to prevent evaporation, and the covers will contain the agitators and circulation pump. They will be supplied with connections for pumping in the feed to fill them from outside the cell through the drum cover. The radioactive/cold cesium and strontium mixtures will be added to the tanks after they have been filled in the cells by pumping the solutions in and allowing the tanks to mix and reach equilibrium. A Cole-Parmer guided-wave radar level probe will be used in the primary tank for checking the tank liquid level during operation. This will provide a secondary check on the flowrate of feed salt to the CSTRs.

TPB Feed tank: This tank is a simple 4 L covered beaker with no stirring as the total value for a Test 1 experiment is 2.5 L. The pump and vessel located outside the cell. For Test 2, the 264 h experiment and the volume for filling the CSTRs to start the run is about the equivalent of 284 h of operation or 8.86 L, so a 10 L carboy will be sufficient for the total required for TPB feeding. A check-valve has been added to the line from the pump to the CSTR on the hot-cell side of the line to prevent any chance of backflow from the vessel back through the line.

Feed tank for Dilution Water and MST: This tank is located outside the cell and is stirred to keep the MST is suspension. The tank and pump are placed outside the cell. This tank is a 10 L plastic stirred vessel with a working volume of 7 L for Tests 1A, 1B, and 1C, and a ~40L vessel with a working volume of 31 L for Test 2. This tank is outside the cell and feeds through the cell wall by peristaltic pump. A check-valve has been added to the line from the pump to the CSTR on the hot-cell side of the line to prevent any chance of backflow from the vessel back through the line.

Wash Water Recycle hold/feed tank: This tank has a 25 L maximum working volume, a 10 L minimum volume, and must be stirred to maintain a well-mixed volume as the wash water is added during the slurry washing step. This tank also is covered and purged with nitrogen, and has about an 8-L freeboard. The tank will be 12” diameter by about 18” high constructed from SS tubing. A Lightnin mixer with a 3” diameter, angled, TriClover fitting, will support the agitator. Fittings and attachments for Recycle-wash-water tank are shown in the design drawings CERS-CSTR-DW23and -DW24. A check-valve has been added to the line from the pump to the CSTR on the hot-cell side of the line to prevent any chance of backflow from the vessel back through the line.

Wash Water Feed Tank: This tank is required for Test 2, and not for Test 1. It needs to hold the volume of wash water for 1 slurry wash at 3.776 L for each wash (at 50% excess TPB) and this is 18.88 L for a 5 L slurry wash. Therefore, a 20-L carboy will work fine for the wash water, to be filled before each slurry wash step. A check-valve has been added to the line from the pump to the CSTR on the hot-cell side of the line to prevent any chance of backflow from the vessel back through the line.

Loaded Washed Slurry Hold Tank: The washed slurry hold tank needs to contain 5 L of 10% slurry from the transfer of each batch of slurry washed. During Test 1, no slurry is washed during the runs, so it is not required. During Test 2, three batches of slurry will be washed and a possible fourth may be washed, for a total of 20-L capacity required. The tank will be 10" ID x 30" high or 12" diameter x 21" high to contain the sludge with an extra capacity of about 10 L in case of unforeseen needs or requirements. The tank will be stirred and have a bottom drain. It will sit on a scale in Cell A, and contain about 21 kg of sludge at the 20-L level. Piping will be SS and go through cell B for sampling and disposal and the tank will be purged with nitrogen.

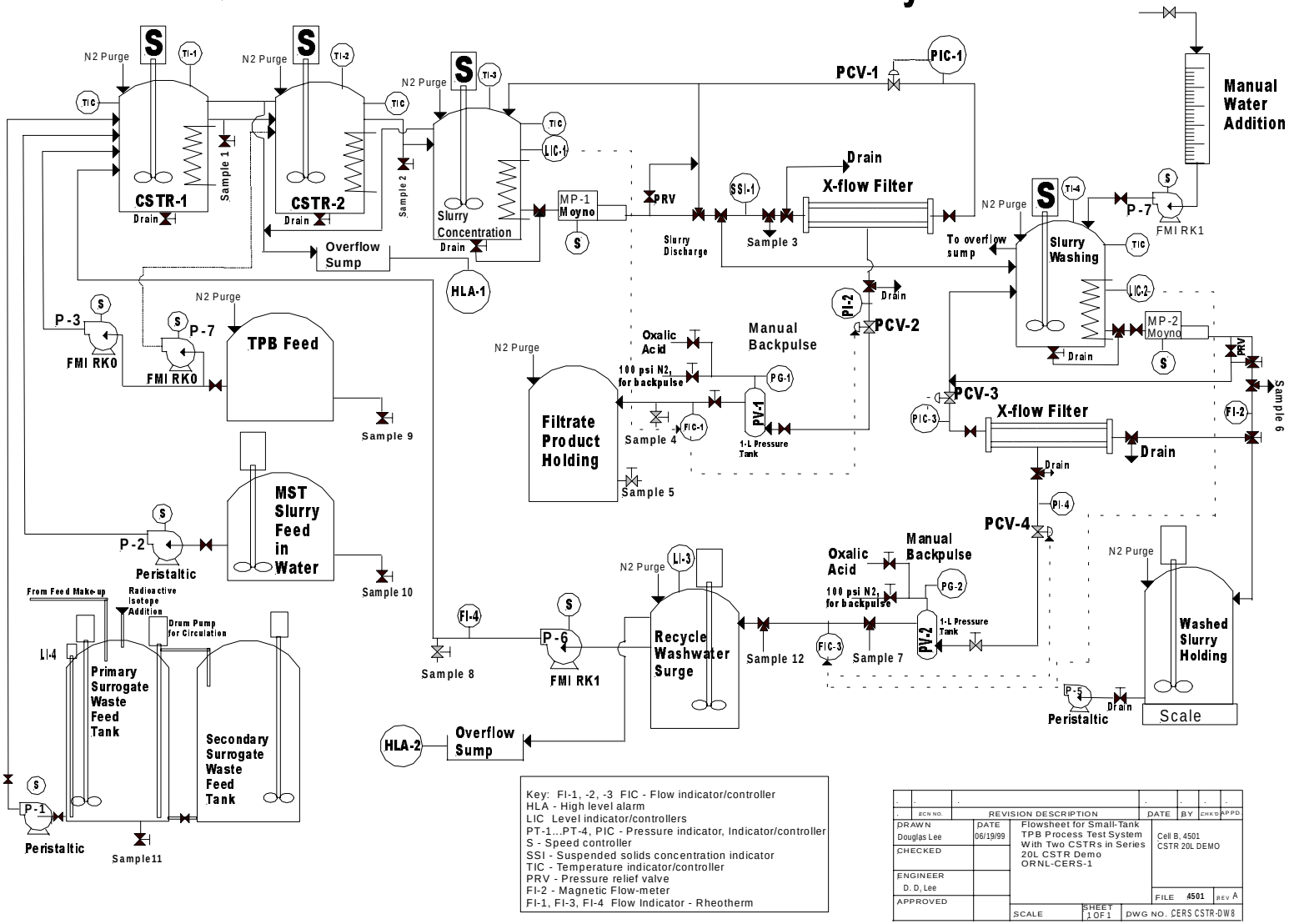
Product Filtrate Hold Tank: This tank is a day tank, used to contain one days' product filtrate liquid for sampling ease and easy removal from the hot cell. One days' filtrate is 32.4 L so a 10-gal tank will be sufficient for a day of filtrate collection. Once a day, the filtrate will be pumped to a 30-gal drum for storage, since most of the radioactivity will have been removed. One drum will be sufficient for each part of Test 1, and four drums will be required for Test 2. The day tank will be a polypropylene 10-gal carboy with a lid with fittings for inlet, outlet to peristaltic pump for transfer to storage drum, sampling, and nitrogen purge.

Surfynol 420 Feed tank: This tank is a simple 0.25-0.5 L covered beaker with no stirring as the amount required for a Test 1 experiment is 69.45 g. The pump (a syringe pump due to the small volumetric flow rate) and vessel are located outside the cell. For Test 2, the 264 h experiment requires about 246.5 g of Surfynol 420 for the entire run. Therefore, a 250-500 mL beaker is sufficient for a feed tank. The line size should be as small as practical to minimize the volume of material in the lines between the tank and the CSTR.

Drawing Numbers for SR CSTR Project

CERS CSTR-DW1	CSTR Cross-section, General view
CERS CSTR-DW2	CSTR 1 Top Flange
CERS CSTR-DW3	CSTR 2 and 3 Top Flange
CERS CSTR-DW4	Concentration Vessel, Cross-section, General View
CERS CSTR-DW5	Wash Vessel General View
CERS CSTR-DW6	Concentration Vessel Top Flange
CERS CSTR-DW7	Wash Vessel Top Flange
CERS CSTR-DW8	Flowsheet for Small-Tank TPB Process Test System with two CSTRs in series
CERS CSTR-DW9	Flowsheet for Small-Tank TPB Process Test System with three CSTRs in series
CERS CSTR-DW10	CSTR Flow Diagram
CERS CSTR-DW11	CSTR2 and 3, Cross-section through TPB and feed lines
CERS CSTR-DW12	Slurry Wash Vessel Cross-Section through Slurry in and out
CERS CSTR-DW13	Wash Vessel Cross-section Through Wash Water In
CERS CSTR-DW14	Concentration Vessel Cross-section Through Slurry in and out
CERS CSTR-DW15	Concentration Vessel Cross-section Through Cooling lines.
CERS CSTR-DW16	CSTR1 Cross-section through MST+ Dilution Water, Cooling , and outlets
CERS CSTR-DW17	CSTR1 Cross-section through TPB and Feed lines
CERS CSTR-DW18	Flowsheet for Slurry Concentration Loop
CERS CSTR-DW19	Flowsheet for Slurry Wash Loop
CERS CSTR-DW20	CSTR1, 2, and 3 Coils
CERS CSTR-DW21	Concentration Vessel Coils
CERS CSTR-DW22	Wash Vessel Coil
CERS CSTR-DW23	Recycle Washwater Hold and feed Tank
CERS CSTR-DW24	Recycle Washwater Hold and feed Tank (Top)
CERS CSTR-DW25	Simulant Salt Solution Feed Tanks

Flowsheet for Small-Tank TPB Process Test System

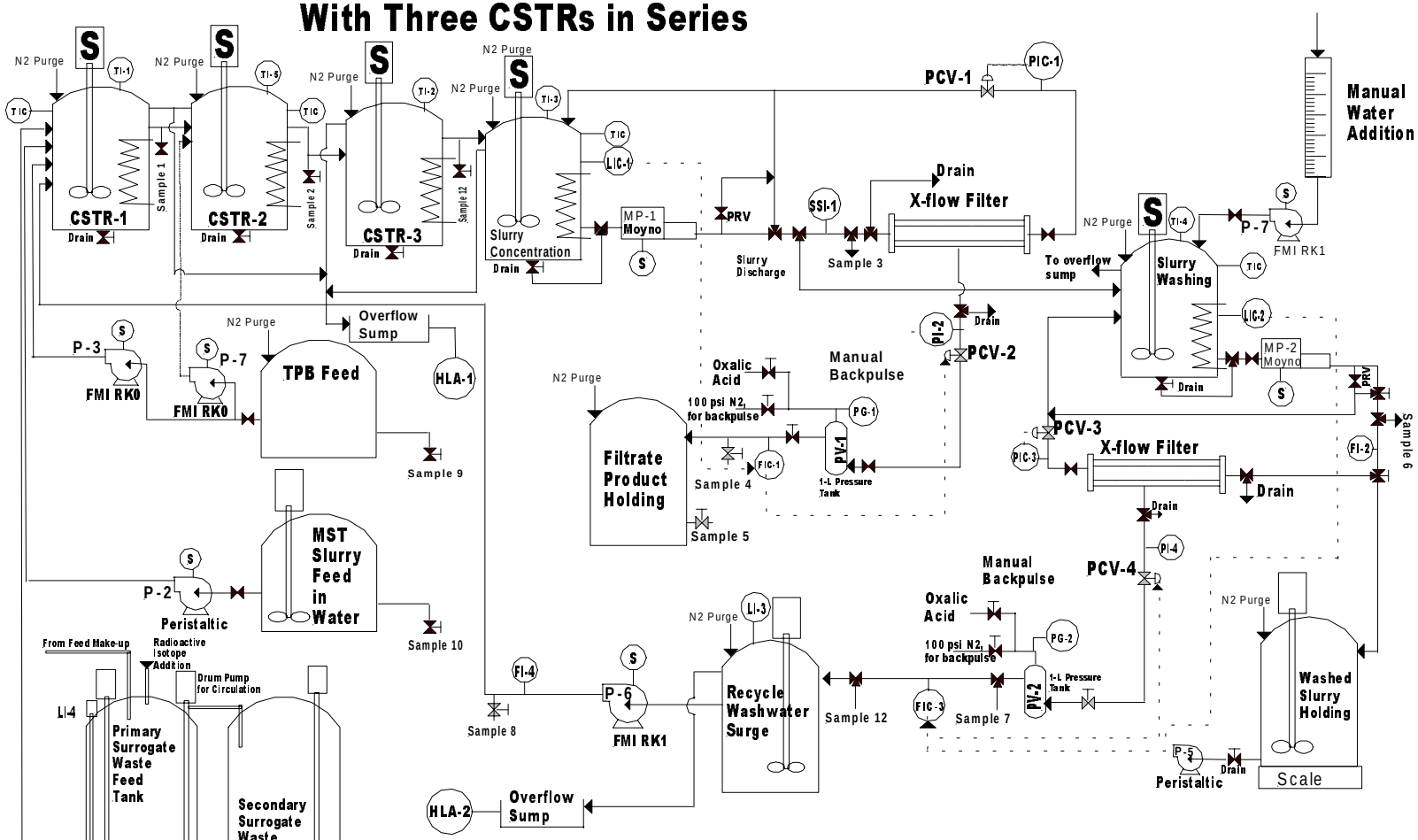


Key: FI-1, -2, -3 FIC - Flow indicator/controller
HLA - High level alarm
LIC Level indicator/controllers
PT-1...PT-4, PIC - Pressure indicator, Indicator/controller
S - Speed controller
SSI - Suspended solids concentration indicator
TIC - Temperature indicator/controller
PRV - Pressure relief valve
FI-2 - Magnetic Flow-meter
FI-1, FI-3, FI-4 Flow Indicator - Rheotherm

ECN NO.	REVISION DESCRIPTION	DATE	BY	CHK'D	APPD

DRAWN Douglas Lee	DATE 06/19/99	Flowsheet for Small-Tank TPB Process Test System With Two CSTRs in Series 20L CSTR Demo ORNL-CERS-1	Cell B, 4501 CSTR 20L DEMO
CHECKED			
ENGINEER D. D. Lee			FILE 4501 REV A
APPROVED		SCALE / SHEET 1 OF 1	DWG NO. CERS CSTR-DW8

Flowsheet for Small-Tank TPB Process Test System With Three CSTRs in Series



Key: FI-1, -2, -3 FIC - Flow indicator/controller
HLA - High level alarm
LIC Level indicator/controllers
PT-1...PT-4, PIC - Pressure indicator, Indicator/controller
S - Speed controller
SSI - Suspended solids concentration indicator
TIC - Temperature indicator/controller
PRV - Pressure relief valve
FI-2 - Magnetic Flow-meter
FI-1, FI-3, FI-4 Flow Indicator - Rheotherm

ECN NO.	REVISION DESCRIPTION	DATE	BY	CHK'D	APPD.

DRAWN Douglas Lee	DATE 06/19/99	Flowsheet for Small-Tank TPB Process Test System With Three CSTRs in Series 20L CSTR Demo ORNL-CERS-1	Cell B, 4501 CSTR 20L DEMO
CHECKED			
ENGINEER D. D. Lee			FILE 4501 REV A
APPROVED		SCALE	SHEET 1 OF 1 DWG NO. CERS CSTR-DW9

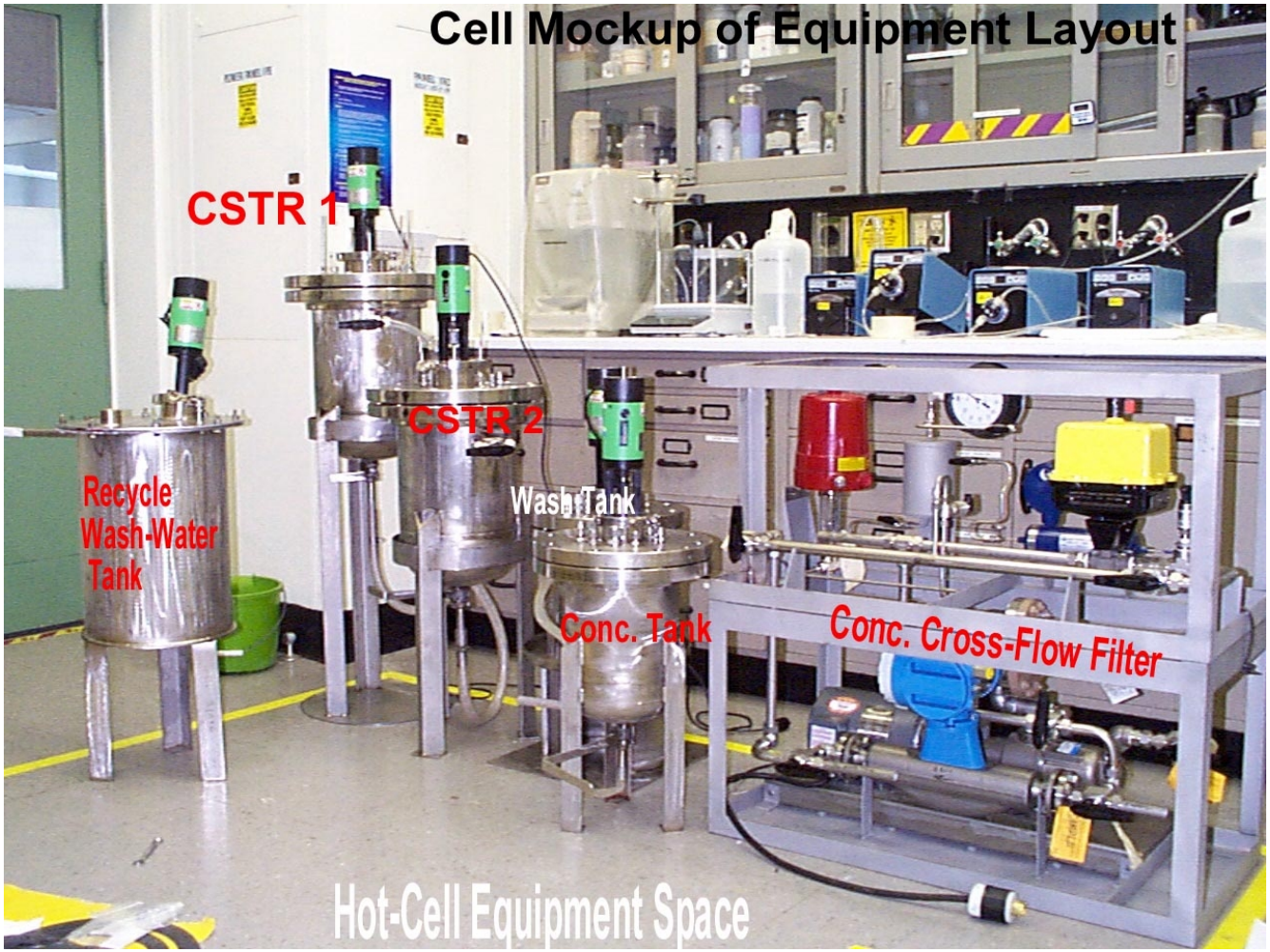
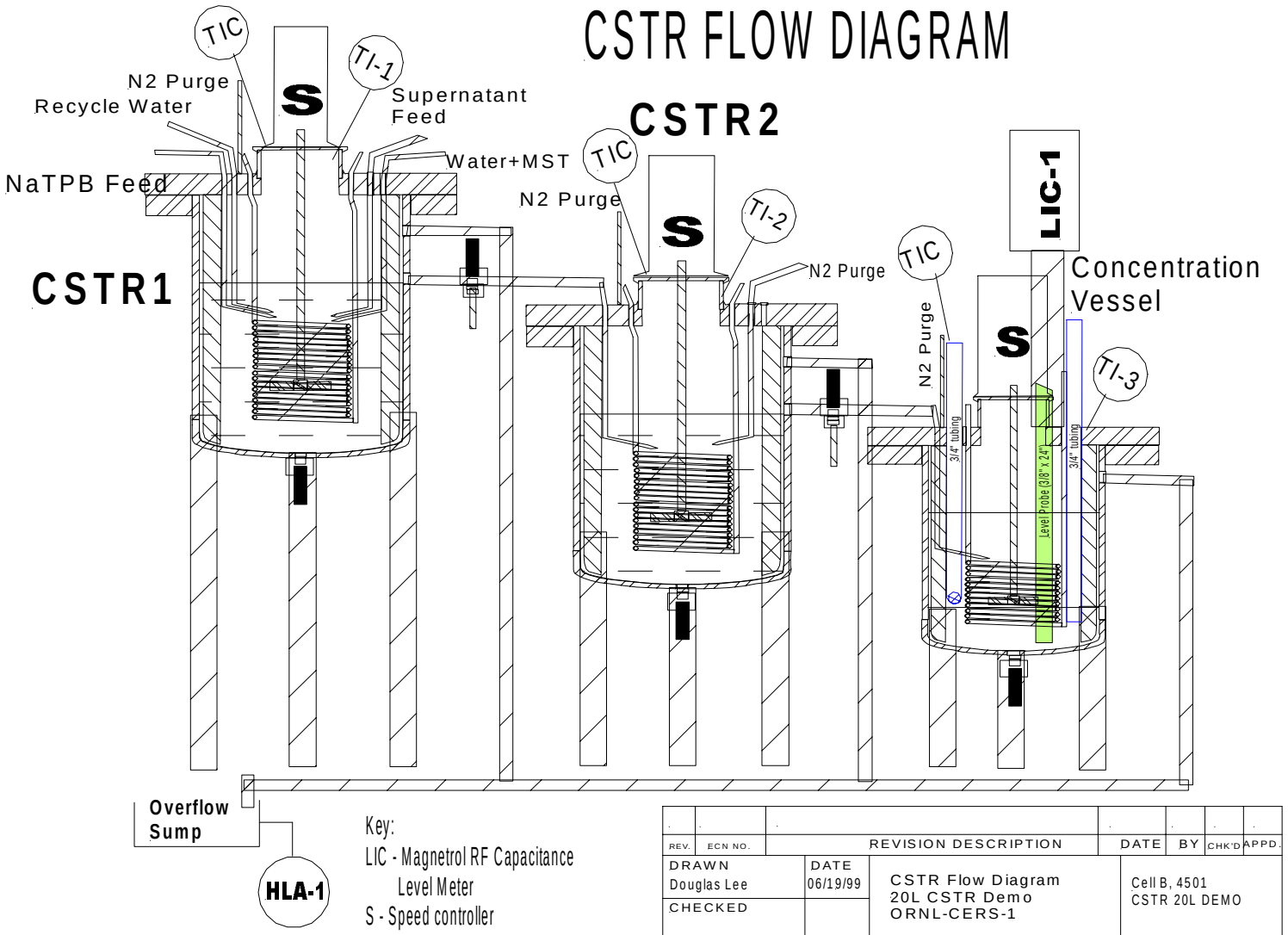
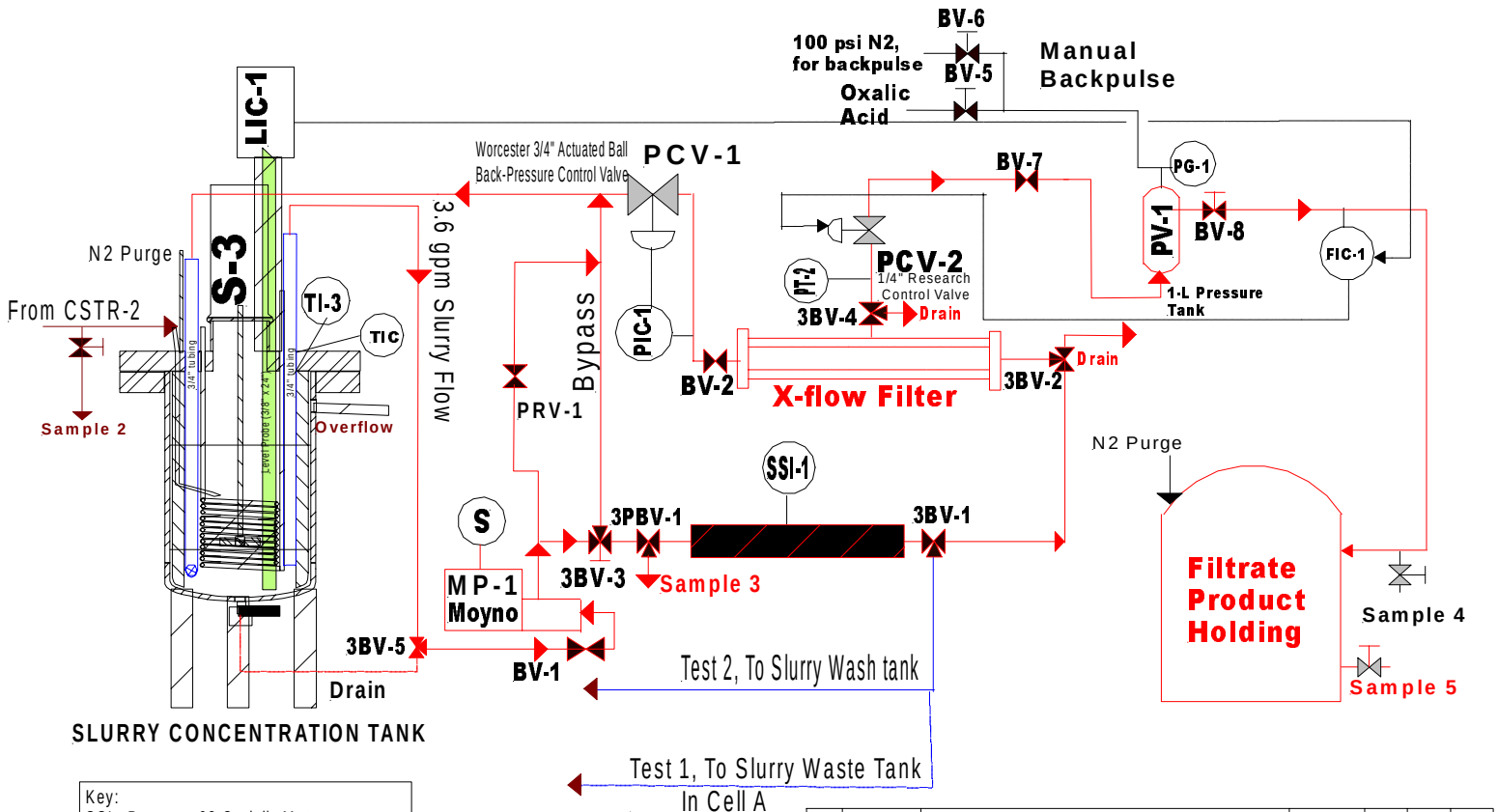


Photo of the equipment before it was installed in the hot cell.


CSTR FLOW DIAGRAM
CSTR1
CSTR2
TIC
TI-1
TI-2
TI-3
S
LIC-1
N2 Purge
Recycle Water
Supernatant Feed
NaTPB Feed
Water+MST
N2 Purge
N2 Purge
N2 Purge
Concentration Vessel
3/4" tubing
Level Probe (3/8" x 2.2")
3/4" tubing
Overflow Sump
HLA-1
Key:
LIC - Magnetrol RF Capacitance Level Meter
S - Speed controller
TI/TIC - Temperature indicator/controller
HLA- High Level Alarm
REV.
ECN NO.
REVISION DESCRIPTION
DATE
BY
CHK'D
APPD.
DRAWN
Douglas Lee
DATE
06/19/99
CHECKED
ENGINEER
D. D. Lee
APPROVED
CSTR Flow Diagram
20L CSTR Demo
ORNL-CERS-1
Cell B, 4501
CSTR 20L DEMO
FILE 4501
REV A
SCALE
SHEET 1 OF 1
DWG NO.CERS CSTR-DW10

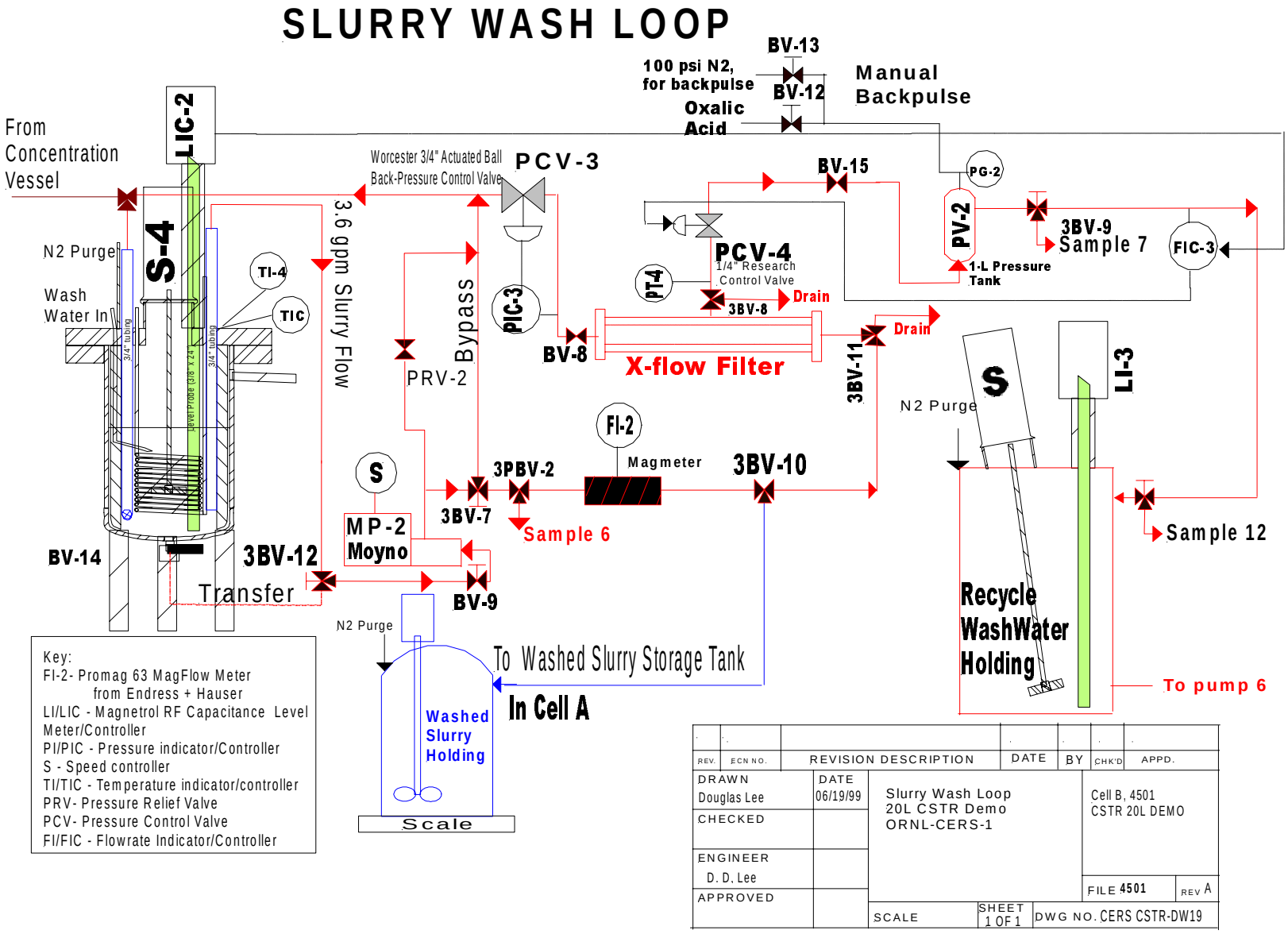
SLURRY CONCENTRATION LOOP

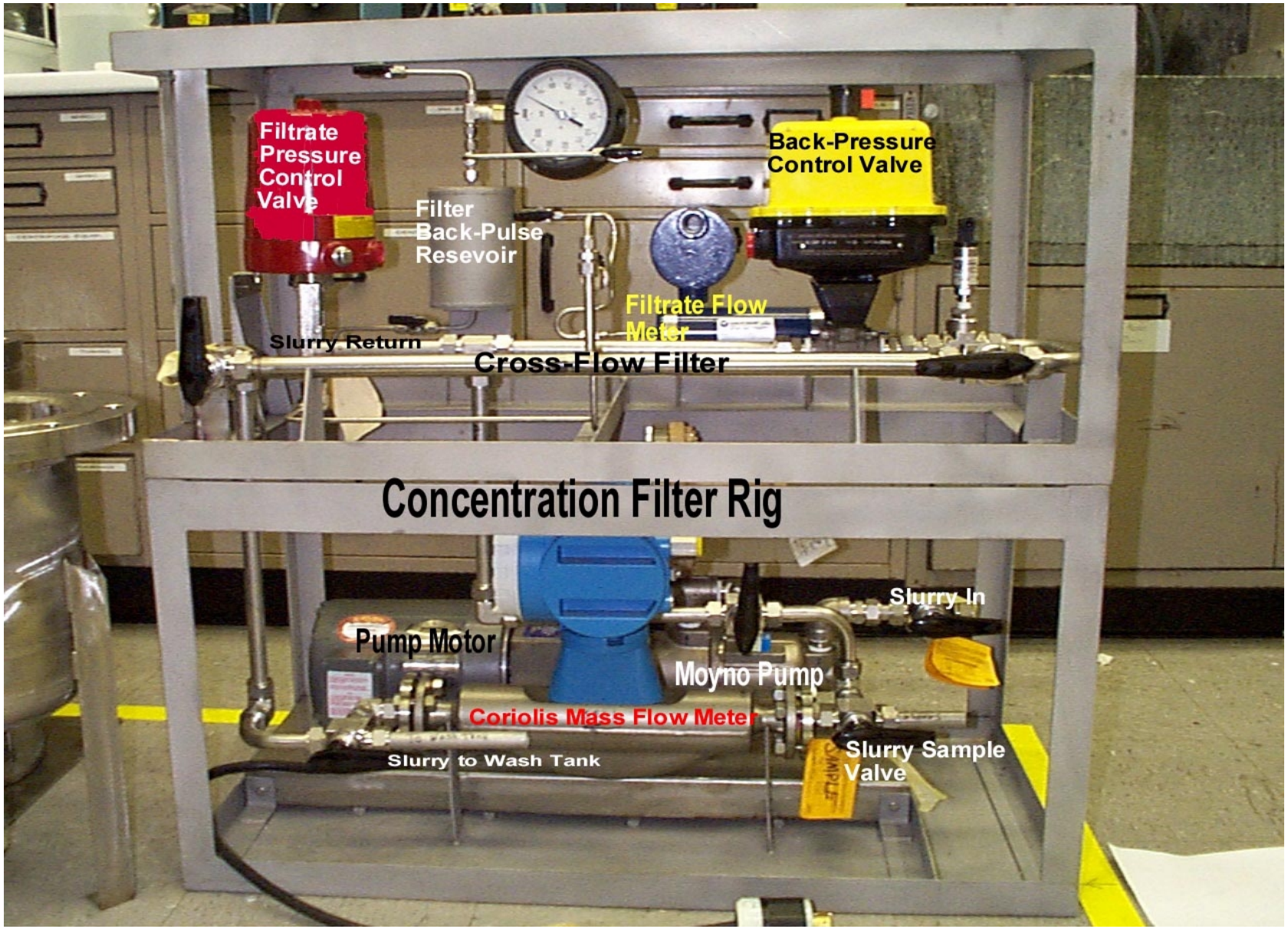


Key:
SSI - Promass 63 Coriolis Meter from Endress + Hauser
LIC - Magnetrol RF Capacitance Level Meter
PI/PIC - Pressure indicator/controller
S - Speed controller
TIC - Temperature indicator/controller
PRV- Pressure Relief Valve
PCV- Pressure Control Valve
FI -1 - Flowrate Indicator- Rheotherm

REV.	ECN NO.	REVISION DESCRIPTION	DATE	BY	CHK'D	APPD.

DRAWN Douglas Lee	DATE 06/19/99	Slurry Concentration Loop 20L CSTR Demo ORNL-CERS-1	Cell B, 4501 CSTR 20L DEMO
CHECKED			
ENGINEER D. D. Lee			FILE 4501 REV A
APPROVED		SCALE	SHEET 1 OF 1 DWG NO. CERS CSTR-DW18


SLURRY WASH LOOP
From Concentration Vessel
LIC-2
S-4
N2 Purge
Wash Water In
TI-4
TIC
BV-14
3BV-12
Transfer
3.6 gpm Slurry Flow
Worcester 3/4" Actuated Ball Back-Pressure Control Valve
PCV-3
PIC-3
Bypass
PRV-2
BV-8
X-flow Filter
PCV-4
1/4" Research Control Valve
PT4
3BV-8
Drain
BV-13
BV-12
100 psi N2, for backpulse
Oxalic Acid
Manual Backpulse
BV-15
PG-2
PV-2
1-L Pressure Tank
3BV-9
Sample 7
FIC-3
3BV-11
Drain
N2 Purge
S
LI-3
Sample 12
Recycle WashWater Holding
To pump 6
FI-2
Magmeter
3BV-10
3PBV-2
3BV-7
Sample 6
MP-2 Moyno
BV-9
N2 Purge
To Washed Slurry Storage Tank
In Cell A
Washed Slurry Holding
Scale
Key:
FI-2- Promag 63 MagFlow Meter from Endress + Hauser
LI/LIC - Magnetrol RF Capacitance Level Meter/Controller
PI/PIC - Pressure indicator/Controller
S - Speed controller
TI/TIC - Temperature indicator/controller
PRV- Pressure Relief Valve
PCV- Pressure Control Valve
FI/FIC - Flowrate Indicator/Controller
REV. ECN NO. REVISION DESCRIPTION DATE BY CHK'D APPD.
DRAWN Douglas Lee
DATE 06/19/99
CHECKED
ENGINEER D. D. Lee
APPROVED
Slurry Wash Loop
20L CSTR Demo
ORNL-CERS-1
Cell B, 4501
CSTR 20L DEMO
FILE 4501
REV A
SCALE
SHEET 1 OF 1
DWG NO. CERS CSTR-DW19


Filtrate Pressure Control Valve
Filter Back-Pulse Resevoir
Back-Pressure Control Valve
Filtrate Flow Meter
Slurry Return
Cross-Flow Filter
Concentration Filter Rig
Slurry In
Pump Motor
Moyno Pump
Coriolis Mass Flow Meter
Slurry to Wash Tank
Slurry Sample Valve

Design of CSTR 20-L Vessels and Associated Equipment Workaid

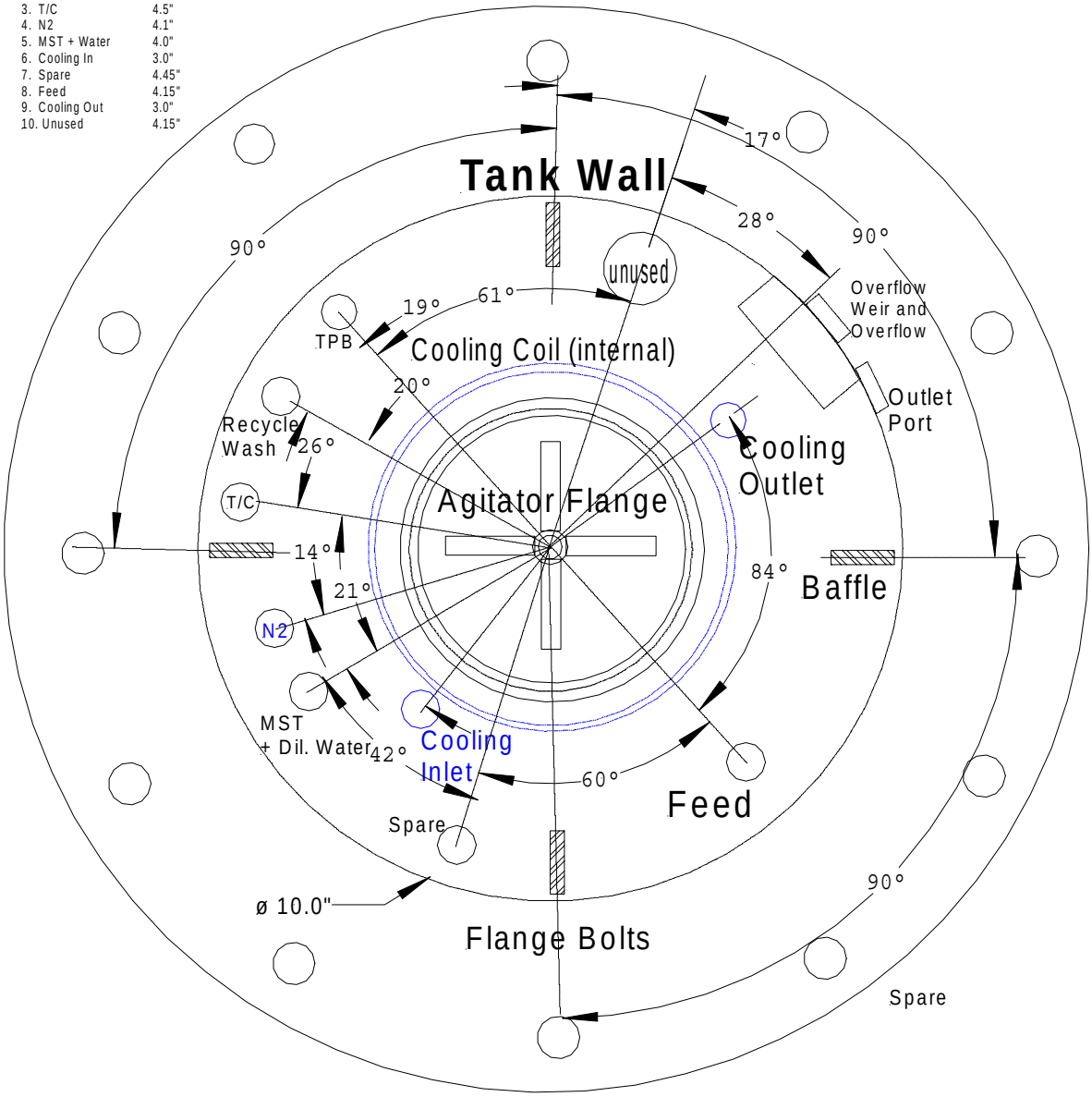
The following drawings were produced to give to the fabricators for construction of the vessels. They have been modified to include the as-built modifications. Some of the changes shown were made to allow better manipulator access. Others, such as the position of the slurry outlet on the CSTRs, were necessitated by the results of volume calibration of the vessels. The port position was changed to allow the vessel to contain the required 15L of slurry during operation for 10.4 H of residence time. The stilling wells were not added as originally drawn. Weirs were added to the CSTRs, the concentration vessel, and wash vessel to allow the tanks to reach a higher volume before they overflowed from the overflow protection fitting. The overflow was lower on the vessel than planned due the thickness of the slip-on flange that was welded in place.

The vessels were constructed with spherical pipe caps with slightly less internal diameter than the eliptical head used in the drawings. The spherical heads resulted in about a 0.6L loss of volume for the same height from the bottom of the vessel compared to the eliptical head for the 10” CSTRs.

Notes--Opening distances from center of flange to center of opening:

1.	TPB	4.45"
2.	Recycle Wash	4.35"
3.	T/C	4.5"
4.	N2	4.1"
5.	MST + Water	4.0"
6.	Cooling In	3.0"
7.	Spare	4.45"
8.	Feed	4.15"
9.	Cooling Out	3.0"
10.	Unused	4.15"

CSTR 1 Top Flange



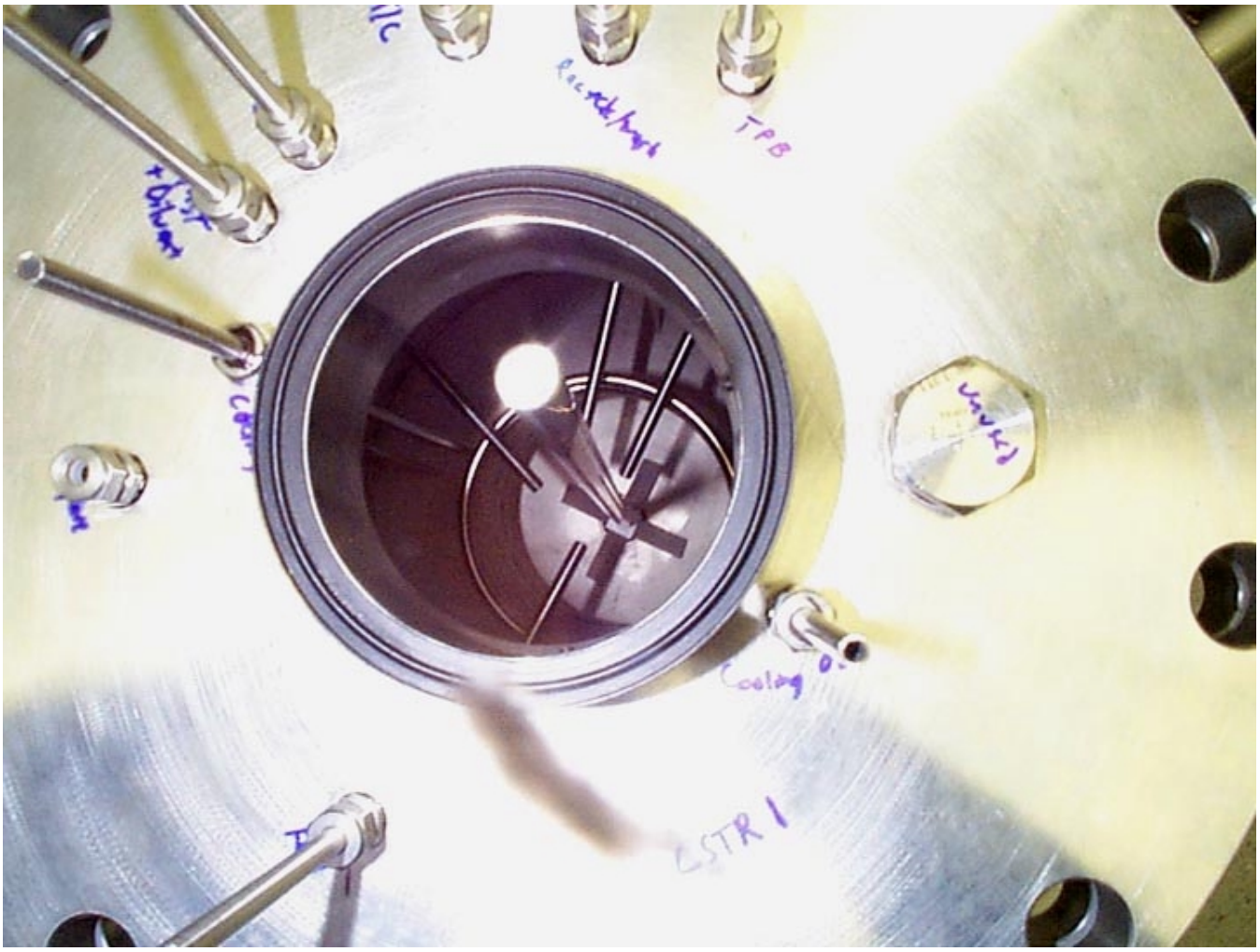
Notes:
1. Holes for T/C, N2, cooling in and out, TPB, MST+Dil Water, recycle wash, Feed, and spare, are 1/4" NPT adapters to 1/4" tube fittings drilled to allow tube to pass through.
2. Unused is 1" NPT drilled and plugged.
3. Baffles at 90 degrees from each other

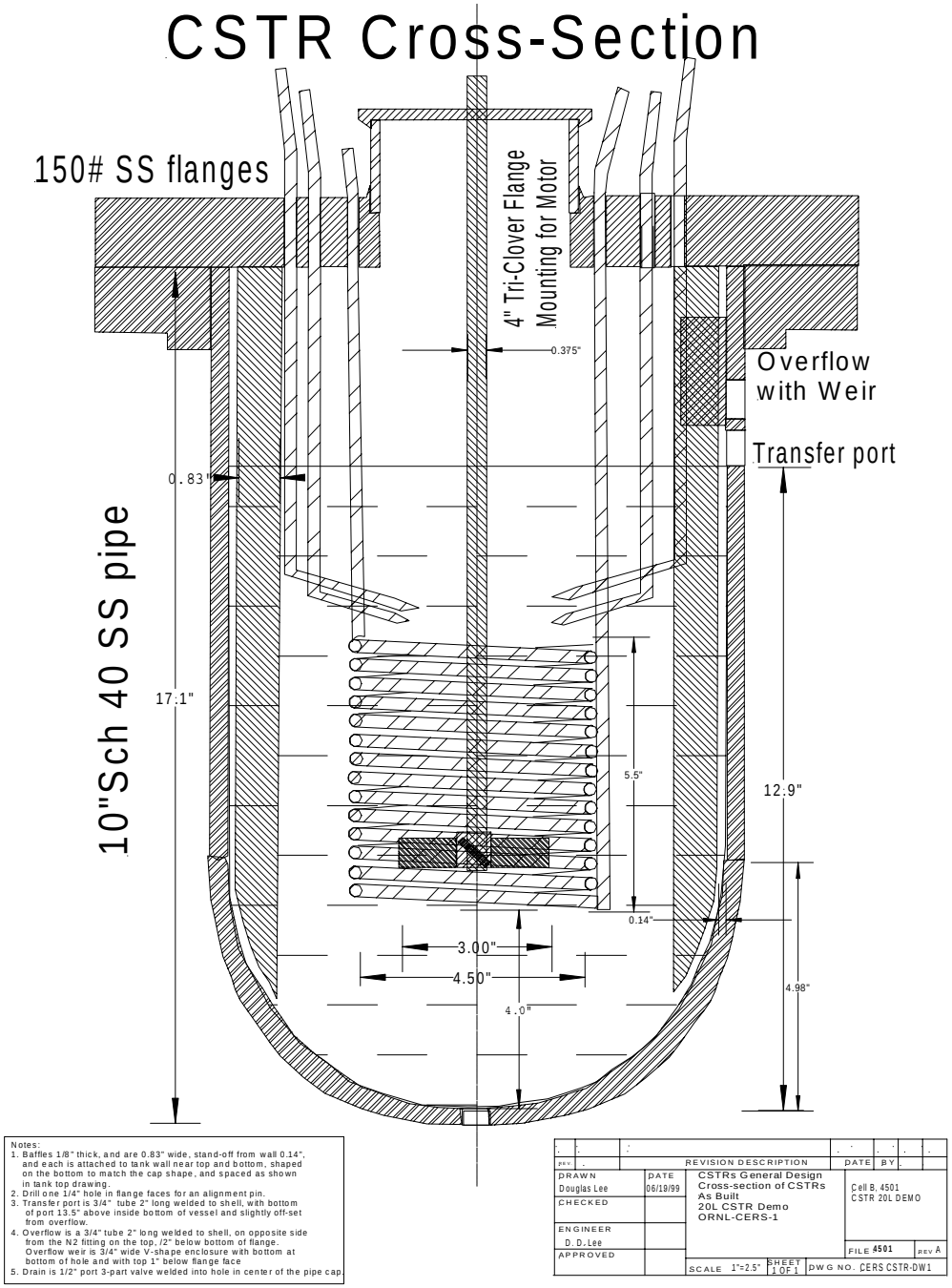
REV.	ECN NO.	REVISION DESCRIPTION	DATE	BY	CHK'D	APPD.

DRAWN Douglas Lee	DATE 06/19/99	CSTR 1 Top Flange 20L CSTR Demo ORNL-CERS-1	Cell B, 4501 CSTR 20L DEMO
CHECKED			
ENGINEER D. D. Lee			FILE 4501 REV A
APPROVED		SCALE 1"=2.5" SHEET 1 OF 1	DWG NO. CERS CSTR-DW2

Top view of CSTR 1 Flange through the agitator flange opening. The picture shows the relative positions of the agitator nd feed tubes

.

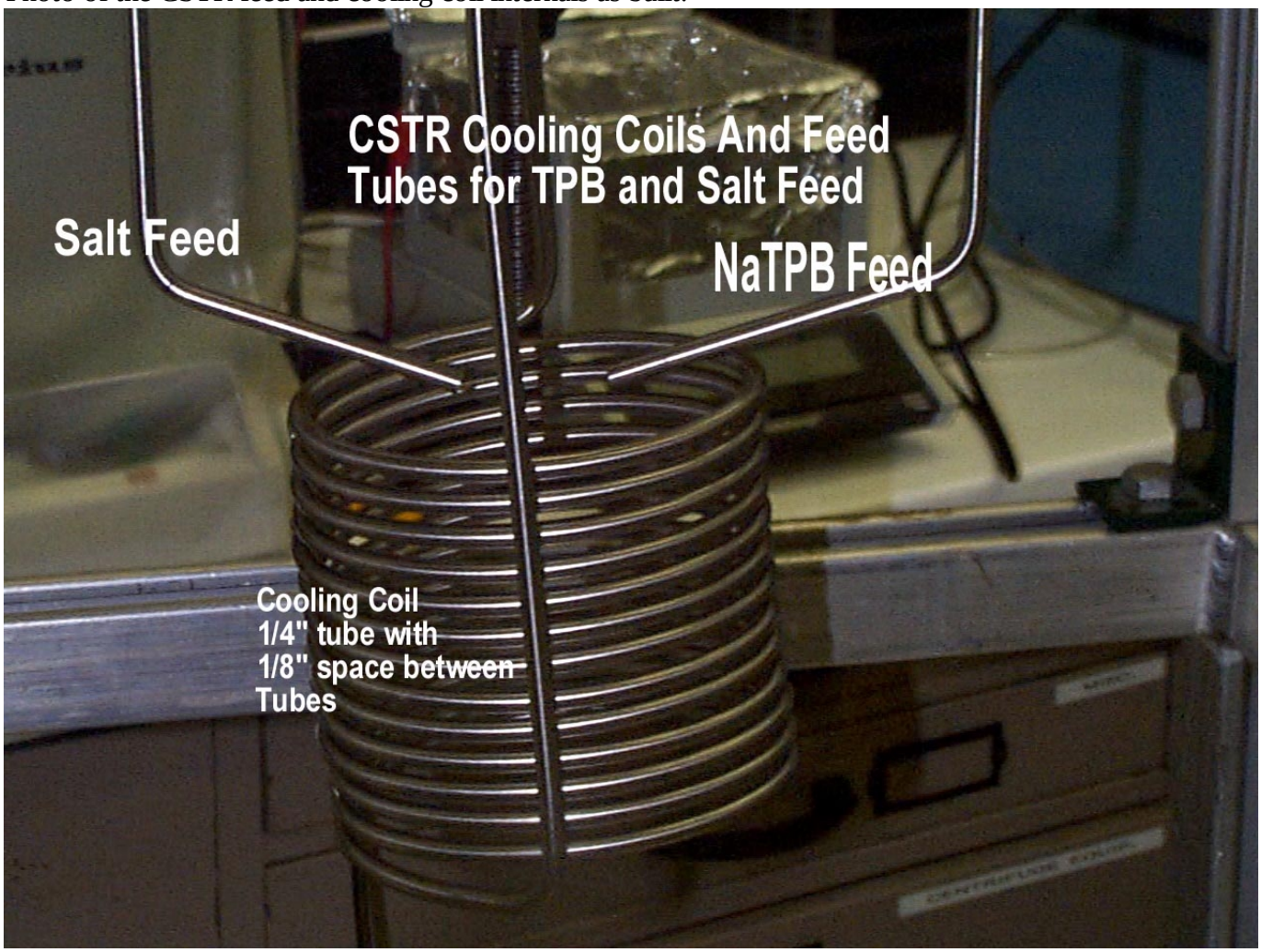




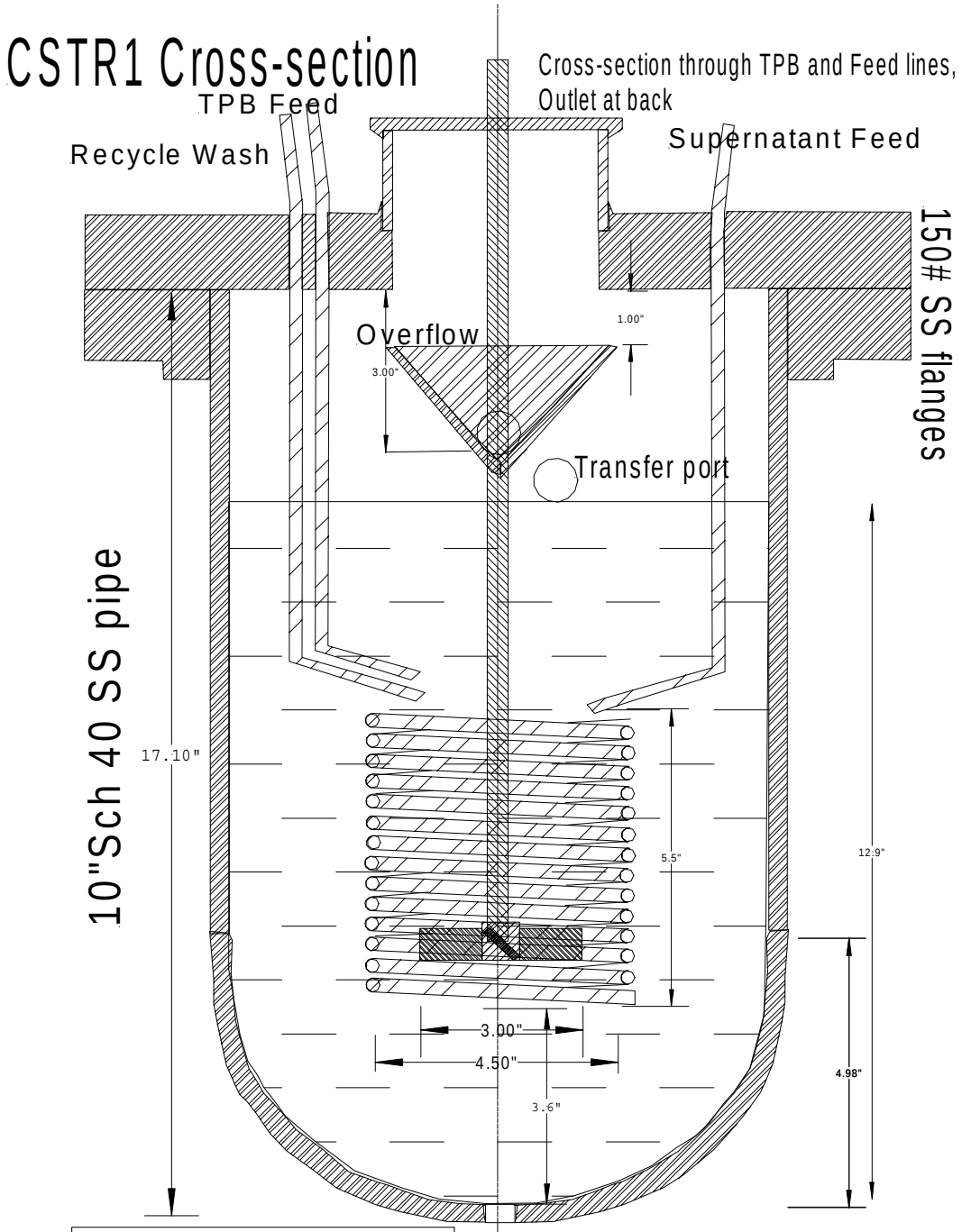
Top view of CSTR 1 Flange through the agitator flange opening. The picture shows the relative positions of the agitator nd feed tubes

.

Photo of the CSTR feed and cooling coil internals as built.



Design of CSTR 20-L Vessels and Associated Equipment Workaid



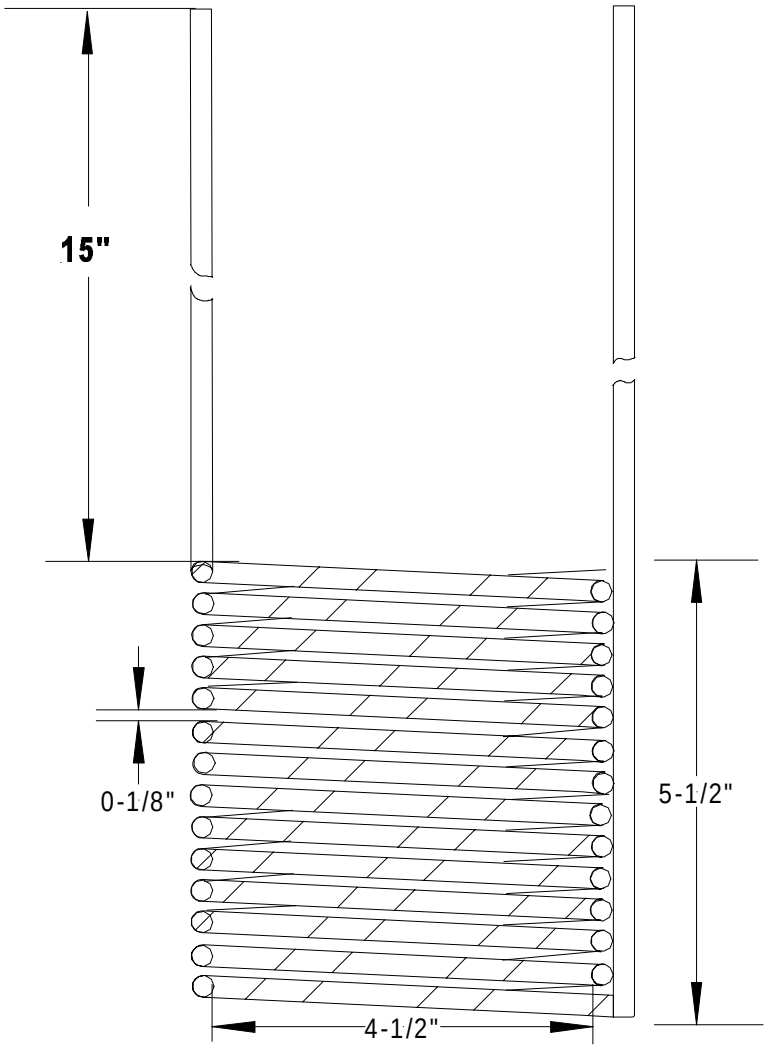
Notes:
1. Baffles 1/8" thick, and are 0.83" wide, stand-off from wall 0.14", and each is attached to tank wall near top and bottom, shaped on the bottom to match the cap shape, and spaced as shown in tank top drawing.
2. Drill one 1/4" hole in flange faces for an alignment pin.
3. Transfer port is 3/4" tube 2" long welded to shell, with bottom of port 13.5" above inside bottom of vessel and slightly off-set from overflow.
4. Overflow is a 3/4" tube 2" long welded to shell, on opposite side from the N2 fitting on the top, /2" below bottom of flange. Overflow weir is 3/4" wide V-shape enclosure with bottom at bottom of hole and with top 1" below flange face
5. Drain is 1/2" port 3-part valve welded into hole in center of the pipe cap.

REV.	REVISION DESCRIPTION	DATE	BY	CHK'D	APPD.

DRAWN Douglas Lee	DATE 06/19/99	CSTR1 Cross-section of Vessel Through Feed and and TPB inlets, as built 20L CSTR Demo ORNL-CERS-1	Cell B, 4501 CSTR 20L DEMO
CHECKED			
ENGINEER D. D. Lee			FILE 4501 REV A
APPROVED		SCALE 1"=2.5" SHEET 1 OF 1	DWG NO. CERS CSTR-DW17

Photos of the CSTR slurry outlet and overflow weir and their placement in the CSTRs.

CSTR1, 2, and 3 COILS

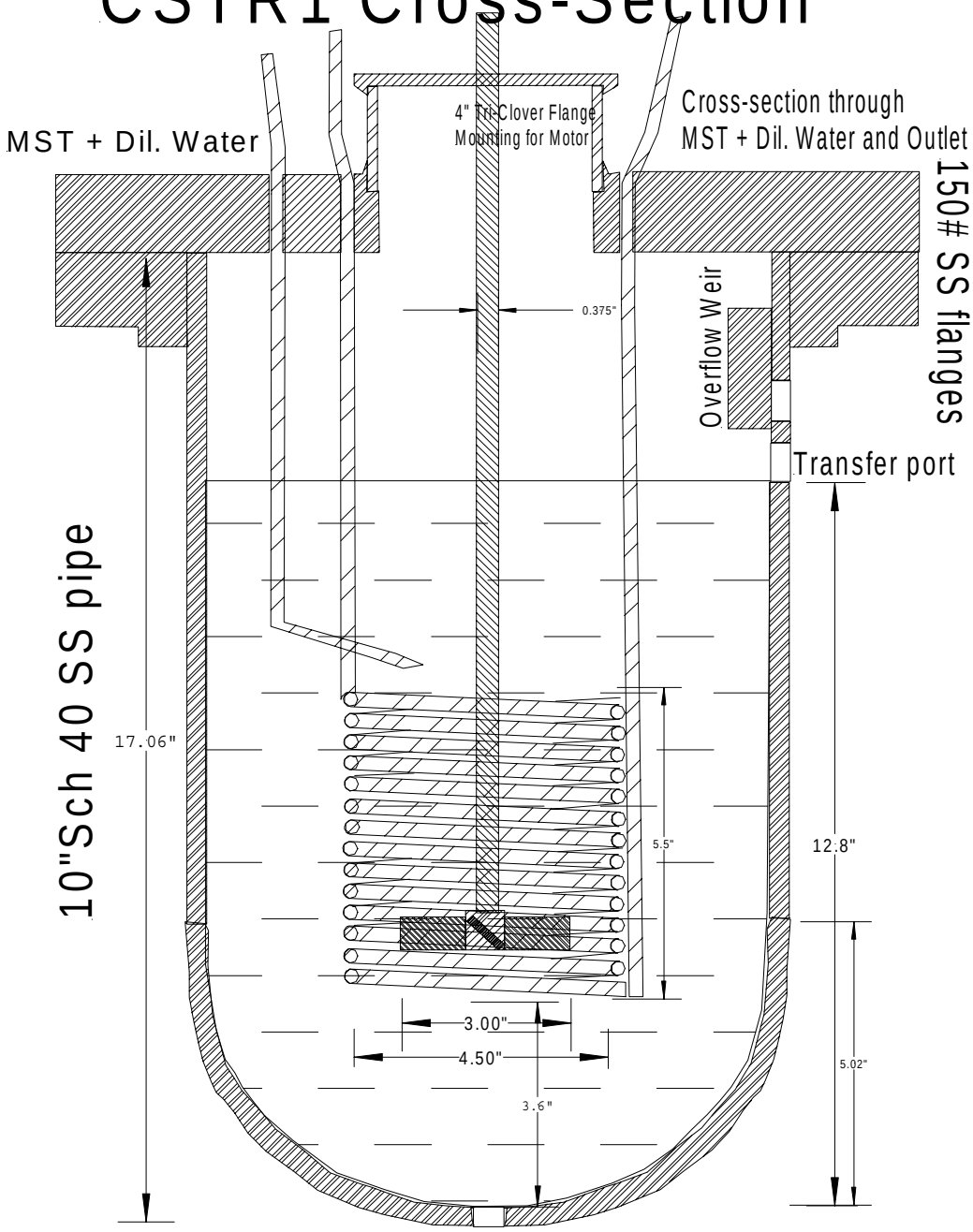


Notes: Use 1/4" SS tubing, thin wall, with the length for the top of the coil to the top of the stub 15" or more. Roll the tubing so the inner diameter is 4 1/2" with 1/8" space between tubes in the coil. This should be about 13 1/2 coils for the 5 1/2" of coils.

REV.	ECN NO.	REVISION DESCRIPTION	DATE	BY	CHK'D	APPD.

DRAWN Douglas Lee	DATE 06/19/99	CSTR1,2, and 3 Coils 20L CSTR Demo ORNL-CERS-1	Cell B, 4501 CSTR 20L DEMO
CHECKED			
ENGINEER D. D. Lee			FILE 4501 REV A
APPROVED		SCALE 1"=2.5" SHEET 1 OF 1	DWG NO. CERS CSTR-DW20

CSTR1 Cross-Section



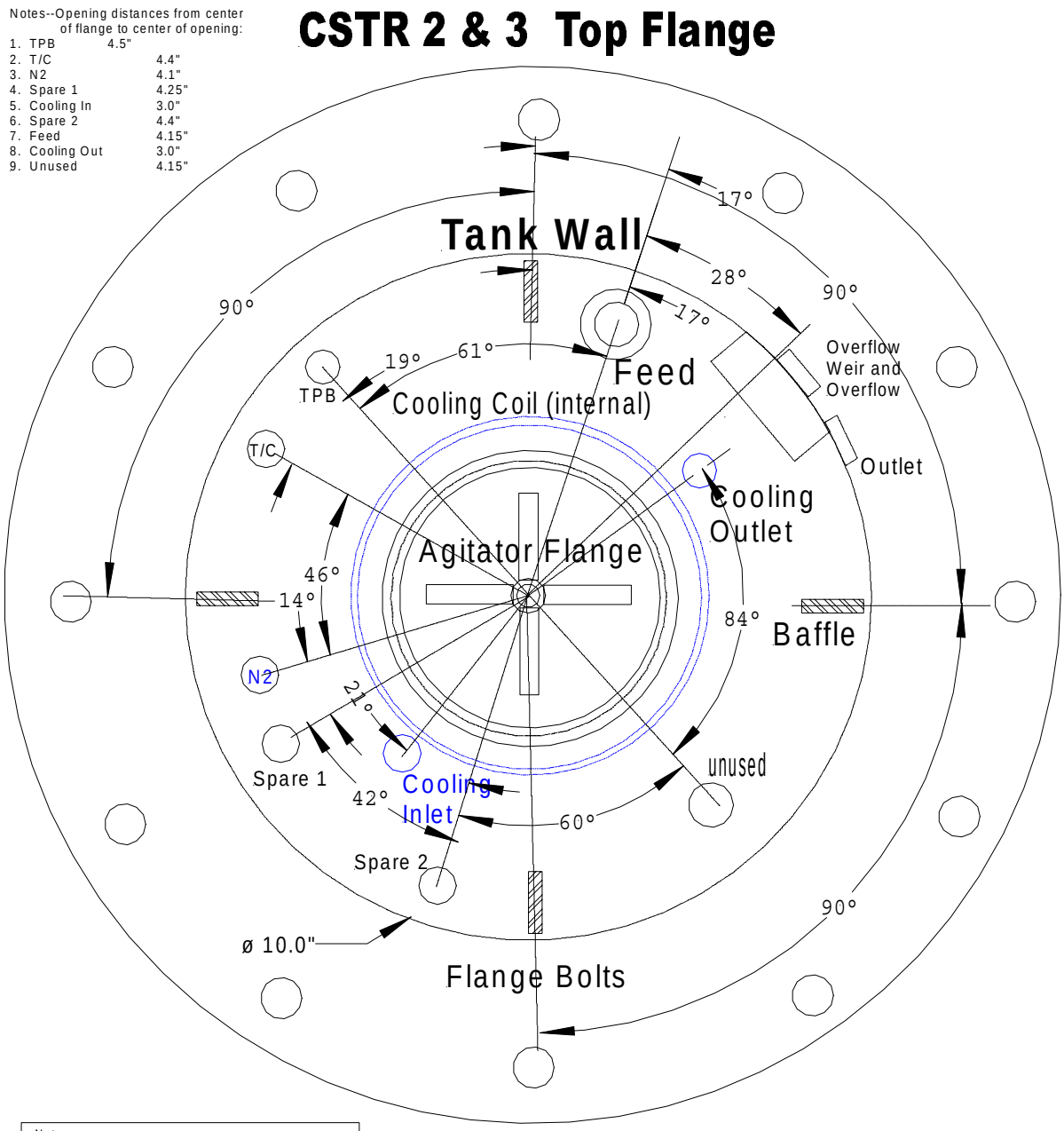
Notes:
1. Baffles 1/8" thick, and are 0.83" wide, stand-off from wall 0.14", and each is attached to tank wall near top and bottom, shaped on the bottom to match the cap shape, and spaced as shown in tank top drawing.
2. Drill one 1/4" hole in flange faces for an alignment pin.
3. Transfer port is 3/4" tube 2" long welded to shell, with bottom of port 13.5" above inside bottom of vessel and slightly off-set from overflow.
4. Overflow is a 3/4" tube 2" long welded to shell, on opposite side from the N2 fitting on the top, 1/2" below bottom of flange. Overflow weir is 3/4" wide V-shape enclosure with bottom at bottom of hole and with top 1" below flange face
5. Drain is 1/2" port 3-part valve welded into hole in center of the pipe cap.

REV.	REVISION DESCRIPTION	DATE	BY	CHK'D	APPD.

DRAWN Douglas Lee	DATE 06/19/99	CSTR1 Cross-section of Vessel Through outlets 20L CSTR Demo As Built ORNL-CERS-1	Cell B, 4501 CSTR 20L DEMO
CHECKED			
ENGINEER D. D. Lee			FILE 4501 REV A
APPROVED		SCALE 1"=2.5" SHEET 1 OF 1	DWG NO. CERS CSTR-DW16

Notes--Opening distances from center of flange to center of opening:

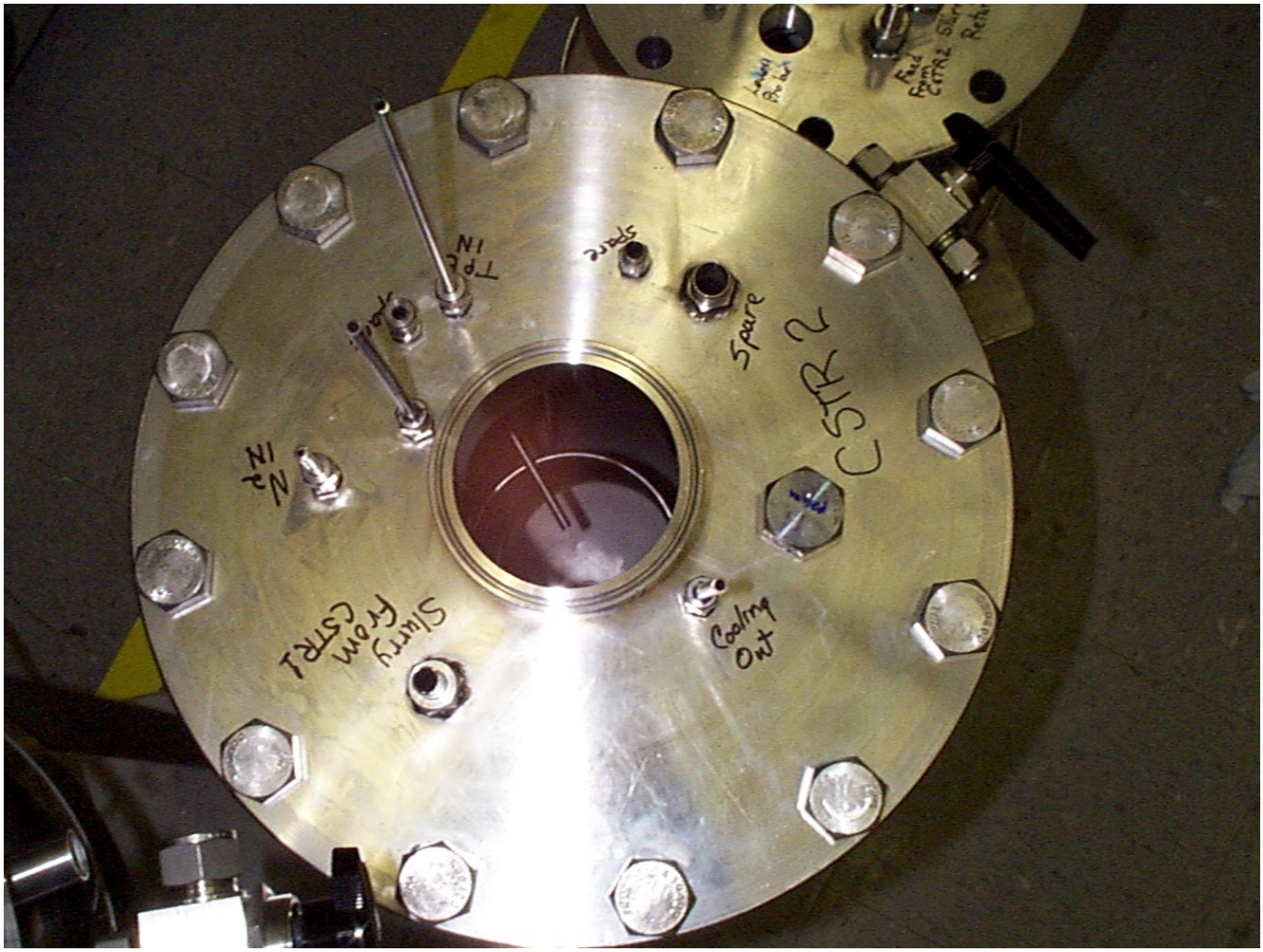
1.	TPB	4.5"
2.	T/C	4.4"
3.	N2	4.1"
4.	Spare 1	4.25"
5.	Cooling In	3.0"
6.	Spare 2	4.4"
7.	Feed	4.15"
8.	Cooling Out	3.0"
9.	Unused	4.15"



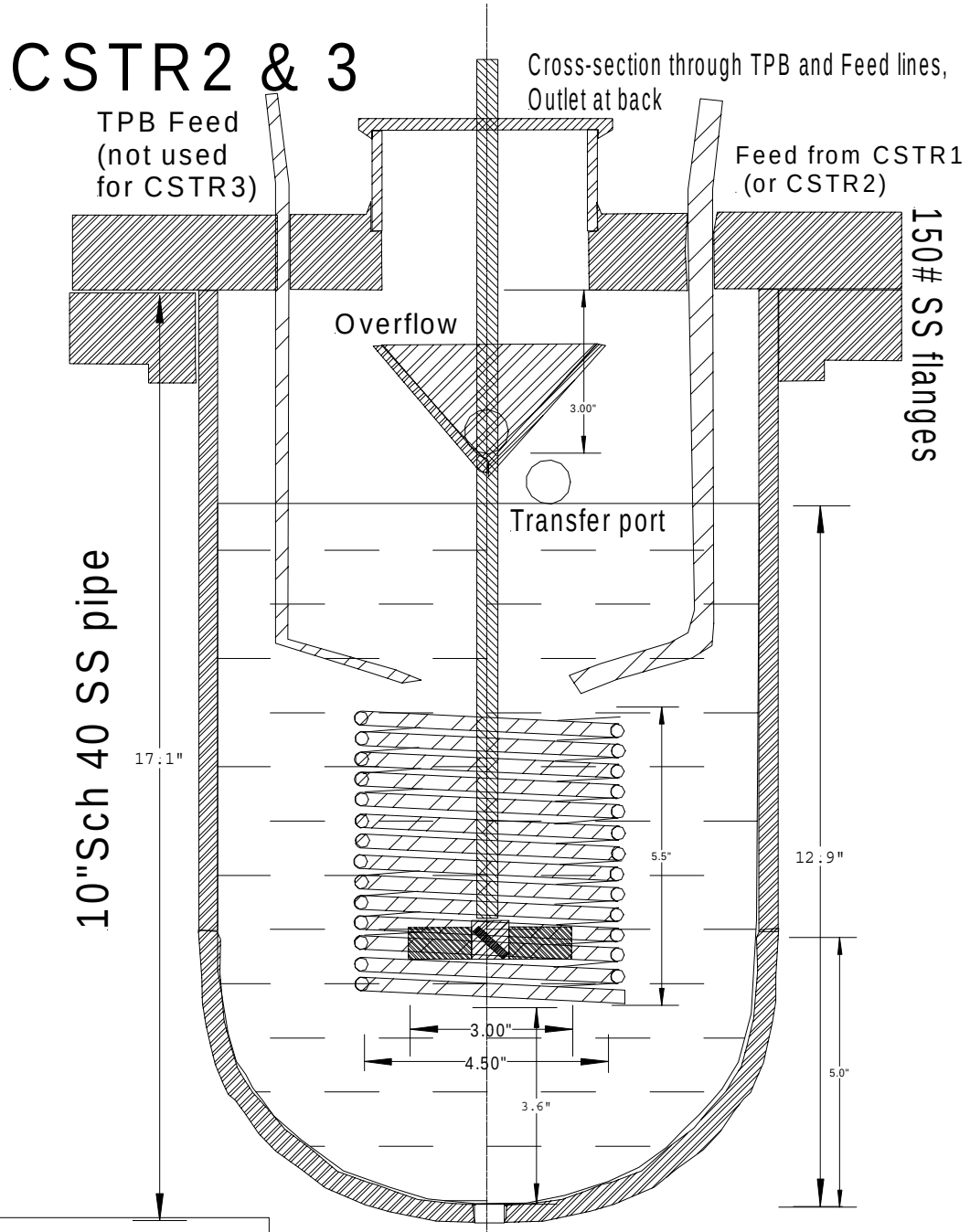
Notes:
1. Holes for T/C, N2, cooling in and out, TPB, and spares are 1/4" NPT adapters to 1/4" tube fittings drilled to allow tube to pass through using teflon ferrules.
2. Unused is 1" NPT drilled and plugged.
3. Feed is 1/2" tube fitting drilled out for tube to pass through and welded to flange.

REV.	ECN NO.	REVISION DESCRIPTION	DATE	BY	CHK'D	APPD.

DRAWN Douglas Lee	DATE 06/19/99	CSTR2 and 3 Top Flange 20L CSTR Demo ORNL-CERS-1	Cell B, 4501 CSTR 20L DEMO
CHECKED			
ENGINEER D. D. Lee			FILE 4501 REV A
APPROVED		SCALE 1"=2.5" SHEET 1 OF 1	DWG NO. CERS CSTR-DW3



CSTR2 Flange as built ready to install in the hot cell



Notes:
1. Baffles 1/8" thick, and are 0.83" wide, stand-off from wall 0.14", and each is attached to tank wall near top and bottom, shaped on the bottom to match the cap shape, and spaced as shown in tank top drawing.
2. Drill one 1/4" hole in flange faces for an alignment pin.
3. Transfer port is 3/4" tube, 2" long welded to shell. ,
4. Overflow is a 3/4" tube 2" long welded to shell, on opposite side from the N2 fitting on the top, 1/2" below bottom of flange.
5. Drain is 1/2" port 3-part valve welded into hole in center of the pipe cap.
6. Feed from CSTR 1 or 2 is 1/2" tube fitting, drilled through and welded to top flange.

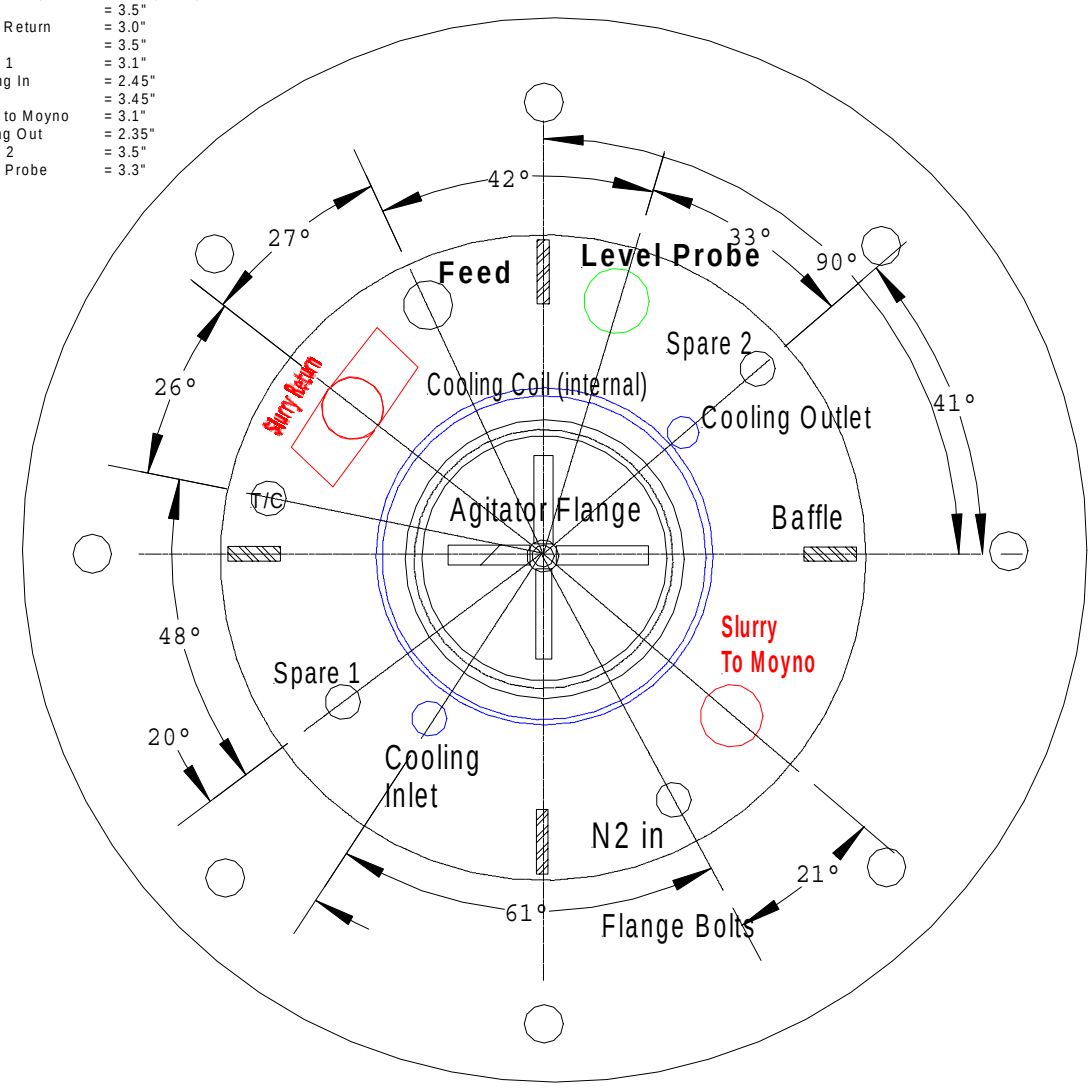
REV.	ECN NO.	REVISION DESCRIPTION	DATE	BY	APPD.

DRAWN Douglas Lee	DATE 06/19/99	CSTR2 and 3 Cross-section of Vessel 20L CSTR Demo (As Built) ORNL-CERS-1	Cell B, 4501 CSTR 20L DEMO
CHECKED			
ENGINEER D. D. Lee			FILE 4501 REV A
APPROVED		SCALE 1"=2.5" SHEET 1 OF 1	DWG NO. CERS CSTR-DW11

Concentration Vessel Top Flange

Notes--Opening distances from center of flange to center of opening:

1. Feed = 3.5"
2. Slurry Return = 3.0"
3. T/C = 3.5"
4. Spare 1 = 3.1"
5. Cooling In = 2.45"
6. N2 In = 3.45"
7. Slurry to Moyno = 3.1"
8. Cooling Out = 2.35"
9. Spare 2 = 3.5"
10. Level Probe = 3.3"

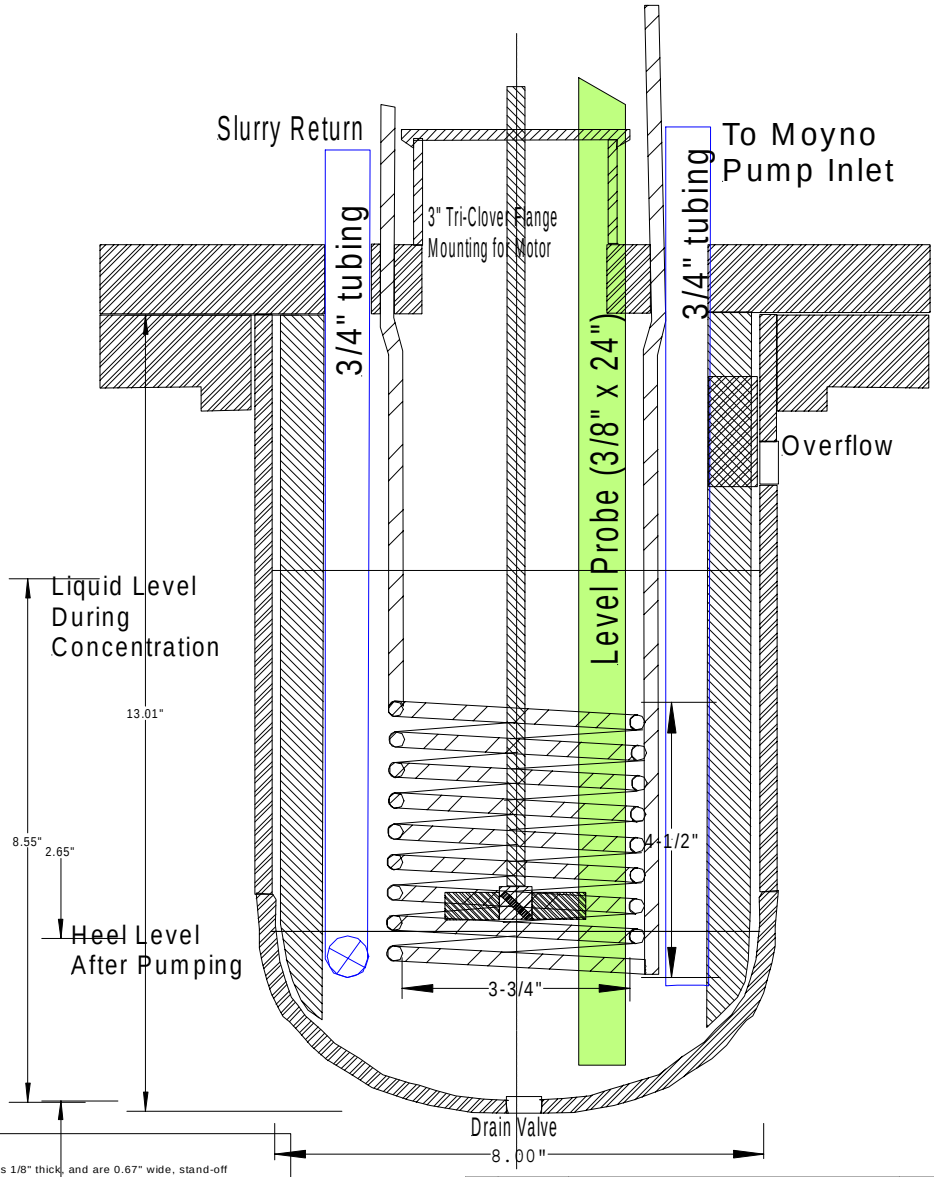


Notes:
1. Holes for T/C, N2, cooling in and out, and spares, are 1/4" NPT adapters to 1/4" tube fittings drilled to allow tube to pass through.
2. Feed is 1/2" tube fitting welded in place, drilled to allow tube to pass through
3. Level Probe is 1" NPT .
4. To Moyno and Slurry Return are holes for 3/4" tube fittings, with fittings drilled to allow tube to pass through.
5. Baffles at 90 degrees from each other

REV.	ECN NO.	REVISION DESCRIPTION	DATE	BY	CHK'D	APPD.

DRAWN Douglas Lee	DATE 06/19/99	Concentration Vessel Top Flange 20L CSTR Demo ORNL-CERS-1	Cell B, 4501 CSTR 20L DEMO
CHECKED			
ENGINEER D. D. Lee			FILE 4501 REV A
APPROVED		SCALE 1"=2.5" SHEET 1 OF 1	DWG NO. CERS CSTR-DW6

Concentration Vessel Cross-Section

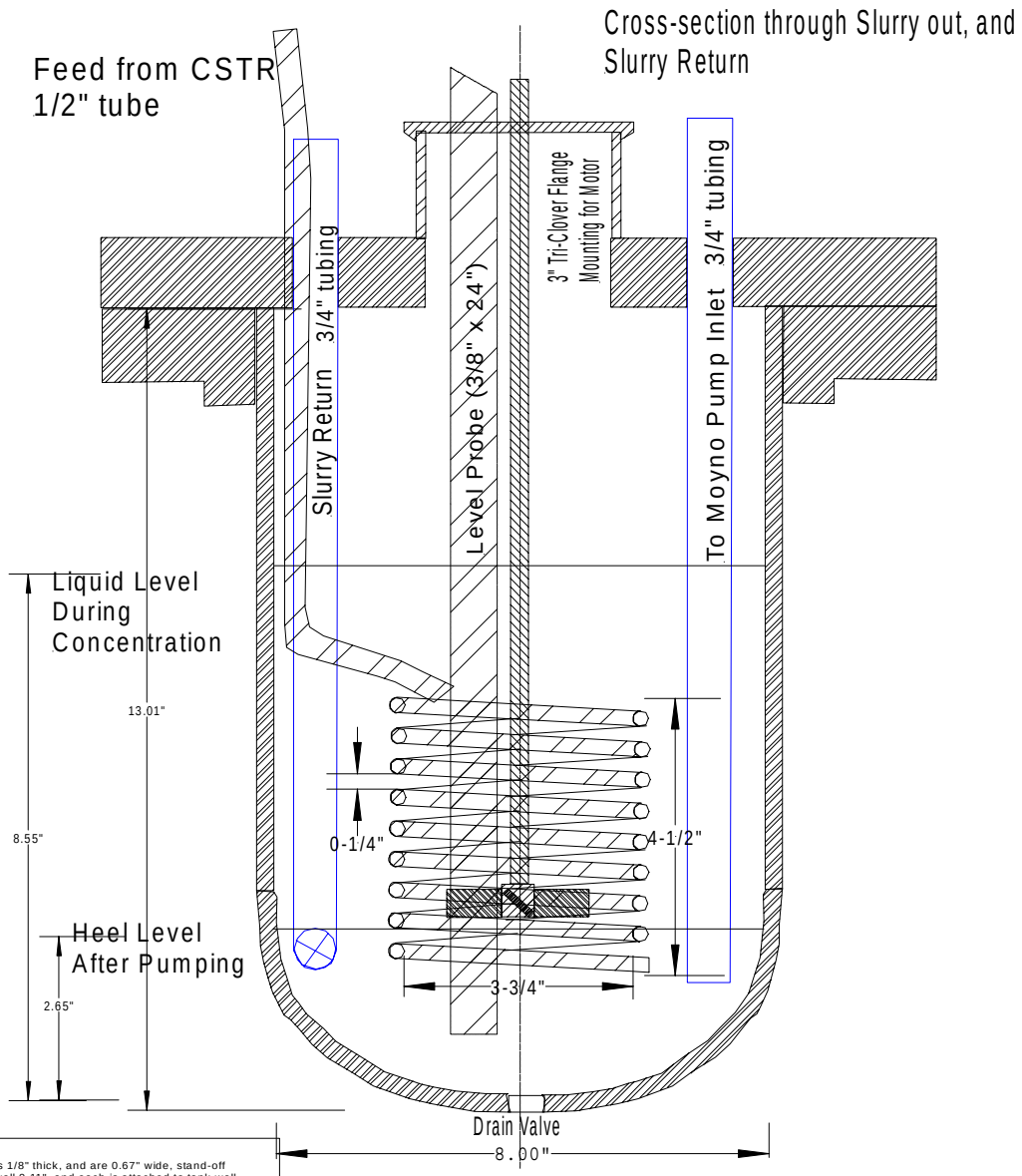


Notes:
1. Baffles 1/8" thick, and are 0.67" wide, stand-off from wall 0.11", and each is attached to tank wall near top and bottom, shaped on the bottom to match the cap shape, and spaced as shown in tank top drawing.
2. Feed tube is 1/2" tube through a 1/2'tube fitting welded to the flange and drilled through.
3. Drill one 1/4" hole in flange faces for an alignment pin.
4. To Moyno tube is 3/4" and reaches to 1" from bottom; Slurry Return tube is 3/4" with TEE at bottom with TEE parallel to tank wall, 2" from tank bottom.
5. Overflow is a 3/4" tube 2" long welded to shell, 180 degrees from the N2 fitting on the top, 1/2" below bottom of flange. Overflow weir is 3/4" wide V-shape enclosure with bottom at bottom of hole and with top 1" below flange face
6. Drain is 1/2" port 3-part valve welded into hole in center of the pipe cap.

REV.	ECN NO.	REVISION DESCRIPTION	DATE	BY	CHK'D	APPD.

DRAWN Douglas Lee	DATE 06/19/99	Concentration Vessel Cross-section General View 20L CSTR Demo (as Built) ORNL-CERS-1	Cell B, 4501 CSTR 20L DEMO
CHECKED			
ENGINEER D. D. Lee			FILE 4501 REV A
APPROVED		SCALE 1"=2.5" SHEET 1 OF 1	DWG NO. CERS CSTR-DW4

Concentration Vessel Cross-Section

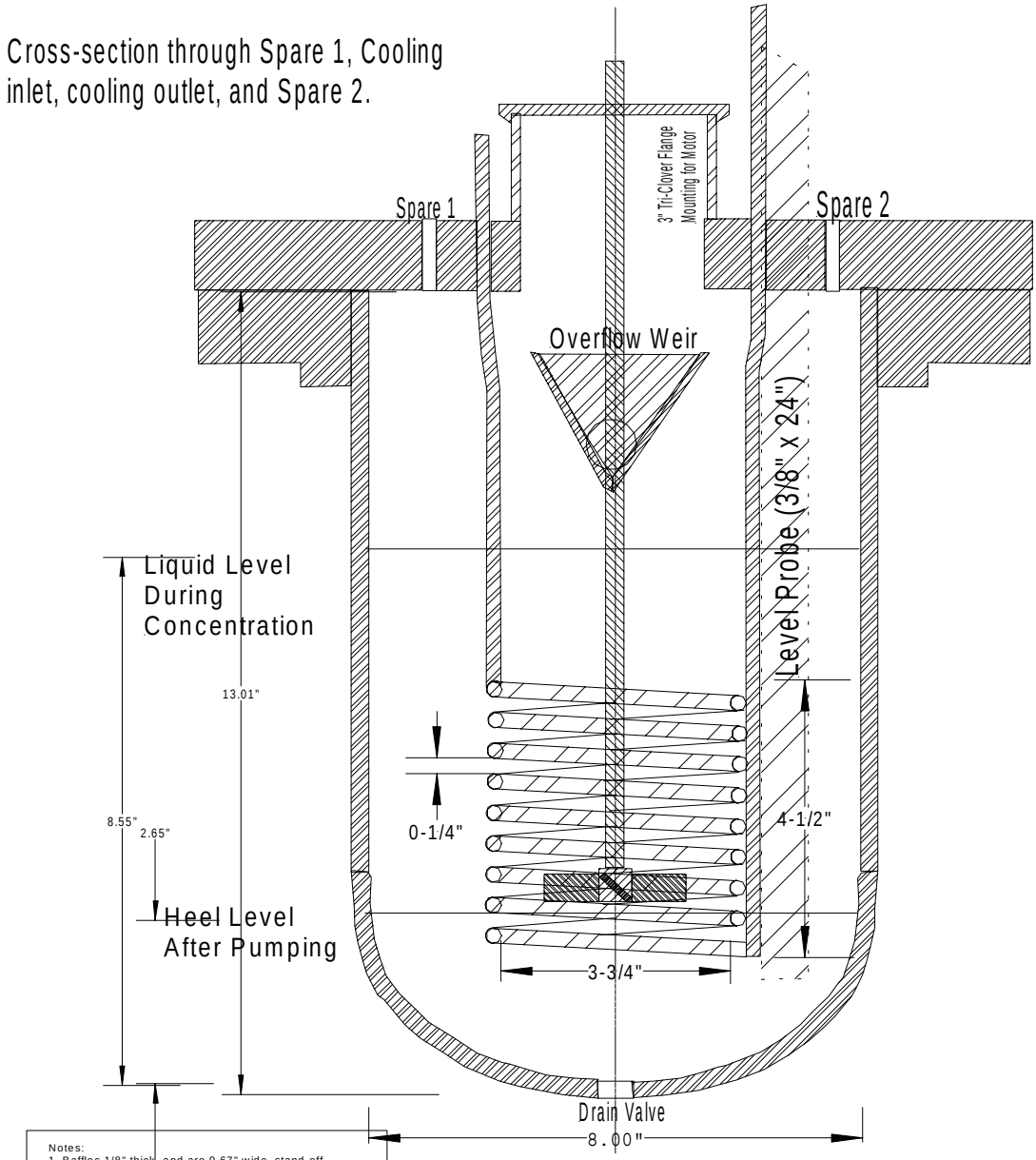


Notes:
1. Baffles 1/8" thick, and are 0.67" wide, stand-off from wall 0.11", and each is attached to tank wall near top and bottom, shaped on the bottom to match the cap shape, and spaced as shown in tank top drawing.
2. Feed tube is 1/2" tube through a 1/2'tube fitting welded to the flange and drilled through.
3. Drill one 1/4" hole in flange faces for an alignment pin.
4. To Moyno tube is 3/4" and reaches to 1" from bottom; Slurry Return tube is 3/4" with TEE at bottom with TEE parallel to tank wall, 2" from tank bottom.
5. Overflow is a 3/4" tube 2" long welded to shell, opposite from the N2 fitting on the top, 1/2" below bottom of flange. Overflow weir is 3/4" wide V-shape enclosure with bottom at bottom of hole and with top 1" below flange face
6. Drain is 1/2" port 3-part valve welded into hole in center of the pipe cap.

REV.	ECN NO.	REVISION DESCRIPTION	DATE	BY	CHK'D	APPD.

DRAWN Douglas Lee	DATE 06/19/99	Concentration Vessel Cross-section Through Slurry in and out 20L CSTR Demo (As Built) ORNL-CERS-1	Cell B, 4501 CSTR 20L DEMO
CHECKED			
ENGINEER D. D. Lee			FILE 4501 REV A
APPROVED		SCALE 1"=2.5" SHEET 1 OF 1	DWG NO. CERS CSTR-DW14

Concentration Vessel Cross-Section

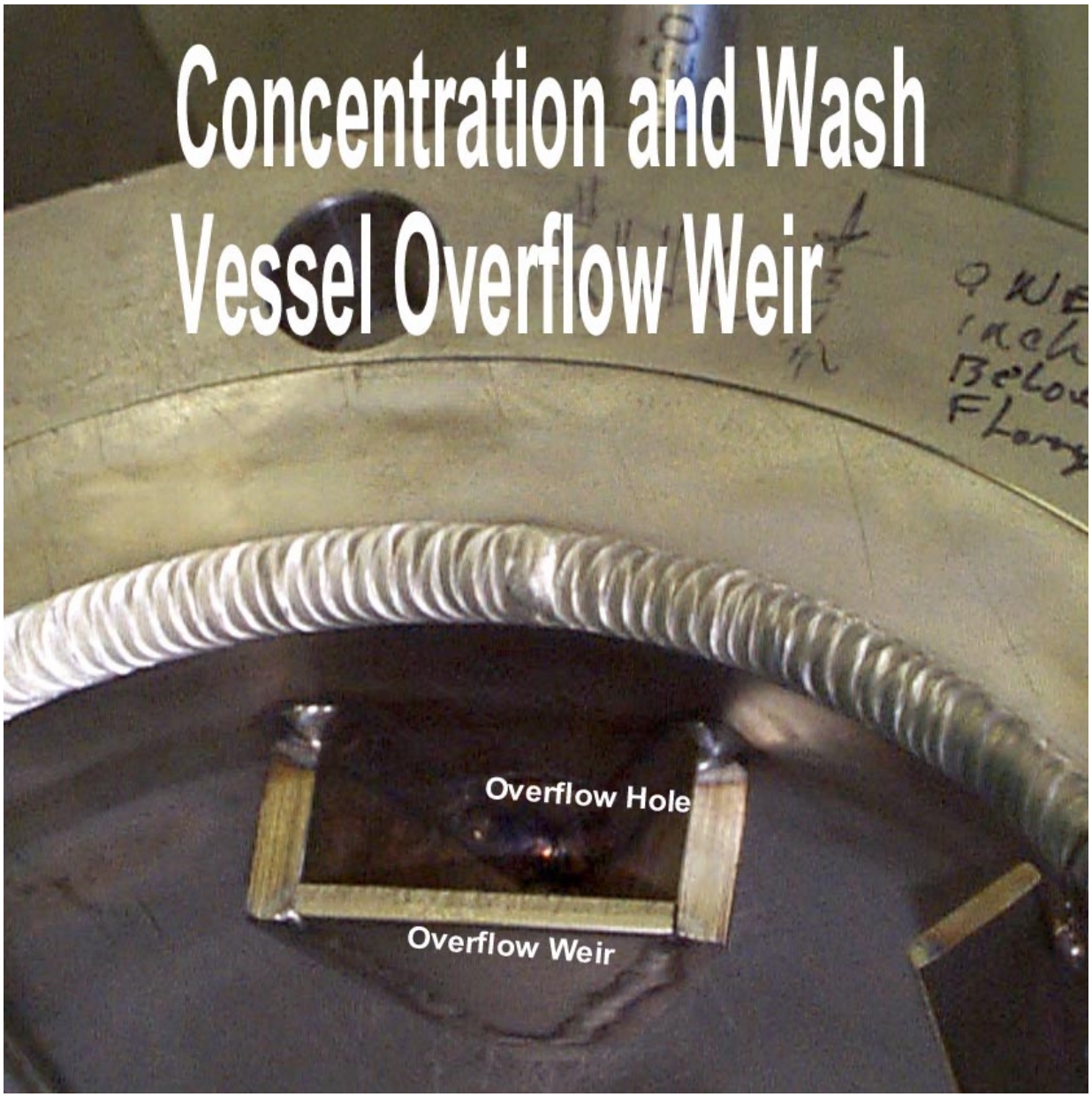


Notes:
1. Baffles 1/8" thick, and are 0.67" wide, stand-off from wall 0.11", and each is attached to tank wall near top and bottom, shaped on the bottom to match the cap shape, and spaced as shown in tank top drawing.
2. Feed tube is 1/2" tube through a 1/2"tube fitting welded to the flange and drilled through.
3. Drill one 1/4" hole in flange faces for an alignment pin.
4. To Moyno tube is 3/4" and reaches to 1" from bottom; Slurry Return tube is 3/4" with TEE at bottom with TEE parallel to tank wall, 1-2" from tank bottom.
5. Overflow is a 3/4" tube 2" long welded to shell, on opposite side of tank from the N2 fitting on the top, 1/2" below bottom of flange. Overflow weir is 3/4" wide V-shape enclosure with bottom at bottom of hole and with top 1" below flange face
6. Drain is 1/2" port 3-part valve welded into hole in center of the pipe cap.

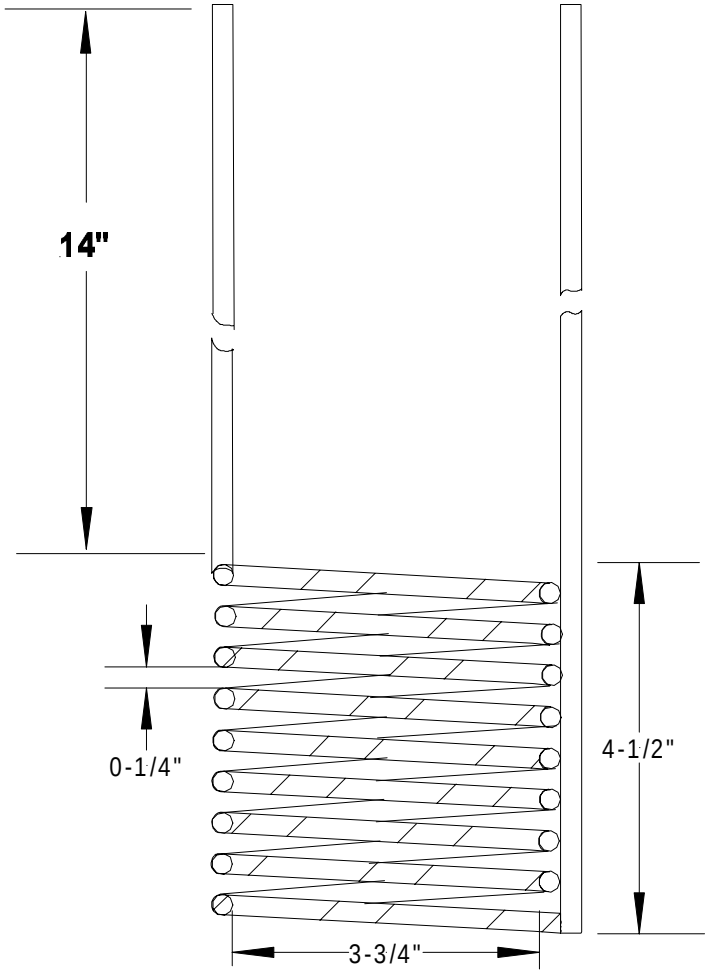
REV.	ECN NO.	REVISION DESCRIPTION	DATE	BY	CHK'D	APPD.

DRAWN Douglas Lee	DATE 06/19/99	Concentration Vessel Cross-section of Vessel Through Cooling Lines 20L CSTR Demo, (as Built) ORNL-CERS-1	Cell B, 4501 CSTR 20L DEMO
CHECKED			
ENGINEER D. D. Lee			FILE 4501 REV A
APPROVED		SCALE 1"=2.5" SHEET 1 OF 1	DWG NO. CERS CSTR-DW15

Photo of the Concentration and Wash Vessel Overflow Weir.



Concentration Vessel Coil



Notes: Use 1/4" SS tubing, thin wall, with the length for the top of the coil to the top of the stub 14" or more. Roll the tubing so the inner diameter is 3 3/4" with 1/4" space between tubes in the coil. This should be about 8 1/2 coils for the 4 1/2" of coils.

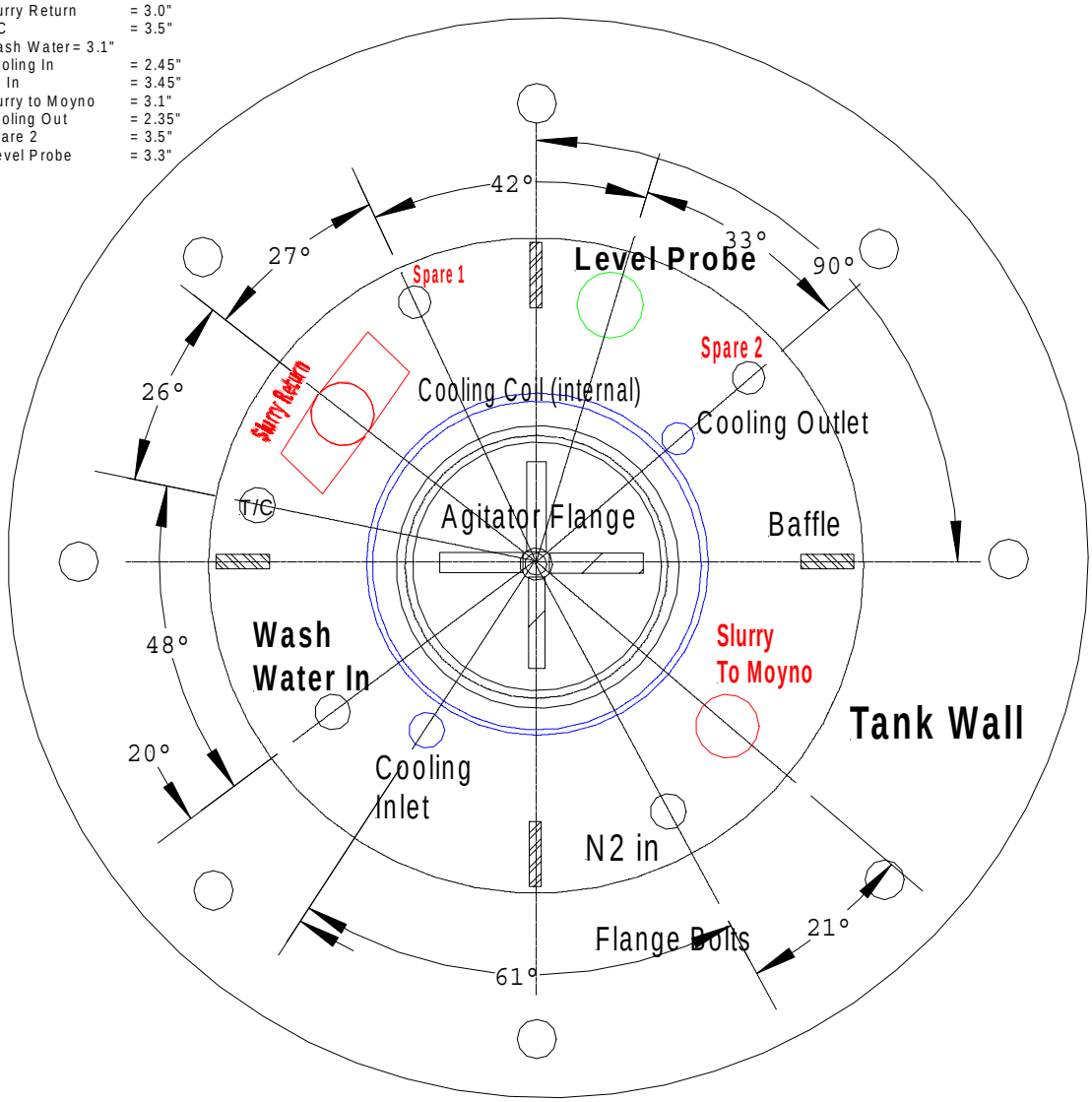
REV.	ECN NO.	REVISION DESCRIPTION	DATE	BY	CHK'D	APPD.

DRAWN Douglas Lee	DATE 06/19/99	Concentration Vessel Coil 20L CSTR Demo ORNL-CERS-1	Cell B, 4501 CSTR 20L DEMO
CHECKED			
ENGINEER D. D. Lee			FILE 4501 REV A
APPROVED		SCALE 1"=2" SHEET 1 OF 1	DWG NO. CERS CSTR-DW21

Wash Vessel Top Flange

Notes--Opening distances from center of flange to center of opening:

1. Spare 1 = 3.5"
2. Slurry Return = 3.0"
3. T/C = 3.5"
4. Wash Water = 3.1"
5. Cooling In = 2.45"
6. N2 In = 3.45"
7. Slurry to Moyno = 3.1"
8. Cooling Out = 2.35"
9. Spare 2 = 3.5"
10. Level Probe = 3.3"

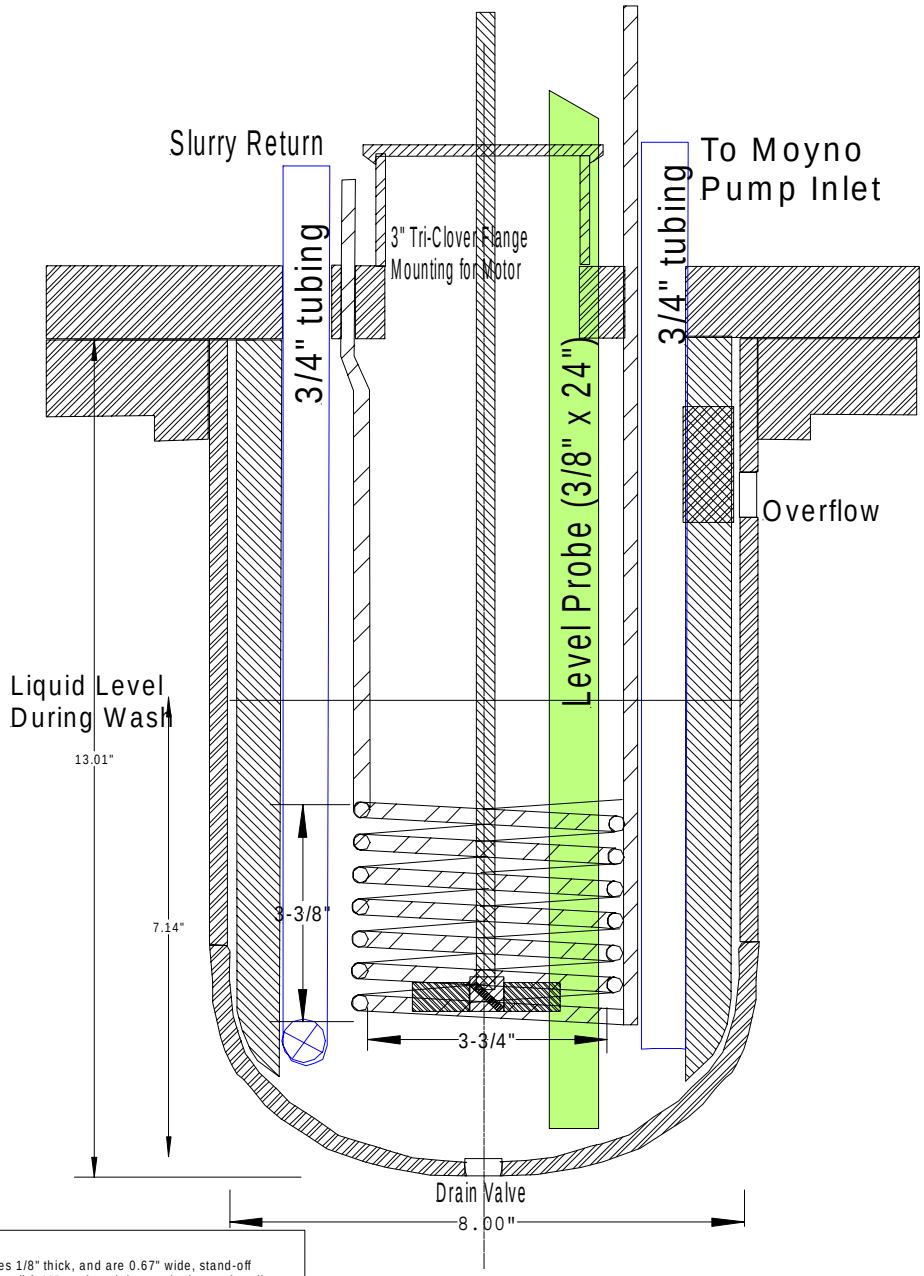


Notes:
1. Holes for T/C, N2, cooling in and out, Feed, Wash water, and spares, are 1/4" NPT adapters to 1/4" tube fittings drilled to allow tube to pass through.
2. Level Probe is 1" NPT .
3. To Moyno and Slurry Return are holes for 3/4" tube fittings, with fittings drilled to allow tube to pass through.
4. Baffles at 90 degrees from each other

REV.	ECN NO.	REVISION DESCRIPTION	DATE	BY	CHK'D	APPD.

DRAWN Douglas Lee	DATE 06/19/99	Wash Vessel Top Flange 20L CSTR Demo ORNL-CERS-1	Cell B, 4501 CSTR 20L DEMO
CHECKED			
ENGINEER D. D. Lee			FILE 4501 REV A
APPROVED		SCALE 1"=2.5" SHEET 1 OF 1	DWG NO. CERS CSTR-DW7

Wash Vessel General View

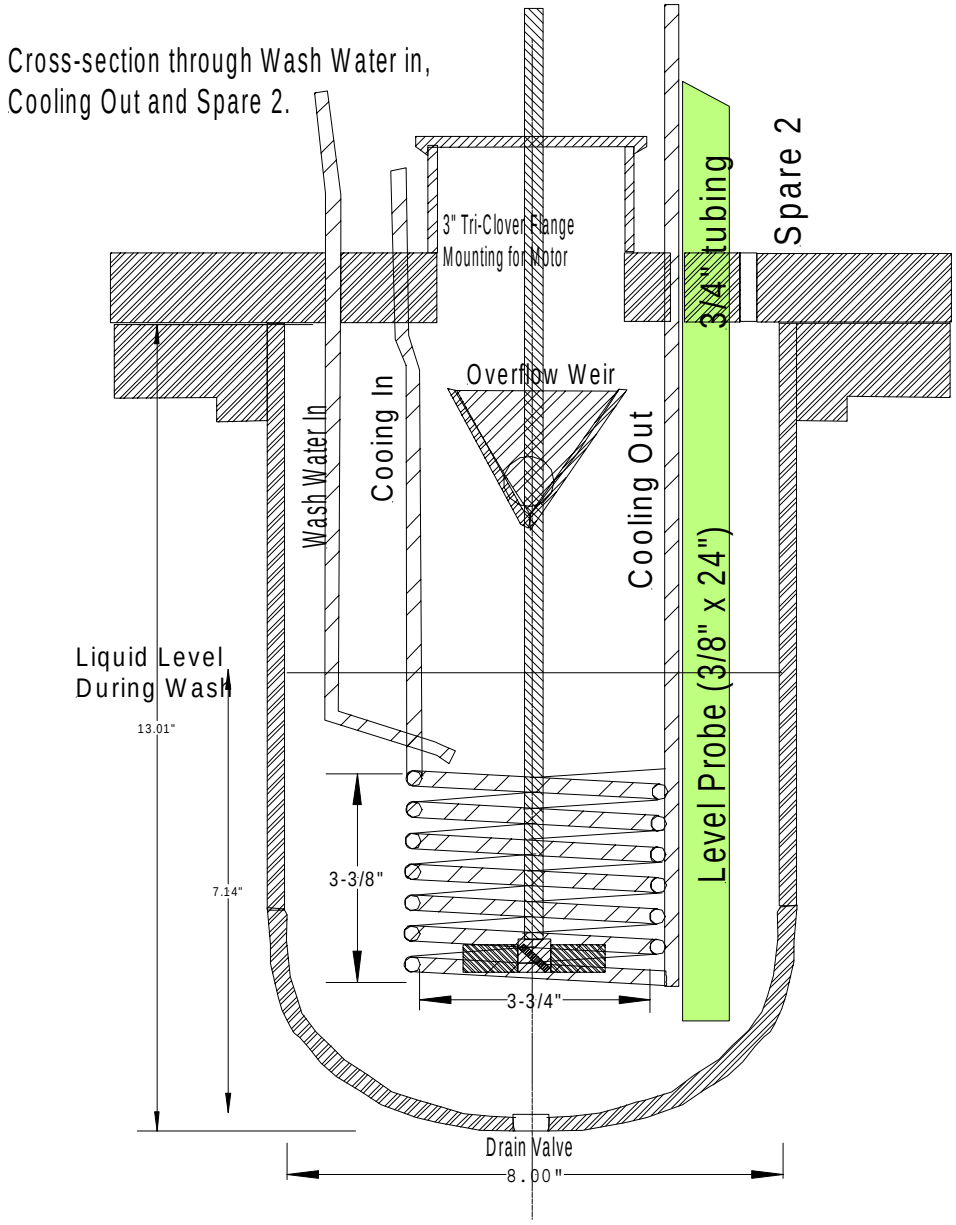


Notes:
1. Baffles 1/8" thick, and are 0.67" wide, stand-off from wall 0.11", and each is attached to tank wall near top and bottom, shaped on the bottom to match the cap shape, and spaced as shown in tank top drawing.
2. Drill one 1/4" hole in flange faces for an alignment pin.
3. To Moyno tube is 3/4" and reaches to 1" from bottom; Slurry Return tube is 3/4" with TEE at bottom with TEE parallel to tank wall, 1-2" from tank bottom.
4. Overflow is a 3/4" tube 2" long welded to shell, 180 degrees from the N2 fitting on the top, 1/2" below bottom of flange. Overflow weir is 3/4" wide V-shape enclosure with bottom at bottom of hole and with top 1" below flange face
5. Drain is 1/2" port 3-part ball valve welded into the pipe cape center.

REV.	ECN NO.	REVISION DESCRIPTION	DATE	BY	CHK'D	APPD.

DRAWN Douglas Lee	DATE 06/19/99	Wash Vessel General View Cross-section of Vessel 20L CSTR Demo ORNL-CERS-1	Cell B, 4501 CSTR 20L DEMO
CHECKED			
ENGINEER D. D. Lee			FILE 4501 REV A
APPROVED		SCALE 1"=2.5" SHEET 1 OF 1	DWG NO. CERS CSTR-DW5

Wash Vessel Cross-Section

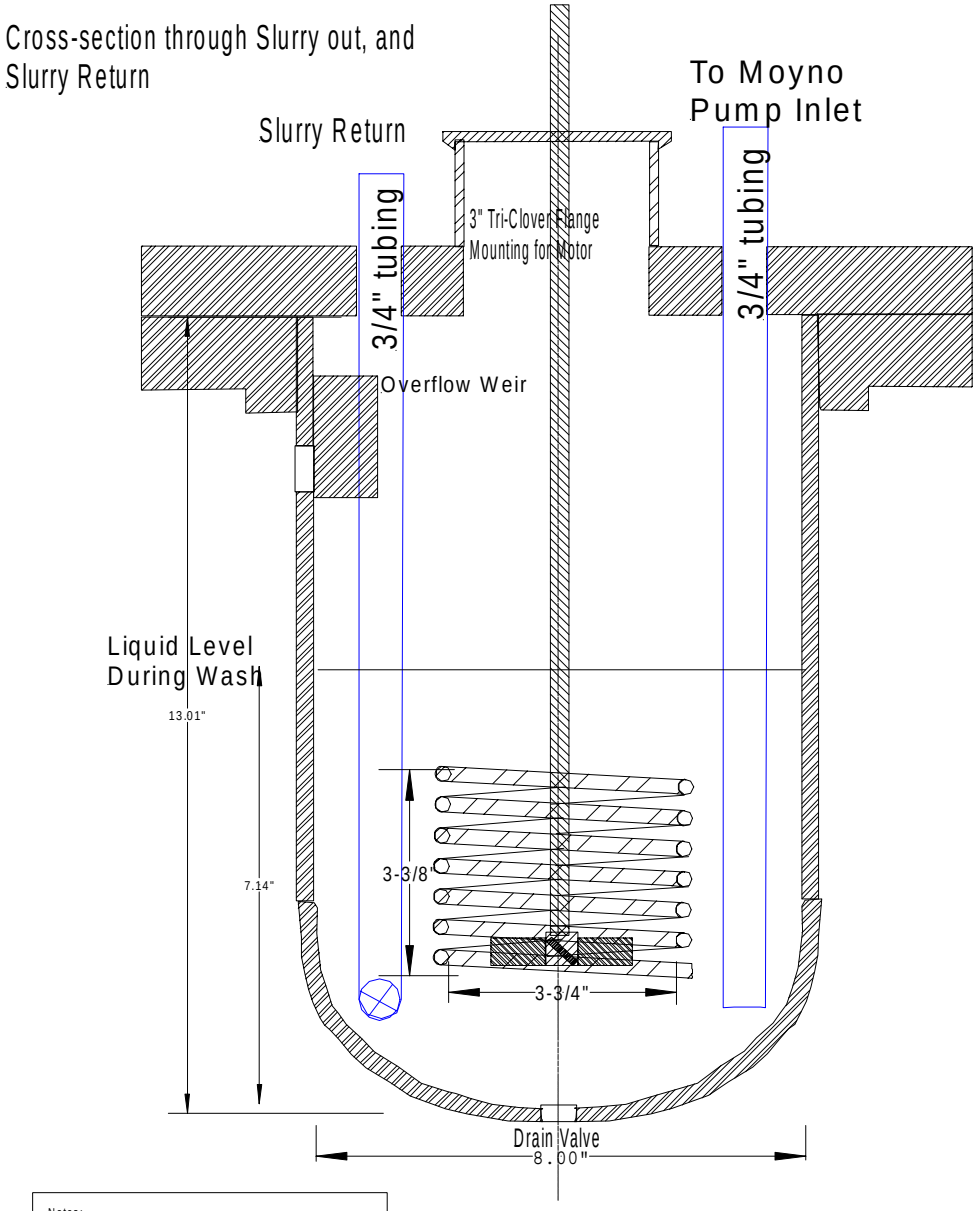


Notes:
1. Baffles 1/8" thick, and are 0.67" wide, stand-off from wall 0.11", and each is attached to tank wall near top and bottom, shaped on the bottom to match the cap shape, and spaced as shown in tank top drawing.
2. Drill one 1/4" hole in flange faces for an alignment pin.
3. Wash water line is 1/4" SS with opening 1.5" below liquid surface and 3/4" inside cooling coil.
4. Overflow is a 3/4" tube 2" long welded to shell, 180 degrees from the N2 fitting on the top, 1/2" below bottom of flange. Overflow weir is 3/4" wide V-shape enclosure with bottom at bottom of hole and with top 1" below flange face.
5. Drain is 1/2" hole for welded valve fitting.
6. Agitator is mounted 2.25" from bottom of tank.

REV.	REVISION DESCRIPTION	DATE	BY	CHK'D	APPD.

DRAWN Douglas Lee	DATE 06/19/99	Wash Vessel Cross-section Through Wash water in, 20L CSTR Demo(As Built) ORNL-CERS-1	Cell B, 4501 CSTR 20L DEMO
CHECKED			
ENGINEER D. D. Lee			FILE 4501 REV A
APPROVED		SCALE 1"=2.5" SHEET 1 OF 1	DWG NO. CERS CSTR-DW13

Wash Vessel Cross-Section

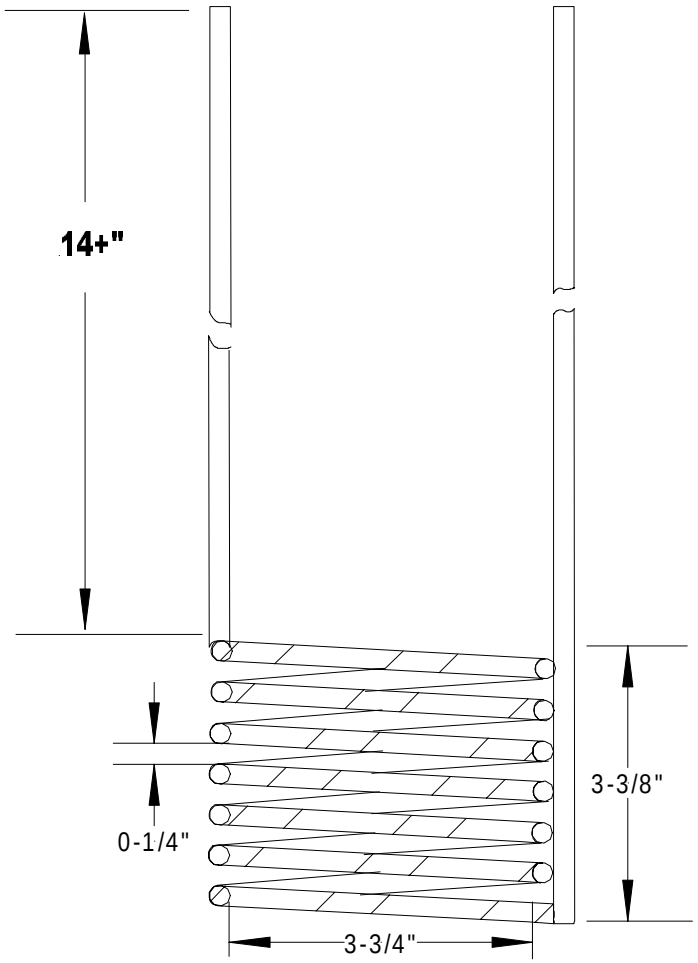


Notes:
1. Baffles 1/8" thick, and are 0.67" wide, stand-off from wall 0.11", and each is attached to tank wall near top and bottom, shaped on the bottom to match the cap shape, and spaced as shown in tank top drawing.
2. Drill one 1/4" hole in flange faces for an alignment pin.
3. To Moyno tube is 3/4" and reaches to 1" from bottom; Slurry Return tube is 3/4" with TEE at bottom with TEE parallel to tank wall, 2" from tank bottom.
4. Overflow is a 3/4" tube 2" long welded to shell, 180 degrees from the N2 fitting on the top, 1/2" below bottom of flange. Overflow weir is 3/4" wide V-shape enclosure with bottom at bottom of hole and with top 1" below flange face.
5. Drain is 1/2" hole for welded valve fitting.

REV.	REVISION DESCRIPTION	DATE	BY	CHK'D	APPD.

DRAWN Douglas Lee	DATE 06/19/99	Wash Vessel Cross-section Through Slurry in and out 20L CSTR Demo (As Built) ORNL-CERS-1	Cell B, 4501 CSTR 20L DEMO
CHECKED			
ENGINEER D. D. Lee			FILE 4501 REV A
APPROVED		SCALE 1"=2.5" SHEET 1 OF 1	DWG NO. CERS CSTR-DW12

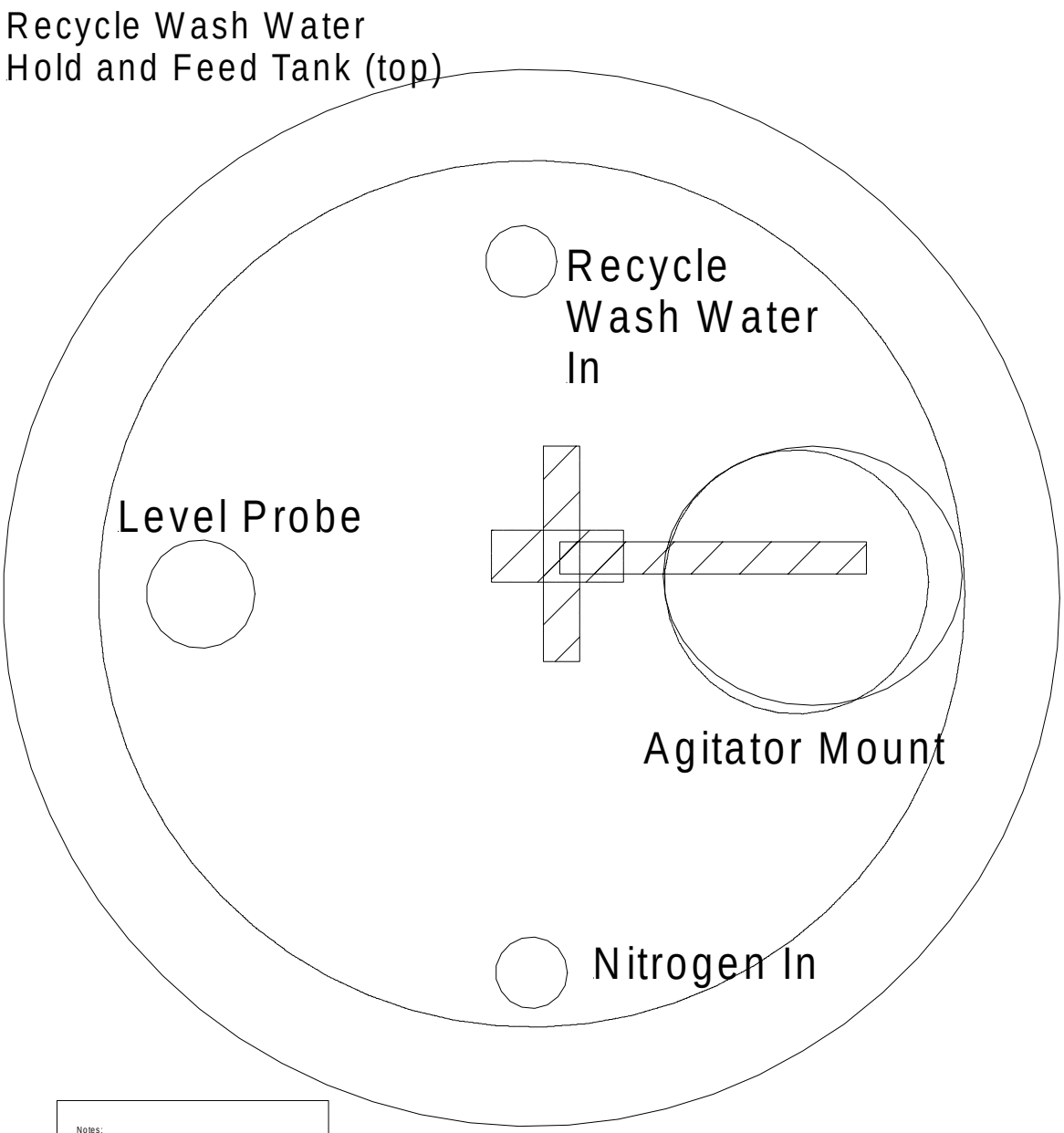
WASH VESSEL COIL



Notes: Use 1/4" SS tubing, thin wall, with the length for the top of the coil to the top of the stub 14" or more. Roll the tubing so the inner diameter is 3 3/4" with 1/4" space between tubes in the coil. This should be about 6 1/2 coils for the 3 3/8" of coils.

REV.	ECN NO.	REVISION DESCRIPTION	DATE	BY	CHK'D	APPD.

DRAWN Douglas Lee	DATE 06/19/99	Wash Vessel Coil 20L CSTR Demo ORNL-CERS-1	Cell B, 4501 CSTR 20L DEMO
CHECKED			
ENGINEER D. D. Lee			FILE 4501 REV A
APPROVED		SCALE 1"=2" SHEET 1 OF 1	DWG NO. CERS CSTR-DW22

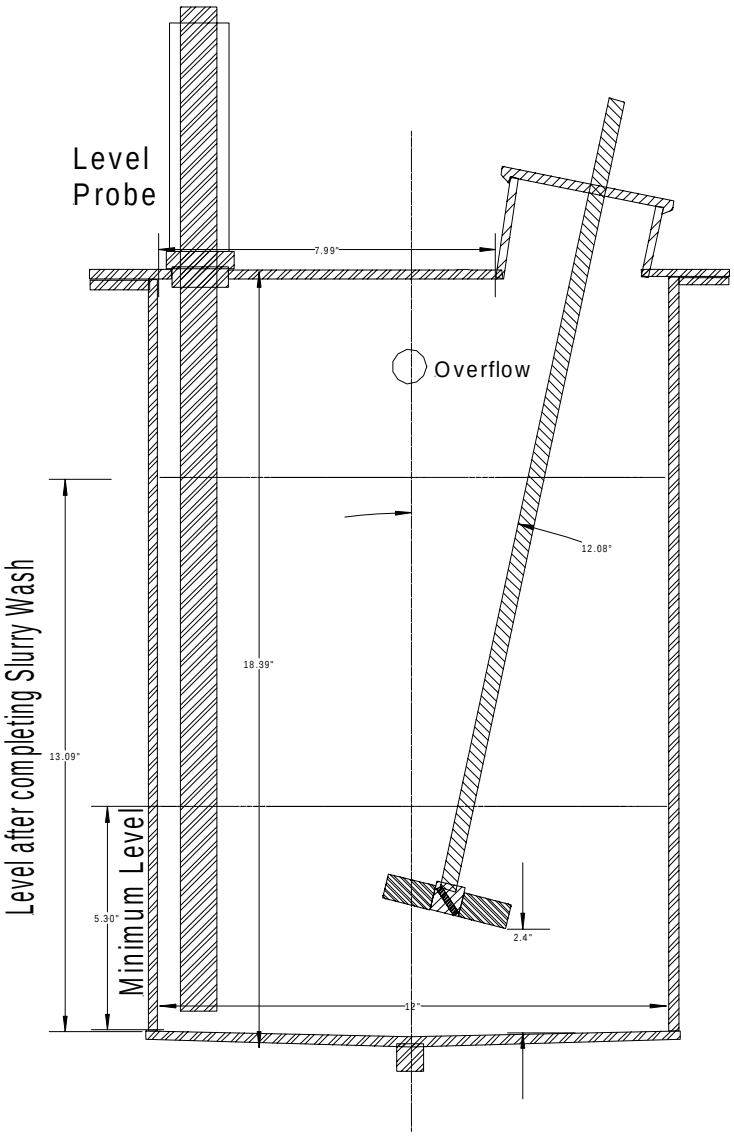


Notes:
1. Construct from SS tubing, 12" ID, with 1/8or 3/16" plate for top and bottom, with bottom slightly dished drainage. Use Viton gasket between flanges.
2. Agitator mount is 3" TriClover fitting set at an 11 degree angle from the horizontal and at the edge of the tank, 1/2" from the inside edge of the tank.
3. Drill one 1/4" hole in flange faces for an alignment pin.
4. Overflow is a 3/4" tube 2" long welded to shell, on opposite side from the N2 fitting on the top, 3/4" below bottom of flange.
4. Level Probe is 1" NPT female fitting to accept 1" male NPT pipe for probe mount.
5. Drain is 1/2" port valve welded onto stub tube in hole in center of the bottom plate.
6. Nitrogen in and Recycle Wash Water in are 1/4" tube fittings welded to the flange top and drilled through for the tubes.

REV.	ECN NO.	REVISION DESCRIPTION	DATE	BY	CHK'D	APPD.

DRAWN Douglas Lee	DATE 06/19/99	Recycle Wash Water Hold and Feed Tank (top) 20L CSTR Demo ORNL-CERS-1	Cell B, 4501 CSTR 20L DEMO
CHECKED			
ENGINEER D. D. Lee			FILE 4501 REV A
APPROVED		SCALE 1"=2" SHEET 1 OF 1	DWG NO. CERS CSTR-DW24

Recycle Wash Water Hold and Feed Tank



Notes:
1. Construct from SS tubing, 12" ID, with 1/8or 3/16" plate for top and bottom, with bottom slightly dished drainage. Use Viton gasket between flanges.
2. Agitator mount is 3" TriClover fitting set at an 11 degree angle from the horizontal and at the edge of the tank, 1/2" from the inside edge of the tank.
3. Drill one 1/4" hole in flange faces for an alignment pin.
4. Overflow is a 3/4" tube 2" long welded to shell, on opposite side from the N2 fitting on the top, 3/4" below bottom of flange.
4. Level Probe is 1" NPT female fitting to accept 1" male NPT pipe for probe mount.
5. Drain is 1/2" port valve welded onto stub tube in hole in center of the bottom plate.

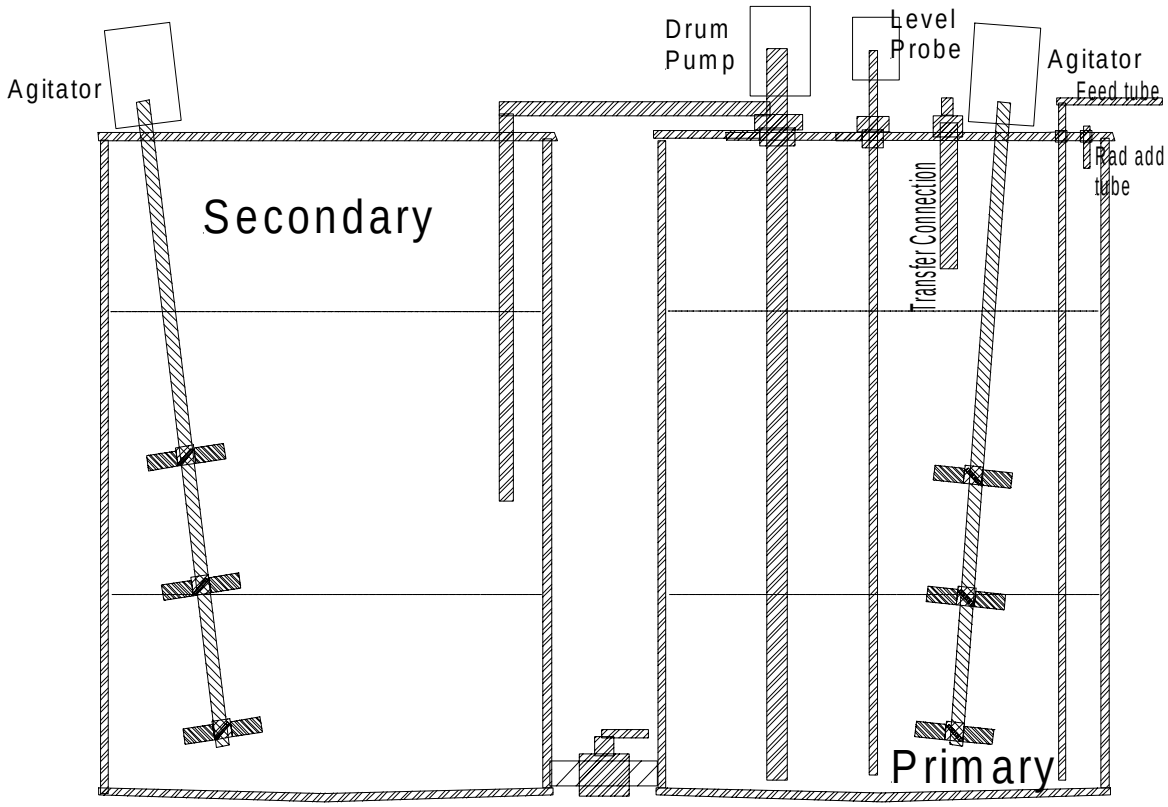
REV.	ECN NO.	REVISION DESCRIPTION	DATE	BY	CHK'D	APPD.

DRAWN Douglas Lee	DATE 06/19/99	Recycle Wash Water Hold and Feed Tank 20L CSTR Demo ORNL-CERS-1	Cell B, 4501 CSTR 20L DEMO
CHECKED			
ENGINEER D. D. Lee			FILE 4501 REV A
APPROVED		SCALE 1"=3" SHEET 1 OF 1	DWG NO. CERS CSTR-DW23

Recycle
Wash-Water
Tank

CSTR System Workaid

Simulant Salt Solution Feed Tanks



Notes:
1. Construct from SS 55 gal open head drums.
2. Agitators are rim mount with agitators through holes in head.
3. Feed tube is 1/4" SS through drum head.
4. Level probe mounts on 3/4" female pipe fitting in drum head.
5. Drum pump mounts on drum head and discharges to second drum head through 3/4" line
6. Connection between drums is 1-1/2" SS with 1-1/2" valve.
7. Transfer connection is 3/4" through Drum head.
8. Radioactive component addition port is 1/4" tube through a 1/4" drilled through tube fitting

REV.	REVISION DESCRIPTION	DATE	BY	CHK'D	APPD.
.	.	.	.	.	.

DRAWN Douglas Lee	DATE 6/19/99	Simulant Salt Solution Feed Tanks 20L CSTR Demo ORNL-CERS-1	Cell B, 4501 CSTR 20L DEMO
CHECKED			
ENGINEER D. D. Lee			FILE 4501 REV A
APPROVED		SCALE	SHEET 1 OF 1 DWG NO. CERS CSTR-DW25

Appendix A.3

INSTRUMENTATION DATA SHEETS AND LOOP I/O DATABASE

WORKAID NUMBER: CERS-08
7/29/99

Instrumentation Data Sheets and Loop I/O Database

Author

T.R. Tipton

Signature _____ **Date** _____

Approvals

D.D. Lee, Project Group Leader for ORNL CSTR Demonstration

Signature _____ **Date** _____

R.T. Jubin, Chemical and Energy Research Section Section Head

Signature _____ **Date** _____

Workaid Number: CERS-08 Revision: 1

Instrumentation Data Sheets

Instrument: FI-2 - Slurry Wash Loop Process Flow

NOTE: Vendor calibrated on site.

Range

0 - 5 GPM

Calibration

Input (%)	Output (mA)	GPM
0	N/A	N/A
20	N/A	N/A
40	N/A	N/A
60	N/A	N/A
80	N/A	N/A
100	N/A	N/A

Controller Settings

Set Point	N/A
P(roportional)	N/A
I(ntegral)	N/A
Scan Rate	1 sec

Alarm Limits

HIHI	5.00
HI	5.00
LO	0.00
LOLO	0.00

Process Reaction PD vs PV

Dead Time	N/A
Tau	N/A
Process Gain	N/A

Workaid Number: CERS-08 Revision: 1

<u>Instrument:</u> FI-4 - Recycle Wash Water Loop Feed Flow

NOTE: Instrument not available.

Range

0 - 25 mL/m

Calibration

Input (%)	Output (mA)	mL/m
0	N/A	N/A
20	N/A	N/A
40	N/A	N/A
60	N/A	N/A
80	N/A	N/A
100	N/A	N/A

Controller Settings

Set Point	N/A
P(roportional)	N/A
I(ntegral)	N/A
Scan Rate	1 sec

Alarm Limits

HIHI	25.00
HI	25.00
LO	0.00
LOLO	0.00

Process Reaction PD vs PV

Dead Time	N/A
Tau	N/A
Process Gain	N/A

Workaid Number: CERS-08 Revision: 1

<u>Instrument:</u> FIC-1 - Slurry Concentration Filtrate Flow

Range

0 - 40 mL/m

Calibration

Output (% Open)	mL/m (Intellution)	mL/m (Protuner)	mL/m (Graduated Cylinder)
80	9.1	8.8	8.0
85	12.1	11.8	N/A
87	14.0	13.7	13.0
89	17.0	16.8	15.6
91	21.8	21.5	19.75
92	24.3	23.9	N/A
93	27.3	26.9	25.5
95	35.3	34.8	33.5
95.5	37.6	37.0	36.0

Controller Settings

Set Point	N/A
P(roportional)	2035.57%
I(ntegral)	.255 min
Scan Rate	.05 sec

Alarm Limits

HIHI	40.00
HI	40.00
LO	0.00
LOLO	0.00

Process Reaction PD vs PV

Dead Time	.87 sec
Tau	15.29 sec
Process Gain	13.25

<u>Instrument:</u> FIC-3 - Slurry Wash Loop Filtrate Flow

*NOTE: System dynamics prohibit use of this element for cotrol purposes. Flow values are approximate.

Range

0 - 50 mL/m

Calibration

Output (% Open)	mL/m (Intellution)	mL/m (Protuner)	mL/m (Graduated Cylinder)
N/A	N/A	N/A	N/A

Controller Settings

Set Point	N/A
P(roportional)	N/A
I(ntegral)	N/A
Scan Rate	N/A

Alarm Limits

HIHI	N/A
HI	N/A
LO	N/A
LOLO	N/A

Process Reaction PD vs PV

Dead Time	N/A
Tau	N/A
Process Gain	N/A

<u>Instrument:</u> HLA-1 - CSTR-2 Sump High Level Alarm

Calibration

Input (%)	Output (mA)	ON/OFF
0	4.0	OFF
100	20.0	ON

Controller Settings

Set Point	N/A
P(roportional)	N/A
I(ntegral)	N/A
Scan Rate	1 sec

Alarm Limits

HIHI	N/A
HI	N/A
LO	N/A
LOLO	N/A

Process Reaction PD vs PV

Dead Time	N/A
Tau	N/A
Process Gain	N/A

Workaid Number: CERS-08 Revision: 1

<u>Instrument:</u> HLA-2 - Recycle Washwater Sump High Level Alarm

Calibration

Input (%)	Output (mA)	ON/OFF
0	4.0	OFF
100	20.0	ON

Controller Settings

Set Point	N/A
P(roportional)	N/A
I(ntegral)	N/A
Scan Rate	1 sec

Alarm Limits

HIHI	N/A
HI	N/A
LO	N/A
LOLO	N/A

Process Reaction PD vs PV

Dead Time	N/A
Tau	N/A
Process Gain	N/A

Workaid Number: CERS-08 Revision: 1

<u>Instrument:</u> LI-3 - Recycle Wash Water Surge Tank Level Controller

***NOTE: This instrument is not available for calibration.**

Range

0 - 18 In

Calibration

Input (%)	Output (mA)	Inches
0	4.0	
20	7.2	
40	10.4	
60	13.6	
80	16.8	
100	20.0	

Controller Settings

Set Point	N/A
P(roportional)	N/A
I(ntegral)	N/A
Scan Rate	1 sec

Alarm Limits

HIHI	18.00
HI	18.00
LO	0.00
LOLO	0.00

Process Reaction PD vs PV

Dead Time	N/A
Tau	N/A
Process Gain	N/A

Workaid Number: CERS-08 Revision: 1

<u>Instrument:</u> LI-4 - Salt Feed Tank Level Indicator

***NOTE: This instrument is not available for calibration.**

Range

0 - 34 In

Calibration

Input (%)	Output (mA)	Inches
0	4.0	
20	7.2	
40	10.4	
60	13.6	
80	16.8	
100	20.0	

Controller Settings

Set Point	N/A
P(roportional)	N/A
I(ntegral)	N/A
Scan Rate	1 sec

Alarm Limits

HIHI	34.00
HI	34.00
LO	0.00
LOLO	0.00

Process Reaction PD vs PV

Dead Time	N/A
Tau	N/A
Process Gain	N/A

Workaid Number: CERS-08 Revision: 1

<u>Instrument:</u> LIC-1 - Slurry Concentration Tank Level Controller

Range

1.10 - 12.00 In

Calibration

Input (L)	Inches (Calc)	Inches (Computer)
Heel	N/A	N/A
Heel	N/A	N/A
3.00	4.52	4.3 - 4.6
4.00	5.62	5.5 - 5.8
5.00	6.89	6.7 - 7.0
6.00	8.12	7.9 - 8.1
7.00	9.33	9.0 - 9.4
8.00	10.51	10.1 - 10.2

Controller Settings

Set Point	
P(roportional)	11.36%
I(ntegral)	94.204 min
Scan Rate	1 sec

Alarm Limits

HIHI	10.00
HI	9.50
LO	7.00
LOLO	3.00

Process Reaction PD vs PV

Dead Time	0
Tau	0
Process Gain	.000121 sec

Workaid Number: CERS-08 Revision: 1

<u>Instrument:</u> LIC-2 - Slurry Wash Tank Level Controller

Range

1.00 - 29.00 In

Calibration

Input (L)	Inches (Calc)	Inches (Computer)
Heel	N/A	N/A
Heel	N/A	N/A
3.00	4.47	
4.00	5.71	
5.00	6.94	
6.00	8.21	
7.00	9.38	
8.00	10.62	

Controller Settings

Set Point	N/A
P(roportional)	42%
I(ntegral)	30 min
Scan Rate	.05 sec

Alarm Limits

HIHI	10.00
HI	9.00
LO	7.00
LOLO	3.00

Process Reaction PD vs PV

Dead Time	N/A
Tau	N/A
Process Gain	N/A

Workaid Number: CERS-08 Revision: 1

<u>Instrument:</u> PCV-1 - Slurry Concentration Loop Backpressure Valve

Range

0 - 100 %

Calibration

Input (%)	Output (mA)	% OPEN
0	4.0	0.6
20	7.2	19.5
40	10.4	39.2
60	13.6	58.7
80	16.8	78.3
100	20.0	98.1

Controller Settings

Set Point	N/A
P(roportional)	N/A
I(ntegral)	N/A
Scan Rate	1 sec

Alarm Limits

HIHI	N/A
HI	N/A
LO	N/A
LOLO	N/A

Process Reaction Input to valve vs valve position

Dead Time	.29 sec
Tau	0.38 sec
Hysterisis	3.75%

Workaid Number: CERS-08 Revision: 1

<u>Instrument:</u> PCV-2 - Slurry Concentration Filtrate Flow Valve (Note: FIC-1 Control Valve)

Range

0 - 100%

Calibration

Input (%)	Output (mA)	% OPEN
0	4.0	0.1
20	7.2	19.5
40	10.4	39.3
60	13.6	58.9
80	16.8	78.9
100	20.0	98.7

Controller Settings

Set Point	N/A
P(roportional)	N/A
I(ntegral)	N/A
Scan Rate	1 sec

Alarm Limits

HIHI	N/A
HI	N/A
LO	N/A
LOLO	N/A

Process Reaction Input to valve vs valve position

Dead Time	.13 sec
Tau	.21 sec
Hysterisis	0.60%

<u>Instrument:</u> PCV-3 - Slurry Wash Loop Backpressure Valve

Range

0 - 100%

Calibration

Input (%)	Output (mA)	% OPEN
0	4.0	-0.1
20	7.2	19.2
40	10.4	39.1
60	13.6	59.1
80	16.8	78.8
100	20.0	98.4

Controller Settings

Set Point	N/A
P(roportional)	N/A
I(ntegral)	N/A
Scan Rate	1 sec

Alarm Limits

HIHI	N/A
HI	N/A
LO	N/A
LOLO	N/A

Process Reaction Input to valve vs valve position

Dead Time	0.20 sec
Tau	0.35 sec
Hysterisis	1.43%

Workaid Number: CERS-08 Revision: 1

<u>Instrument:</u> PCV-4 - Slurry Concentration Filtrate Flow Valve (Note: FIC-3 Control Valve)

Range

0 - 100%

Calibration

Input (%)	Output (mA)	% OPEN
0	4.0	-0.3
20	7.2	19.4
40	10.4	39.6
60	13.6	59.4
80	16.8	79.3
100	20.0	99

Controller Settings

Set Point	N/A
P(roportional)	N/A
I(ntegral)	N/A
Scan Rate	1 sec

Alarm Limits

HIHI	N/A
HI	N/A
LO	N/A
LOLO	N/A

Process Reaction Input to valve vs valve position

Dead Time	0.4 sec
Tau	0.88 sec
Hysterisis	0.24%

Workaid Number: CERS-08 Revision: 1

<u>Instrument:</u> PI-2 - Slurry Wash Loop Filtrate Flow Pressure

Note: Not field tested - Factory calibration only

Range

0 - 100 PSIG

Calibration

Input (%)	Output (mA)	PSIG
0	N/A	N/A
20	N/A	N/A
40	N/A	N/A
60	N/A	N/A
80	N/A	N/A
100	N/A	N/A

Controller Settings

Set Point	N/A
P(roportional)	N/A
I(ntegral)	N/A
Scan Rate	1 sec

Alarm Limits

HIHI	N/A
HI	N/A
LO	N/A
LOLO	N/A

Process Reaction PD vs PV

Dead Time	N/A
Tau	N/A
Process Gain	N/A

Workaid Number: CERS-08 Revision: 1

<u>Instrument:</u> PI-4 - Slurry Wash Loop Filtrate Flow Pressure

Note: Not field tested - Factory calibration only

Range

0 - 200 PSIG

Calibration

Input (%)	Output (mA)	PSIG
0	N/A	N/A
20	N/A	N/A
40	N/A	N/A
60	N/A	N/A
80	N/A	N/A
100	N/A	N/A

Controller Settings

Set Point	N/A
P(roportional)	N/A
I(ntegral)	N/A
Scan Rate	1 sec

Alarm Limits

HIHI	N/A
HI	N/A
LO	N/A
LOLO	N/A

Process Reaction PD vs PV

Dead Time	N/A
Tau	N/A
Process Gain	N/A

<u>Instrument:</u> PIC-1 - Slurry Concentration Loop Cross-Flow Backpressure

Note: Not field tested - Factory calibration only

Range

0 - 200 PSIG

Calibration

Input (%)	Output (mA)	PSIG
0	N/A	N/A
20	N/A	N/A
40	N/A	N/A
60	N/A	N/A
80	N/A	N/A
100	N/A	N/A

Controller Settings

Set Point	N/A
P(roportional)	657.90%
I(ntegral)	0.0125 min
Scan Rate	.05 sec

Alarm Limits

HIHI	N/A
HI	N/A
LO	N/A
LOLO	N/A

Process Reaction PD vs PV

Dead Time	0.36 sec
Tau	0
Process Gain	0.969

Workaid Number: CERS-08 Revision: 1

<u>Instrument:</u> PIC-3 - Slurry Wash Loop Cross-Flow Filter Backpressure

Note: Not field tested - Factory calibration only

Range

0 - 200 PSIG

Calibration

Input (%)	Output (mA)	PSIG
0	N/A	N/A
20	N/A	N/A
40	N/A	N/A
60	N/A	N/A
80	N/A	N/A
100	N/A	N/A

Controller Settings

Set Point	N/A
P(roportional)	452.80%
I(ntegral)	0.0125 min
Scan Rate	.05 sec

Alarm Limits

HIHI	N/A
HI	N/A
LO	N/A
LOLO	N/A

Process Reaction PD vs PV

Dead Time	N/A
Tau	N/A
Process Gain	N/A

Workaid Number: CERS-08 Revision: 1

<u>Instrument:</u> SSI-1D - Slurry Concentration Loop Density

NOTE: Vendor calibrated on site.

Range

0.0 - 1.3 g/cc

Calibration

Input (%)	Output (mA)	g/cc
0	N/A	N/A
20	N/A	N/A
40	N/A	N/A
60	N/A	N/A
80	N/A	N/A
100	N/A	N/A

Controller Settings

Set Point	N/A
P(roportional)	N/A
I(ntegral)	N/A
Scan Rate	1 sec

Alarm Limits

HIHI	N/A
HI	N/A
LO	N/A
LOLO	N/A

Process Reaction PD vs PV

Dead Time	N/A
Tau	N/A
Process Gain	N/A

<u>Instrument:</u> SSI-1F - Slurry Concentration Loop Flow

NOTE: Vendor calibrated on site.

Range

0.0 - 5.0 GPM

Calibration

Input (%)	Output (mA)	GPM
0	N/A	N/A
20	N/A	N/A
40	N/A	N/A
60	N/A	N/A
80	N/A	N/A
100	N/A	N/A

Controller Settings

Set Point	N/A
P(roportional)	N/A
I(ntegral)	N/A
Scan Rate	1 sec

Alarm Limits

HIHI	N/A
HI	N/A
LO	N/A
LOLO	N/A

Process Reaction PD vs PV

Dead Time	N/A
Tau	N/A
Process Gain	N/A

Workaid Number: CERS-08 Revision: 1

<u>Instrument:</u> SSI-1T - Slurry Concentration Loop Temperature

NOTE: Vendor calibrated on site.

Range

0 - 100 degC

Calibration

Input (%)	Output (mA)	degC
0	N/A	N/A
20	N/A	N/A
40	N/A	N/A
60	N/A	N/A
80	N/A	N/A
100	N/A	N/A

Controller Settings

Set Point	N/A
P(roportional)	N/A
I(ntegral)	N/A
Scan Rate	1 sec

Alarm Limits

HIHI	35.00
HI	30.00
LO	0.00
LOLO	0.00

Process Reaction PD vs PV

Dead Time	N/A
Tau	N/A
Process Gain	N/A

Workaid Number: CERS-08 Revision: 1

<u>Instrument:</u> TIC-1 - CSTR - 1 Temp Controller (Single Loop Controller)

Range

Calibration

Input (%)	Output (mA)	Temperture degC
0	N/A	N/A
20	N/A	N/A
40	N/A	N/A
60	N/A	N/A
80	N/A	N/A
100	N/A	N/A

Controller Settings

Set Point	N/A
P(roportional)	N/A
I(ntegral)	N/A
Scan Rate	1 sec

Alarm Limits

HIHI	N/A
HI	N/A
LO	N/A
LOLO	N/A

Process Reaction PD vs PV

Dead Time	N/A
Tau	N/A
Process Gain	N/A

Workaid Number: CERS-08 Revision: 1

<u>Instrument:</u> TIC-2 - CSTR - 2 Temp Controller (Single Loop Controller)

Range

Calibration

Input (%)	Output (mA)	Temperature degC
0	N/A	N/A
20	N/A	N/A
40	N/A	N/A
60	N/A	N/A
80	N/A	N/A
100	N/A	N/A

Controller Settings

Set Point	N/A
P(roportional)	N/A
I(ntegral)	N/A
Scan Rate	1 sec

Alarm Limits

HIHI	N/A
HI	N/A
LO	N/A
LOLO	N/A

Process Reaction PD vs PV

Dead Time	N/A
Tau	N/A
Process Gain	N/A

Workaid Number: CERS-08 Revision: 1

<u>Instrument:</u> TIC-3 - Slurry Concentration Tank Temp Controller (Single Loop Controller)

Range

Calibration

Input (%)	Output (mA)	Temperature degC
0	N/A	N/A
20	N/A	N/A
40	N/A	N/A
60	N/A	N/A
80	N/A	N/A
100	N/A	N/A

Controller Settings

Set Point	N/A
P(roportional)	N/A
I(ntegral)	N/A
Scan Rate	1 sec

Alarm Limits

HIHI	N/A
HI	N/A
LO	N/A
LOLO	N/A

Process Reaction PD vs PV

Dead Time	N/A
Tau	N/A
Process Gain	N/A

Workaid Number: CERS-08 Revision: 1

<u>Instrument:</u> TIC-4 - Slurry Wash Tank Temp Controller (Single Loop Controller)

Range

Calibration

Input (%)	Output (mA)	Temperature degC
0	N/A	N/A
20	N/A	N/A
40	N/A	N/A
60	N/A	N/A
80	N/A	N/A
100	N/A	N/A

Controller Settings

Set Point	N/A
P(roportional)	N/A
I(ntegral)	N/A
Scan Rate	1 sec

Alarm Limits

HIHI	N/A
HI	N/A
LO	N/A
LOLO	N/A

Process Reaction PD vs PV

Dead Time	N/A
Tau	N/A
Process Gain	N/A

Workaid Number: CERS-08 Revision: 1

<u>Instrument:</u> TI-1 - CSTR - 1
Temperature - Type K Thermocouple

Note: Not field tested - Factory calibration only

Range

0 - 1000 degC

Calibration

Input (%)	Output (mA)	Temperature degC
N/A	N/A	N/A

Controller Settings

Set Point	N/A
P(roportional)	N/A
I(ntegral)	N/A
Scan Rate	1 sec

Alarm Limits

HIHI	N/A
HI	N/A
LO	N/A
LOLO	N/A

Process Reaction PD vs PV

Dead Time	N/A
Tau	N/A
Process Gain	N/A

Workaid Number: CERS-08 Revision: 1

<u>Instrument:</u> TI-2 - CSTR - 2
Temperature - Type K Thermocouple

Note: Not field tested - Factory calibration only

Range

0 - 1000 degC

Calibration

Input (%)	Output (mA)	Temperature degC
N/A	N/A	N/A

Controller Settings

Set Point	N/A
P(roportional)	N/A
I(ntegral)	N/A
Scan Rate	1 sec

Alarm Limits

HIHI	N/A
HI	N/A
LO	N/A
LOLO	N/A

Process Reaction PD vs PV

Dead Time	N/A
Tau	N/A
Process Gain	N/A

Workaid Number: CERS-08 Revision: 1

<u>Instrument:</u> TI-3 - Slurry Concentration Tank Temperature - Type K Thermocouple

Note: Not field tested - Factory calibration only

Range

0 - 1000 degC

Calibration

Input (%)	Output (mA)	Temperature degC
N/A	N/A	N/A

Controller Settings

Set Point	N/A
P(roportional)	N/A
I(ntegral)	N/A
Scan Rate	1 sec

Alarm Limits

HIHI	N/A
HI	N/A
LO	N/A
LOLO	N/A

Process Reaction PD vs PV

Dead Time	N/A
Tau	N/A
Process Gain	N/A

Workaid Number: CERS-08 Revision: 1

<u>Instrument:</u> TI-4 - Slurry Wash Tank Temperature - Type K Thermocouple

Note: Not field tested - Factory calibration only

Range

0 - 1000 degC

Calibration

Input (%)	Output (mA)	Temperature degC
N/A	N/A	N/A

Controller Settings

Set Point	N/A
P(roportional)	N/A
I(ntegral)	N/A
Scan Rate	1 sec

Alarm Limits

HIHI	N/A
HI	N/A
LO	N/A
LOLO	N/A

Process Reaction PD vs PV

Dead Time	N/A
Tau	N/A
Process Gain	N/A

Workaid Number: CERS-08 Revision: 1

Vessel: Slurry Concentration Tank (Reference ORNL Dwg. No. CERS-CSTR-DW4).
Note: Following Calcs were performed to verify empirical data. Slurry Wash Tank and Recycle Washwater Tank were calibrated by the same method.

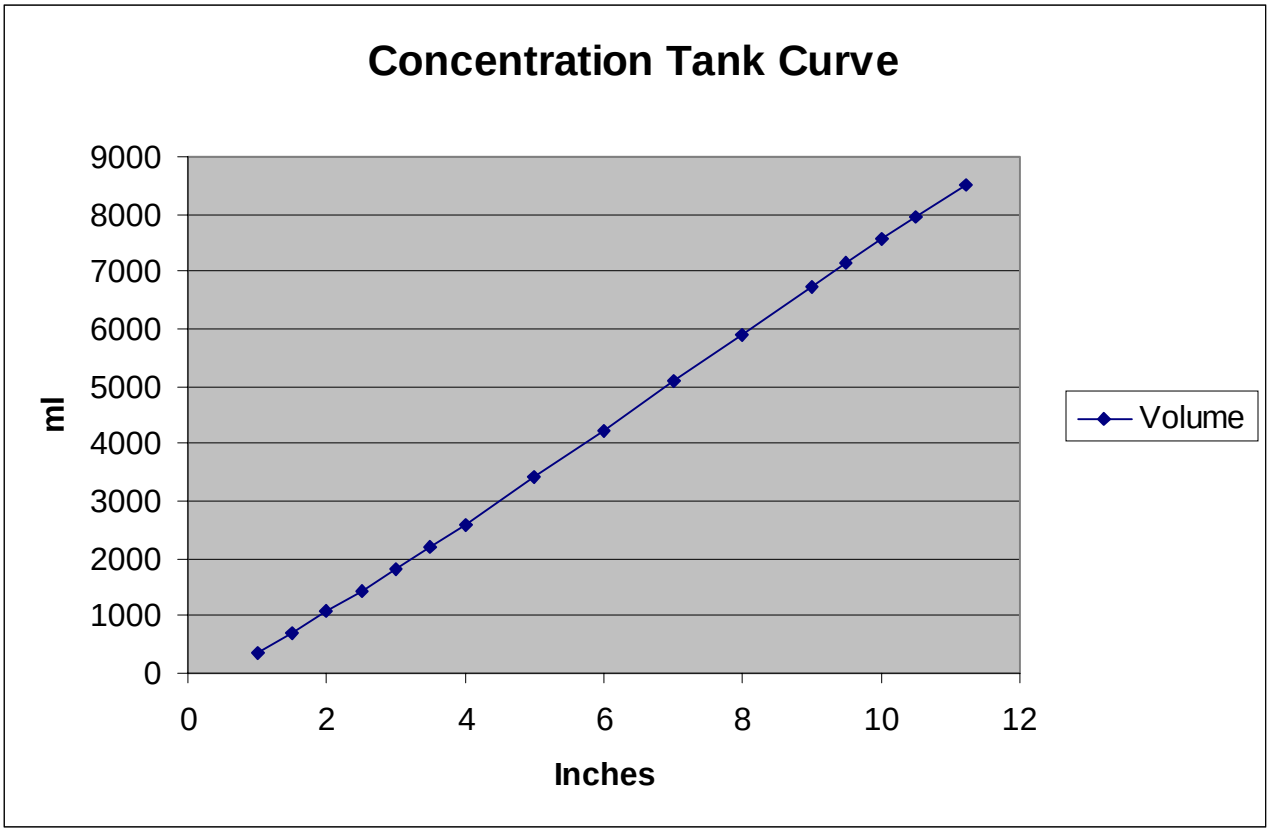
Description: The Slurry Concentration Tank is an 8" diameter stainless steel vessel with a total height of 13.01". Operating ranges are from 1.10" to 12.00". Normal operating level is 8.55".

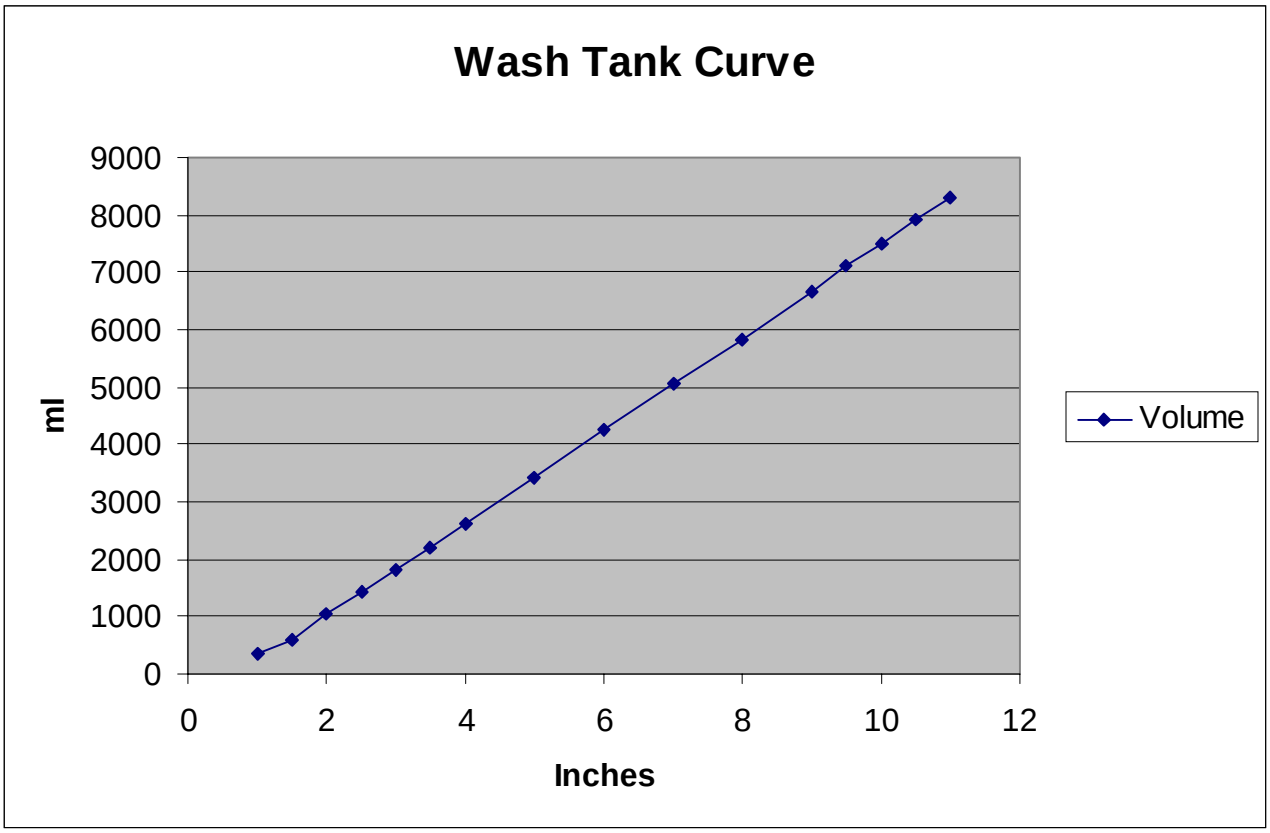
Inside the tank are 1/8" x .67" baffles (4),
3/4" tubes (2), a 7/8" level probe and 1/4" coiled tubing.

Distance from Bottom of Vessel (Inches)	Volume (mL)
1.909	1000
3.247	2000
4.517	3000
5.619	4000
6.89	5000
8.122	6000
9.325	7000
10.507	8000
11.214	8500

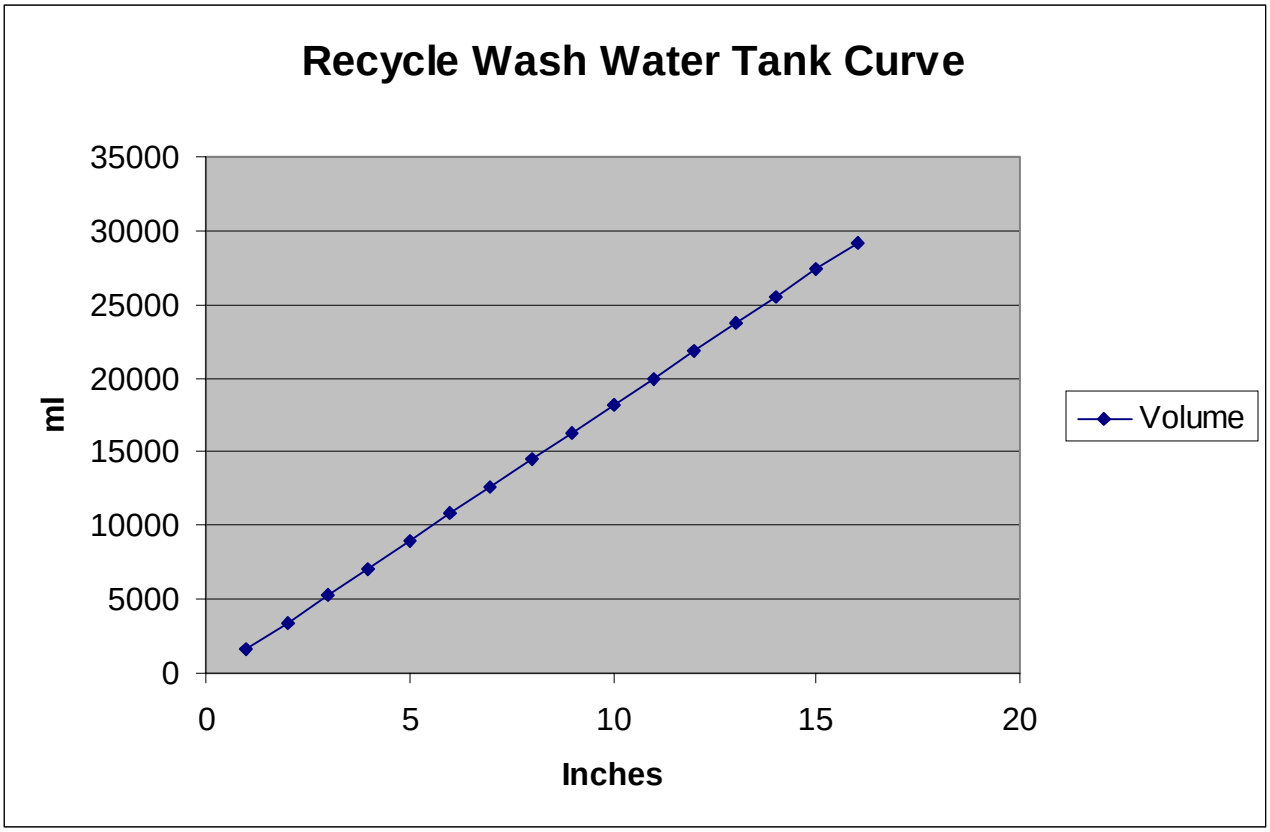
Calculated volumes below were used to verify the above calibration curve.

Volume	Description
50.265482	8" Dia tank - per inch
0.883573	3/4" tubing (2)
0.335000	baffle - (4)
0.578249	1/4" cooling coil (below 7.15")
0.601320	7/8" Lvl probe
0.098175	1/4" straight tubing above 7.15" (2)
2.398143	displacement per inch below 7.15 in.
47.867340	8" Dia tank - per inch less displacement
0.027701	cf/in below 7.15"
1.034495	8" Dia tank displacement per inch above 7.15 in.
49.230987	8" Dia tank volume above 7.15 in
0.028490	cf/in above 7.15"
0.20720353	gal/cf below 7.15"
0.213106357	gal/cf above 7.15"
0.784265363	liters/in below 7.15"
0.80660756	liters/in above 7.15"
784.2653628	mL/in below 7.15"
806.6075601	mL/in above 7.15"


Concentration Tank Curve
9000
8000
7000
6000
5000
4000
3000
2000
1000
0
ml
0
2
4
6
8
10
12
Inches
Volume


Wash Tank Curve
9000
8000
7000
6000
5000
4000
3000
2000
1000
0
ml
0
2
4
6
8
10
12
Inches
Volume

Workaid Number: CERS-08 Revision: 1



Loop I/O Data Base

Record Number	Loop Tag Name	Type	Loop Description	Scan Rate	I/O Type	I/O Address	Signal Conditioning	Low EGU	High EGU	EGU	AI Smoothing	AI Lo	AI LoLo	AI HI	AI HIHI
1	FI-2	AI	Slurry Wash Feed Flow	1	TPI	1:2:AI:4	TSCS	0	5	GPM	0	0	0	5	5
2	FI-4	AI	Recycle Washwater Flow	1	TPI	1:1:AI:8	TSCS	0	25	mL/M	0	0	0	25	25
3	FIC-1	AI	Concentration Filtrate Flow	0.05	TPI	1:1:AI:9	TSCS	0	40	mL/M	0	0	0	40	40
4	FIC-3	AI	Wash Filtrate Flow Rate	0.05	TPI	1:1:AI:10	TSCS	0	25	mL/M	0	0	0	25	25
5	HLA-1	AI	CSTR-2 Sump High Level Alarm	1	TPI	1:1:AI:15	TSCS	-5	5		0	-5	-5	2.5	0
6	HLA-2	AI	Recycle Washwater Sump High	1	TPI	1:1:AI:16	TSCS	-5	5		0	-5	-5	2.5	0
7	LI-3	AI	Recycle Washwater Level Indi	1	TPI	1:1:AI:7	TSCS	0	18	in	0	0	0	18	18
8	LI-4	AI	Salt Feed Tank Level Indicat	1	TPI	1:2:AI:9	TSCS	0	34	in	0	0	0	34	34
9	LIC-1	AI	Concentration Level Indicato	1	TPI	1:1:AI:5	TSCS	1.1	12	in	15	3	7	9.5	10
10	LIC-2	AI	Wash tank Level Indicator	0.05	TPI	1:1:AI:6	TSCS	1.1	12	in	0	3	7	9.5	10
11	PCV-1IN	AI	Concentration Post Cross Flo	1	TPI	1:2:AI:5	TSCS	0	100	%OPN	0	0	0	100	100
12	PCV-1OUT	AO	Concentration Post Cross Flo	----	TPI	1:3:AO:3	LIN	-150	100	%OPN	--	--	----	----	----
13	PCV-2IN	AI	Concentration Cross Flow Pre	1	TPI	1:1:AI:3	TSCS	0	100	%OPN	0	0	0	100	100
14	PCV-2OUT	AO	Concentration Cross Flow Pre	----	TPI	1:3:AO:1	LIN	-150	100	%OPN	--	--	----	----	----
15	PCV-3IN	AI	Slurry Wash Post Cross Flow	1	TPI	1:2:AI:6	TSCS	0	100	%OPN	0	0	0	100	100
16	PCV-3OUT	AO	Slurry Wash Post Cross Flow	----	TPI	1:3:AO:4	LIN	-150	100	%OPN	--	--	----	----	----
17	PCV-4IN	AI	Slurry Wash Cross Flow Pres	1	TPI	1:1:AI:4	TSCS	0	100	%OPN	0	0	0	100	100
18	PCV-4OUT	AO	Slurry Wash Cross Flow Pres	----	TPI	1:3:AO:2	LIN	-150	100	%OPN	--	--	----	----	----
19	PG_PIDPCV1	PG		1	---	----	----	----	----	----	--	--	----	----	----
20	PG_PIDPCV3	PG		1	---	----	----	----	----	----	--	--	----	----	----
21	PG_SCAN	PG		30	---	----	----	----	----	----	--	--	----	----	----
22	PI-2	AI	Concentration Cross Flow Pre	1	TPI	1:1:AI:2	TSCS	0	100	PSIG	0	0	0	100	100
23	PI-2/PIC-1DIF	CA	DIFFERENCE BETWEEN PIC-1 AND	----	---	----	----	-5	1		--	--	----	----	----
24	PI-2/PIC-1DIF_LO	AI	PI-2/PIC-1DIF Pressure Lo	1	SIM	60		-1	1		0	-1	-1	0.02	1
25	PI-2/PICDIF_AO	AO	Writes PI-2/PIC-1DIF TO SIM	----	SIM	60		-1	1		--	--	----	----	----

26	PI-4	AI	Slurry Wash Cross Flow Press	1	TPI	1:2:AI:8	TSCS	0	100	PSIG	0	0	0	100	100
27	PI-4/PIC-3DIF	CA	DIFFERENCE BETWEEN PIC-3 AND	----	---	----	----	-5	1		--	--	----	----	----
28	PI-4/PIC-3DIF_AO	AO	Writes PI-4/PIC-3DIF TO SIM	----	SIM	61		-1	1		--	--	----	----	----
29	PI-4/PIC-3DIF_LO	AI	PI-4/PIC-3DIF Pressure Lo	1	SIM	61		-1	1		0	-1	-1	0	1
30	PIC-1	AI	Concentration Post Cross Flo	0.05	TPI	1:1:AI:1	TSCS	0	200	PSIG	0	0	0	200	200
31	PIC-1DIFFEED	AI	Concentration Post Cross Flo	1	TPI	1:1:AI:1	TSCS	0	200	PSIG	0	0	0	200	200
32	PIC-3	AI	Slurry Wash Post Cross Flow	0.05	TPI	1:2:AI:7	TSCS	0	200	PSIG	0	0	0	200	200
33	PIC-3DIFFEED	AI	Slurry Wash Post Cross Flow	1	TPI	1:2:AI:7	TSCS	0	200	PSIG	0	0	0	200	200
34	PID1A/M	DO	PCV-1 Auto Manual	----	SIM	00:00	----	----	----	----	--	--	----	----	----
35	PID1SP	AO	PIDPCV-1 SETPOINT	----	SIM	12		0	100		--	--	----	----	----
36	PID2A/M	DO	PCV-2 Auto Manual	----	SIM	00:01	----	----	----	----	--	--	----	----	----
37	PID3A/M	DO	PCV-3 Auto Manual	----	SIM	00:02	----	----	----	----	--	--	----	----	----
38	PID3SP	AO	PIDPCV-3 SETPOINT	----	SIM	2		0	100		--	--	----	----	----
39	PID4A/M	DO	PCV-4 Auto Manual	----	SIM	00:03	----	----	----	----	--	--	----	----	----
40	PIDPCV-1	PI	----	----	---	----	----	0	200		--	--	----	----	----
41	PIDPCV-2A	PI	----	----	---	----	----	1.1	12		--	--	----	----	----
42	PIDPCV-2B	PI	----	----	---	----	----	0	40		--	--	----	----	----
43	PIDPCV-2LEV/FLW	DR	Level/Flow toggle indicator	----	SIM	00:04	----	----	----	----	--	--	----	----	----
44	PIDPCV-3	PI	----	----	---	----	----	0	100		--	--	----	----	----
45	PIDPCV-4A	PI	----	----	---	----	----	1.1	12		--	--	----	----	----
46	PIDPCV-4B	PI	----	----	---	----	----	0	25		--	--	----	----	----
47	PIDPCV-4LEV/FLW	DR	Level/Flow toggle indicator	----	SIM	00:05	----	----	----	----	--	--	----	----	----
48	PIDPCV1MNL	AO	Manual Control out For PCV-1	----	SIM	4		-150	100	%OPN	--	--	----	----	----
49	PIDPCV3MNL	AO	Manual Control out For PCV-3	----	SIM	6		-150	100	%OPN	--	--	----	----	----
50	SETPOINT2FLOW	AO	PCV-2 Flow Setpoint	----	SIM	8		0	40		--	--	----	----	----
51	SETPOINT2LEVEL	AO	PCV-2 Level Setpoint	----	SIM	9		1.1	12		--	--	----	----	----
52	SETPOINT4FLOW	AO	PCV-4 Flow Setpoint	----	SIM	10		0	25		--	--	----	----	----
53	SETPOINT4LEVEL	AO	PCV-4 Level Setpoint	----	SIM	11		1.1	12		--	--	----	----	----

54	SSI-1D	AI	Concentration Feed Density	1	TPI	1:2:AI:1	TSCS	0	1.3	g/cc	0	0	0	1.3	1.3
55	SSI-1F	AI	Concentration Feed Flow	1	TPI	1:2:AI:2	TSCS	0	5	GPM	0	0	0	5	5
56	SSI-1T	AI	Concentration Feed Temperatu	1	TPI	1:2:AI:3	TSCS	0	100	degC	0	0	0	30	35
57	TI-1	AI	CSTR-1 Temperature	1	TPI	1:1:AI:11	TSCS	0	1,000	degC	0	0	0	1,000.00	1,000.00
58	TI-2	AI	CSTR-2 Temperature	1	TPI	1:1:AI:12	TSCS	0	1,000	degC	0	0	0	1,000.00	1,000.00
59	TI-3	AI	Concentration Temperature	1	TPI	1:1:AI:13	TSCS	0	1,000	degC	0	0	0	1,000.00	1,000.00
60	TI-4	AI	Slurry Washing Temperature	1	TPI	1:1:AI:14	TSCS	0	1,000	degC	0	0	0	1,000.00	1,000.00

Calculation Parameter Database

Record Number	Loop Tag Name	Loop Description	CA Input A	CA Input B	CA Input C	CA Output Expression
1	PI-2/PIC-1DIF	DIFFERENCE BETWEEN PIC-1 AND PI-2	PIC-1.F_CV	PI-2.F_CV	0.9	((A-B)-(A*C))
2	PI-4/PIC-3DIF	DIFFERENCE BETWEEN PIC-3 AND PI-4	PIC-3.F_CV	PI-4.F_CV	0.9	((A-B)-(A*C))

Controller Tuning Parameter Database

Record Number	Loop Tag Name	Type	PID Output Low	PID Output High	PID Set Point Address	PID Prop Band	PID Reset Time	PID Rate	PID Output Reverse	PID Bumpless
1	PIDPCV-1	PI	0	100	PID1SP.F_CV	657.9	0.0125	0	YES	BALANCE
2	PIDPCV-2A	PI	0	40	SETPOINT2LEVEL.F	11.358	94.204	0	YES	BALANCE
3	PIDPCV-2B	PI	0	100	PIDPCV-2A.F_CV	2,035.00	0.255	0	NO	BALANCE
4	PIDPCV-3	PI	0	100	PID3SP.F_CV	424.5	0.013	0	YES	BALANCE
5	PIDPCV-4A	PI	0	100	SETPOINT4LEVEL.F	42	30	0	YES	BALANCE
6	PIDPCV-4B	PI	0	100	PIDPCV-4A.F_CV	969	0.5618	0	NO	BALANCE

Appendix B

CSTR 20-L DEMONSTRATION COSTS

APPENDIX B. CSTR COSTS

This is a listing of the costs during construction and operation of the 20-L CSTR System for Savannah River.

Table B1. Mechanical Items

Purchase Order #	Mechanical Items	# of Items	Purchase Date	Cost	Total	Date Received	Planned Cost
4500005612	Labmaster G2L05R w/speed display/3"mount	1	03/11/99	$ 1,581.00	Mixers	05/14/99	
4500005616	Labmaster G2L05R w/speed display/4"mount	3	03/11/99	$ 5,874.00	$ 7,455.00	04/16/99	$ 13,000.00
4500005671	E+H Coriolis meter 63F-S15AAW00A25A3G	1	03/12/99	$ 5,595.00		04/23/99	
4500005671	E+H Transmitter DZL363-551F311	1	03/12/99	$ 2,620.00		04/23/99	
4500005732	E+H Promag 33, 33F-T15MD1ED11D21A	1	03/12/99	$ 2,665.00		04/05/99	
4500005812	Moyno 500 pumps 33304	2	03/17/99	$ 5,410.00	$ 5,410.00	03/29/99	$ 10,000.00
4500005976	Pressure transmitters	10	03/18/99	$ 2,500.00	Meters	04/23/99	$ 33,000.00
4500005982	INTEK flow meters	3	03/18/99	$ 8,265.00		04/29/99	
4500005984	Pressure control Valves	2	03/18/99	$ 4,846.00		05/06/99	
4500006028	Level meters	3	03/18/99	$ 4,980.00	$ 28,666.00	04/15/99	
4500006311	Level control valves for filter systems	2	03/29/99	$ 4,675.00		04/18/99	
4500006682	Impellers for reactors	6	04/06/99	$ 1,526.00		04/27/99	
4500006691	Back Pressure Regulators	2	04/09/99	$ 2,156.00		04/23/99	
4500006731	Cross-Flow Filter Rigs	2	04/08/99	$ 10,000.00		05/14/99	$ 10,000.00
4500008183	Replacement Baldor Inverter; returned $100	1	05/21/99	$ 100.00		05/28/99	
4500009195	Pressure GaugeTransducer Model P2-200-I	4	06/26/99	$ 1,000.00		07/01/99	
4700061238	Tandem pump heads-54845-607	4	03/12/99	$ 791.20		04/01/99	
4700061238	Piston pump heads 54845-609	2	03/12/99	$ 1,113.20		04/01/99	
4700061238	Piston pump heads 54845-610	2	03/12/99	$ 1,113.20		04/01/99	
4700061238	Peristaltic pump 54845-686	2	03/12/99	$ 1,803.20		04/01/99	
4700061238	Piston pump 54845-690	2	03/12/99	$ 2,012.96	Lab pumps	04/01/99	
4700061238	Piston pump heads 54845-610	2	03/12/99	$ 1,113.20	$ 17,645.14	04/01/99	$ 15,700.00
4700061251	Labmaster Mixer 58957-092	2	03/11/99	$ 2,625.68		04/13/99	
4700061770	Temp bath 13270-615	3	03/15/99	$ 7,072.50		03/28/99	
4700061770	Temp Bath 13270-540	1	03/15/99	$ 2,419.00	Temp Baths	03/28/99	
4700061770	Temp Probe 13270-655	3	03/15/99	$ 427.80	$ 9,919.30	04/12/99	$ 6,000.00
4700061829	Poly Tank 5Gal, 60453-025	2	03/15/99	$ 48.00		03/22/99	
4700062845	Analy. Balance 11273-708	1	03/17/99	$ 2,636.00	$ 2,636.00	03/24/99	
4700063824	Peristaltic pump 54845-686	2	03/12/99	$ 1,803.20		04/01/99	
4700063824	Piston pump 54845-690	2	03/12/99	$ 2,012.96	Lab pumps	04/01/99	
4700066185	Balances for tank weight	2	03/26/99	$ 577.50		04/01/99	
4700071376	Piston pump heads 54845-609	2	04/12/99	$ 1,113.20		04/24/99	
4700076717	Water Purification Cartriges	2	05/03/99	$ 1,062.50		05/06/99	
4700078498	Sage syringe pump	1	05/04/99	$ 1,449.00		05/13/99	
4700088055	Bench Scale, 110lb capacity	2	06/08/99	$ 866.25		06/08/99	
	Magnetrol level probe; Fluid Flow of TN	5	07/23/99	$ 2,563.37		07/23/99	
	Level probe; Venture	5	07/27/99	$ 1,651.20		07/27/99	
	MECHANICAL ITEMS COST		**TOTAL**	**$ 100,068.12**			**$ 87,700.00**

Table. B2. P & E Purchases

Purchase Order	Company or Vendor Purchased From	Description of P & E Items for Reactors	# of Items	Purchase Date	Cost	Date Received
4500008200	Drum-Mates, Inc.	variable speed mixer	2	06/11/99	$ 2,514.00	06/11/99
4700058913	Apex Supply Company Inc.	pipe, stainless steel, 24'	2	03/08/99	$ 122.08	03/13/99
4700058914	Apex Supply Company Inc.	pipe, stainless steel, 24'	5	03/08/99	$ 498.50	03/13/99
4700058915	Apex Supply Company Inc.	flange, stainless steel, 10"	3	03/08/99	$ 776.94	03/13/99
4700058916	Apex Supply Company Inc.	flange, blind, stainless steel, 10"	3	03/08/99	$ 631.05	03/13/99
4700058918	Apex Supply Company Inc.	flange, stainless steel slip-on, raised face	1	03/08/99	$ 165.97	03/13/99
4700058919	Apex Supply Company Inc.	blind flange, stainless steel, 8"	1	03/08/99	$ 120.46	03/13/99
4700058920		hex nut, stainless steel, 3/4-10 18-8	10	03/08/99	$ 5.80	03/13/99
4700058920		hex nut, stainless steel, 7/8-9 18-8	40	03/08/99	$ 56.80	03/13/99
4700058921		hex cap screw, stainless steel, 3/4-10x3-1/2 18-8	10	03/08/99	$ 23.10	03/13/99
4700059014	Royal Brass & Hose	hex cap screw, stainless steel, 7/8-9 x 3 3/4"	65	03/08/99	$ 137.15	03/13/99
4700060563	Apex Supply Company Inc.	pipe cap, 10" stainless steel butt weld	3	03/11/99	$ 349.95	03/16/99
4700060564	Apex Supply Company Inc.	pipe cap, 8" stainless steel butt weld	1	03/11/99	$ 106.26	03/16/99
4700061905	Royal Brass & Hose	hex nut, stainless steel, 7/8-9 18-8	40	03/06/99	$ 56.80	03/26/99
4700063611		flange, 10" stainless steel	1	03/19/99	$ 258.98	03/24/99
4700063612	Apex Supply Company Inc.	flange, 10" blind stainless steel	1	03/19/99	$ 210.35	03/24/99
4700063613	Apex Supply Company Inc.	pipe cap, 10" stainless steel butt weld	1	03/19/99	$ 116.65	03/24/99
4700063614	Royal Brass & Hose	hex nut, stainless steel, 7/8-9 18-8	20	03/19/99	$ 28.40	03/29/99
4700063780	Roden Electrical Supply	3 phase transformer	1	03/19/99	$ 315.55	03/24/99
4700064414	Apex Supply Company Inc.	flange, stainless steel, 8"	1	03/23/99	$ 165.97	03/28/99
4700064415	Apex Supply Company Inc.	pipe cap, stainless steel butt weld, 8"	1	03/23/99	$ 106.26	03/28/99
4700064417	Apex Supply Company Inc.	flange, stainless steel slip-on, 8"	1	03/23/99	$ 120.46	03/28/99
4700065740		hex nut screw, stainless steel, 3/4-10x3-1/2 18-8	10		$ 23.10	
4700065741		hex nut, stainless steel, 3/4-10 18-8	10		$ 5.80	
4700068925	Ridge Valve & Fitting Co.	valve, 3 way, tube fitting ends, 3/4"	3	04/06/99	$ 637.80	04/11/99
4700071093	Ridge Valve & Fitting Co.	valve, ball, "40" series, stainless, 3/4" swagelok	6	04/12/99	$ 913.74	04/19/99
4700071093	Ridge Valve & Fitting Co.	valve, ball, "40" series, stainless, 1/4" swagelok	8	04/12/99	$ 488.40	04/19/99
4700071094	Ridge Valve & Fitting Co.	valve, ball, "42"series, stainless, 1/4" swagelok	20	04/12/99	$ 1,044.00	04/26/99
4700071095	Ridge Valve & Fitting Co.	valve, 3 way, 3/4" swagelok	9	04/12/99	$ 1,840.50	04/17/99
4700071487	Apex Supply Company Inc.	process gauge	2	04/13/99	$ 148.56	04/18/99
4700072105	Royal Brass & Hose	washer, flat, stainless steel, 1.8750x.813 3/4 18-8	24	04/14/99	$ 7.92	04/17/99
4700072106	Royal Brass & Hose	washer, flat, stainless steel, 2.250 x.969 7/8 18-8	32	04/14/99	$ 14.08	04/24/99
4700073551	Oneal Steel Inc.	angle, stainless steel	40.17 ft.	04/20/99	$ 82.75	04/26/99
4700073767	Apex Supply Company Inc.	tubing, stainless steel, seamless, 1/4 .035	100	04/20/99	$ 126.00	04/25/99

Table. B2. P & E Purchases, cont'd

Purchase Order	Company or Vendor Purchased From	Description of P & E Items for Reactors	# of Items	Purchase Date	Cost	Date Received
4700077040	Ridge Valve & Fitting Co.	connector, male, stainless steel	35	04/29/99	$ 216.60	05/06/99
4700077344	Ridge Valve & Fitting Co.	connector, male, stainless steel	9	04/29/99	$ 43.20	05/06/99
4700077506	Ridge Valve & Fitting Co.	valve, 3-way, stainless steel, 3/4"	3	04/29/99	$ 637.80	05/04/99
4700077796	Ridge Valve & Fitting Co.	union, tube, stainless steel	1	04/30/99	$ 29.11	05/07/99
4700077799	Ridge Valve & Fitting Co.	set, ferrule, teflon	40	04/30/99	$ 99.30	05/07/99
4700078351	Ridge Valve & Fitting Co.	connector, hose, stainless steel	12	05/04/99	$ 74.76	05/11/99
4700078352	Apex Supply Company Inc.	plug, 1" stainless steel	2	05/04/99	$ 7.30	05/09/99
4700080457	Roden Electrical Supply	wire, 4 conductor	500 ft.	05/11/99	$ 60.00	05/11/99
4700080458	Roden Electrical Supply	wire, 2 conductor	500 ft.	05/11/99	$ 65.00	05/11/99
4700083356	Ridge Valve & Fitting Co.	connector, hose, stainless steel	12	05/20/99	$ 74.76	06/01/99
4700084405	Ridge Valve & Fitting Co.	union tees, needle valves, & 1/4" stainless nuts	30	05/25/99	$ 307.84	06/04/99
4700084801	Apex Supply Company Inc.	bushing, stainless steel, screwed, 1"x3/4"	4	05/26/99	$ 23.64	06/03/99
4700085737	Dixie Industrial Supply	replacement cutter	3	05/28/99	$ 9.81	06/02/99
4700088548	Royal Brass & Hose	3/8 T-316 standard	100	06/09/99	$ 9.00	06/14/99
4700088809	Royal Brass & Hose	1/4-20x72, 3/8-16x72, & 1/2-13x72 18-8 stainless	18	06/10/99	$ 177.54	06/16/99
4700089208	Ridge Valve & Fitting Co.	union elbow stainless & union tee stainless	14	06/11/99	$ 557.26	06/22/99
4700089563	Roden Electrical Supply	1/2" L/T con tubing, LT conn 90d tubing, & sealtite	140	06/14/99	$ 129.80	06/14/99
4700089564	Roden Electrical Supply	LEV 5269-C fem connector	20	06/14/99	$ 81.00	06/24/99
4700091002	Ridge Valve & Fitting Co.	Connector, female	4	06/18/99	$ 85.60	06/28/99
4700091275	Apex Supply Company Inc.	tubing, 1/4" x .028	20ft	06/21/99	$ 98.40	06/29/99
4700091581	Ridge Valve & Fitting Co.	elbow, union, stainless	10	06/22/99	$ 388.10	07/01/99
4700091581	Ridge Valve & Fitting Co.	ferrule, back, stainless	10	06/22/99	$ 18.80	07/01/99
4700091581	Ridge Valve & Fitting Co.	ferrule, front, stainless	10	06/22/99	$ 21.30	07/01/99
4700091581	Ridge Valve & Fitting Co.	nut, stainless, 3/4"	10	06/22/99	$ 51.60	07/01/99
4700091581	Ridge Valve & Fitting Co.	connector, male, stainless steel	8	06/22/99	$ 135.60	07/01/99
4700091581	Ridge Valve & Fitting Co.	tee, union, 316 stainless	4	06/22/99	$ 183.08	07/01/99
4700092597	Ridge Valve & Fitting Co.	plug, male elbow, & union elbow	9	06/25/99	$ 94.81	07/07/99
4700092598	Ridge Valve & Fitting Co.	elbow, union, stainless	4	06/25/99	$ 43.48	07/07/99
4700099305	V W R Scientific	tubings of various sizes	8	07/25/99	$ 204.97	08/02/99
es: 0000009462		drum, carbon steel, 02-112-5885	2	06/15/99	$ 120.10	06/15/99
es: 0000010480		drum, carbon steel, 02-112-6400	1	07/22/99	$ 42.19	07/22/99

Table. B2. P & E Purchases, cont'd

Purchase Order Reference #	Company or Vendor Purchased From	Description of P & E Items for Reactors	# of Items	Purchase Date	Cost	Date Received
969667039	Knoxville Rubber & Gasket Co.	Gaskets, viton ring, 10" & 8"		03/12/99	$ 203.28	03/15/99
969667051	Knoxville Rubber & Gasket Co.	gasket, viton ring, 1/8" thick	2	03/24/99	$ 34.32	03/26/99
923762639	Rodem Inc.	clamps, ferrule, gaskets	8	03/26/99	$ 311.37	03/28/99
923762712	Rodem Inc.	gaskets	7	03/26/99	$ 149.56	03/28/99
970290706	Sears Roebuck	(tools) flashligh, screwdriver, hammer	9	03/25/99	$ 75.78	03/28/99
643727930	Rodem Inc.	ferrules, clamps, gaskets	16	04/07/99	$ 373.26	04/08/99
329005075	Newark Electronics		24	04/07/99	$ 207.24	04/08/99
910127760	Rodem Inc.	gaskets	7	04/09/99	$ 18.21	04/11/99
021104682	Omega Engineering, Inc.	probes, low noise	12	04/08/99	$ 374.13	04/11/99
329008070	Newark Electric	(electric supplies) 1951/3T-250-BLK PL25001	1	04/14/99	$ 84.08	04/15/99
329001382	Newark Electric	(electric supplies) 5440/4-100	1	04/20/99	$ 350.25	04/21/99
329001829	Newark Electric	electric supplies	49	04/21/99	$ 605.87	04/22/99
329002537	Newark Electric	electric supplies	25	04/22/99	$ 131.65	04/23/99
329004488	Newark Electric	electric supplies	5	04/27/99	$ 63.65	04/28/99
461089958	Rodem Inc.	gaskets	6	05/04/99	$ 14.63	05/05/99
960261174	Cole-Parmer Instrument Co.	frame, flowtube, cartridge valve	12	05/07/99	$ 1,642.85	05/10/99
000014460	Rodem Inc.	fuses	10	05/13/99	$ 12.14	05/16/99
000014635	Rodem Inc.	locking male cap & locking female connector	4	05/13/99	$ 47.13	05/16/99
969667018	Knoxville Rubber & Gasket Co.	clear braid	417 ft.	05/14/99	$ 249.43	05/17/99
007936486	MSC Industrial Direct Co., Inc.	case hoods one size Kimberly Clark Kleenguard	1	05/17/99	$ 108.49	05/20/99
329003718	Newark Electric	electric supplies	30	05/17/99	$ 431.85	05/18/99
000127893	McMaster-Carr Supply Co.	TYVEK-QC accessories	10	05/18/99	$ 109.58	05/20/99
000113866	McMaster-Carr Supply Co.	drum pump	2	05/19/99	$ 1,651.68	05/21/99
007977528	MSC Industrial Supply Co.	hold cap clamp & oil-impreg brnz flng brg	18	05/20/99	$ 94.36	05/23/99
000126387	McMaster-Carr Supply Co.	hose clamp, hose fitting, hose nipple, steel hexagon	7	05/21/99	$ 79.12	05/24/99
141010012	Edwards Supply Co. Inc.	conductor & SJ cord	600 ft.	05/01/99	$ 213.75	05/23/99
968311432	Cole-Parmer Instrument Co.	wave guide, level transmitter, panel meter, line cord	4	05/26/99	$ 749.47	05/28/99
000183508	McMaster-Carr Supply Co.	swivel leveling mount with bolts	9	05/26/99	$ 668.79	05/28/99
000180206	McMaster-Carr Supply Co.	swivel leveling mount with bolts	1	05/27/99	$ 76.34	05/30/99
175487820	Home Depot	drill, wrenches, batteries, bit, & C-clamp	9	05/27/99	$ 187.34	05/30/99
000129924	McMaster-Carr Supply Co.	swivel leveling mount with bolts	4	06/01/99	$ 89.27	06/03/99
001128812	Newark Electronics	RN55C-249	100 ft.	06/03/99	$ 25.00	06/04/99

Table. B2. P & E Purchases, cont'd

Purchase Order	Company or Vendor Purchased From	Description of P & E Items for Reactors	# of Items	Purchase Date	Cost	Date Received
021104770	Omega Engineering Inc.	extension wire & quick disconnect T/C connectors	13	06/07/99	$ 261.81	06/09/99
175404034	Home Depot	batteries & 1/2 CHULK	4	06/08/99	$ 34.92	06/10/99
286007653	Black & Decker	extension, back pad, snakellight camplte, straig bld,. Jig	10	06/08/99	$ 43.08	06/09/99
020049634	Lowe's	flowering del, engineer's ha, spring links, female ball, bra	13	06/10/99	$ 43.76	06/13/99
000175610	McMaster-Carr Supply Co.	stainless steel shaft & mixing propeller	2	06/11/99	$ 167.59	06/14/99
400220124	Ridge Valve & Fitting Co.	valve & seal kit	2	06/11/99	$ 708.65	06/13/99
400230020	Ridge Valve & Fitting Co.	bar handle kit	2	06/11/99	$ 431.71	06/18/99
400220090	Ridge Valve & Fitting Co.	valve	4	06/11/99	$ 261.68	06/13/99
161010035	Edwards Supply Co. Inc.	Leviton twist locks	30	06/13/99	$ 271.35	06/14/99
969667069	Knoxville Rubber & Gasket Co. I	tubing: clear braided, red poly, clear poly; nickel anti-sei	1000ft.; 1	06/15/99	$ 200.15	06/17/99
400230103	Ridge Valve & Fitting Co.	union elbow, union tee, male connector	14	06/15/99	$ 287.30	06/18/99
973355747	Sears Roebuck	tape, carburetor, paint brushes, ext bar fl	6	06/15/99	$ 22.93	06/17/99
680424656	Grainger	GPM transfer pump	1	06/16/99	$ 601.98	06/18/99
001168714	Newark Electric	electric supplies	8	06/16/99	$ 98.38	06/17/99
175313764	Home Depot	blade, keys, surge strip, ext. pole, organizer, lamp, swag	27 items	06/17/99	$ 231.01	06/20/99
400230145	Ridge Valve & Fitting Co.	bar handle kit	10	06/17/99	$ 620.73	06/18/99
773245917	Springfield Cop	std platform truck & cart	2	06/18/99	$ 376.23	06/21/99
		P & E ITEMS COST	**TOTAL**		**$ 30,409.01**	
Res = Reservation Identification Number						

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Table B3. Chemicals for Operation

Purchase Order #	Chemicals for feed make-up	# of Items	Purchase Date	Cost	Date Received	Lot Numbers	Purity, %
4500007139	Cesium 137	2	04/22/99	$ 2,360.00	05/11/99		
4500007319	NaTPB	4	05/01/99	$ 5,074.20	05/07/99	EU 09009MS	99.00%
4500008589	Strontium-85	1	06/10/99	$ 1,396.00	06/17/99	99MS1M10	99.88%
4500009626	Strontium 85, Package of 10 mCi	3	07/19/99	$ 4,188.00	07/22/99		
4700072170	Sodium Sulfate EM-SX0760-5	1	04/14/99	$ 229.37	04/20/99	38362902, 126754/1159	99.00%
4700072171	Potassium Nitrate MK702802	7	04/14/99	$ 133.24	04/20/99	7028KXTG (3), 7028KXD	99.00%
4700072172	Sodium Sulfate EM-SX0760-5	1	04/14/99	$ 229.37	04/20/99	38362902, 126754/1159	99.00%
4700072173	Sodium Fluoride MK763604	2	04/14/99	$ 111.66	04/20/99	7636 M42619	99.60%
4700072174	Sodium Chloride JT4058-1	2	04/14/99	$ 37.92	04/20/99	H4794058012C +$N0146	99.90%
4700072175	Sodium Phosphate dibasic MK791404	3	04/14/99	$ 67.98	04/20/99	7914KXNY	99.90%
4700072235	Sodium Molybdate JT3764-1	1	04/14/99	$ 107.24	04/20/99	H4793764012F +$M376	99.90%
4700072236	Sodium Meta Silicate JT3868-1	2	04/14/99	$ 99.08	05/07/99	K15161 & M16144	99.90%
4700072237	Sodium Nitrite EM-SX0665-7	2	04/14/99	$ 885.64	04/20/99	38253840	97.00%
4700072238	Sodium Oxalate EM-SX0685-2	3	04/14/99	$ 73.59	05/06/99	38190834	99.50%
4700072239	Sodium Nitrate JT3770-7	5	04/14/99	$ 1,140.70	04/27/99	M14156	99.80%
4700072240	Aluminum Nitrate JT0528-9	1	04/14/99	$ 614.38	05/18/99	M25146	98-102%
4700072242	Sodium Hydroxide MK771222	3	04/14/99	$ 905.55	04/23/99	M22635	Purified
4700081911	Nitric Acid, JT9607-4	1	05/15/99	$ 127.14	06/22/99	Jt9607-4	69-71%
4700082968	Ethylene Glycol MK500119	1	05/19/99	$ 155.46	05/19/99		
4700084266	Sodium Carbonate RE Mono JT3598-7	1	05/24/99	$ 152.93	05/24/99		
4700087522	Sodium Carbonate Monohydrate EM SX0405-3	1	06/05/99	$ 76.80	06/11/99	37310823	99.00%
4700087523	Ethylene Glycol	1	06/05/99	$ 72.45	06/11/99		
4700087524	Sodium Carbonate mono hydrate EM SX0405-3	1	06/05/99	$ 22.50	06/11/99		
4700092931	Oxalic Acid, Dihydra EM-OX0280-2	2	06/27/99	$ 67.18	07/07/99		
4700094457	Sodium Hydroxide MK771220	1	07/01/99	$ 109.86	07/12/99		
4700094458	Potassium Nitrate MK702802	2	07/01/99	$ 34.92	07/12/99		
4700094459	Aluminum Nitrate JT0528-9	1	07/01/99	$ 614.38	07/12/99		
4700094460	Sodium Phosphate MK791404	1	07/01/99	$ 22.60	07/12/99		
4700094461	Sodium Nitrate JT3770-7	1	07/01/99	$ 236.36	07/12/99		
4700094761	Sodium Molybdate, Di JT3764-1	1	07/02/99	$ 110.72	07/16/99		
4700094762	Cupric Sulfate, 5-Hy JT1844-1	1	07/02/99	$ 56.48	07/16/99		
4700094763	Arsenic Trioxide, Powder	1	07/02/99	$ 236.32	07/16/99		
4700094764	Sodium meta-Silicate JT3868-1	1	07/02/99	$ 49.54	07/16/99		
4700094765	Lead Nitrate, Crstal JT2322-4	1	07/02/99	$ 30.28	07/16/99		

Table B3. Chemicals for Operation, cont'd.

Purchase Order #	Chemicals for feed make-up	# of Items	Purchase Date	Cost	Date Received	Lot Numbers	Purity, %
4700094766	Zinc Nitrate, 6-Hydr JT4344-1	1	07/02/99	$ 31.01	07/16/99		
4700094767	Mercuric Nitrate, Mo JT2614-4	1	07/02/99	$ 95.64	07/16/99		
4700094768	Ferric Nitrate, 9-Hy JT2018-1	1	07/02/99	$ 47.70	07/16/99		
4700094769	Stannous Chloride, D EM-SX0885-3	1	07/02/99	$ 25.17	07/16/99		
4700094770	Calcium Nitrate, 4-H JT1395-1	1	07/02/99	$ 35.29	07/16/99		
4700094771	Cadmium Nitrate, 4-H JT1226-4	1	07/02/99	$ 87.00	07/16/99		
4700094772	Lanthanum Nitrate, n MKE02057	1	07/02/99	$ 56.56	07/16/99		
4700094773	2-Propanol, (isopropyl) JT9084-6	1	07/02/99	$ 13.84	07/16/99		
4700094774	Cobalt Nitrate, 6-Hy MK454402	1	07/02/99	$ 24.81	07/16/99		
4700094775	Biphenyl, Practical JTC278	1	07/02/99	$ 23.84	07/16/99		
4700094776	Methanol, (methyl alcohol) JT9076-1	1	07/02/99	$ 8.87	07/16/99		
4700094777	Phenol GR EM-PX0510-1	1	07/02/99	$ 41.24	07/16/99		
4700094778	Benzene, (Benzol), T EM-BX0220-13	1	07/02/99	$ 19.09	07/16/99		
Aldrich: 04752	Palladium (II) Nitrate, Hydrate	2	07/08/99	$ 368.80	07/09/99		
Aldrich: B7830	Rhodium (II) Nitrate Solution	2	07/08/99	$ 978.60	07/09/99		
Aldrich: 07225	Ruthenium (III) Chloride hydrate	2	07/08/99	$ 260.00	07/09/99		
Aldrich: 05672	Diphenylmercury	16	08/02/99	$ 450.60	08/03/99		
Aldrich: 06385	Sodium Dichromate	1	07/08/99	$ 30.45	07/08/99		
Aldrich: 08599	Sodium Selenate	1	07/08/99	$ 13.60	07/08/99		
Aldrich: C3169	Triphenylborane-sodium adduct	13	07/08/99	$ 308.10	07/08/99		
Aldrich: C3170	Phenylboranic acid	2	07/08/99	$ 278.30	07/08/99		
Aldrich: C3168	Diphenylborinic acid ethanolamine ester	4	07/08/99	$ 466.80	07/08/99		
Aldrich:	Sodium Tetraphenyl Borate	16	08/03/99	$ 140.95	08/03/99		
Already in Hous	Cesium Chloride			No Charge			
Already in Hous	Strontium nitrate (Baker Analyzed)			No Charge		Lot 626131	99.90%
	Surfynol# 420 Surfactant		04/14/99	No Charge	04/21/99	BP11738	
	CHEMICAL ITEMS COST		**TOTAL**	**$ 23,636.10**			

Table B4 Miscellaneous Expenses

Purchase Order #	Miscellaneous Items	# of Items	Purchase Date	Cost	Date Received
Res: 0000006574	Bag, Polyethylene, Clear 15-005-1237	1	03/17/99	$ 160.65	03/24/99
Res: 0000006663	Boots, Polyvinyl Chloride 12-010-4307	100	03/21/99	$ 234.00	03/29/99
Res: 0000007094	Bag, polyethylene sq. fold 15-005-0731	50	04/01/99	$ 66.50	04/01/99
Res: 0000007331	Bag, Polyethylene, Clear 15-005-1290	2	04/09/99	$ 157.16	04/09/99
Res: 0000007345	Bag, Laundry, Nylon 08-020-0204	4	04/09/99	$ 92.92	04/09/99
Res: 0000007345	Bag, Polyethylene, Clear 15-005-1237	4	04/09/99	$ 642.60	04/09/99
Res: 0000008583	Boots, Polyvinyl Chloride 12-010-4307	100	05/15/99	$ 234.00	05/17/99
Res: 0000009403	Tubing, Polyethylene 15-282-3420	1	06/13/99	$ 0.05	06/14/99
Res: 0000009403	Tubing, Polyethylene 15-282-1980	1	06/13/99	$ 101.34	06/13/99
Res: 0000009403	Tubing, Polyethylene 15-282-2000	1	06/13/99	$ 106.70	06/14/99
Res: 0000009482	Boots, Plastic 12-010-4307	100	06/17/99	$ 234.00	06/17/99
Res: 0000009609	Sheet, Vinyl, Reinforced 07-450-8720	1	06/18/99	$ 377.43	06/21/99
Res: 0000009853	Boots, Polyvinyl Chloride 12-010-4307	100	06/26/99	$ 234.00	06/28/99
Res: 0000009853	Sleeve, 18" Elastic Top 12-016-6800	100	06/26/99	$ 97.00	06/28/99
Res: 0000009904	Bag, polyethylene sq. fold 15-005-0681	20	06/27/99	$ 59.60	06/28/99
Res: 0000009904	Bag, polyethylene sq. fold 15-005-0731	50	06/27/99	$ 66.50	06/28/99
Res: 0000009904	Bag, polyethylene sq. fold 15-005-0701	20	06/27/99	$ 34.60	06/28/99
Res: 0000010075	Bag, Polyethylene, RAD 15-005-1290	2	07/07/99	$ 157.16	07/07/99
Res: 0000010085	Bag, Polyethylene 15" 15-005-0701	10	07/07/99	$ 17.30	07/07/99
Res: 0000010623	Boots, Polyvinyl Chloride 12-010-4307	100	07/25/99	$ 234.00	07/25/99
Res: 0000010623	Stocknet, Cotton, Knit Tube 12-040-9307	1	07/25/99	$ 16.68	07/25/99
Res: 0000010624	Glove Liners, Cotton 12-020-9500	12	07/25/99	$ 61.32	07/25/99
Res: 0000010625	Bag, Polyethylene sq. fold 15-005-0701	20	07/25/99	$ 34.60	07/25/99
Res: 0000010625	Bag, Polyethylene sq. fold 15-005-0731	50	07/25/99	$ 66.50	07/25/99
Res: 0000011218	Belt, Leather, 42" 12-010-0970	2	07/27/99	$ 18.02	07/27/99
Res: 0000011230	Belt, Leather, 42" 12-010-0975	1	07/28/99	$ 9.01	07/28/99
Res: 0000011281	Belt, Leather, 42" 12-010-0970	1	07/28/99	$ 9.01	07/28/99
4500006135	Lab VIEW 5.1 Upgrade, for Windows 98	1	04/05/99	$ 295.00	04/05/99
4700055946	Rad Gloves ATCP1812/M-9	144	03/01/99	$ 177.12	03/01/99
4700055946	Rad Gloves ATCP1812/M-10	144	03/01/99	$ 177.12	03/01/99
4700058603	PCWIN95-UPG PROG MGM	1	03/08/99	$ 179.67	03/19/99
4700058628	Inkjet transparency	2	03/08/99	$ 47.32	03/26/99
4700061387	Rad Gloves ATCP1812/M-9	144	03/13/99	$ 177.12	03/29/99
4700061387	Rad Gloves ATCP1812/M-10	144	03/13/99	$ 177.12	03/29/99
4700062646	Cartridge, Ink, & InkJ; JTFJF50000	1	03/17/99	$ 14.67	03/22/99

Table B4 Miscellaneous Expenses, cont'd

Purchase Order #	Miscellaneous Items	# of Items	Purchase Date	Cost	Date Received
4700062646	Cartridge, InkJet, R: NUKRF172	1	03/17/99	$ 22.40	03/22/99
4700062653	Cartridge, Print Black for DJ	2	03/17/99	$ 50.94	03/27/99
4700065784	Book, Laboratory Record	6	03/25/99	$ 184.56	03/30/99
4700066883	Cheesecloth lint free	120	03/29/99	$ 355.20	04/01/99
4700066884	Tyvek suits	25	03/31/99	$ 93.50	04/01/99
4700066884	Tyvek suits	25	03/31/99	$ 94.00	04/01/99
4700068211	Cheeseclothe Grade 60	1	04/01/99	$ 659.28	04/01/99
4700069965	Computer	1	04/08/99	$ 2,617.00	04/23/99
4700071912	Monitor, PR	1	04/14/99	$ 480.94	04/23/99
4700073841	Network EN104 Ether	2	04/20/99	$ 88.12	04/30/99
4700078665	0.22 Micron Filter	2	05/04/99	$ 258.72	05/06/99
4700079117	Tygon tubing	19	05/03/99	$ 864.23	05/14/99
4700081573	Vinyl gloves	10	05/13/99	$ 112.50	05/17/99
4700081912	Stirrer, W/Ring Stand	1	05/15/99	$ 703.80	05/17/99
4700081915	Bottle Carboy, Autocl	1	05/16/99	$ 153.18	05/18/99
4700081915	Bottle Carboy, PP	1	05/16/99	$ 145.93	05/18/99
4700081916	Bottle, Narrow Mouth	3	05/16/99	$ 229.32	05/21/99
4700081917	Iomega Zip 100 MB USB	1	05/16/99	$ 138.34	05/17/99
4700081918	Cartridge Iomega ZI	2	05/16/99	$ 209.30	05/17/99
4700082988	Ink jet papers	6	05/19/99	$ 162.57	05/24/99
4700083135	Wipers, Kaydry	1	05/19/99	$ 163.88	05/24/99
4700083135	Bottle, WM, PP	3	05/19/99	$ 114.93	05/20/99
4700083803	CD ROM Writable, External 24X 6X	1	05/21/99	$ 470.00	05/21/99
4700083805	CD ROM Internal Writable	1	05/21/99	$ 241.55	05/21/99
4700084349	CD, Recordable	20	05/24/99	$ 112.40	05/25/99
4700084351	Cable Internal Audi	2	05/24/99	$ 2.00	05/25/99
4700087708	Paper, Ink Jet, 20#	3	06/07/99	$ 20.79	06/08/99
4700088596	BTL. WM, PP, 60 mL, PK12	3	06/09/99	$ 145.32	06/14/99
4700088596	GHP Acrodisc syringe	3	06/09/99	$ 1,014.90	06/14/99
4700088596	Syringe Bulk CS-850	1	06/09/99	$ 143.27	06/14/99
4700088596	SYR, Bulk, NS, LUER 20	1	06/09/99	$ 160.79	06/14/99
4700089274	Tape, Masking, Painters Grade	10	06/13/99	$ 19.60	06/13/99
4700090458	Rad Gloves ATCP1812/M-9	144	06/16/99	$ 177.12	06/17/99

Table B4 Miscellaneous Expenses, cont'd

Purchase Order #	Miscellaneous Items	# of Items	Purchase Date	Cost	Date Received
4700090458	Rad Gloves ATCP1812/M-10	144	06/16/99	$ 177.12	06/17/99
4700091148	0.45 Micron Filter	2	06/18/99	$ 258.72	6/28,29/1999
4700091506	N 99.97% minimum purity (gas)	4	06/21/99	$ 27.20	06/21/99
4700092225	BTL. WM, PP, 125mL, PK12	4	06/23/99	$ 210.44	06/24/99
4700092927	Rad Gloves ATCP1812/M-9	144	06/26/99	$ 177.12	07/01/99
4700092927	Rad Gloves ATCP1812/M-10	144	06/26/99	$ 177.12	07/01/99
4700092930	Cylinders, screw closure, beakers, tubing	19	06/27/99	$ 833.53	07/07/99
4700093974	P-10 Gas, 200 cu. Ft cylinder	4	06/30/99	$ 232.00	06/30/99
4700094038	Cartridge, Ink, Colo	3	06/30/99	$ 99.87	07/01/99
4700094038	Cartridge, Ink, Black	3	06/30/99	$ 97.17	07/01/99
4700094227	Mixed Gas, 90% Ar, 10%CH4,	4	07/01/99	$ 253.76	07/01/99
4700094750	Clear Sleeve	12	07/02/99	$ 299.88	07/16/99
4700095361	Pipette, Ele, Epp4850	1	07/08/99	$ 556.60	07/16/99
4700096136	Binders	8	07/13/99	$ 62.28	07/21/99
4700096370	N 99.97% minimum purity (gas)	6	07/13/99	$ 42.54	07/13/99
4700096656	Tips, Epp, F/4850, Bulk, 5mL	1	07/14/99	$ 71.10	07/22/99
4700098694	Rad Gloves ATCP1812/M-9	432	07/22/99	$ 531.36	07/22/99
4700098694	Rad Gloves ATCP1812/M-10	432	07/22/99	$ 531.36	07/22/99
4700099306	Paper, Ink Jet	4	07/25/99	$ 27.72	07/25/99
4700099445	Nitrite electrode	1	07/26/99	$ 486.00	08/02/99
4700099445	Electrode, Reference	1	07/26/99	$ 112.00	08/02/99
4700099446	Nitrate/ite Outer fill soln	1	07/26/99	$ 56.70	08/02/99
4700099447	Sodium Nitrite STD	1	07/26/99	$ 87.10	08/02/99
4700100475	Bottle, WM, PP, 60mL	3	07/28/99	$ 145.32	07/28/99
4700100475	Bottle, WM, PP, 125mL	3	07/28/99	$ 148.62	07/28/99
4700100475	Autoclavable Carboy with Handles	1	07/28/99	$ 145.93	07/28/99
4700100475	Beaker, Polyp.,Disp Pk-100, 50mL	2	07/28/99	$ 28.62	07/28/99
4700100475	Beaker, Polypropylene, 100 ml Disposable	2	07/28/99	$ 39.14	07/28/99
4700100475	Beaker, 150 mL, Polyprop. Disp. Pk/100	2	07/28/99	$ 67.00	07/28/99
4700100508	Nitrite, Ionic STR ADJ 475 mL	8	07/28/99	$ 396.00	07/28/99
4700101133	Nitrogen, Cylinder, 99.97% minimum purity	6	07/30/99	$ 42.54	08/02/99
4700101425	Bottle, Narrow Mouth	2	08/01/99	$ 152.88	08/01/99
4700101425	Beaker, Polypropylene, disp Pk 50	1	08/01/99	$ 20.36	08/01/99

Table B4 Miscellaneous Expenses, cont'd

Purchase Order #	Miscellaneous Items	# of Items	Purchase Date	Cost	Date Received
4700102196	Rad Gloves ATCP1812/M-9	432	08/04/99	$ 531.36	08/04/99
4700102196	Rad Gloves ATCP1812/M-10	432	08/04/99	$ 531.36	08/04/99
4700102258	Nitrite Ionic, STR ADJ 475 mL	2	08/04/99	$ 99.00	08/04/99
4700102712	Sterilizing Pan	1	08/05/99	$ 110.81	08/05/99
4700102712	Large Wide-Mouth Bottle	4	08/05/99	$ 153.24	08/05/99
4700107279	Clear Sleeve	12	08/19/99	$ 299.88	08/26/99
4700107279	Protector Sleeve	1	08/19/99	$ 102.53	08/26/99
4700108617	VWRbrand Traceable	3	08/24/99	$ 38.91	09/01/99
4700110793	Nitrogen, Cylinder, 99.97% minimum purity	8	08/31/99	$ 56.72	08/31/99
	MISCELLANEOUS ITEMS COST		**TOTAL**	**$ 23,697.08**	
Res = Reservation Identification Number					

Table B5. SR 20-L CSTR Demo Total Costs.

CSTR Cost Reports		Total
CSTR Mechanical Cost Report	$	100,068.12
CSTR P & E Cost Report	$	30,409.01
CSTR Chemical Cost Report	$	23,636.10
CSTR Miscellaneous Cost Report	$	23,697.08
Total Costs	**$**	**177,810.31**

Appendix C

PREPARATION OF SIMULATED WASTE SOLUTIONS FOR CSTR TEST 2

PREPARATION OF SIMULATED WASTE SOLUTIONS FOR CSTR TEST 2

The following procedure was used for the preparation of a simulated waste solution for CSTR Test2 and was based upon the average simulated waste composition as described in the SRS document SCR-TR-99-00116 which is authored by D. D. Walker. According to Walker, the average solution represents a blend of all soluble waste in the SRS tank farms. The average simulated waste formulation has all the major waste components; however, there are a number of hazardous, minor components such as chromium were not included.

Based on the SCR-TR-99-00116, only high purity chemicals were purchased for CSTR Test 2. Table 1 and 2 provides the Lot #s and purity of the chemical that were used.

Table 1. Chemicals used in simulated waste solution preparation for CSTR Tests 2

Chemical	Formula	Lot number[a]	Purity
Potassium nitrate	KNO_3	7028KXTG(3), 7028KXDK(4)	99.0
Sodium hydroxide	NaOH	M22635	Purified
Sodium nitrate	$NaNO_3$	M14156	99.8
Sodium nitrite	$NaNO_2$	38253840	97.0
Aluminum nitrate nonohydrate	$AL(NO_3)_3 \cdot 9H_2O$	M25146	98 – 102
Sodium carbonate monohyrate	$Na_2CO_3 \cdot H_2O$	37310823	99.5
Sodium sulfate	Na_2SO_4	38362902, 126754/115904	99.0
Sodium chloride	NaCL	H4794058012C +$N01467C4	99.9
Sodium fluoride	NaF	7636 M42619	99.6
Sodium monohydrogen phosphate heptahydrate	$Na_2HPO_4 \cdot 7H_2O$	7914KXNY	99.9
Sodium oxalate	$Na_2C_2O_4$	38190834	99.5
Sodium metasilicate nonohydrate	$Na_2SiO_3 \cdot 9H_2O$	K15161 & M16144	99.9
Sodium molybdate monohydrate	$Na_2MoO_4 \cdot H_2O$	H4793764012F +$M37620F6	99.9

Table 2 Other chemical and radioactive additives

Sodium tetraphenylborate	NaTPB	EU 099009MS	>99.6
Cesium chloride	CsCl		99.9
Strontium nitrate	$Sr(NO_3)_2$	626131	99.9
Uranium oxide	UO_2	93.34 isotopic Wt. %	99.9

The calculate average simulated supernatant (6.4 *M* Na^+) for 1-L batches is given in Table 3. The calculated masses of the compounds that are needed to prepare 223 L of extra salt feed for CSTR Test 2 are given in Table 4.

Table 3. Average simulated supernatant (6.4 *M* Na^+) for 1-L batches

Comp.	Molarity (mol/L)	Compound	Molecular Weight (g/mol)	Mass (g)
K^+	0.0171	KNO_3	101.1	1.719
$OH^{-\bullet}$	3.611	NaOH	40.00	144.46[a]
$NO_3^{-\bullet}$	1.388[b]	$NaNO_3$	84.99	117.97
$NO_2^{-\bullet}$	0.594	$NaNO_2$	69.00	40.98
$AlO_2^{-\bullet}$	0.354	$AL(NO_3)_3 \cdot 9H_2O$	375.14	132.80
$CO_3^{2-\bullet}$	0.183	$Na_2CO_3 \cdot H_2O$	124.01	22.69
$SO_4^{2-\bullet}$	0.171	Na_2SO_4	142.04	24.29
$Cl^{-\bullet}$	0.0286	NaCl	58.44	1.671
$F^{-\bullet}$	0.0366	NaF	41.99	1.537
$HPO_4^{2-\bullet}$	0.0114	$Na_2HPO_4 \cdot 7H_2$	268.09	3.056
$C_2O_4^{2-\bullet}$	0.0091	$Na_2C_2O_4$	134.00	1.219
$SiO_3^{2-\bullet}$	0.0046	$Na_2SiO_3 \cdot 9H_2O$	284.2	1.307
$MoO_4^{2-\bullet}$	0.00023	$Na_2MoO_4 \cdot 2H_2O$	241.95	0.056
H_2O				796.25
UO_2^{2+}	*0.0000043*	*$UO_2(NO_3)_2$*	*(Appendix C-2)*	
Sr^{2+}	*0.0000123*	*$Sr(NO_3)_2$*	*(Appendix C-3)*	
Cs^+	*0.00016*	*CsCl*	*(Appendix C-4)*	

[a]The actual mass of NaOH that was required for a 1-L batch of 5.6 *M* Na^+ average simulated salt solution for a 1-L batch was 126.40 g which actually yielded a 3.16 molar solution of NaOH rather than the stated, targeted molar concentration of1.91. Based on the 3.16 molar solution, a 1-L batch of 6.4 *M* Na^+ average simulated salt solution required 3.611 mol of NaOH [(6.4/5.6)(3.16)].

[b]The target molarity for $NO_3^{\bullet}$ is 2.45 mol/L.

$NO_3^{\bullet}$ was added as $NaNO_3$ per 1-L batch

2.45mol – 3(0.354 mol) = 1.388 mol

The density given in the tables on pages 6 and 7 of HLW-SDT-99-0041 for Salt Solution Feed **is 1.29 g/mL**. The calculated water mass needed for 1L = 1290 g total - 493.755g salts = **796.245 g**

Table 4. Extra simulated supernatant (6.4 *M* Na^+) needed for Test 2

Comp.	Molarity (mol/L)	Compound	Molecular Weight (g/mol)	Mass (g/L)	Mass (g/223L)
K^+	0.0171	KNO3	101.1	1.729	383.3
$OH^{-\bullet}$	3.611	NaOH	40.00	144.46[a]	32,214.6
$NO_3^{-\bullet}$	1.388[b]	$NaNO_3$	84.99	117.97	26,307.3
$NO_2^{-\bullet}$	0.594	$NaNO_2$	69.00	40.98	9,138.5
$AlO_2^{-\bullet}$	0.354	$AL(NO_3)_3 \cdot 9H_2O$	375.14	132.80	29,614.4
$CO_3^{2-\bullet}$	0.183	$Na_2CO_3 \cdot H_2O$	124.01	22.69	5,059.9
$SO_4^{2-\bullet}$	0.171	Na_2SO_4	142.04	24.29	5,416.7
$Cl^{-\bullet}$	0.0286	NaCl	58.44	1.671	372.6
F^-	0.0366	NaF	41.99	1.537	342.8
$HPO_4^{2-\bullet}$	0.0114	$Na_2HPO_4 \cdot 7H_2O$	268.09	3.056	681.5
$C_2O_4^{2-\bullet}$	0.0091	$NA_2C_2O_4$	134.00	1.219	271.8
$SiO_3^{2-\bullet}$	0.0046	$Na_2SiO_3 \cdot 9H_2O$	284.2	1.307	291.5
$MoO_4^{2-\bullet}$	0.00023	$Na_2MoO_4 \cdot 2H_2O$	241.95	0.056	12.5
H_2O				796.25	177,564

(The measured density before the sludge and catalyst slurries were added was 1.2851 kg/L. The mass of the chemicals divided by the density showed that 224 L of salt feed were actually prepared.)

CSTR TEST 2 FLOW RATES TO CSTR-1
(from HLW-SDT-99-01nn; 6/16/99)

Salt Feed:	*21.5 mL/min (1290 mL/h)*
Water + MST	*2.14 mL/min (128.4 mL/h)*
Recycle water	*6.86 mL/min (411.6 mL/h)*
NaTPB	*0.68 mL/min (40.8 mL/h)*
Extra NaTPB	*0.40 mL/min (24.0 mL/h)*
Total	*31.58 mL/min (1894.8 mL/h)*

The feed preparations for Test 2 were for a 264 h test. However, since the residence time was shortened in the CSTRs from 10.5 to 8.2 h, the test time was also shortened to 230 h which was enough test time to allow all the objectives of the test to be achieved.

TOTAL SALT FEED NEEDED FOR TEST 2

Salt feed for CSTRs and Slurry Concentration Tank at start of test

[2(15.4 L) + 3 L][(21.5)/(31.58] = **23.01 L (6.08 gal)**

Salt feed during test
(21.5)(60 min/h)(264 h)(10^{-3}L/mL) = **340.6 L (90 gal)**

Extra salt feed that will remain in the mixing tanks at end of test

24 L (6.34 gal)

Total salt feed needed = 23L + 340.6 L + 24L = *387.6 L (102.4 gal)*

TOTAL MST + WATER NEEDED (SEE Appendix C-1)

(2.14 mL/min)(60 min/h)(264 h)(10^{-3}L/mL) = **33.9 L (9.0 gal)**

SLURRY WASH RECYCLE WATER NEEDED (See Appendix C-5)

(6.86 mL/min)(60 min/h)(264 h)(10^{-3}L/mL) = **108.7 L (28.7gal)**

NaTPB NEEDED

(0.68 mL/min)(60 min/h)(264 h)(10^{-3}L/mL) = **10.8 L (2.85 gal)**

EXTRA NATPB NEEDED (supply line connected to the MST + Water line to CSTR-1)

(0.4 mL/min)(60 min/h)(264 h)(10^{-3}L/mL) = **6.34 L (1.67 gal)**

ACTUAL SALT FEED PREPARATION FOR TEST 2

- 123.5 L of salt feed (MST treated) which were prepared for Test 1b (not conducted) were used. The density was 1.288 kg/L

- 49.5 L of salt feed (of MST treated) in the Hot Cell "A" feed tanks which were not used in Test 1a were used. This solution contained ^{137}Cs and ^{85}Sr and contained ^{235}U. The density was 1.288 kg/L.

- 224 L of MST treated salt feed (see Table 4) were prepared for use in Test 2. However; *only 200.7 L were added to the feed tanks*. The density was 1.2851 kg/L.

- 1.75 L (1.909 kg) of the 50-50 mixture of HM - Purex sludge were added to the feed tanks. The density was 1.0909 kg/L.

- A total of 8.2 L of catalyst which were provided by M. J. Barnes of the SRS was added. The estimated density of the catalyst was about 1 kg/L.

- ***The total volume of salt feed in the two 55 gal tanks in Hot Cell "A" at the start of Test 2, which included the sludge and catalysts, was 384 L (101.5 gal). The calculated density of the feed was 1.281 kg/L.***

Method used in Preparation of Salt Solutions for Test

Aluminum nitrate yields an acidic solution in water and can react with sodium nitrite to form NO_2 gas. To avoid the decomposition of the sodium nitrite, the order of addition was to dissolve the sodium hydroxide first in about half the water needed for the feed preparation followed by the aluminum nitrate. Sodium nitrite was then added only after the aluminum nitrate was completely dissolved in the sodium hydroxide.

1. The amounts of chemical used to prepare 224 L (59.2 gal) of salt solution are given in Table 4.
2. A 100 gal plastic mixing drum was cleaned and rinsed well with deionized water and allowed to dry. The drum had an agitator attached to the drum lip. At the start of a preparation, the top of the drum was covered with a sheet of plastic. It was placed over the top of the drum in a concave position to allow condensate from the heat of chemical dissolution vapor to drip back into the drum. The plastic was also arranged (tapped) around the agitator in a way that did not interfere with mixing (kept away from the mixing shaft).
3. The chemicals were weighed with calibrated balances, and weighed in separate tarred containers before adding them to the mixing drum. First, about half of the deionized water was added to the drum, and then the NaOH was slowly added with gentle mixing. The dissolution of NaOH in water is exothermic and generates heat. The container drum was kept sealed with the plastic as much as possible while the solution was hot to avoid loss of water by evaporation. The solution was allowed to cool to room temperature before adding the other chemicals.
4. The aluminum nitrate nonohydrate was added next and mixed well until it dissolved.
5. The remaining chemicals and water were then added with continuous mixing. The $Na_2SiO_3 \bullet 9H_2O$ was weighed in a plastic beaker and dissolved in water before adding it to the drum. *If solid $Na_2SiO_3 \bullet 9H_2O$ were added to the salt feed directly, it would be extremely slow to get into solution.* The salt solution was then mixed for about 2 h.
6. MST solids were added to the salt solution to remove tramp strontium. Needed $\geq$ 0.5 g MST per liter of salt solution.
   (224 L)(0.5 g MST/L) = 112 g MST
   Total suspended solids in MST slurry = 160.6 g/L (Appendix C-1)
   (112g)/(160.6 g/L) **= 0.697 L MST slurry**
   - While mixing and keeping the MST solids of the slurry in suspension, pull 800 mL of MST slurry into a 1000 mL plastic bottle.
   - Allowed the MST particles to settle over a two day period and separated the supernatant from the solids by decantation.. Weighed the solids and then flushed the solids from the bottle into the salt feed drum with salt feed.
   - Mixed the MST in the salt solution for two days to sorb the tramp strontium.
7. Filtered the solution through large 0.45 µm cartridge filters to remove the MST.
8. Took a 20 mL sample of the salt solution and have it analyzed for strontium and salt for the constituents.
9. The density of the salt solution was determined to be 1.285 g/mL with a 1000 mL volumetric flask.
10. Transferred 200.7 L of the MST treated and filtered salt solutions to the feed tanks in Hot Cell "A"

U Preparation and Addition to Salt Feed Tanks in Hot Cell "A"

The concentration of uranium in the final salt feed solution needed to be about 1 mg U/L for Test 2.

(1 mg U/L)(384 L) = **384 mg U needed**

49.5 L from Test 1a already contained the needed uranium (49.5 mg U)

[(384 – 49.5) mg U]/(35 mg U/mL) = 9.557 mL stock

(see Appendix C-2 for the uranium stock solution preparation)

- **Added 9.666 mL stock solution (338.3 mg U)** to a tarred 60 mL plastic bottle which contained 50.85 g of 0.1 *M* HNO_3 and mixed well on a Vortex mixer.

- This solution was poured into the salt feed tanks via a 1/4"stainless steel with a funnel attachment in Hot Cell "A". Afterwards, the funnel and line were rinsed with 25 mL of 0.1 *M* HNO_3.

Concentrations of U in 6.4 and 4.7 *M* Na^+ feed solutions:

[(338.3 +49.5)mg U]/384 L **= 1.01 mg U/L and 0.943 mg ^{235}U/L** in 6.25 *M* Na^+ feed solution

[(338.3 +49.5)mg U]/564 L = **0.688 mg U/L and 0.642 mg ^{235}U/L** in 4.5 *M* Na^+ feed solution

Sr Preparation and Addition to Salt Feed Tanks in Hot Cell "A"

•Added 7.384 mL of the cold Sr stock solution (see Appendix C-3) to a 60 mL tared plastic bottle which contained 35 mL of 0.1 *M* HNO_3 and mixed well.

(7.384 mL)(2.0536 mg Sr/mL) = 15.16 mg Sr

49.5 L of the salt feed in the drums in Hot Cell "A" which were not used in Test 1 and were used. The concentration of strontium for this feed was 0.049 mg/L.

(49.5 L)(0.049 mg Sr/L) = 2.43 mg Sr

Total Sr in salt feed (excluding the strontium in the sludge) =
15.16 + 2.43 = **17.59 mg Sr**

(17.59 mg Sr)/(384 L) = **0.046 mg Sr/L in 6.25 *M* Na^+ feed solution**

(17.59 mg Sr)/(564 L) = **0.031 mg Sr/L in 4.5 *M* Na^+ feed solution**

- **Received 30 mCi ^{85}Sr on 7/23/99; added to cold strontium solution, and then added to Salt Feed Solution on 7/27/99; ($t_{1/2}$ = 64.8 days); as SrCl in 7.3236 mL of 0.5 *M* HCl; unshielded radiation = 200 mR/h/mCi; <u>specific data calibrated for 7/29/99 (on this date the mCi ^{85}Sr/mL was 4.096); used the calibration date for ^{85}Sr decay considerations.</u>**

- In Hot Cell "A" 30 mCi ^{85}Sr were added to the cold strontium solution and mixed well on a vortex mixer. Afterwards, the solution was poured into the salt feed tanks via a 1/4"stainless steel with a funnel attachment. The funnel and line were rinsed with 50 mL of 0.1 *M* HNO_3.

  The 49.5 L of salt feed also contained ^{85}Sr.

  (49.5 L)(72.2 μCi ^{85}Sr/L) = 3,574 μCi ^{85}Sr on 6/24/99

  Corrected for decay for use in Test 2 to 7/29/99
  $^{85}Sr_{(7/29/99)}$ = 3,574 μCi $e^{(-\ln 2/64.8\ d)(35\ d)}$ = **2,458 μCi**

  Total μCi ^{85}Sr available on 7/29/99 for Test 2 = 32,458

  (32,458 μCi ^{85}Sr)/(384 L) = **84.53 μCi ^{85}Sr/L (or 0.0845 μCi /mL) in 6.25 *M* Na^+ feed solution**

  (32,458 μCi ^{85}Sr)/(564 L) = **57.55 μCi ^{85}Sr/L (or 0.0576 μCi /mL) in 4.5 *M* Na^+ feed solution**

  The ***effective specific activity of strontium*** which was added to the feed solution prior to the sludge being added.

  (32,458 μCi ^{85}Sr)/17.59 mg Sr = **1,845 μCi ^{85}Sr/mg Sr**

- Cold strontium that was present as $SrCO_3$ in the sludge, which was added to the feed solution, complicated our understanding of the strontium removal behavior with MST. It was not clear how fast the cold strontium would equilibrate with the traced strontium.

  1.75 L (1.909 g) of a 50-50 mixture of HM and PUREX sludge were added to the salt feed. Both HM and Purex fractions had 15.4 wt.% total solids (See Tables 5 and 6)

  (0.154) (1.909 kg)(1000 g/kg) = 294 g total solids
  Wt.% $SrCO_3$ on a dry weight basis was 0.42% for HM and 0.034% for PUREX sludge.

  [(0.0042 + 0.00034)/2](294 g)(1000 mg/g) = 667 mg $SrCO_3$
  Strontium in sludge that was added to salt feed
  (667 mg $SrCO_3$)[(87.63 g Sr/mol)/(147.64 g$SrCO_3$/mol) = 396 mg Sr

  396 mg Sr/384 L = 1.03 mg Sr/L in 6.25 *M* Na^+ salt feed solution
  396 mg Sr/564 L = 0.70 mg Sr/L in 4.5 *M* Na^+ salt feed solution in CSTRs and Slurry Concentration Tank

Concentration of Total Strontium in feed Solutions

[(396 + 17.6)mg Sr]/384 L = **1.08 mg Sr/L in 6.25 *M* Na^+ salt feed solution**
[(396 + 17.6)mg Sr]/564 L = **0.73 mg Sr/L in 4.5 *M* Na^+ salt feed solution**

- Hydrous metal oxide of iron, zirconium, neodymium, and manganese in the sludge are ion exchangers that can sorb strontium which makes the interpretation of strontium removal behavior more difficult. These sorbents can compete with MST for the strontium. The same is true for the hydrous metal oxides of iron, tin, lanthanum, cerium in the Enhanced Comprehensive Catalyst (ECC) system and for some of these elements in combination with silicon, molybdenum, and phosphate.

Cs Preparation and Addition to Salt Feed Tanks in Hot Cell "A"

Weighed 62.31 g of the ^{137}Cs stock solution (0.042832 Ci ^{137}Cs/g) in a tared plastic 60 mL plastic bottle. To this bottle was added a cold cesium solution (9.0532 g of CsCl dissolved in 37.53 g deionized water) which was then mixed well on a vortex mixer (see Appendix C-4). The solution was poured into the salt feed tanks via a 1/4"stainless steel with a funnel attachment. The funnel and line were rinsed with 50 mL of water.

(62.31 g)(0.042832 Ci ^{137}Cs/g) = **2.669 Ci ^{137}Cs**

Ci ^{137}Cs in the 49.5 L of salt feed not used in Test 1a

(49.5L)(7.5 mCi/L)(10^{-3} L/mL) = **0.371 Ci ^{137}Cs**

Total Ci ^{137}Cs in salt feed was 3.040

(3,040 mCi ^{137}Cs)/(384 L) = **7.917 mCi ^{137}Cs/L**

Total Cesium:

Fission Product Cs

(3.04 Ci ^{137}Cs)/(86.4 Ci/g ^{137}Cs) = 0.0352 g ^{137}Cs

The Cs-137 is estimated to be about 23% of the total fission product cesium.

(0.23)X = 0.0352; X = **0.153 g Cs**

Cold Cs

(9.0532 g of CsCl)[(133 g Cs/mol)/(168.47 g CsCl/mol)] = **7.147 g Cs**

The 49.5 L of salt feed from Test 1a also contained cold Cs.

(49.5 L)(0.0176 g cold Cs/L) = **0.87 g Cs**

Sludge Cs (See Table 5 and 6)

Wt.% $CsNO_3$ on a dry weight basis was 0.029% for HM and 0.0029% for PUREX sludge.

[(0.00029 + 0.000029)/2](294 g) = 0.047 g $CsNO_3$

Cesium in sludge that was added to salt feed

(0.047g $CsNO_3$)[(133 g cs/mol)/(195 g$SrCO_3$/mol) = **0.032 g Cs**

Total mass of cesium in salt feed

7.147 + 0.87 + 0.153 + 0.032 **= 8.202 g Cs**

Concentration of cesium in feed Solutions

8,202 mg Cs/384 L = **21.36 mg Cs/L in 6.25 *M* Na^+ salt feed solution**
8,202 mg Cs/564 L = **14.54 mg Cs/L in 4.5 *M* Na^+ salt feed solution**

Molarity of cesium in 6.4 *M* Na^+ salt feed solution

[(8.202 g Cs)/(133 g/mol)]/(384 L) **= 0.000161 mol/L Cs**

- After the Sr, U, and were equilibrated for 24 h, a 30 mL sample was taken for analysis. Half of the sample was filtered through a 0.45 μ filtered. Filtered and unfiltered samples were sent to analytical chemistry for ICP-MS analysis. A portion of the filtered sample was analyzed by Delayed Neuron Counting. The objective was to make sure that all the uranium which was added was in solution. Also, this was a comparative check of ICP-MS and DNC analyses. One-mL samples of each were taken for "in-house" gamma counting.

Preparation and Addition Sludge to Salt Feed Tanks in Hot Cell "A"

1.75 L (1,909 g) of a 50-50 mixture of HM and PUREX sludge (see Table 6 and 7) were added to the salt feed. Both HM and Purex sludge fractions had 15.4 wt.% total solids. The density of the sludge mixture was 1.091 g/mL. The pH was ~10.5. The sludge was poured into the salt feed tanks in Hot Cell "A". via a 3/4"stainless steel attachment with a funnel which was located in from of the cell in room 103. Afterwards, the funnel and line were rinsed with a weighed amount of the salt feed.

Preparation and Addition Catalyst to Salt Feed Tanks in Hot Cell "A"

Table 8 is a modification of Table 1 of SRT-LWP-99-00099 which was obtained from M. J. Barnes of WSRC. It gives a description of the components of a modified ECC (Enhanced Comprehensive Catalyst) system which was prepared at ORNL by M. J. Barnes and K. A. L. Prettel of WSRC. According to M. J. Barnes, the modified ECC system should have provided the best opportunity for catalytic decomposition of sodium tetraphenylborate (NaTPB) in Test 2. In previous work at the SRS it was determined that the primary active components of the catalyst system in SRS waste were palladium, copper, tetraphenylborate decomposition product intermediates, and mercury. In addition to those catalysts, the modified ECC system has a number of other potential catalysts such as Ni, and Ag. A total of 8.2 L was added to the salt feed tanks in Hot Cell "A". via a 3/4"stainless steel attachment with a funnel which was located in from of the cell in room Afterwards, the funnel and line were rinsed with a weighed amount of the salt feed.

Summary of Salt Feed Solution Preparation for CSTR Test 2

Table 5 gives the composition of some of the primary salt constituents in the salt feed after the sludge and catalytic solution had been added. Those solution decreased the concentration of the Na^+ and K^+ from 6.4 and 0.0171 molar to 6.25 and 0.0167 molar. The compositions of the catalytic constituents in the salt feed are given in Table 8.

The test duration was 230 h, which provided for about 28 CSTR-volume turnovers. Both CSTRs were filled prior to testing. About 3L salt feed solution were added to the Slurry Concentration Tank. To fill the tanks as stated above, salt feed was pumped into CSTR-1 at a rate of 21.5 mL/min and water pumped at a rate 9.68mL/min to provide a total rate of 31.18 mL/min. The extra water flow needed to represent the Extra NaTPB was not used because the Extra NaTPB system which was pumped into the MST water line was added at the last moment before the test actually started.

Table 6 Composition of 50 – 50 mixture of HM and PUREX SLUDGE*

Solids (wt%)	HM Sludge	PUREX Sludge	50-50 Mixture Sludge Used in Test 2
Insoluble Solids	12.9	12.5	12.7
Soluble Solids	2.87	3.28	3.08
Total Solids	15.4	15.4	15.4
Component (wt% dry basis)			
$Al(OH)_3$	25.8	10.9	18.35
$BaSO_4$	0.23	0.45	0.34
$Ca_3(PO_4)_2$	0.10	0.23	0.17
$CaCO_3$	1.52	4.57	3.05
$CaSO_4$	0	0.42	0.21
Cr_2O_3	0.23	0.36	0.30
$CsNO_3$	0.029	0.0029	0.016
CuO	0.056	0.16	0.108
$Fe(OH)_3$	26.7	45.5	36.10
HgO	0	0	0
KNO_3	0.019	0.017	0.018
KOH	0.18	0.35	0.265
MgO	0.35	0.22	0.285
MnO_2	6.86	6.50	6.68
Mn_3O_4	0.77	0.73	0.75
Na_2CO_3	0.11	0.13	0.12
Na_2SO_4	0.49	0.15	0.32
Na_3PO_4	0.042	0.011	0.027
NaCl	0.49	1.37	0.93
NaF	0.27	0.24	0.255
NaI	0	0.024	0.012
$NaNO_2$	11.9	11.9	11.9
$NaNO_3$	1.28	1.28	1.28
NaOH	1.41	3.11	2.26
Nd_2O_3	2.41	0.22	1.315
$Ni(OH)_2$	1.35	4.00	2.675
$PbSO_4$	0.23	0.45	0.34
SiO_2	5.20	1.48	3.34
$SrCO_3$	0.42	0.034	0.227
Zeolite	9.63	1.47	5.55
ZnO	0.044	0.30	0.172
ZrO_2	1.90	3.40	2.65

*Table 5 is a modification of Table 1 in SRT-WHM-99-004 which was obtained from M. R. Poirier of SRS.

Table 7. Composition of 50 – 50 mixture of HM and PUREX SLUDGE*

Solids (wt%)	50-50 Mixture of HM and PUREX Sludge Used in Test 2	Mass of Comp Added to Salt Feed (g)	Mass of Cations Added to Salt Feed (g)
Insoluble Solids	12.7	242.5	
Soluble Solids	3.08	58.8	
Total Solids	**15.4**	**294**	
Component **(wt% dry basis)**			
$Al(OH)_3$	18.35	59.95	20.73
BaSO_4	0.34	1.00	0.588
$Ca_3(PO_4)_2$	0.17	0.50	0.194
$CaCO_3$	3.05	8.97	3.592
$CaSO_4$	0.21	0.617	0.182
Cr$_2O_3$	0.30	0.882	0.604
$CsNO_3$	0.016	0.047	0.032
CuO	0.108	0.318	0.254
$Fe(OH)_3$	36.10	105.84	0.553
HgO	0	0	0
KNO_3	0.018	0.053	0.020
KOH	0.265	0.779	0.543
MgO	0.285	0.838	0.505
MnO_2	6.68	19.64	12.41
Mn_3O_4	0.75	2.205	1.588
Na_2CO_3	0.12	0.353	0.153
Na_2SO_4	0.32	0.941	0.305
Na_3PO_4	0.027	0.079	0.033
NaCl	0.93	2.734	1.076
NaF	0.255	0.750	0.411
NaI	0.012	0.035	0.005
$NaNO_2$	11.9	34.99	11.66
$NaNO_3$	1.28	3.763	1.018
NaOH	2.26	6.644	3.820
Nd_2O_3	1.315	3.866	3.315
Ni$(OH)_2$	2.675	7.865	4.980
PbSO_4	0.34	1.000	0.683
SiO_2	3.34	9.820	4.588
$SrCO_3$	0.227	0.667	0.396
Zeolite	5.55	16.32	
ZnO	0.172	0.506	0.407
ZrO_2	2.65	7.791	5.768

* Table 5 is a modification of Table 1 in SRT-WHM-99-004 which was obtained from M. R. Poirier of SRS.

Table 8 Composition of Modified Enhanced Comprehensive Catalyst System

Stock Solution Batch #: MJB-ORNL-xxxx-1	Compound	Solvent	Stock Species Target Concentration (mg/L)	Volume of Stock Solution Required (mL)[g]	Species Concentration in 6.4 M Na^+ Feed (mg/L)[h]	Species Concentration in 4.7 M Na^+ Slurry (mg/L)[h]
Pd	$Pd(NO_3)_2$	0.1 M HNO_3	13010	494	16.7	11.6
Cu	$Cu(SO_4) \cdot 5H_2O$	water	3710	494	4.8	3.3
2PB/1PB	$(C_6H_5)_2BOH$[d] $(C_6H_5)B(OH)_2$	1.0 M NaOH	62500 62500	990	161 161	110 110
3PB[a]	$(C_6H_5)_3B$	0.1 M NaOH	-	1205 g	161	110
Hg	$Hg(NO_3)_2 \cdot H_2O$	0.1 M HNO_3	2200	494	2.9	2.0
Diphenyl mercury[b]	$(C_6H_5)_2Hg$	pure	-	74.5 g	194	132
Benzene[c]	C_6H_6	pure	-	358 g	932	635
Mo/Cr/Si/Se/As	$Na_2MoO_4 \cdot 2H_2O$ Na_2CrO_4 $Na_2SiO_3 \cdot 9H_2O$ Na_2SeO_4 As_2O_3	0.1 M NaOH	12010 60070 16020 1000 41.2	494	15.2 77.9 20.9 1.3 0.048	10.3 53 14.2 0.89 0.033
Zn/Pb/Fe	$Zn(NO_3)_2 \cdot 4H_2O$ $Pb(NO_3)_2$ $Fe(NO_3)_3 \cdot 9H_2O$	0.1 M HNO_3	8820 1200 2600	494	11.4 1.5 3.3	7.8 1.02 2.2
Sn	$SnCl_2$	0.1 M HNO_3	2100	494	2.7	1.8
Ca/La/Co	$Ca(NO_3)_2 \cdot 4H_2O$ $La(NO_3)_3 \cdot 6H_2O$ $Co(NO_3)_2 \cdot 6H_2O$	0.1 M HNO_3	12210 50.1 40.0	494	15.8 0.066 0.048	10.5 0.045 0.037
Cd/Ce	$Cd(NO_3)_2 \cdot 4H_2O$ $Ce(NO_3)_3 \cdot 6H_2O$	0.1 M HNO_3	401 301	494	0.48 0.38	0.33 0.25
Rh	$Rh(NO_3)_3$	0.1 M HNO_3	1400	494	1.8	1.2
Ag	$AgNO_3$	0.1 M HNO_3	6810	494	8.8	6.0
Ru	$RuCl_3 \cdot xH_2O$	0.1 M HNO_3	5410	494	7.0	4.8
Inactive Organics	isopropanol methanol	0.1 M NaOH	50100 5010	494	64.6 6.7	44 4.6
Phenol[e]	C_6H_5OH	pure	-	62.0 g	162	110
Biphenyl[f]	$(C_6H_5)_2$	pure	-	74.5 g	194	132

[a]3PB was purchased as a ~6 wt. % solution (in 0.1 M NaOH) of triphenylborane sodium hydroxide adduct. It was added as purchased (i.e., stock solution not prepared). The quantity required (1205 g) was the total weight of the as received solution.
[b]Diphenylmercury was added as a pure compound (i.e., stock solution not prepared).
[c]Benzene was added as a pure compound (i.e., stock solution not prepared).
[d]2PB was purchased as diphenylborinic acid ethanolamine complex. No pretreatment is performed on it prior to preparation of the stock solution.
[e]Phenol is added as a pure compound (i.e., stock solution not prepared).
[f]Biphenyl is added as a pure compound (i.e., stock solution not prepared).
[g]Volume of each stock solution required assumed the volume of 6.4 M Na^+ feed solution was 365 L (96 gal).
[h]The actual volume of feed solution was 384 L (101.4 gal)

Table 9. Average simulated supernatant with sludge and catalyst added for CSTR Test 2

Comp.	Molarity (mol/L)	Compound
Na^+	6.25	(Composite of all Na salts)
K^+	0.0167	KNO3
$OH^{-\bullet}$	3.526	NaOH
$NO_3^{-\bullet}$	1.319	$NaNO_3$
$NO_2^{-\bullet}$	0.566	$NaNO_2$
$AlO_2^{-\bullet}$	0.337	$AL(NO_3)_3 \cdot 9H_2O$
$CO_3^{2-\bullet}$	0.174	$Na_2CO_3 \cdot H_2O$
$SO_4^{2-\bullet}$	0.163	Na_2SO_4
$Cl^{-\bullet}$	0.0273	NaCl
$F^{-\bullet}$	0.0348	NaF
$HPO_4^{2-\bullet}$	0.0108	$Na_2HPO_4 \cdot 7H_2O$
$C_2O_4^{2-\bullet}$	0.0086	$Na_2C_2O_4$
$SiO_3^{2-\bullet}$	0.0048	$Na_2SiO_3 \cdot 9H_2O$
$MoO_4^{2-\bullet}$	0.00038	$Na_2MoO_4 \cdot 2H_2O$
Cs^+	*0.00016*	*CsCl*
UO_2^{2+}	*0.0000043*	*$UO_2(NO_3)_2$*
Sr^{2+}	*0.0000123*	*$Sr(NO_3)_2$*

<u>NaTPB Preparation for CSTR Test 2</u>

Data from the tables on pages 6 and 7 of HLW-SDT-99-0041.

(0.52 gal/min)(3.7854 L/gal)(60 min/h) = 118.105 L/h

(49.4 lb/h)(0.45359 kg/lb)(1000 g/kg) = 22,407 g NaTPB/h

[(22,407 g NaTPB)/(342.23 g/mol)]/118.105 L = **0.5545 mol NaTPB /L**

Total Na^+ is 0.65 mol/L

Extra Na^+ as NaOH = 0.65 – 0.5545 = **0.0955 mol/L**

<u>Total NaTPB Feed Needed for Test 2</u>

(0.68 mL/min)(60 min/h)(264 h)(10^{-3}L/mL) = **<u>10.77 L (2.845 gal)</u>**

(0.40 mL/min)(60 min/h)(264 h)(10^{-3}L/mL) = **<u>6.34 L (1.674 gal)</u>**

<u>Total needed = 17.11 L (4.52 gal)</u>

<u>NaTPB needed for CSTR Test 2:</u>

(0.5545 mol/L)(17.11 L)(342.23 g/mol) = **3,246.9 g NaTPB**

(0.096 mol Na^+)(17.11 L)(40.00 g NaOH/mol) = **65.7 g NaOH**

Salt wt. = 3,312.6 g

X_{water} = 15,286.6 g water

Actual Preparation of NaTPB for Test 2

Only had 2940 g NaTPB for the test

Added the NaTPB to 59.5 g NaOH and 13,834 g deionized water.

(2940 g NaTPB)/(342.23 g/mol) = 8.591 mol NaTPB

(59.50 g NaOH)/(40 g/mol) = 1.49 mol NaOH

After mixing well, the density was determined to be 1.0336 g/.mL using a 1000 mL volumetric flask.

(2940 g + 59.5 g + 13,834 g)/(1.0336 g/mL) = **16,286 mL NaTPB Feed Solution**

NaTPB molarity = (8.591 mol)/(16.286 L) = **0.528 mol/L**

NaOH molarity = (1.49 mol)/(16.286 L) = **0.0915 mol/L**

Na^+ molarity = [(8.591 + 1.49) mol)]/(16.286 L) = **0.62 mol/L**

Test 2 was run only 230 h and all but 915.3 g (or 886 mL) were used.

Total volume of NaTPB used during test was 16,286 – 886 = **15,400 mL**

Volume used per min during Test 2: (15,400 mL)/[(230 h)(60 min/h)] **1.116 mL/min**

The Extra NaTPB pumping rate was fairly constant during the test at **0.4 mL/min**
The average rate the NaTPB feed was pumped into CSTR 1 was **0.716 mL/min**

<u>MST + Water Needed for CSTR Test 2 (See Appendix C-1)</u>

(340.6 L)(0.548 MST g/L of 6.4 *M* Na^+ feed) = **<u>186.65 g MST for test</u>**

(186.65 g)/[(264 h)(60 min/h] = **0.011783 g MST/min**

<u>The concentration of MST in slurry is 0.1606 g/mL.</u>

(186.65 g MST)/(0.1606 g MST/mL) = **<u>1,162 mL slurry</u>**

(2.14 mL/min)(60 min/h)(264 h) = **33,898 mL (MST + water)**
Need 1,162 mL slurry + 32,736 mL H_2O

Check:
(186.65 g MST)/(33,898 mL) = **or 0.0055 g/mL**
(0.0055 g/mL)(2.14 mL/min) = **0.01177 g/min**

Actual Preparation of MST + Water for Test 2

Prepared 40 L of MST +Water solution (contained 220 g MST). Used all but 10.3 L of the MST + Water solution during the test which lasted 230 h.

Rate used during Test 2

[(40 L- 10.3L)(1000 mL/L)]/[(230 h)(60 min/h)] = **2.15 mL/min**

APPENDIX C-1

Characterization of MST Slurry Sample (Lot # 96QAB281) by *Catherine Mattus*

Density of MST slurry = 1.113 g/mL

Total solids:

For 25 mL sample of slurry

(25 mL)(1.113 g/mL) = 27.825 g

mass of solids after drying = 4.1483 g

(4.1483 g)/(27.825 g) × 100 = 14.91 wt.%

*(1000mL/L)(1.113 g/mL)(0.1491) = **165.95 g total solids/L slurry***

Total suspended solids

(Solids were removed by filtration with a 1.6μm filter and dried at 105°C)

For 23 mL sample of slurry:

(23 mL)(1.113 g/mL) = 25.559g

MST solids removed by filtration and dried at 105°C = 3.6928 g

(3.6928 g)/(25.599 g) x.100 = 14.43 wt.%

*(1000mL/L)(1.113 g/mL)(0.1443) = **160.61 g suspended solids/L slurry***

Dissolved solids

(Solids were removed by filtration with a 1.6µm filter and the filtrate was dried at 105°C)
For 23 mL sample of slurry

(23 mL)(1.113 g/mL) = 25.559g

(0.1844 g)/(25.559 g) × 100 = 0.72.wt.%

(1000mL/L)(1.113 g/mL)(0.0072) = ***8.014 g dissolved solids/L slurry***

Ratio of volume of supernatant mixture to mass of MST in CSTR-1

Using data from table in on page 3 of HLW-SDT-99-01nn:

{[(5.9 lb MST/h)(0.45359 kg/lb)(1000 g/kg)]/(60 min/h)]}/81.386 L/min =
= 0.548 MST g/L of 6.4 *M* Na^+ feed

or (0.548 g MST)/{[(31.18/21.5)](1 L)} = **0.3779 g MST/L of 4.7 *M* Na^+ salt solution**

APPENDIX C-2

URANIUM CONSIDERATIONS FOR CSTR TEST 2

After the salt solution feed has been prepared, mixed for 2 days with MST (≥0.5 g per liter of feed) to remove as much of the tramp strontium as possible, filtered through a 0.45µm cartridge filter, and pumped to the feed mixing and storage tanks in hot cell A, an- amount of uranium was added to obtain a concentration of 1 mg/L. The uranium was added as an acidic (pH 1 – 2) uranyl nitrate solution (uranium precipitates in the pH range of 3.2 to 5). After the uranium was added , it was mixed for two days to equilibrate. Samples were taken which were analyzed by Delayed-Neutron Counting and ICP-MS to check the accuracy of these analyses. Both filtered and unfiltered were analyzed.

Based upon blanks currently being run for ICP-MS, a detection limit of 1 to 0.1 ppb is now possible for normal or deleted uranium and 5 ppt for U-235. However, because of the high salt content of the CSTR test samples, which can cause plugging of the sample delivery system for the ICP-MS, sample dilutions of 100 to 1000 were needed.

ICP-MS example calculations:

If the concentration of uranium in the simulant supernatant in the CSTR is 1 mg/L, and the MST gives of DF of 10 (based on the initial supernatant, the concentration in the filtrate would be 0.1 mg/L or 0.1 µg/mL. According to Dave Hobbs of SRS, the DF should be between 2 and 5.

If 1 mL samples of filtrate were diluted by factors of 100 , 500, and 1000 with water and mixed well, analyses of these samples by the ORNL ICP-MS should give the following results.

For a dilution of 100×:
0.1 μgU-235/100 mL = 0.001 μg/mL or ***1 ppb***

For a dilution of 500×:
0.1 μgU/500 mL = 0.0002 μg/mL or ***0.2 ppb***

For a dilution of 1000×:

0.1 μgU/1000 mL = 0.0001 μg/mL or ***0.1 ppb***

If the MST only gave a DF of 5, which is more likely the case, the values calculated above would increase by a factor of 2

For a dilution of 100×, *a value* ***of 2 ppb*** *would be expected.*
For 500×, *it would be* ***0.4 ppb***
For 1000×, *it would be* ***0.2 ppb***

If the detection limit for the ICP-MP, does prove to be 1 to 5 ppt U-235, the values obtained above would be adequate for determining the DF values for uranium removal in the CSTR tests.

Delayed-neutron counting analyses are conducted by the neutron activation analysis group at the High Flux Isotope Reactor (HFIR) at ORNL. HFIR, which has a pneumatic tube irradiation system, has the highest thermal neutron flux in the world. Delayed-neutron counting depends upon the ^{235}U *content in the uranium. According to David Glasgow of the Neuron Activation Analysis Group, a sample in a "rabbit" that contains 10 ng* ^{235}U *would give 1-2% two-sigma-uncertainty. A 12-% two-sigma uncertainty would be obtained if the* ^{235}U *content were 0.7 ng.*

<u>Example calculation:</u>

If the concentration of uranium in the simulant supernatant in the CSTR is 1 mg/L, and the MST gives a DF of 10, the concentration in the filtrate would be 0.1 mg/L. If uranium (93% U-235) is used in the tests, the concentration of the ^{235}U *in the filtrate would be 0.032 mg/L.*

Assume that 1 mL filtrate were dried in a "rabbit" on silica gel, irradiated, and counted*.*

$(0.1\ \mu g\ U)(0.93\ \mu g\ ^{235}U/\mu g\ U)(10^3\ ng/\mu g) = \mathbf{93\ ng\ ^{235}U}$

High boron content samples can be a problem when irradiated because of the thermal properties of the boron. If the boron content is too high, it could melt the plastic rabbit containing the sample. For the CSTR 1a, 1b, and1c tests the concentration of the boron in the filtrate, according to Roy Jacobs, will be about 4 to 5 mg/L.

$(5\ \mu g\ B/mL)(0.1\ mL\ \text{"rabbit" sample})(10^3\ ng/\mu g) = 500\ ng\ boron$

David Glasgow does not think this amount would be a problem; but our plan is to send him a simulant supernatant sample containing no ^{137}Cs, ^{85}Sr, *or U to make sure that the boron is not a problem. The*

higher concentrations noted by Roy Jacobs for Test 2 solution could be a problem, and samples with boron contents at those levels will also be sent to Glasgow for thermal checks.

If only 0.1-mL of the filtrate can be added to a "rabbit" because of the boron, a DF of 10 for the mass of U-235 would be 9.2 ng, which would still provide a very good 1-2% two-sigma-uncertainty----
(0.1 μg U)(0.93 μg ^{235}U/μg U)(10^3 ng/μg) = ***9.3 ng ^{235}U***

<u>Preparation of Uranium Stock Solution</u>

Uranim oxide (U_3O_8) with an isotopic weight percentage of 93.34 was obtained from Steve Wood (574-6940) at Bldg. 3027. The ID # was U-235-VR. It was weighed in a tarred glass flask and weighed 4.12 g. About 95 mL of 6 *M* HNO_3 was added to the flask which was heated overnight with stirring. The yellow solution was a clear and free of solids. Afterwards, it was filtered with a 0.45 μ filter and added to a 100 mL volumetric flask and brought up to volume with deionized water. After mixing with a Vortex mixer, a sample was taken and sent to analytical chemistry for analysis.

The ICP-MS result was 35,000 mg U/L or <u>35 mg/mL</u>.

(35,000 mg U/L)(0.1 L) = 3,500 mg or 3.5 g U in volumentric flask solution

(3.5 g U){[(3)(235.2) +(8)(16)]/[(3)(235)} = **4.14 g U_3O_8**
This mass matches closely the 4.12 g on Steve Wood's inventory.

Molecular Weight Calculation:
(0.9334)(235) = 219.35
(0.0666)(238) = <u>15.85</u>
235.20 g/mol

APPENDIX C-3

STRONTIUM CONSIDERATION FOR TEST 2

If it is assumed that 1000 counts of ^{85}Sr are needed for a 30min count of 10 mL sample of filtrate for the needed counting accuracy, and if it is assumed that the MST will provide a DF of 176, how much ^{85}Sr will be needed for the test? <u>Sr-85 has a half-life of 64.84 day</u> and decay has to be considered in the calculations.

^{85}Sr NEEDED FOR CSTR TEST 2

A total of 384 L of simulant salt solution feed (6.4 M Na^+) were prepared for Test 2.

Total ^{85}Sr in 4.7 M salt solution:

Even though the 24 L of 6.4 *M* salt solution in the feed tank does not get used in the test, it does have to be considered in the total strontium to be added calculation.

(384 L)(6.4 mol/L) = V(4.7 mol/L)
V = 522.9 L

(100 counts/mL)(1/30 min)(522,900 mL)(31.18/21.5)(100 dpm/cpm)(1 mCi/2.22 $\times 10^9$ dpm) = 0.1139 mCi ^{85}Sr

Total ^{85}Sr needed for the simulant supernatant feed will be based upon a DF of 176.

(0.1139 mCi)(176) = 20.05 **mCi ^{85}Sr**

To account for decay, the above value will be increased by 50 % for one half-life.

(20.07 mCi)(1.5) = **<u>30.07 mCi ^{85}Sr were needed CSRT Test 2.</u>**

<u>Mass of ^{85}Sr = (30 mCi)/(2360 mCi/mg) = 0.0042 mg</u>

Total mCi ^{85}Sr available on 7/29/99 for Test 2 = 32.46 mCi ^{85}Sr

(32.46 mCi ^{85}Sr)/(2360 46 mCi ^{85}Sr/mg Sr) = 0.0138 mg Sr)

<u>Concentration of ^{85}Sr in 4.7 *M* Na^+ feed solution</u>:

(0.0138mg)/(564 L) = **0.000024 Sr mg /L (this amount of Sr is of no significant)**

Mass of Sr needed for test = (0.035 mg/L)(200.867 L) = **19.74 mg Sr.**

<u>Cold Sr Solution Stock Solution Preparation</u>

(see pages 22 of the "Operation Notebook")

- 0.4989g $Sr(NO_3)_2$ were added to a cleaned volumetric flask.

- Added 10 mL of 1.000±0.005 *M* HNO_3 (VWR Scientific Products – Lot # 3708277) and brought volume up to 100 mL with deionized water and mixed well.

- (0.4989 g)[87.12 g Sr/mol)/211.646 g $Sr(NO_3)_2$)] = 0.2054 g Sr
- (205.4 mg Sr)(100 mL) = 2.0536 mg Sr/mL (Cold Sr Stock Solution)

APPENDIX C-4

ORNL PLAN FOR CS DF MEASUREMENTS:

If it is assumed that 1000 counts are needed for a 30min count of 10 mL samples of filtrate for the needed counting accuracy (according to John Keller of the Chemical & Analytical Sciences Division (C&ASD) at ORNL) and if it is assumed that the NaTPB will provide a DF of 40,000, how much ^{137}Cs will be needed for CSTR Test 2?

Total simulant supernatant needed for CSTR Test 2

384 L or 101.5 gal

^{137}Cs in a Liter of Filtrate in Test 2 needed for the needed counting accuracy

(100 counts/mL)(1/30 min)(1000mL)(100 dpm/cpm)(1 mCi/2.22×10^{9} dpm) = 0.00015015 mCi ^{137}Cs

Total ^{137}Cs per Liter of filtrate based upon a DF of 40,000

$C_o/(0.0001515\ mCi) = 40{,}000$

$C_o = (0.0001515)(40{,}000) =$ **6 mCi ^{137}Cs/L**

The amount ^{137}Cs needed has to be based on potential filtrate not the actual filtrate collected in the test.

(384 L)(31.58/21.5) = 564 L potential filtrate

(6 mCi ^{137}Cs/L)(564 L) = **3,384 Ci ^{137}Cs**

APPENDIX C-5

SLURRY WASH RECYCLE SOLUTION SOLUTIONS

During CSTR Test 2 recycle solution were pumped to CSTR-1 at a rate of 6.86 mL/min.

(6.86 mL/min)(1 L/1000 mL)(60 min/h)(264 h) = **108.7 L or 28.71 gal**

The slurry wash recycle solution needed to be 1.195 molar in Na^+; and also needed to be about 0.03303 molar in NaTPB.
(Information from table on page 3 of HLW-SDT-99-01nn)

For 60 % Excess NaTPB

38 lb/h NaTPB = 17,509 g/h
(6.86 gal/min)(60 min/h)(3.7854 L/gal) = 1558.1 L/h
[(17,608 g)/(342.23 g/mol)]/1558.1 L = **0.03302 mol/L soluble NaTPB**

Recycle solution will be 1.195 *M* Na^+
(1.195 mol Na^+/L) - (0.03303 mol Na^+/L) = 1.162 *M* Na^+

Table 5. Estimated Slurry Wash Recycle Solution (1.195 *M* Na^+) for Test 2

Comp.	Molarity (mol/L)	Compound	Molecular Weight (g/mol)	Mass (g/L)
TPB	0.03303	NaTPB	342.23	11.30
OH^-	0.6584	NaOH	40.00	26.35
NO_3^-	0.2531	$NaNO_3$	84.99	21.51
NO_2^-	0.1083	$NaNO_2$	69.00	7.47
AlO_2^-	0.0645	$AL(NO_3)_3 \bullet 9H_2O$	375.14	24.20
CO_3^{2-}	0.0334	$Na_2CO_3 \bullet H_2O$	124.01	4.14
SO_4^{2-}	0.0312	Na_2SO_4	142.04	4.43
Cl^-	0.0052	NaCl	58.4	0.30
F^-	0.0067	NaF	41.99	0.28
HPO_4^{2-}	0.0015	$Na_2HPO_4 \bullet 7H_2O$	268.09	0.40
$C_2O_4^{2-}$	0.0017	$Na_2C_2O_4$	134.00	0.23
SiO_3^{2-}	0.0008	$Na_2SiO_3 \bullet 9H_2O$	284.2	0.23
MoO_4^{2-}	0.00004	$Na_2MoO_4 \bullet 2H_2O$	241.95	0.01

Calculated SG for preparation = 1.0568 g/mL

Appendix D

CSTR 20-L DEMONSTRATION CONTROL SYSTEMS

July 14, 1999

HLW-SDT-99-0204

Trip Report – Small Tank Pilot Scale-up – ORNL June 2 -June 25, 1999

Travelers: Tony Tipton, Jim Firth, Rocci Norcia

Contacts: Doug Lee

Summary:

An Operational Readiness Review for planned testing at the ORNL facility, relative to the Small Tank process, was completed on May 18, 1999. The review turned up four areas requiring additional work and preparation, one specific to Instrumentation and Controls. ORNL responded to the areas of concern with a request to WSRC to provide assistance in correcting their I&C issues, as defined in recovery plan (HLW-SDT-99-0134). WSRC agreed, directing P&CT to take the lead on this initiative, and three people were identified to support this effort. Tony Tipton, was identified as the P&CT point of contact managing the overall effort on location at ORNL. A contract was written to the Controls Company, to assist with evaluating the final control and measuring elements insuring satisfactory equipment operation, and later performing tuning under a load condition. A pre-existing contract was modified to provide support for the Intellution workstation, from resources currently working at SRS.

Mockups for the Slurry Concentration Loop and Slurry Wash Loop equipment was staged in preparation for final installation for the Small Tank Process testing, at the ORNL facility (See Attachment 1 for process layout). The planned efforts by P&CT were broken into three separate activities. Activity 1 - checkout of all final control and measuring elements, beginning 6/2/99 and continued through 6/25/99. Activity 2 - configuration and checkout of the Intellution workstation which began 6/7/99 and also continued through 6/25/99. Activity 3 - evaluation of the entire system during an integrated water runs, to perform a final checkout and tuning of the continuous control loops. This effort completed 6/25/99.

General comments are summarized below:

1. **Activity 1** - Over a three week period, a checkout of all equipment associated with the Slurry Concentration and Slurry Wash Loops was completed (See Attachment 2) to evaluate performance, and recommendations were made to address unsatisfactory conditions where deficiencies existed. Any equipment requiring modification was re-evaluated to insure correct functionality, and a baseline was established for all control elements in preparation for tuning during integrated testing. At the completion of this effort, data was gathered to allow initial tuning constants for the back pressure loops for both systems at close to normal operating conditions.

2. Attachment 1 includes all equipment evaluated for the specific loops, as well as actions required to establish full operability of the control elements prior to running the system. In most cases, control elements required some type of adjustment or repair, but at some point were declared satisfactory and initial tuning could be derived for the integrated testing conducted. See Attachment 2 for Protuner System Analysis data.

3. A question was raised during this exercise regarding the proposed control scheme for the Slurry Concentration Loop. Slurry Concentration Tank level control was discussed as the primary control element, however it was stressed by ORNL that Filtrate Flow rate must be maintained at a specified value. In order for this to happen, it was necessary to control the filtrate flow valve PCV-2 based on the flow-indicating controller FIC-1. It would be difficult to maintain filtrate flow rate based solely on the tank level LIC-1 with the Test 1A configuration because of the surges that will occur when feed is added into the concentration tank. This is due to the closely coupled nature of the system components. Testing demonstrated that the Slurry Concentration Tank had the capability of staying within boundaries for up to an hour without feed, and conversely, the tank could withstand an infeed without filtrate flow for up to an hour. Based on this input, a recommendation was made to use flow control as the primary control loop, with Tank level as a secondary control loop, (see discussions in Attachment 3). However, the final control strategy was then changed as it was determined that for longer term, Level control, based on LIC-1 would probably be the method of choice when the system was fully configured.

4. The final control strategy for the test system, was implemented as cascaded control that uses LIC-1 as the primary (outer) loop that feeds its output to the setpoint of FIC-1, the secondary (inner) loop. During Test 1A, the cascade was broken, leaving a control based on FIC-1. This was configured and tested in an integrated simulated run and proved very stable. The system was left in this configuration for the remainder of the testing exercises.

5. **Activity 2 -** WSRC contract personnel (Firth), began work on 6/8/99 at the Oak Ridge Facility. The workstation had been specified and procured by ORNL and partially configured at this point. ORNL had configured the interface between the MTL product (used to control field I/O) and the Intellution package (used to build I/O database and Graphics). Firth was directed to concentrate efforts with the Intellution package, as the configuration had not been started, building the database and graphics. Field troubleshooting was required to ring out any wiring problems and historical trends were established to capture all data associated with the system. All control algorithms were performed in the Intellution software. Integrated functional testing of the control system and process components was satisfactorily performed. Operator training was provided to ORNL personnel on both shifts to ensure that they understood the control and functionality of the system. The overall effort took approximately two weeks to complete, and a baseline was created prior to running the first test.

6. **Activity 3 -** Final control element tuning constants are listed below. These values include a configuration for controlling filtrate flow rate based on level (LIC-1) or flow (FIC-1).

Loop Tag Name	PID Output Low	PID Output High	PID Set Point Address	PID Prop Band	PID Reset Time	PID Rate	PID Output Reverse
PIDPCV-1	0	100	PID1SP.F_CV	657.9	0.0125	0	YES
PIDPCV-2A	0	40	SETPOINT2LEVEL.F	11.358	94.204	0	YES
PIDPCV-2B	0	100	PIDPCV-2A.F_CV	2,035.00	0.255	0	NO
PIDPCV-3	0	100	PID3SP.F_CV	452.8	0.0125	0	YES
PIDPCV-4A	0	25	SETPOINT4LEVEL.F	50	20	0	YES
PIDPCV-4B	0	100	PIDPCV-4A.F_CV	969	0.5618	0	NO

cc: J. T. Carter, 704-3N
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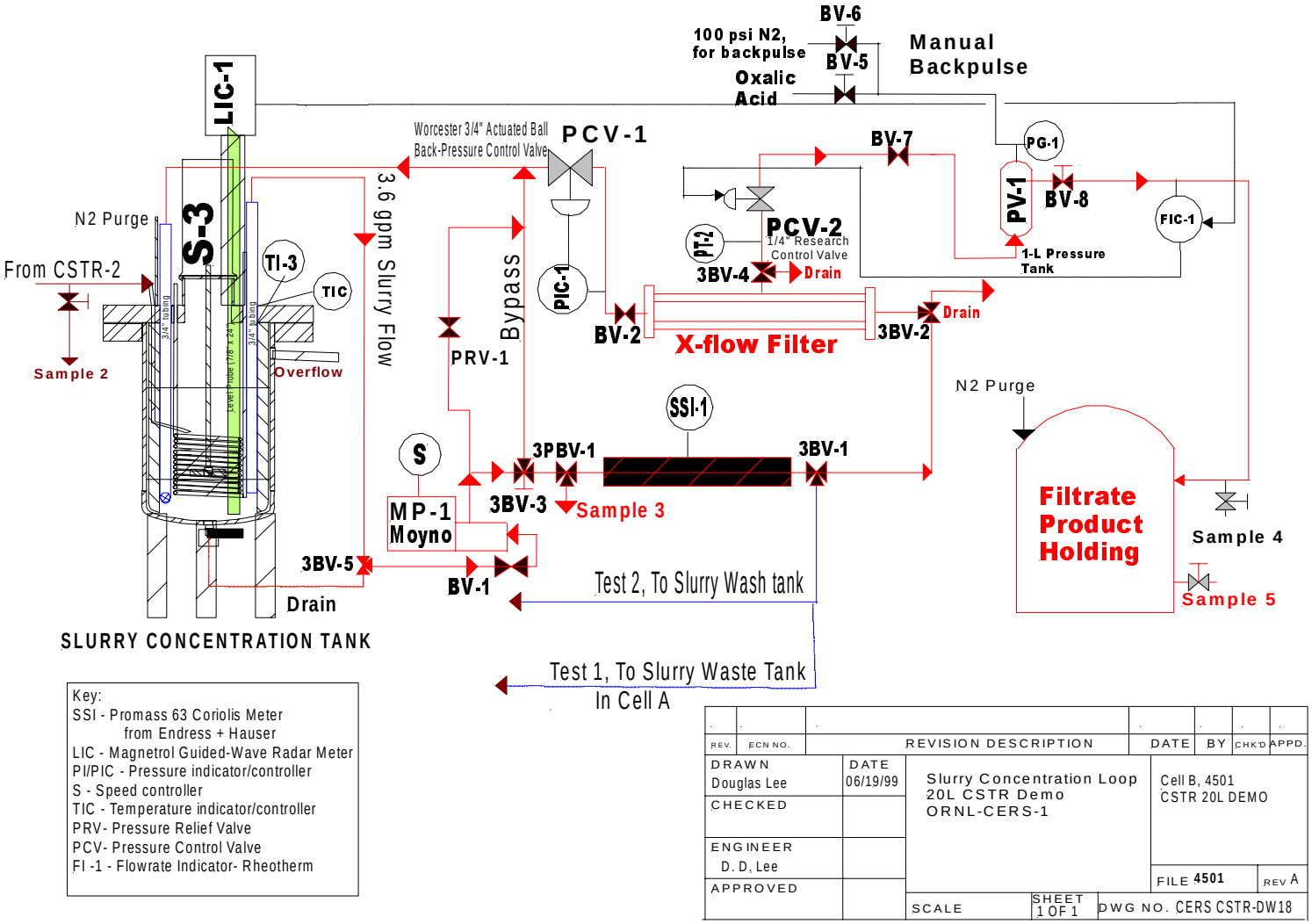
Attachment 1

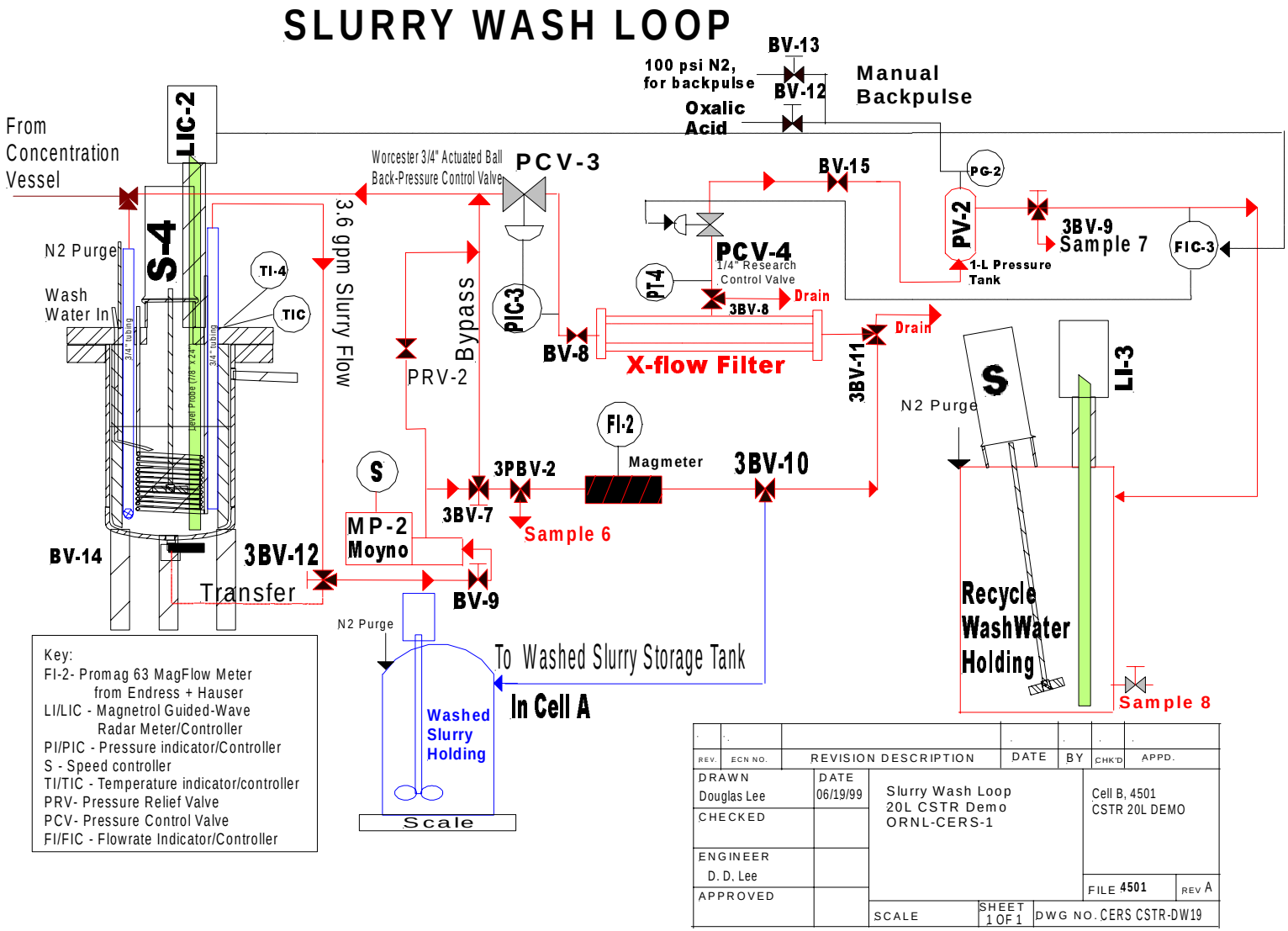
SLURRY CONCENTRATION LOOP



Key:
SSI - Promass 63 Coriolis Meter from Endress + Hauser
LIC - Magnetrol Guided-Wave Radar Meter
PI/PIC - Pressure indicator/controller
S - Speed controller
TIC - Temperature indicator/controller
PRV- Pressure Relief Valve
PCV- Pressure Control Valve
FI -1 - Flowrate Indicator- Rheotherm

REV.	ECN NO.	REVISION DESCRIPTION	DATE	BY	CHK'D	APPD.

DRAWN Douglas Lee	DATE 06/19/99	Slurry Concentration Loop 20L CSTR Demo ORNL-CERS-1	Cell B, 4501 CSTR 20L DEMO
CHECKED			
ENGINEER D. D. Lee			FILE 4501 REV A
APPROVED		SCALE	SHEET 1 OF 1 DWG NO. CERS CSTR-DW18

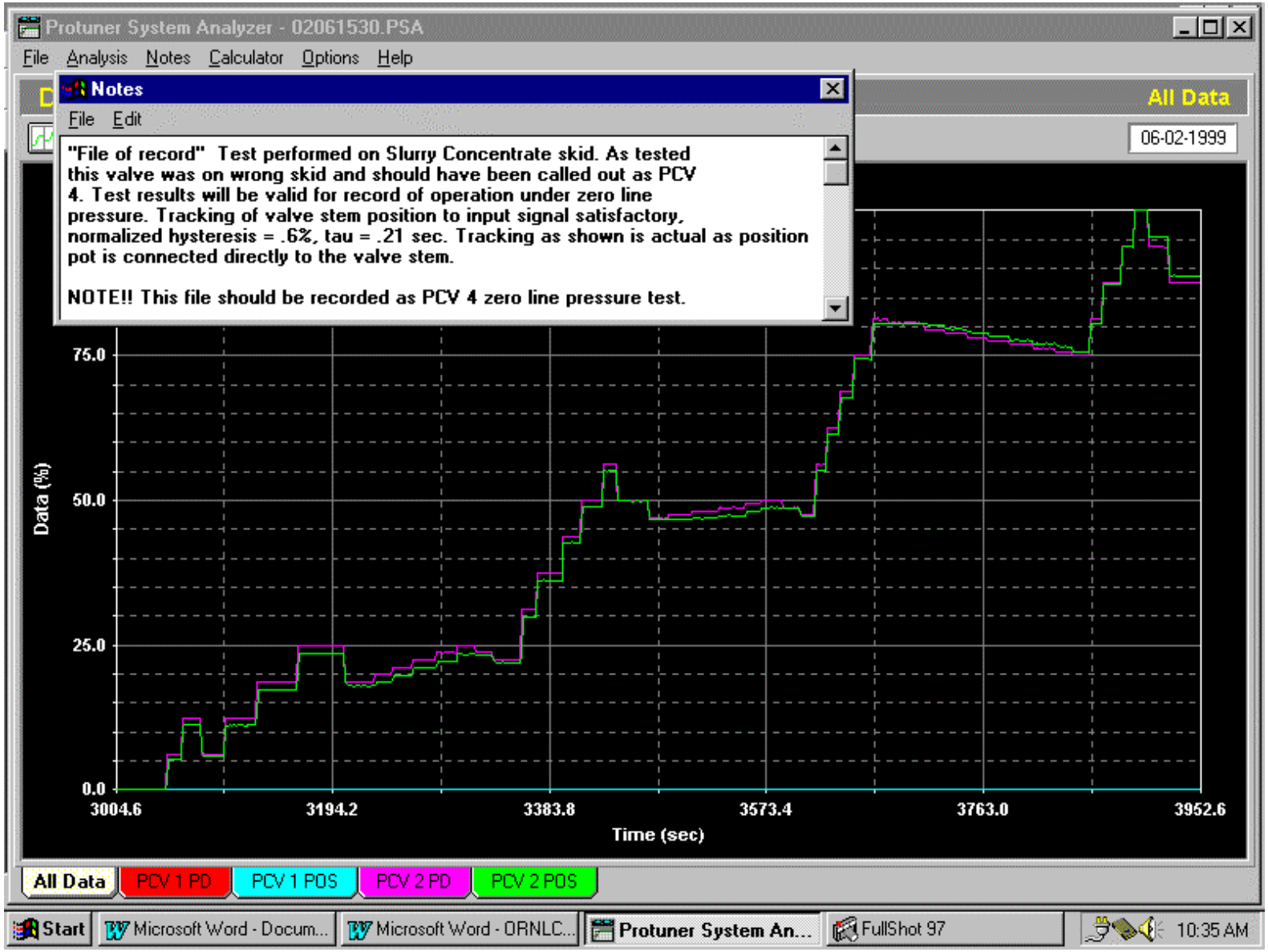

SLURRY WASH LOOP
From Concentration Vessel
LIC-2
S-4
N2 Purge
Wash Water In
TI-4
TIC
BV-14
3BV-12
Transfer
3.6 gpm Slurry Flow
Worcester 3/4" Actuated Ball Back-Pressure Control Valve
PCV-3
Bypass
PRV-2
PIC-3
BV-8
S
MP-2 Moyno
3BV-7
3PBV-2
Sample 6
BV-9
N2 Purge
Washed Slurry Holding
Scale
To Washed Slurry Storage Tank
In Cell A
100 psi N2, for backpulse
BV-13
BV-12
Oxalic Acid
Manual Backpulse
BV-15
PG-2
PV-2
1-L Pressure Tank
3BV-9
Sample 7
FIC-3
PCV-4
1/4" Research Control Valve
PT-4
3BV-8
Drain
X-flow Filter
3BV-11
Drain
FI-2
Magmeter
3BV-10
N2 Purge
S
LI-3
Recycle WashWater Holding
Sample 8
Key:
FI-2- Promag 63 MagFlow Meter from Endress + Hauser
LI/LIC - Magnetrol Guided-Wave Radar Meter/Controller
PI/PIC - Pressure indicator/Controller
S - Speed controller
TI/TIC - Temperature indicator/controller
PRV- Pressure Relief Valve
PCV- Pressure Control Valve
FI/FIC - Flowrate Indicator/Controller
REV. ECN NO. REVISION DESCRIPTION DATE BY CHK'D APPD.
DRAWN Douglas Lee
DATE 06/19/99
CHECKED
ENGINEER D. D. Lee
APPROVED
Slurry Wash Loop
20L CSTR Demo
ORNL-CERS-1
Cell B, 4501
CSTR 20L DEMO
FILE 4501
REV A
SCALE
SHEET 1 OF 1
DWG NO. CERS CSTR-DW19

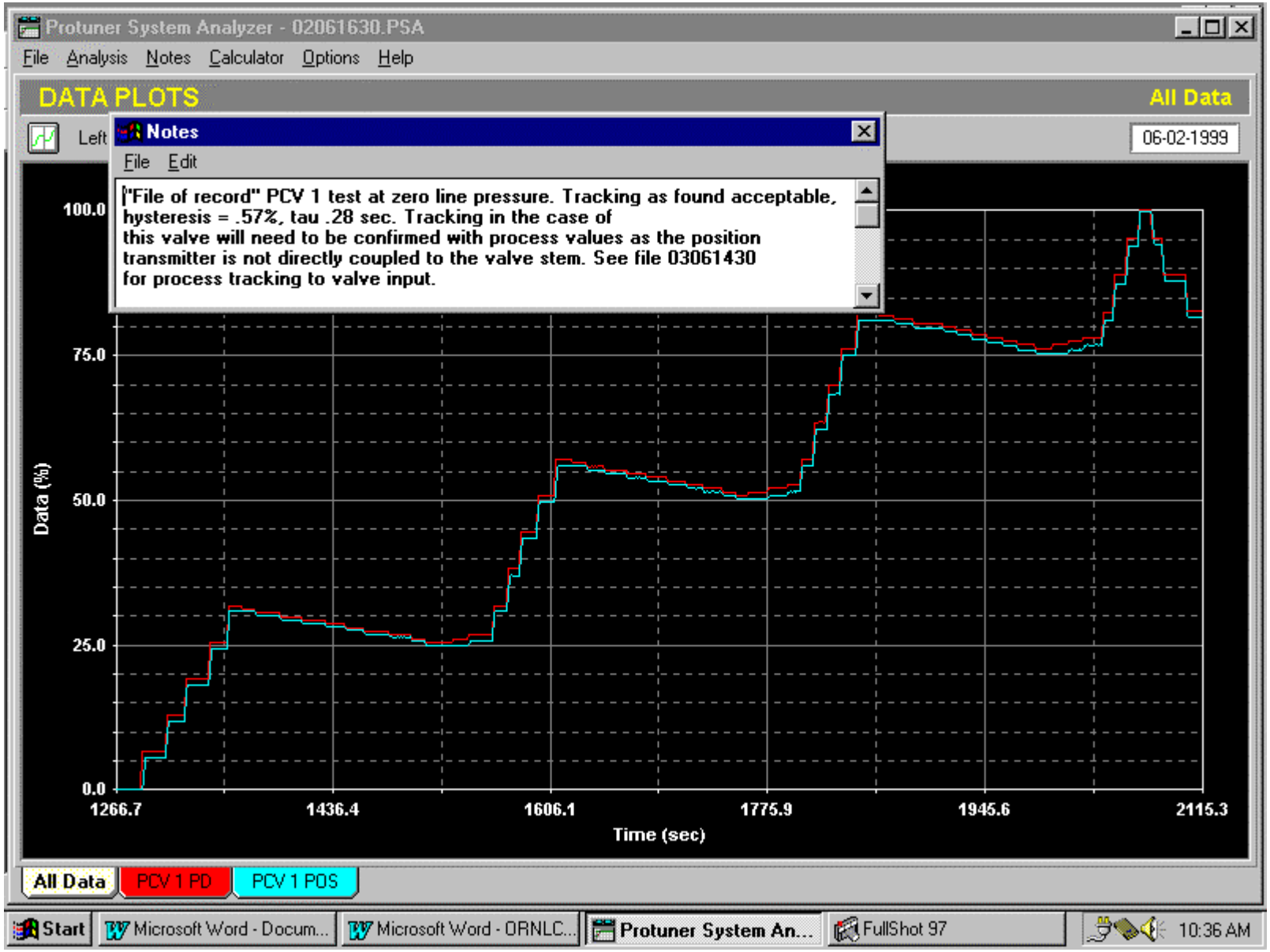
Attachment 2

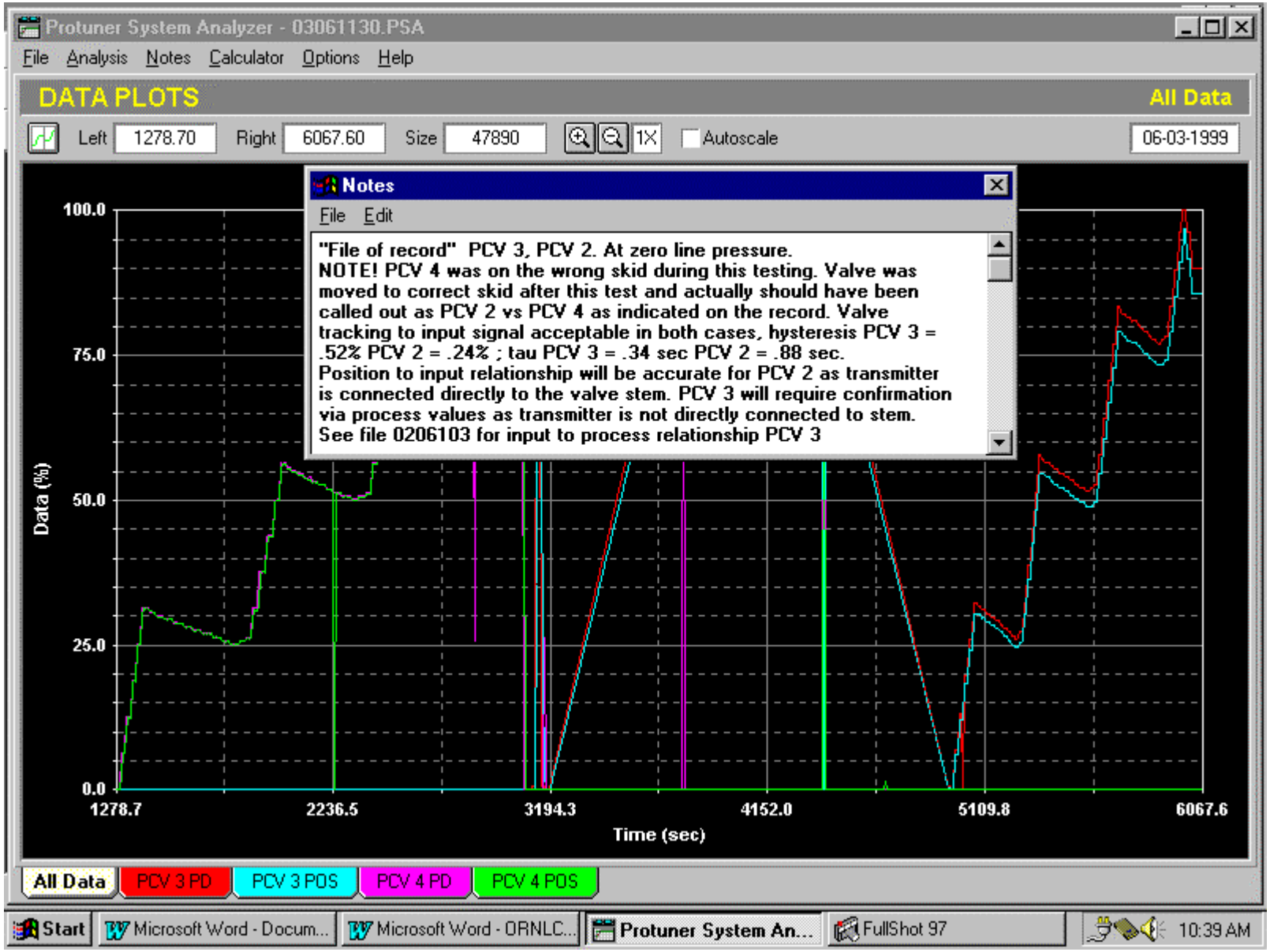
Loop Tag Name	Loop Description	As Found	Comments	*Resolution*	*As Left*	File Name
FIC-1	Concentration Filtrate Flow	Not Functional	Final control element and associated electronics board were serial number mismatched. Additionally, element was being supplied power from two sources, loop power as well as its own power supply.	Matched final control element with electronics board, and removed loop power. Performed calibration.	Functional.	06231110.psa
FI-2	Slurry Wash Feed Flow	Not Functional	Mag flow meter out of calibration.	Vendor contacted, came to ORNL and calibrated meter.	Functional	N/A
FIC-3	Wash Filtrate Flow Rate	Not Functional	Final control element and associated electronics board were serial number mismatched. Additionally, element was being supplied power from two sources, loop power as well as its own power supply.	Matched final control element with electronics board, and removed loop power. **Calibration to be performed prior to Test 2**.	N/A – To be tested prior to Test 2	N/A
FI-4	Recycle Washwater Flow	Not Installed	To be installed prior to Test 2	N/A	Not Installed	N/A
HLA-1	CSTR-2 Sump High Level Alarm	Functional	N/A	N/A	Functional	N/A
HLA-2	Recycle Washwater Sump High	Not Installed	To be installed prior to Test 2	N/A	Not Installed	N/A
LIC-1	Concentration Level Indicator	Functional	Calibration of level indicator shows that the signal is noisy at either end of the scale, and more smooth at midrange.	Added signal filtering in the Intellution control software.	Functional	06231110.psa
LIC-2	Wash tank Level Indicator	Installed	Instrument provides feedback, however calibration will not be performed until prior to Test 2.	Calibrate prior to Test 2.	Installed	N/A
LI-3	Recycle Washwater Level Indicator	Not Installed	To be installed prior to Test 2	N/A	Not Installed	N/A
LI-4	Salt Feed Tank Level Indicator	Functional	This level indicator will be used in a secondary capacity for the Chemists' use. Calibrated by ORNL. Signal is noisy similar to LIC-1.	Added signal filtering in the Intellution control software.	Functional	N/A

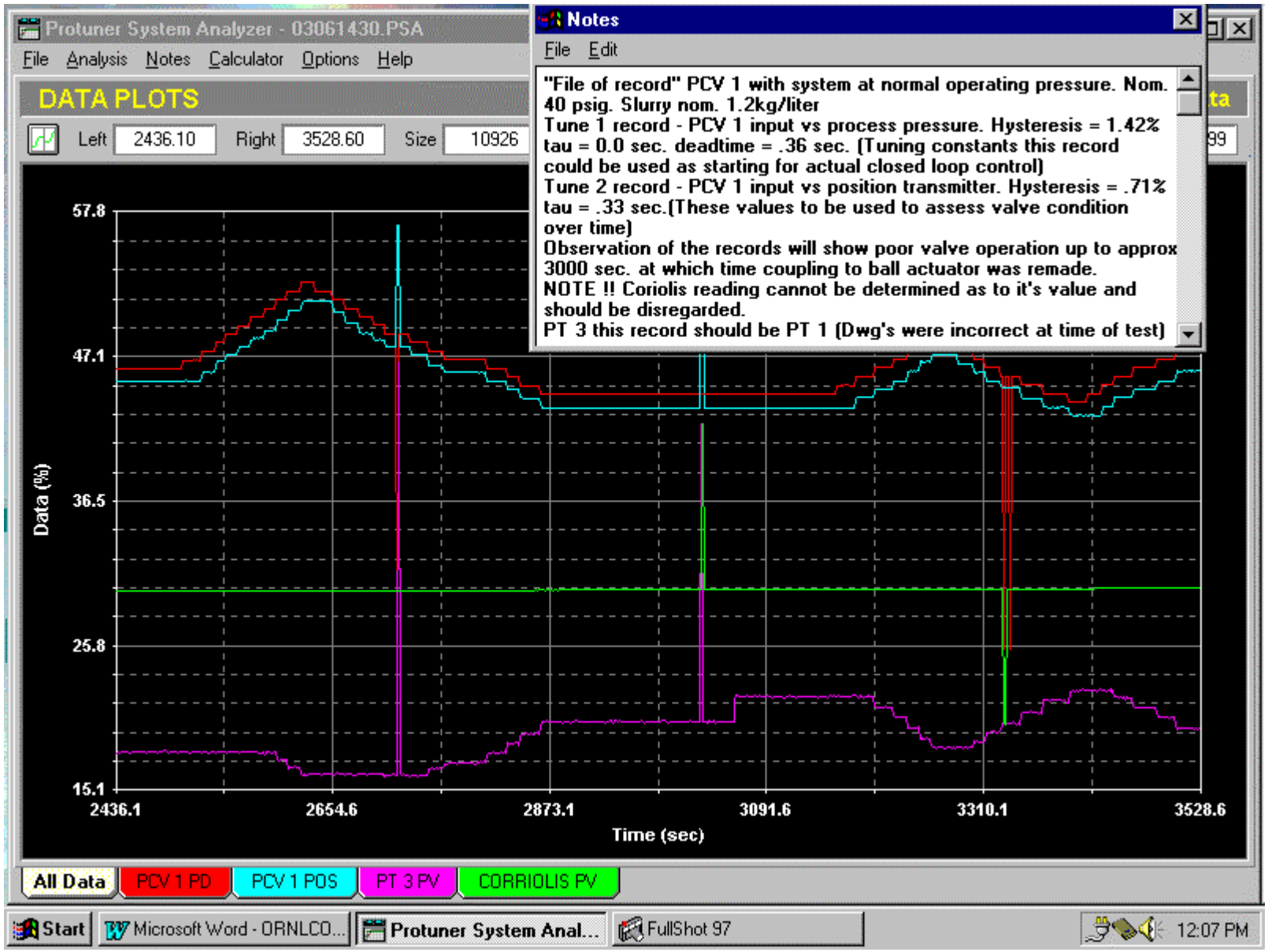
PCV-1	Concentration Post Cross Flow	Functional	Zero line pressure on System	N/A	Functional. Hysteresis = 0.57%, Tau=0.28 sec. With Zero line pressure.	02061630.psa 06171200.psa
PCV-1	Concentration Post Cross Flo	Functional	Normal Operating Pressure – 40psig. Slurry simulate was used to represent the process fluid. Under these conditions, a noticeable amount of hysteresis detected. Hysteresis = 1.42%, Tau = 0.0 sec., Deadtime = 0.36 sec. Suggested to ORNL that coupler be drilled and pinned to the positioner as well as the valve stem.	Tightened connection between valve positioner and coupling device as well as valve stem and coupling device. Additional set screw was installed opposite existing screws for more stability in the connections.	Hysteresis = 0.71%, Tau = 0.33 sec, Deadtime = 0.32 sec.	03061430.psa
PCV-2	Concentration Cross Flow Pre	Functional	Zero line pressure on System	N/A	Functional. Hysteresis = 0.24%, Tau = 0.88 sec. With zero line pressure.	03061130.psa 06171200.psa
PCV-2	Concentration Cross Flow Pre	Partially Functional	Normal Operating Pressure – 40psig. Slurry simulate was used to represent the process fluid. Under these conditions the valve would not open at a controlled rate. Only fully open or fully closed.	Valve trim was damaged. Contacted vendor and had a new one shipped in. Valve now functions correctly.	Functional.	06091545.psa
PCV-3	Slurry Wash Post Cross Flow	Functional	Zero line pressure on System	N/A	Functional. Hysteresis = 0.52%, Tau = .34 sec. With zero line pressure.	03061130.psa 06171200.psa
PCV-3	Slurry Wash Post Cross Flow	Functional	Normal Operating Pressure – 40psig. Slurry simulate was used to represent the process fluid. Under these conditions, process is tightly coupled between flow rate and back pressure control.	N/A	Functional. PCV-3 vs Process Variable – Hysteresis = 2.51%, Tau = 0.27 sec., Deadtime = 0.15 sec. PCV-3 vs Position Transmitter – Hysteresis = .007%, Tau = 0.18 sec.	04061030.psa

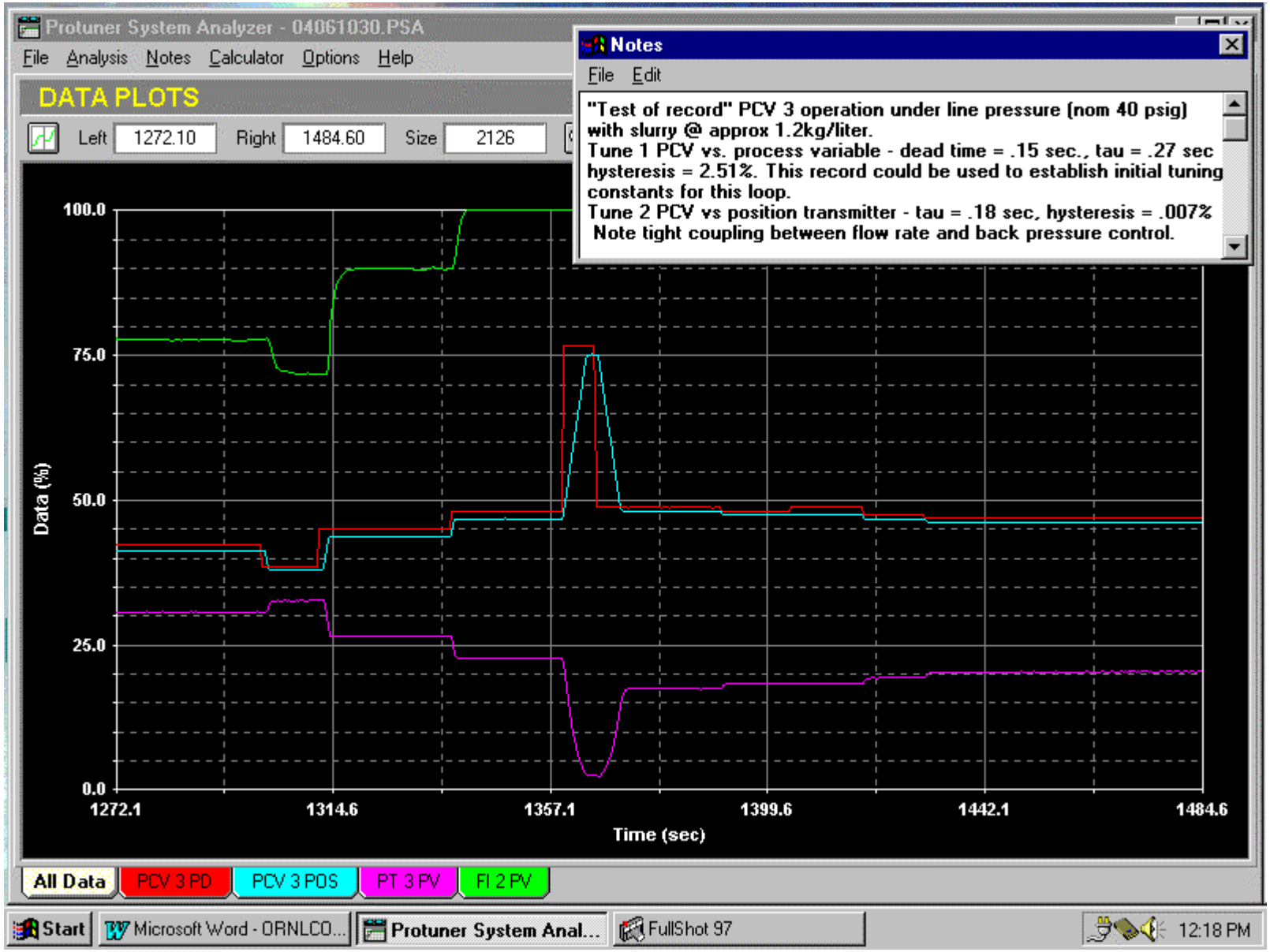
PCV-4	Slurry Wash Cross Flow Press	Output signal not responding	Swapped electronic board with similar valve, however, did not correct problem.	Connected position pot to valve for characterization. Called vendor for assistance with non-responsive output signal. Re-seated all electrical connections on valve positioner. Output signal problem corrected.	Functional. Normalized hysteresis = 0.6%, tau=0.21 sec. With Zero line pressure.	02061530.psa 06171200.psa
PCV-4	Slurry Wash Cross Flow Press	Functional	Normal Operating Pressure – 40psig. Slurry simulate was used to represent the process fluid. Under these conditions, valve operates in the upper ranges of positioning (75% open and higher). This is due to the valve being slightly undersized because of recent test requirements changes that specifies a larger filtrate flow rate. This also applies to PCV-2.	Evaluation determined that operating range of valve could be controlled satisfactorily.	Functional.	04061100.psa (Note: Data is valid for PCV-4)
PIC-1	Concentration Post Cross Flow	Functional	Used factory calibration data.	N/A	Functional	06231110.psa
PI-2	Concentration Cross Flow Pressure	Functional	Used factory calibration data.	N/A	Functional	06231110.psa
PIC-3	Slurry Wash Post Cross Flow	Functional	Used factory calibration data. To be checked out prior to Test 2.	N/A	Functional	N/A
PI-4	Slurry Wash Cross Flow Press	Functional	Used factory calibration data. To be checked out prior to Test 2.	N/A	Functional	N/A
SSI-1	Concentration Feed Density	Not Functional	Corriolis giving one output, but it can't be determined what the data represents, Temperature, Flow, or Density. It is also mounted such that solids could collect in one of the tubes. Needs to be rotated 180 deg.	Vendor came to site, modified wiring terminations, and calibrated the meter.	Functional.	N/A
TI-1	CSTR-1 Temperature	Functional	Used factory calibration data.	N/A	Functional	N/A
TI-2	CSTR-2 Temperature	Functional	Used factory calibration data.	N/A	Functional	N/A
TI-3	Concentration Temperature	Functional	Used factory calibration data.	N/A	Functional	N/A
TI-4	Slurry Washing Temperature	Functional	Used factory calibration data. To be checked out prior to Test 2.	N/A	Functional	N/A

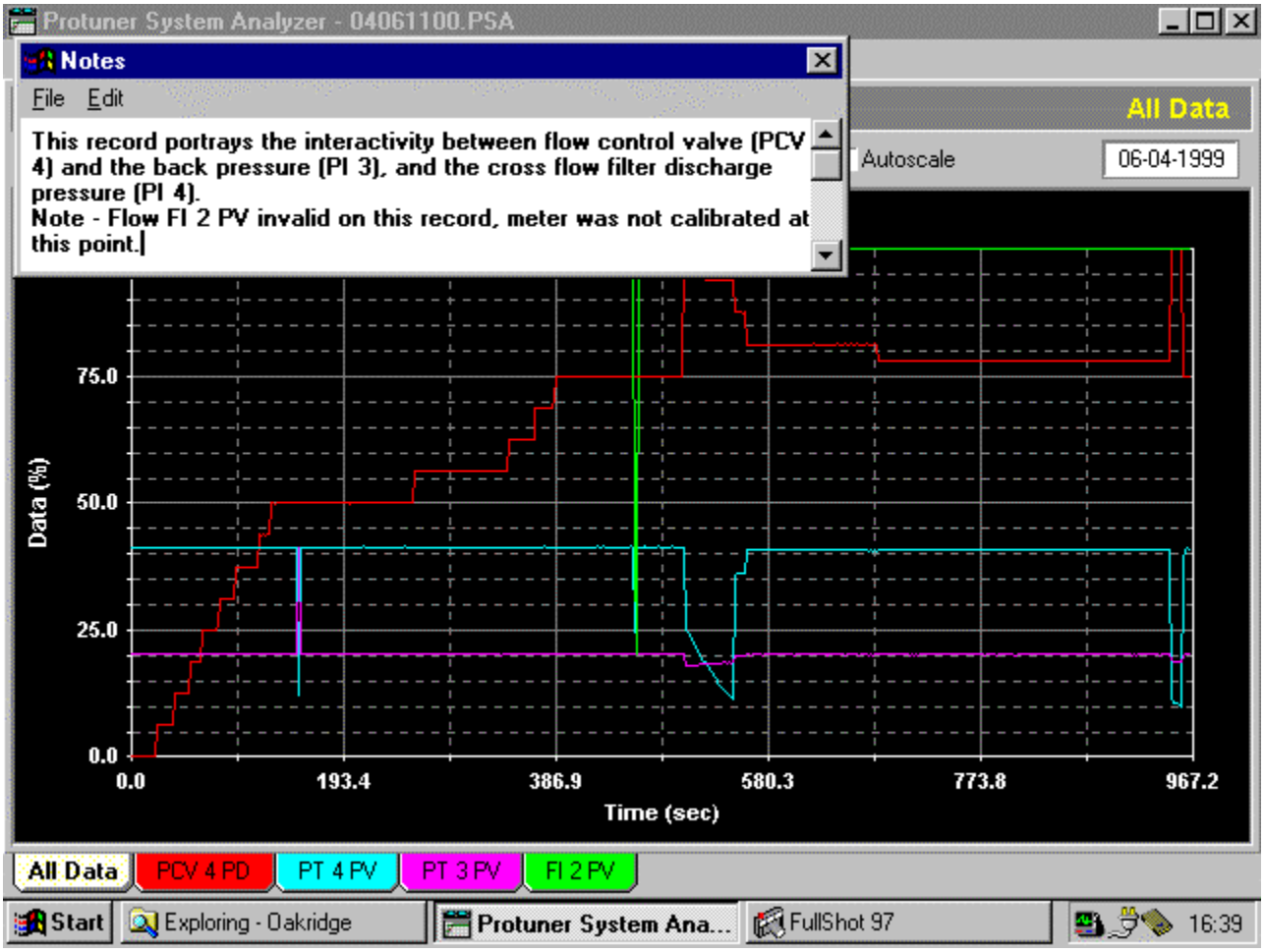

Protuner System Analyzer - 02061530.PSA
File Analysis Notes Calculator Options Help
Notes
File Edit
"File of record" Test performed on Slurry Concentrate skid. As tested this valve was on wrong skid and should have been called out as PCV 4. Test results will be valid for record of operation under zero line pressure. Tracking of valve stem position to input signal satisfactory, normalized hysteresis = .6%, tau = .21 sec. Tracking as shown is actual as position pot is connected directly to the valve stem.
NOTE!! This file should be recorded as PCV 4 zero line pressure test.
All Data
06-02-1999
Data (%)
75.0
50.0
25.0
0.0
3004.6
3194.2
3383.8
3573.4
3763.0
3952.6
Time (sec)
All Data
PCV 1 PD
PCV 1 POS
PCV 2 PD
PCV 2 POS
Start
Microsoft Word - Docum...
Microsoft Word - ORNLC...
Protuner System An...
FullShot 97
10:35 AM

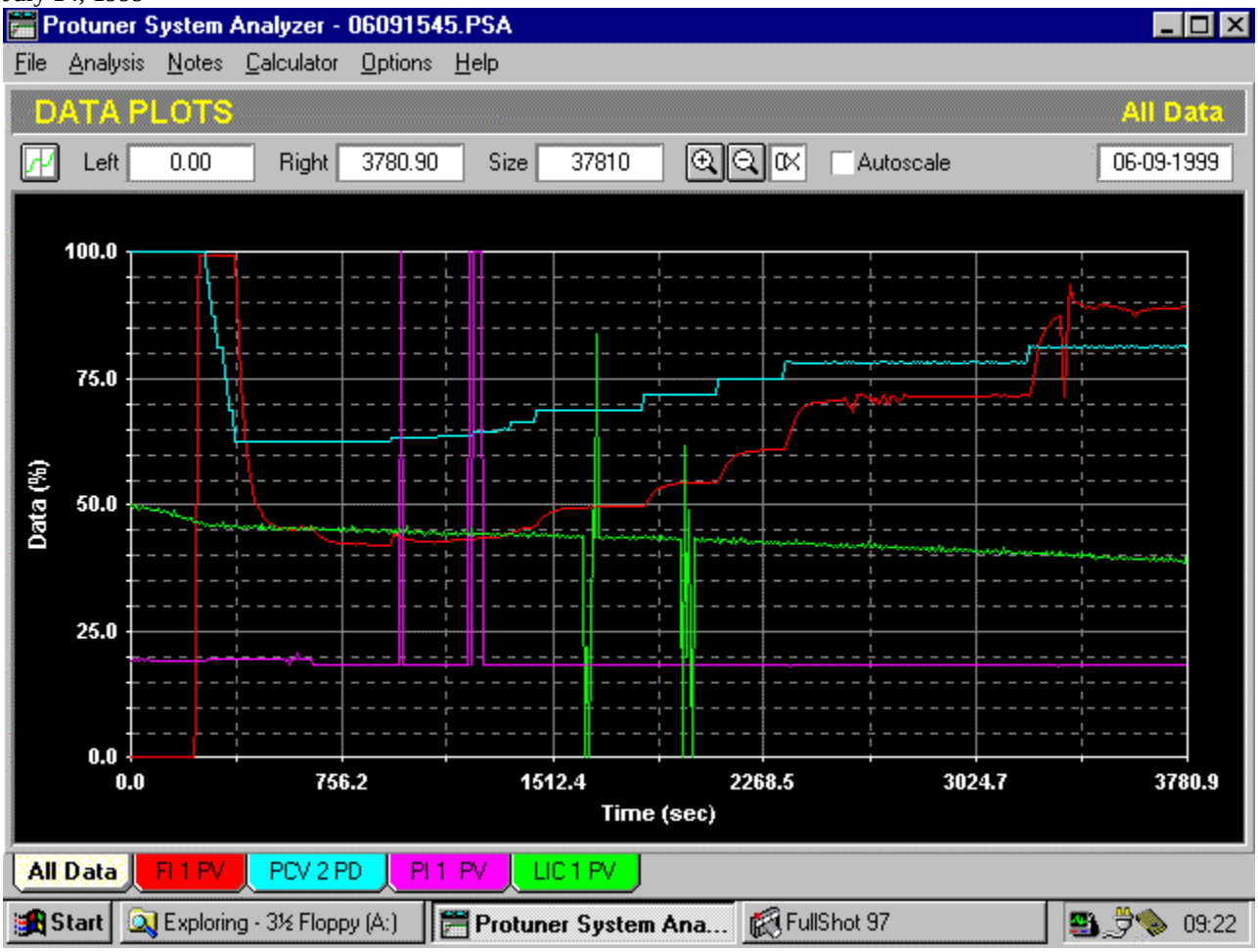

Protuner System Analyzer - 02061630.PSA
File Analysis Notes Calculator Options Help
DATA PLOTS
All Data
Left
06-02-1999
Notes
File Edit
"File of record" PCV 1 test at zero line pressure. Tracking as found acceptable, hysteresis = .57%, tau .28 sec. Tracking in the case of this valve will need to be confirmed with process values as the position transmitter is not directly coupled to the valve stem. See file 03061430 for process tracking to valve input.
100.0
75.0
50.0
25.0
0.0
Data (%)
1266.7
1436.4
1606.1
1775.9
1945.6
2115.3
Time (sec)
All Data
PCV 1 PD
PCV 1 POS
Start
Microsoft Word - Docum...
Microsoft Word - ORNLC...
Protuner System An...
FullShot 97
10:36 AM

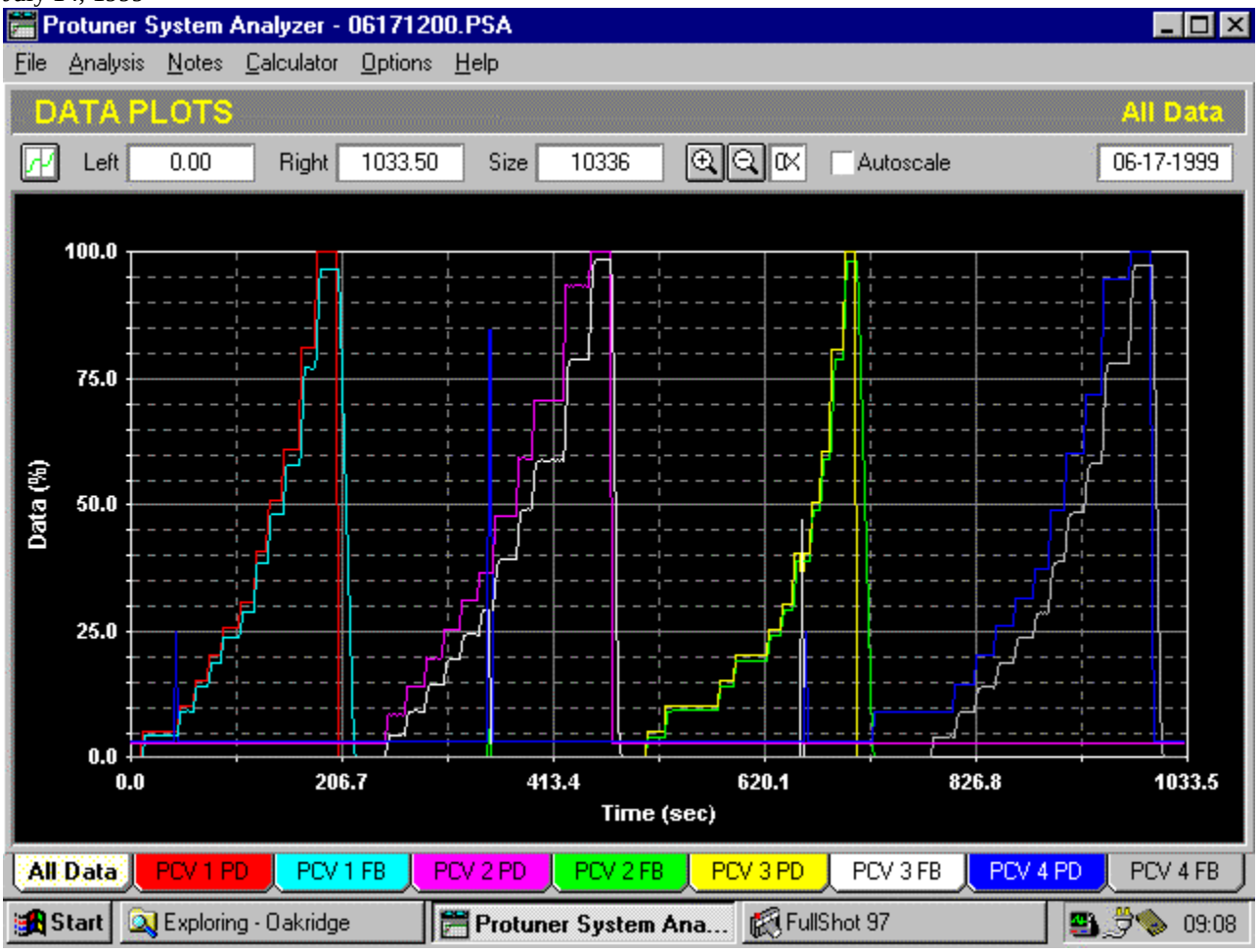

Protuner System Analyzer - 03061130.PSA
File Analysis Notes Calculator Options Help
DATA PLOTS
All Data
Left 1278.70 Right 6067.60 Size 47890 1X Autoscale
06-03-1999
Notes
File Edit
"File of record" PCV 3, PCV 2. At zero line pressure.
NOTE! PCV 4 was on the wrong skid during this testing. Valve was moved to correct skid after this test and actually should have been called out as PCV 2 vs PCV 4 as indicated on the record. Valve tracking to input signal acceptable in both cases, hysteresis PCV 3 = .52% PCV 2 = .24% ; tau PCV 3 = .34 sec PCV 2 = .88 sec.
Position to input relationship will be accurate for PCV 2 as transmitter is connected directly to the valve stem. PCV 3 will require confirmation via process values as transmitter is not directly connected to stem.
See file 0206103 for input to process relationship PCV 3
100.0
75.0
50.0
25.0
0.0
Data (%)
1278.7
2236.5
3194.3
4152.0
5109.8
6067.6
Time (sec)
All Data
PCV 3 PD
PCV 3 POS
PCV 4 PD
PCV 4 POS
Start
Microsoft Word - Docum...
Microsoft Word - ORNLC...
Protuner System An...
FullShot 97
10:39 AM


Protuner System Analyzer - 03061430.PSA
File Analysis Notes Calculator Options Help
DATA PLOTS
Left 2436.10 Right 3528.60 Size 10926
Notes
File Edit
"File of record" PCV 1 with system at normal operating pressure. Nom. 40 psig. Slurry nom. 1.2kg/liter
Tune 1 record - PCV 1 input vs process pressure. Hysteresis = 1.42% tau = 0.0 sec. deadtime = .36 sec. (Tuning constants this record could be used as starting for actual closed loop control)
Tune 2 record - PCV 1 input vs position transmitter. Hysteresis = .71% tau = .33 sec.(These values to be used to assess valve condition over time)
Observation of the records will show poor valve operation up to approx 3000 sec. at which time coupling to ball actuator was remade.
NOTE !! Coriolis reading cannot be determined as to it's value and should be disregarded.
PT 3 this record should be PT 1 (Dwg's were incorrect at time of test)
57.8
47.1
36.5
25.8
15.1
Data (%)
2436.1
2654.6
2873.1
3091.6
3310.1
3528.6
Time (sec)
All Data
PCV 1 PD
PCV 1 POS
PT 3 PV
CORRIOLIS PV
Start
Microsoft Word - ORNLCO...
Protuner System Anal...
FullShot 97
12:07 PM


Protuner System Analyzer - 04061030.PSA
File Analysis Notes Calculator Options Help
DATA PLOTS
Left 1272.10 Right 1484.60 Size 2126
Notes
File Edit
"Test of record" PCV 3 operation under line pressure (nom 40 psig) with slurry @ approx 1.2kg/liter.
Tune 1 PCV vs. process variable - dead time = .15 sec., tau = .27 sec hysteresis = 2.51%. This record could be used to establish initial tuning constants for this loop.
Tune 2 PCV vs position transmitter - tau = .18 sec, hysteresis = .007%
Note tight coupling between flow rate and back pressure control.
100.0
75.0
50.0
25.0
0.0
Data (%)
1272.1
1314.6
1357.1
1399.6
1442.1
1484.6
Time (sec)
All Data
PCV 3 PD
PCV 3 POS
PT 3 PV
FI 2 PV
Start
Microsoft Word - ORNLCO...
Protuner System Anal...
FullShot 97
12:18 PM


Protuner System Analyzer - 04061100.PSA
Notes
File Edit
This record portrays the interactivity between flow control valve (PCV 4) and the back pressure (PI 3), and the cross flow filter discharge pressure (PI 4).
Note - Flow FI 2 PV invalid on this record, meter was not calibrated at this point.
All Data
Autoscale
06-04-1999
Data (%)
75.0
50.0
25.0
0.0
0.0
193.4
386.9
580.3
773.8
967.2
Time (sec)
All Data
PCV 4 PD
PT 4 PV
PT 3 PV
FI 2 PV
Start
Exploring - Oakridge
Protuner System Ana...
FullShot 97
16:39


Protuner System Analyzer - 06091545.PSA
File Analysis Notes Calculator Options Help
DATA PLOTS
All Data
Left 0.00
Right 3780.90
Size 37810
Autoscale
06-09-1999
100.0
75.0
50.0
25.0
0.0
Data (%)
0.0
756.2
1512.4
2268.5
3024.7
3780.9
Time (sec)
All Data
FI 1 PV
PCV 2 PD
PI 1 PV
LIC 1 PV
Start
Exploring - 3½ Floppy (A:)
Protuner System Ana...
FullShot 97
09:22


Protuner System Analyzer - 06171200.PSA
File Analysis Notes Calculator Options Help
DATA PLOTS
All Data
Left 0.00
Right 1033.50
Size 10336
Autoscale
06-17-1999
100.0
75.0
50.0
25.0
0.0
Data (%)
0.0
206.7
413.4
620.1
826.8
1033.5
Time (sec)
All Data
PCV 1 PD
PCV 1 FB
PCV 2 PD
PCV 2 FB
PCV 3 PD
PCV 3 FB
PCV 4 PD
PCV 4 FB
Start
Exploring - Oakridge
Protuner System Ana...
FullShot 97
09:08

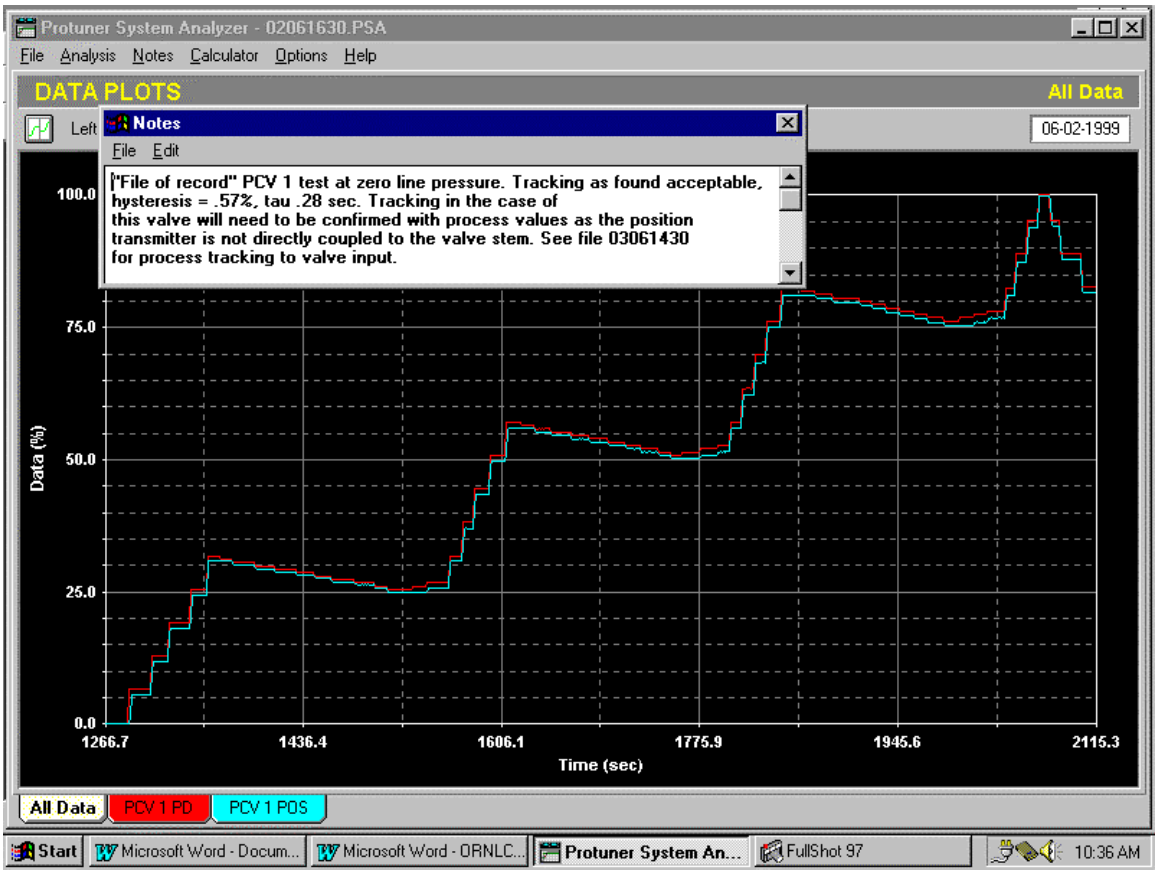
Attachment 3

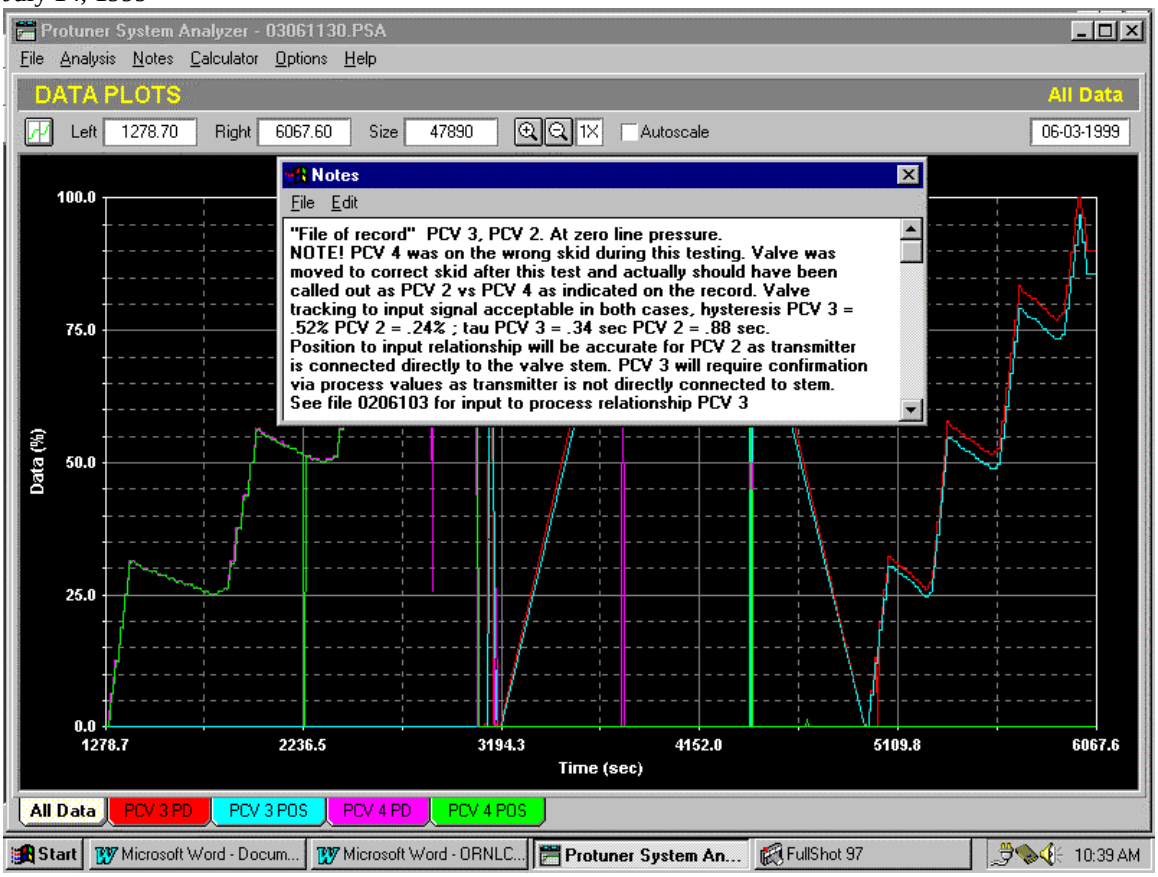
ORNL Control Strategy Status, Discussions, Concerns and Conclusions

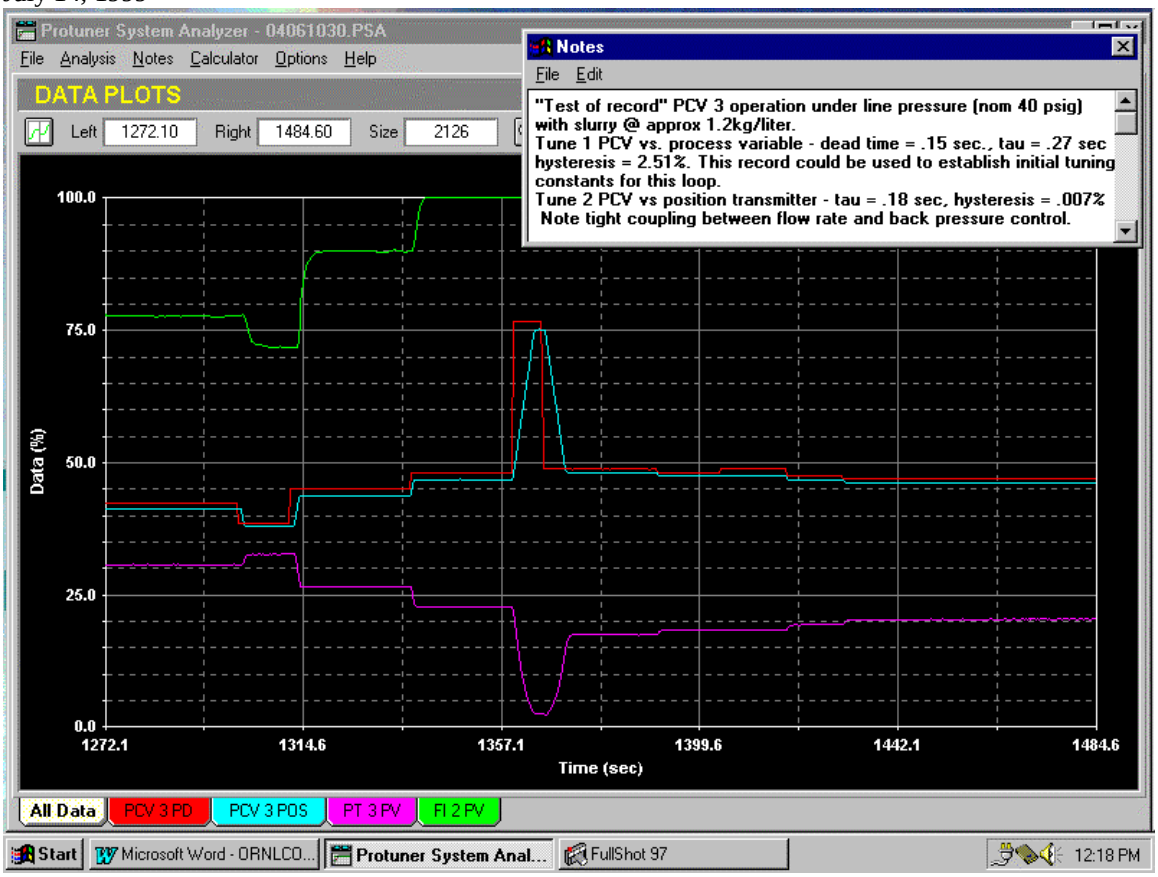
Status Overview

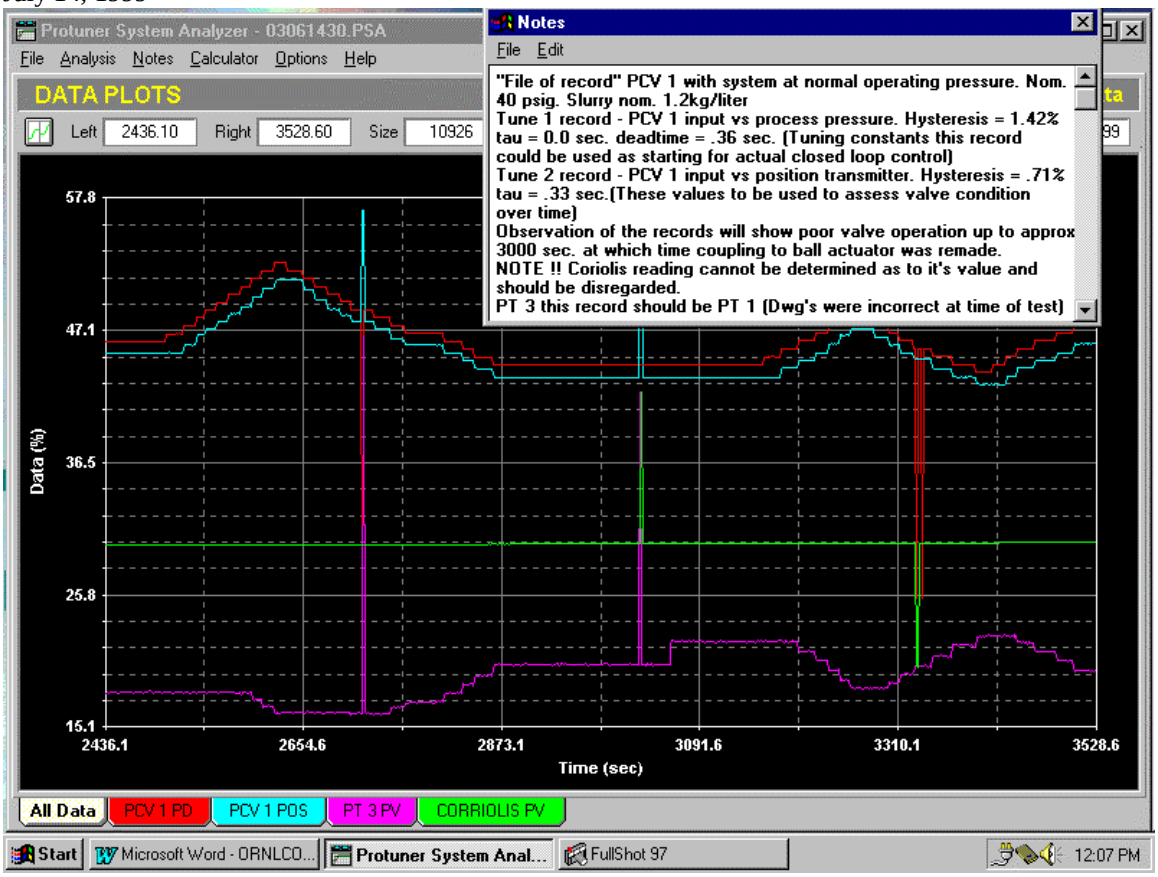
A control strategy for the Small Tank TPB Process test system has been determined based upon a dissertation from Doug Lee of ORNL. Control algorithms have been reviewed on the basis of these control needs and are available in the Intellution FIX control software.

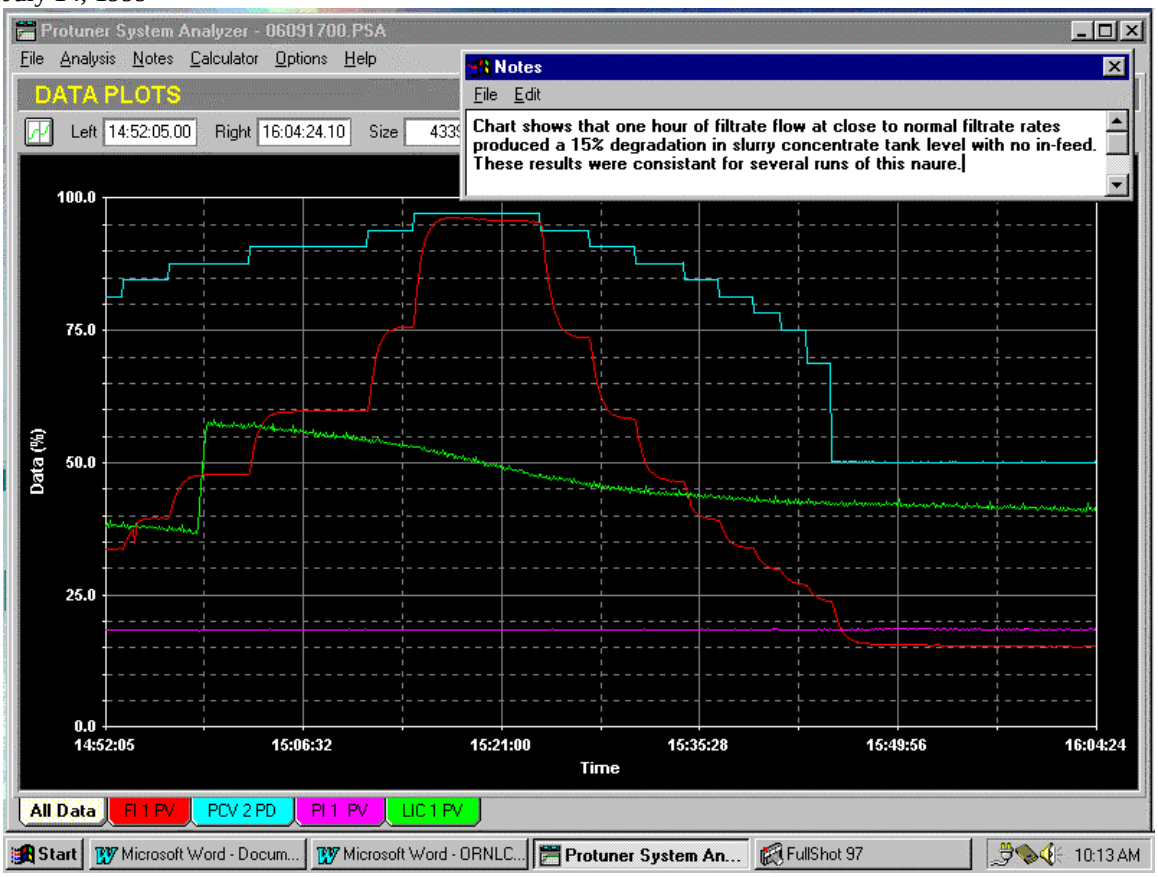
All final control elements have been tested for operability and are functional.




Protuner System Analyzer - 03061130.PSA
File Analysis Notes Calculator Options Help
DATA PLOTS
All Data
Left 1278.70 Right 6067.60 Size 47890 1X Autoscale
06-03-1999
Notes
File Edit
"File of record" PCV 3, PCV 2. At zero line pressure.
NOTE! PCV 4 was on the wrong skid during this testing. Valve was moved to correct skid after this test and actually should have been called out as PCV 2 vs PCV 4 as indicated on the record. Valve tracking to input signal acceptable in both cases, hysteresis PCV 3 = .52% PCV 2 = .24% ; tau PCV 3 = .34 sec PCV 2 = .88 sec.
Position to input relationship will be accurate for PCV 2 as transmitter is connected directly to the valve stem. PCV 3 will require confirmation via process values as transmitter is not directly connected to stem.
See file 0206103 for input to process relationship PCV 3
100.0
75.0
50.0
25.0
0.0
Data (%)
1278.7
2236.5
3194.3
4152.0
5109.8
6067.6
Time (sec)
All Data
PCV 3 PD
PCV 3 POS
PCV 4 PD
PCV 4 POS
Start
Microsoft Word - Docum...
Microsoft Word - ORNLC...
Protuner System An...
FullShot 97
10:39 AM


Protuner System Analyzer - 04061030.PSA
File Analysis Notes Calculator Options Help
DATA PLOTS
Left 1272.10 Right 1484.60 Size 2126
Notes
File Edit
"Test of record" PCV 3 operation under line pressure (nom 40 psig) with slurry @ approx 1.2kg/liter.
Tune 1 PCV vs. process variable - dead time = .15 sec., tau = .27 sec hysteresis = 2.51%. This record could be used to establish initial tuning constants for this loop.
Tune 2 PCV vs position transmitter - tau = .18 sec, hysteresis = .007%
Note tight coupling between flow rate and back pressure control.
100.0
75.0
50.0
25.0
0.0
Data (%)
1272.1
1314.6
1357.1
1399.6
1442.1
1484.6
Time (sec)
All Data
PCV 3 PD
PCV 3 POS
PT 3 PV
FI 2 PV
Start
Microsoft Word - ORNLCO...
Protuner System Anal...
FullShot 97
12:18 PM


Protuner System Analyzer - 03061430.PSA
File Analysis Notes Calculator Options Help
DATA PLOTS
Left 2436.10 Right 3528.60 Size 10926
Notes
File Edit
"File of record" PCV 1 with system at normal operating pressure. Nom. 40 psig. Slurry nom. 1.2kg/liter
Tune 1 record - PCV 1 input vs process pressure. Hysteresis = 1.42% tau = 0.0 sec. deadtime = .36 sec. (Tuning constants this record could be used as starting for actual closed loop control)
Tune 2 record - PCV 1 input vs position transmitter. Hysteresis = .71% tau = .33 sec.(These values to be used to assess valve condition over time)
Observation of the records will show poor valve operation up to approx 3000 sec. at which time coupling to ball actuator was remade.
NOTE !! Coriolis reading cannot be determined as to it's value and should be disregarded.
PT 3 this record should be PT 1 (Dwg's were incorrect at time of test)
57.8
47.1
36.5
25.8
15.1
Data (%)
2436.1
2654.6
2873.1
3091.6
3310.1
3528.6
Time (sec)
All Data
PCV 1 PD
PCV 1 POS
PT 3 PV
CORRIOLIS PV
Start
Microsoft Word - ORNLCO...
Protuner System Anal...
FullShot 97
12:07 PM

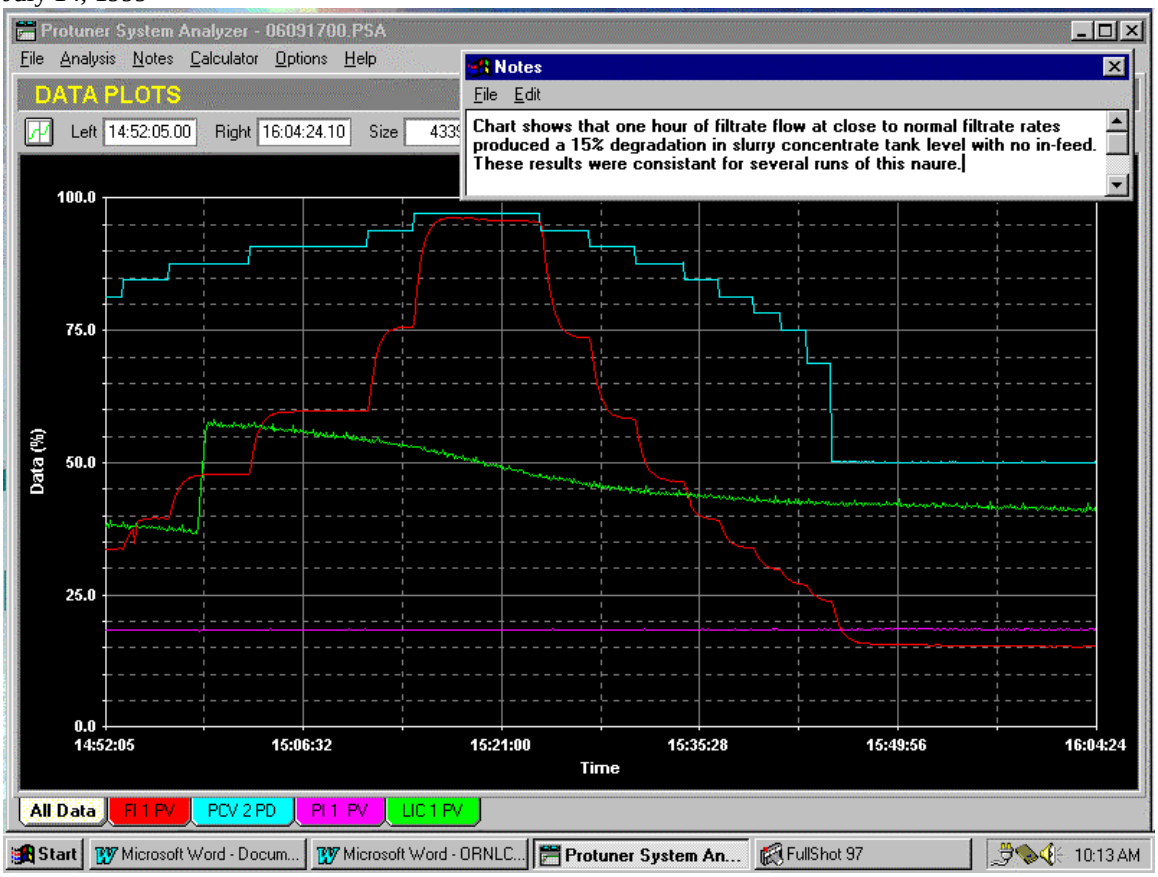


Couplings on Worcester Controls valves (PCV-1 and PCV-3) which had slack between the positioner/coupling and valve stem/coupling have been successfully repaired. PCV-2 required trim replacement has successfully been made.

All primary elements and transmitters in and of themselves have been tested and calibrated with water with the exception of flow indicators FI-3 and FI-4. These final two elements are currently being configured into the system and are expected to be tested by close of business 6/10/99.

Discussion

A dissertation was provided by Doug Lee describing how the Slurry Concentration Loop process needs to function in order for him to properly evaluate the chemistry. The Filtrate Flow rate must be closely maintained at a specified value that, at this point, is determined to be 35mL/M in the Slurry Concentration Loop. In order for this flow rate to be maintained, filtrate flow valve PCV-2 needs to be controlled based on the filtrate flow indicator FI-1. Filtrate flow control and Slurry Concentration Tank level cannot be mutually exclusive. The Slurry Concentration Tank has the capability of staying within boundaries for up to an hour without feed, conversely the tank can withstand an infeed without filtrate flow for up to an hour.

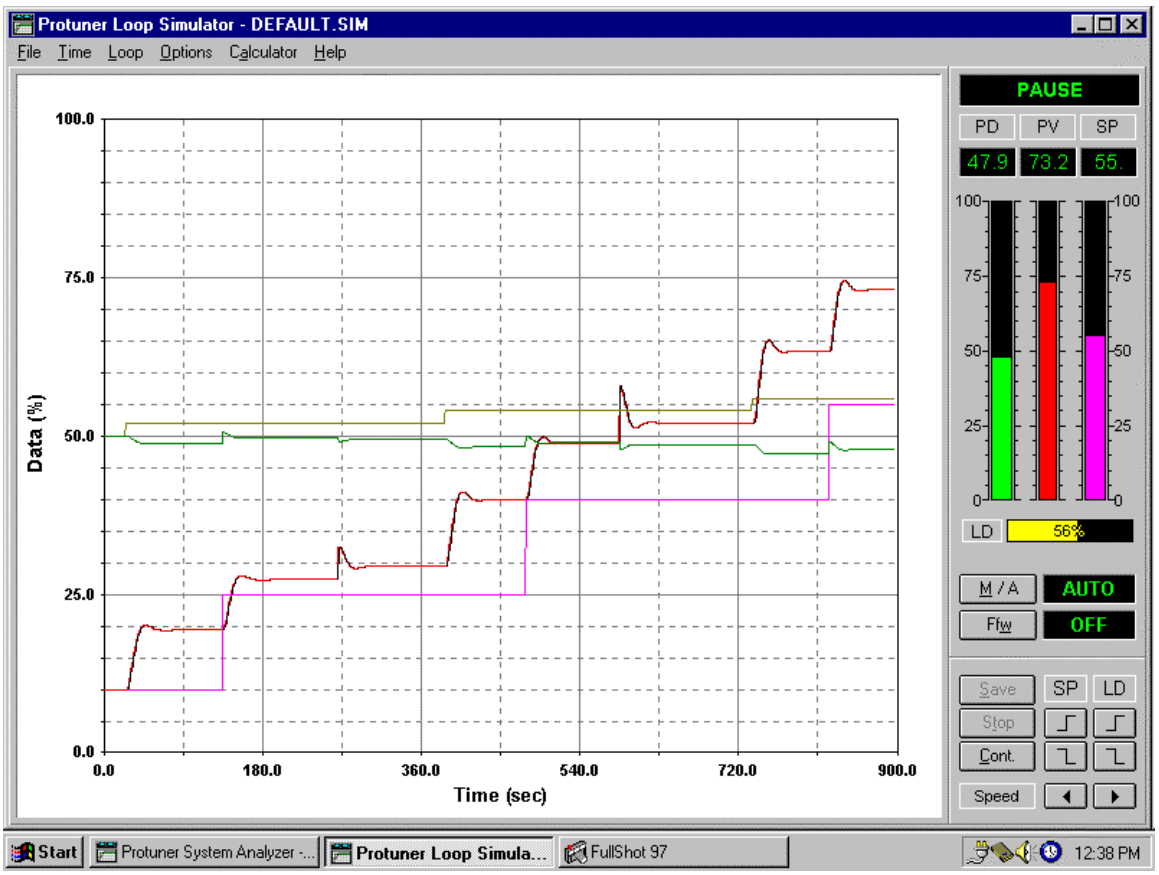


The feed pumps to the CSTRs will be manually operated by a technician. The feed rate to the CSTRs are a rate that must be regulated to satisfy test conditions. Also, the filtrate flow rate must be maintained as previously stated to satisfy test conditions which dictates that the slurry concentration tank be managed as a surge tank. Once the system is started and the appropriate filtrate flow rate has stabilized to satisfy test conditions, and the CSTR feed rate has been stabilized to satisfy test conditions, any deviation from mass balance at the slurry concentration tank will be adjusted according to test conditions at the discretion of the technician running the test. This could be either flow rate to maintain test integrity. Testing has shown that the technician has several hours to make these adjustments. It is our intent to add both alert and alarm limits to the slurry concentration tank and the slurry wash tank level indicators with the Intellution FIX control software.

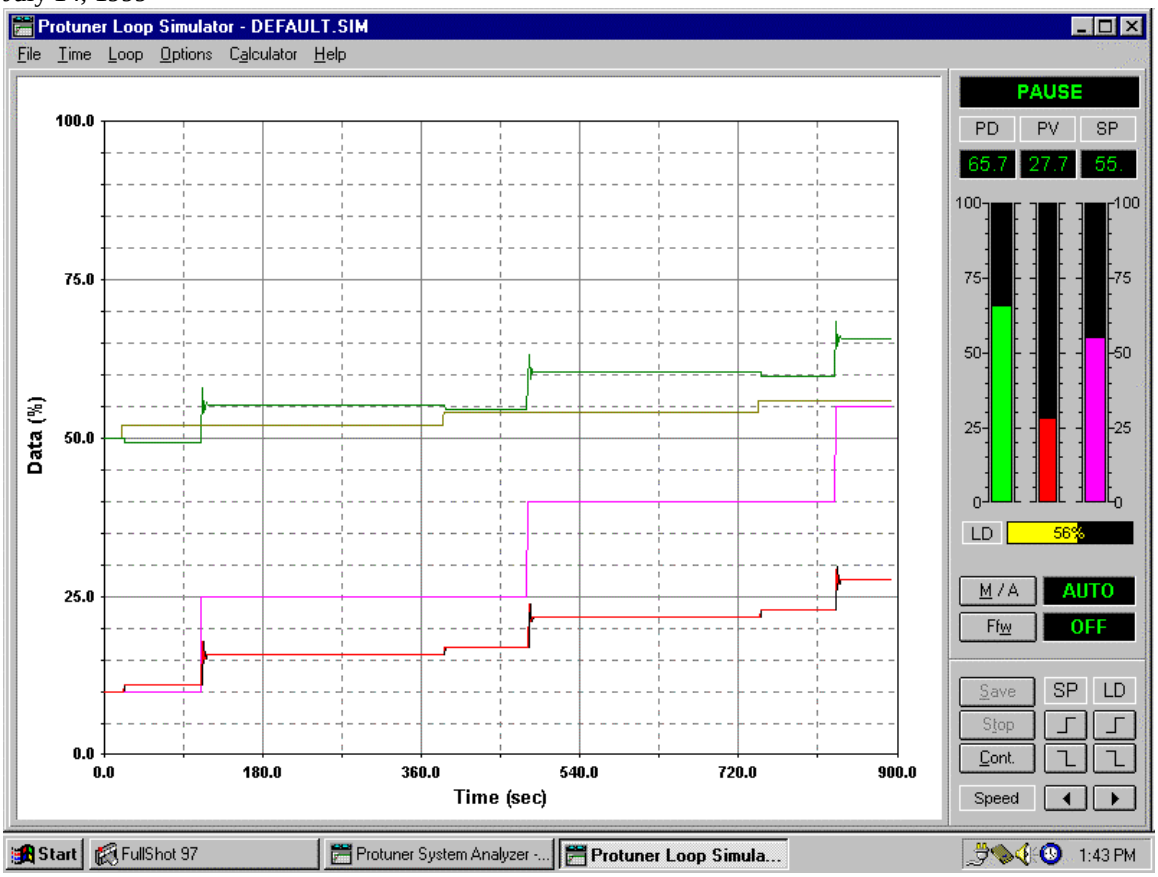
Attachment 4

Turn down estimates for controllers FIC 1 & PIC 1. See following simulation charts. In each case the record as shown depicts the controller operation for changes in the process of -25% of actual value (0-180 sec) the process at actual value (360 – 540 sec) and the process at +25% of actual value (720 – 900 sec). The first perturbation in each window is a load demand change, the second perturbation is a set point change.

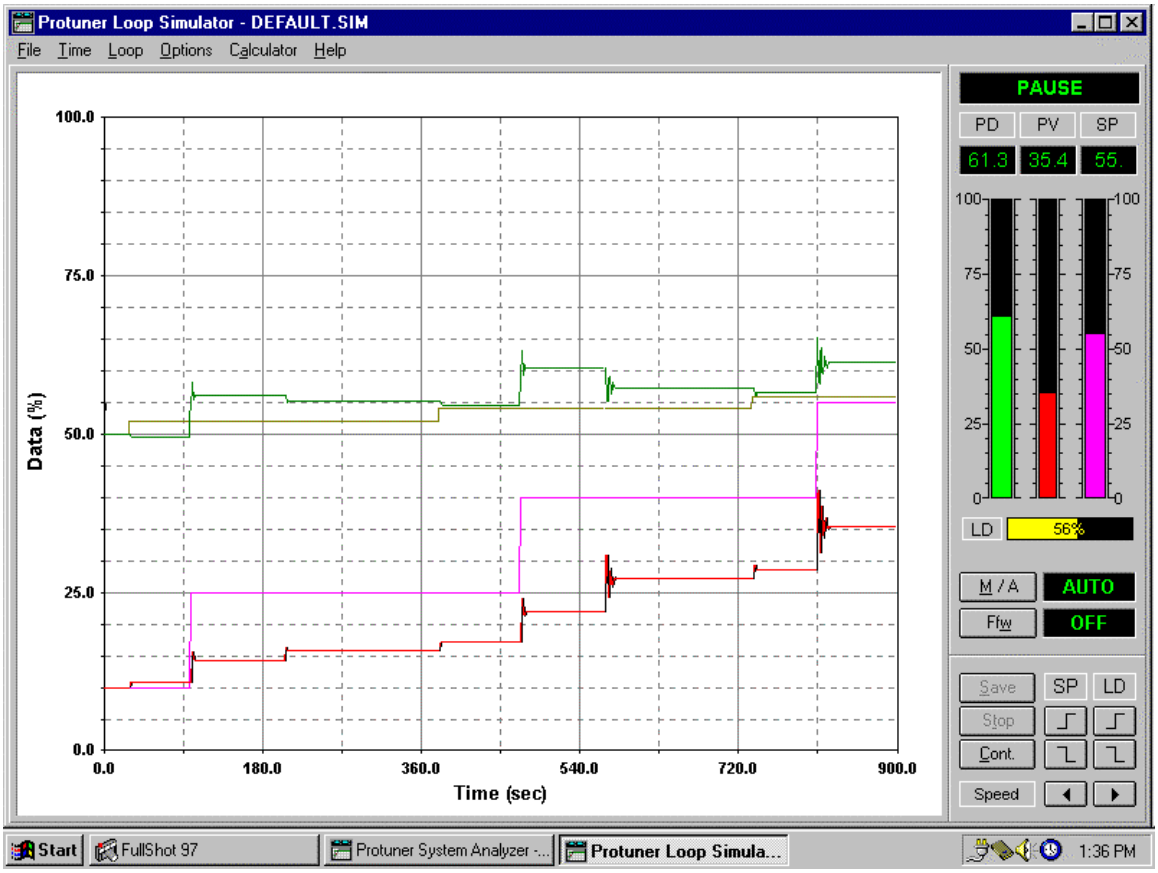
FIC 1 GAIN TURNDOWN



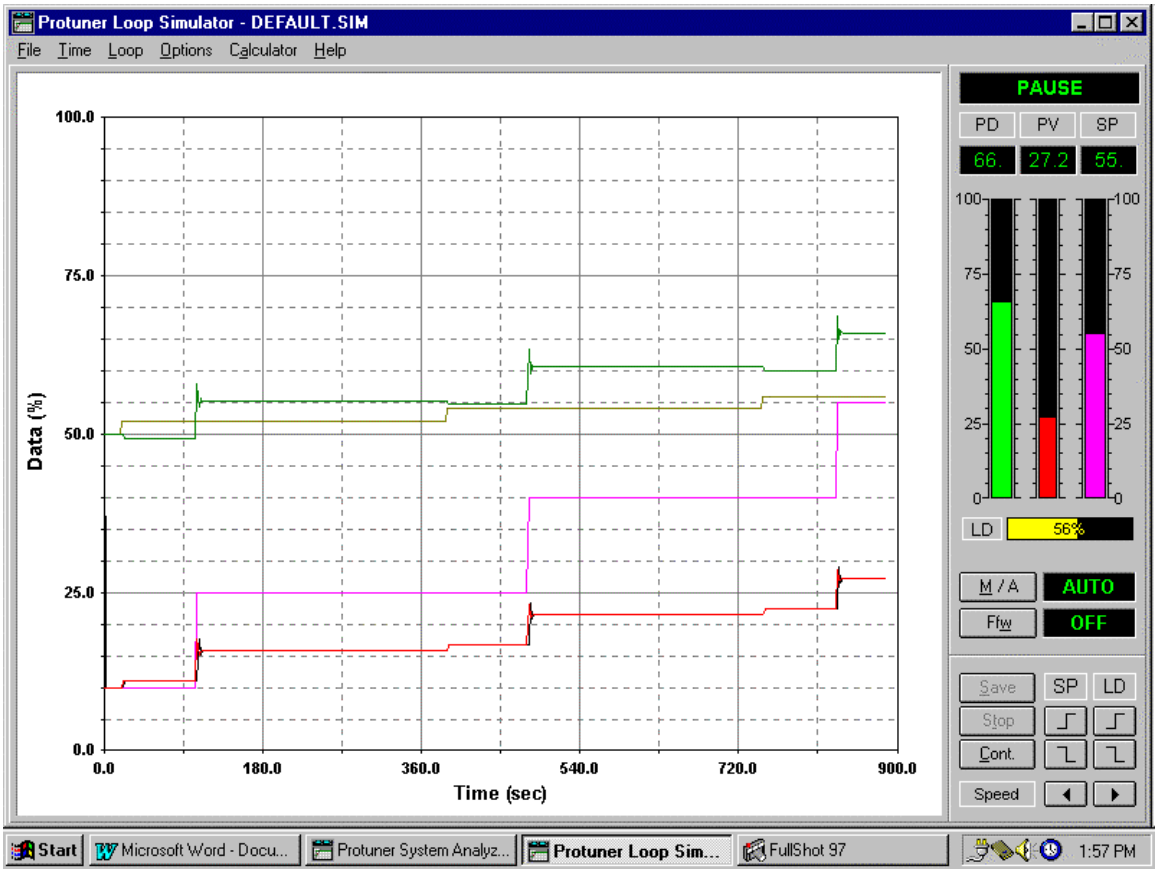
FIC 1 DEAD TIME AND TAU TURNDOWN


Protuner Loop Simulator - DEFAULT.SIM
File Time Loop Options Calculator Help
Data (%)
100.0
75.0
50.0
25.0
0.0
0.0
180.0
360.0
540.0
720.0
900.0
Time (sec)
PAUSE
PD PV SP
65.7 27.7 55.
LD 56%
M / A AUTO
Ffw OFF
Save SP LD
Stop
Cont.
Speed
Start
FullShot 97
Protuner System Analyzer -...
Protuner Loop Simula...
1:43 PM

PIC 1 GAIN TURNDOWN



PIC 1 DEADTIME AND TAU TURN DOWN

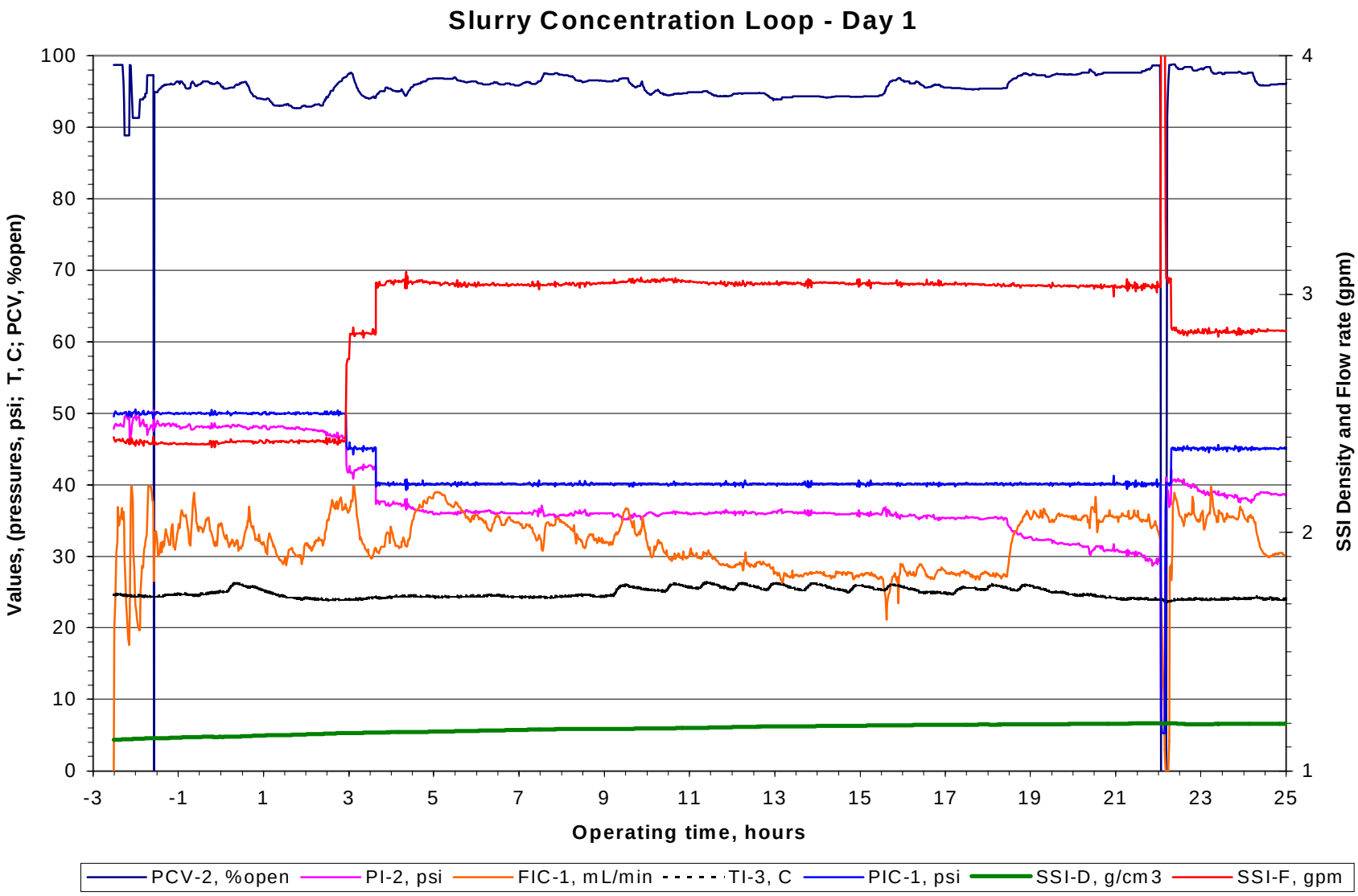


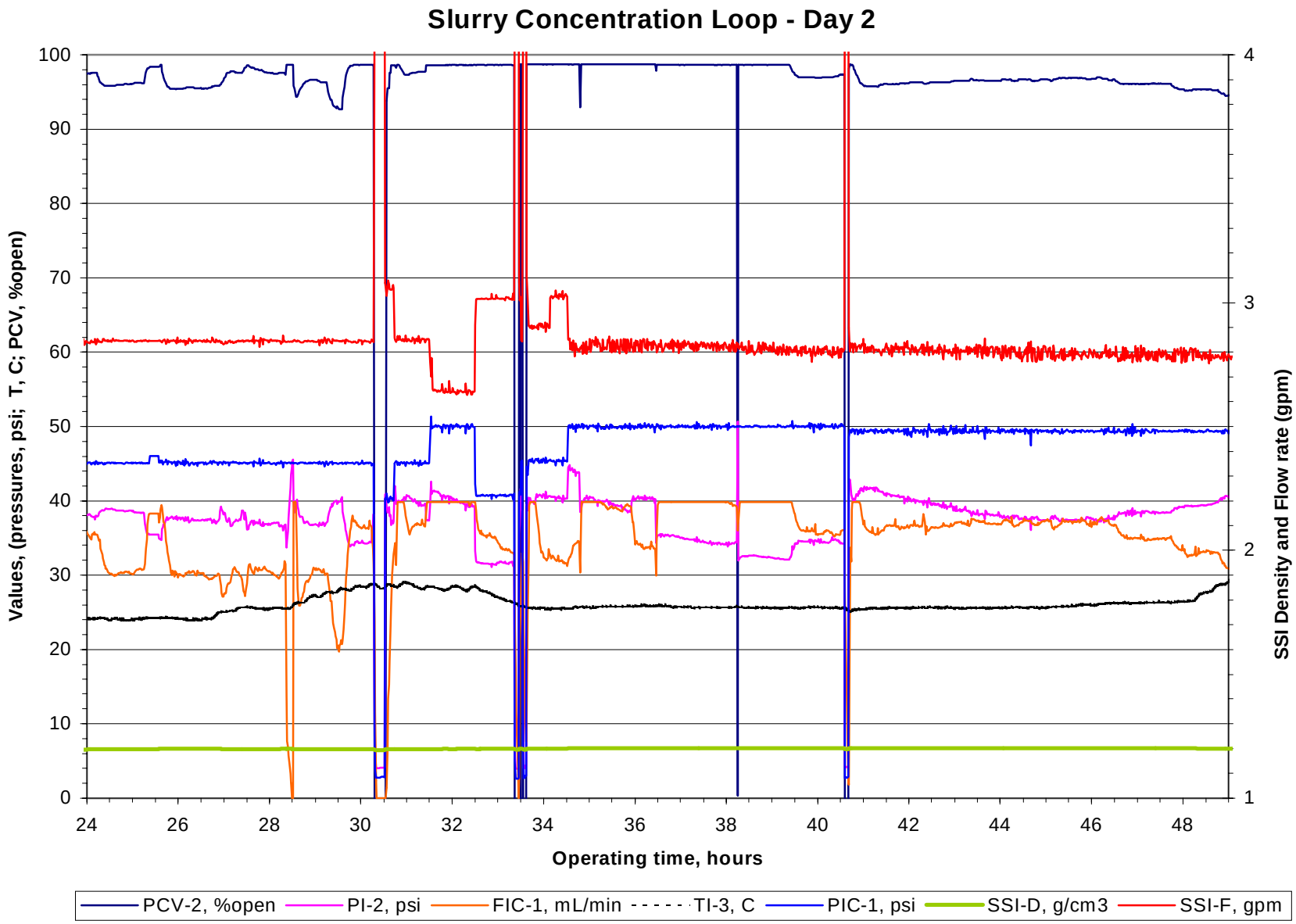
Appendix E

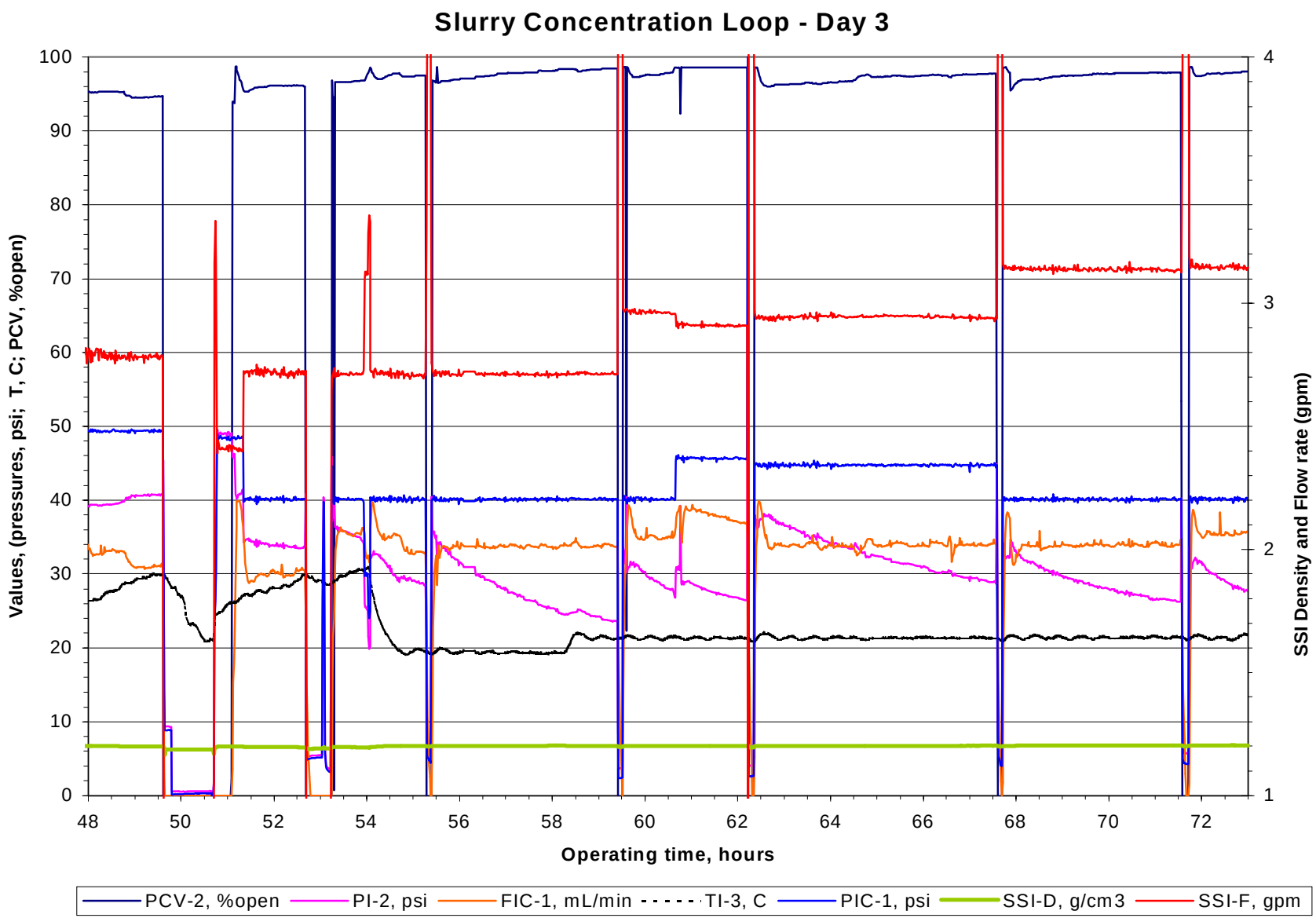
INTELLUTION DATA FOR TEST 2 FOR THE SLURRY CONCENTRATION LOOP

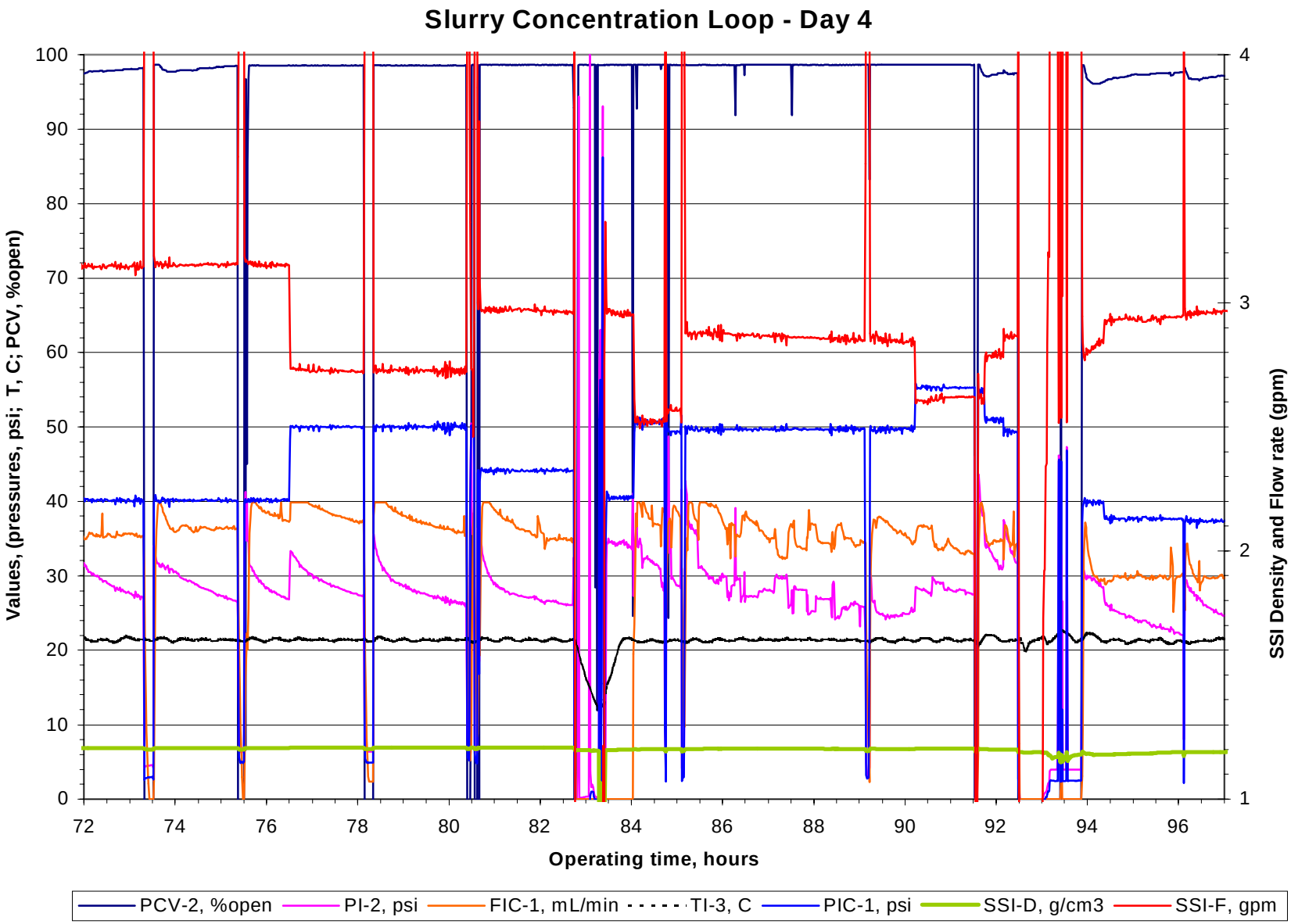
Appendix E Intellution Data for Test 2 for the Slurry Concentration Loop

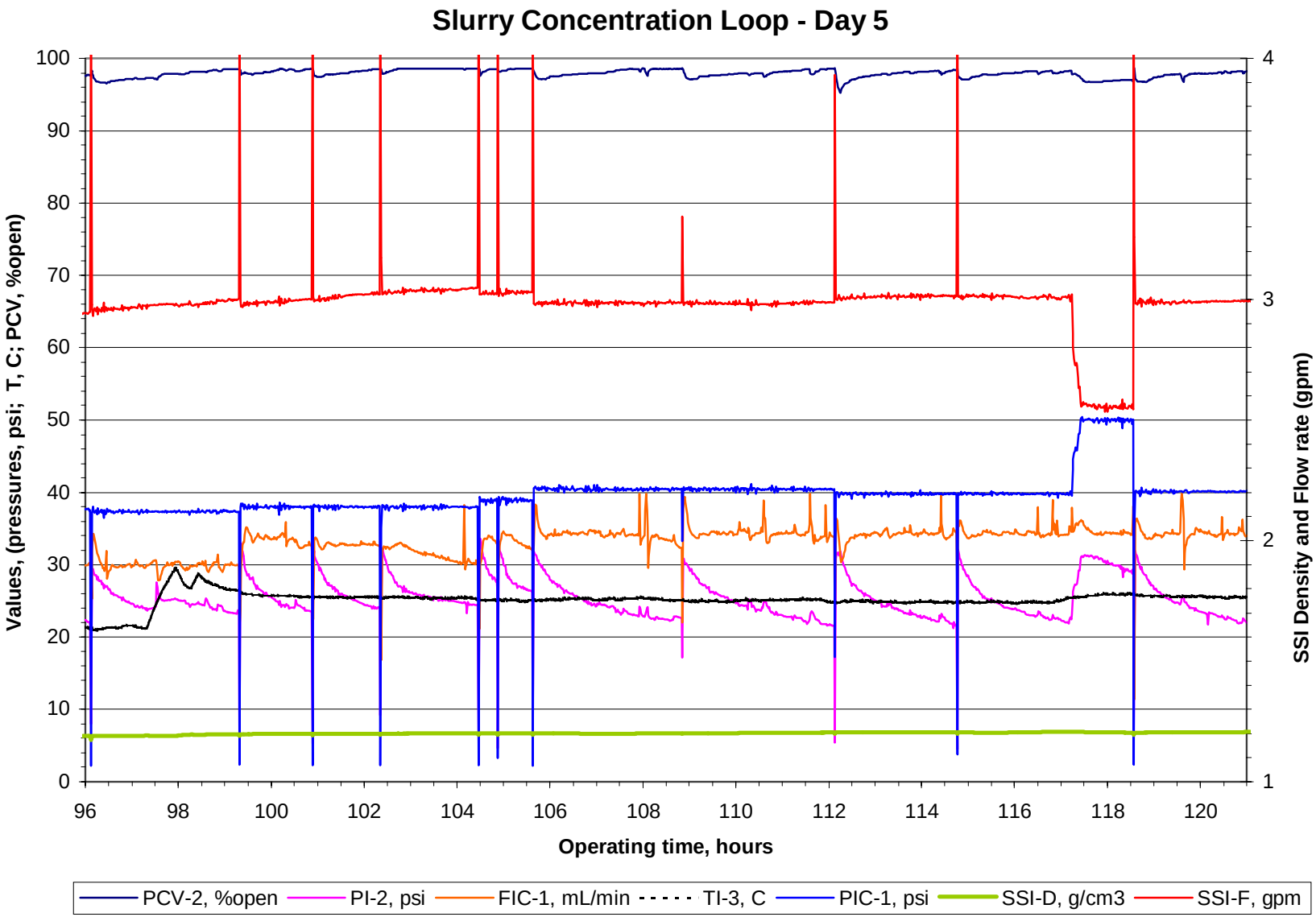
The following graphs show the day-to-day operation of the slurry concentration loop over the Test 2 operating period. It is apparent that the transmembrane pressure drop, PIC-1 – PI-2, increases during the course of operation. To keep the system operating, the back-pulsing of the filter became more and more frequent.

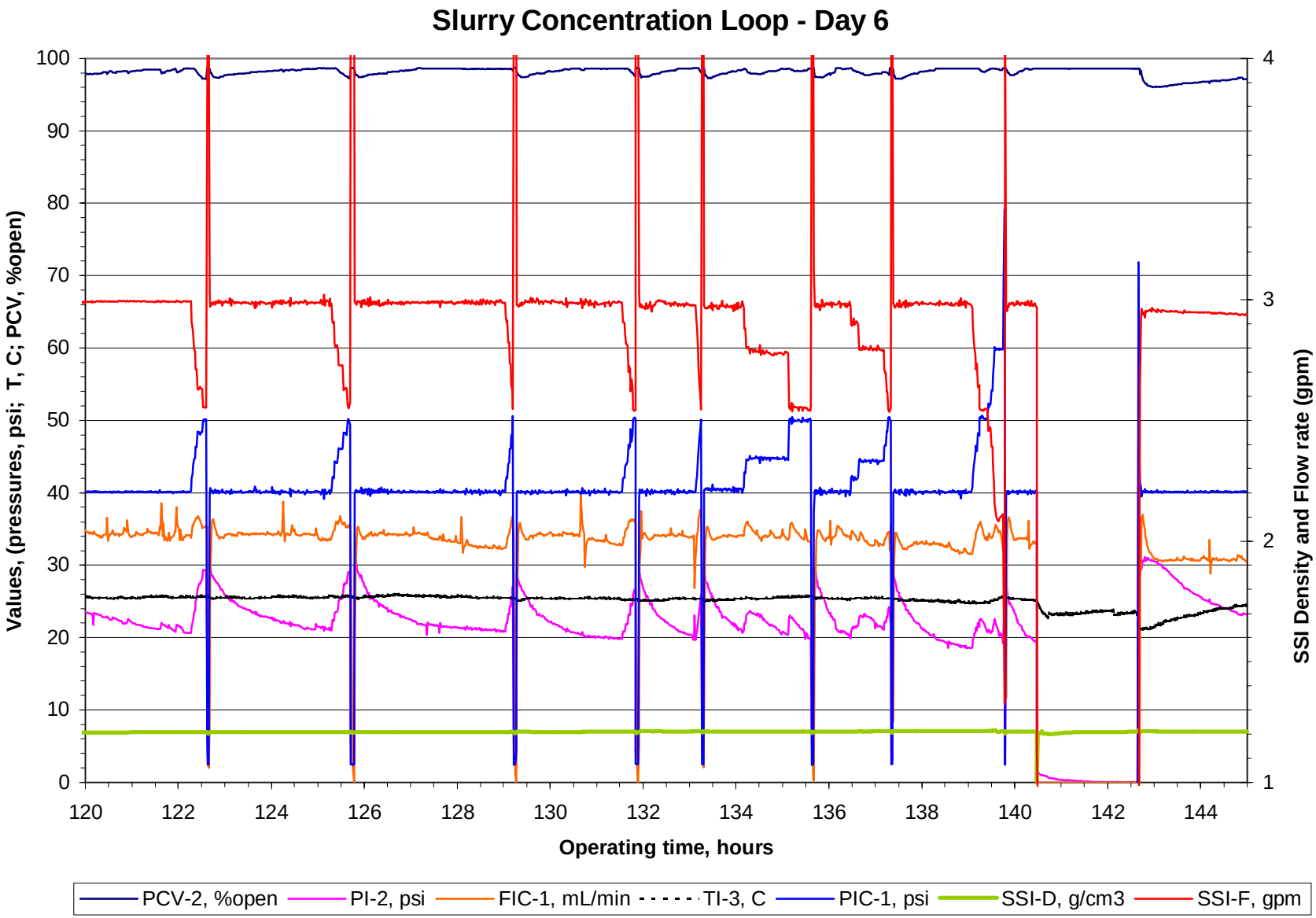

Slurry Concentration Loop - Day 1
Values, (pressures, psi; T, C; PCV, %open)
SSI Density and Flow rate (gpm)
Operating time, hours
100
90
80
70
60
50
40
30
20
10
0
4
3
2
1
-3
-1
1
3
5
7
9
11
13
15
17
19
21
23
25
PCV-2, %open
PI-2, psi
FIC-1, mL/min
TI-3, C
PIC-1, psi
SSI-D, g/cm3
SSI-F, gpm

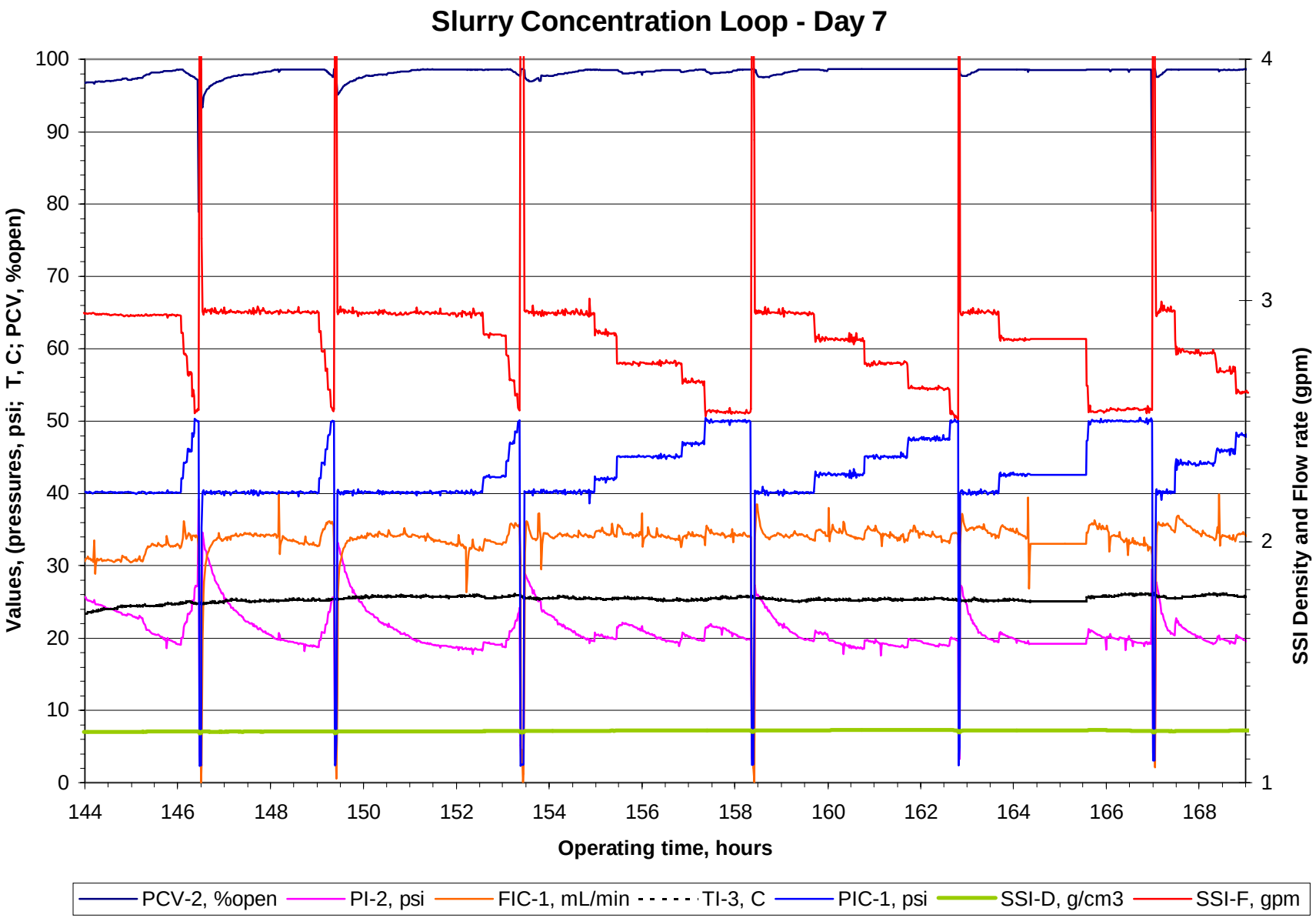

Slurry Concentration Loop - Day 2
Values, (pressures, psi; T, C; PCV, %open)
SSI Density and Flow rate (gpm)
Operating time, hours
0
10
20
30
40
50
60
70
80
90
100
1
2
3
4
24
26
28
30
32
34
36
38
40
42
44
46
48
PCV-2, %open
PI-2, psi
FIC-1, mL/min
TI-3, C
PIC-1, psi
SSI-D, g/cm3
SSI-F, gpm

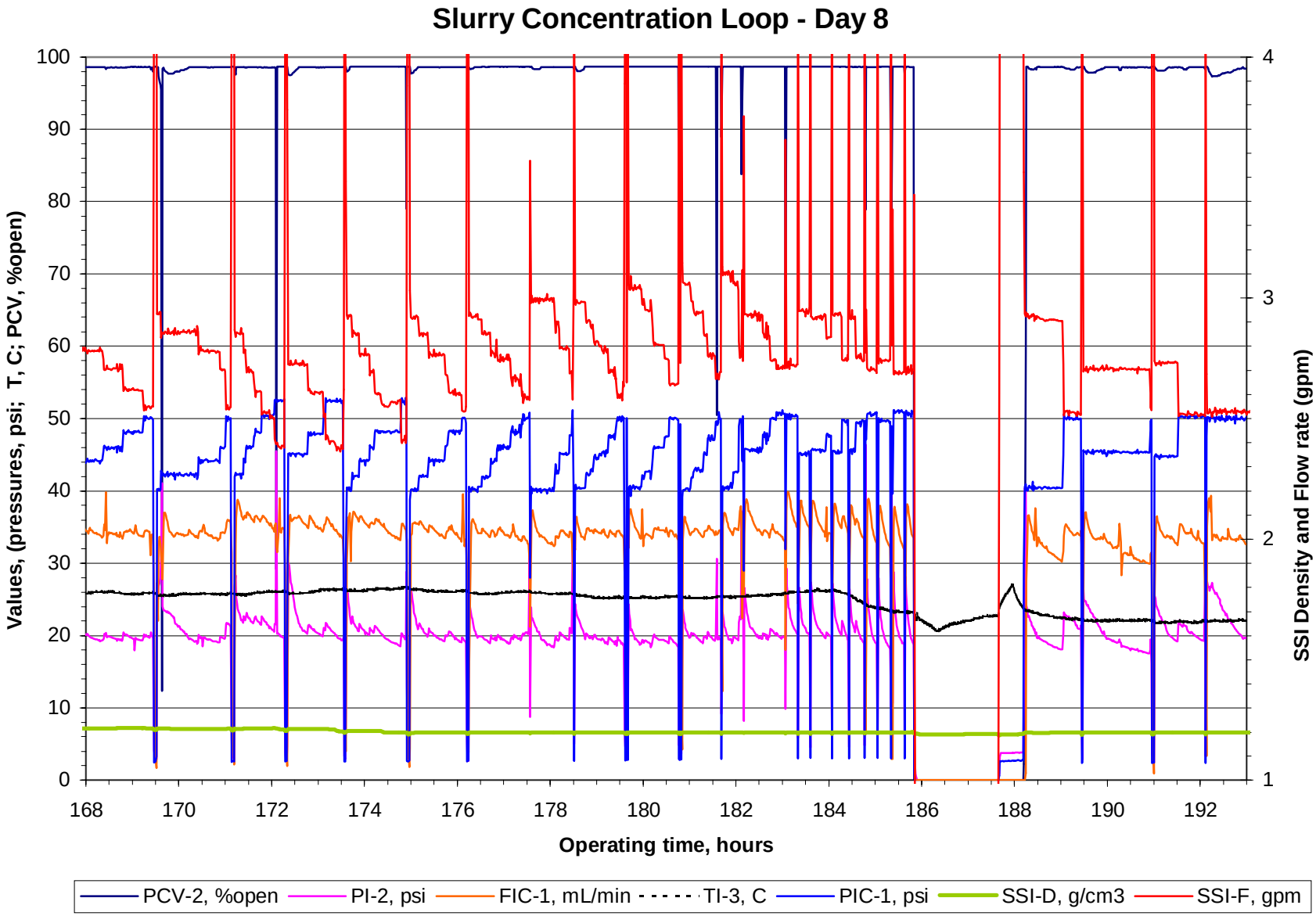

Slurry Concentration Loop - Day 3
Values, (pressures, psi; T, C; PCV, %open)
SSI Density and Flow rate (gpm)
Operating time, hours
0
10
20
30
40
50
60
70
80
90
100
1
2
3
4
48
50
52
54
56
58
60
62
64
66
68
70
72
PCV-2, %open
PI-2, psi
FIC-1, mL/min
TI-3, C
PIC-1, psi
SSI-D, g/cm3
SSI-F, gpm

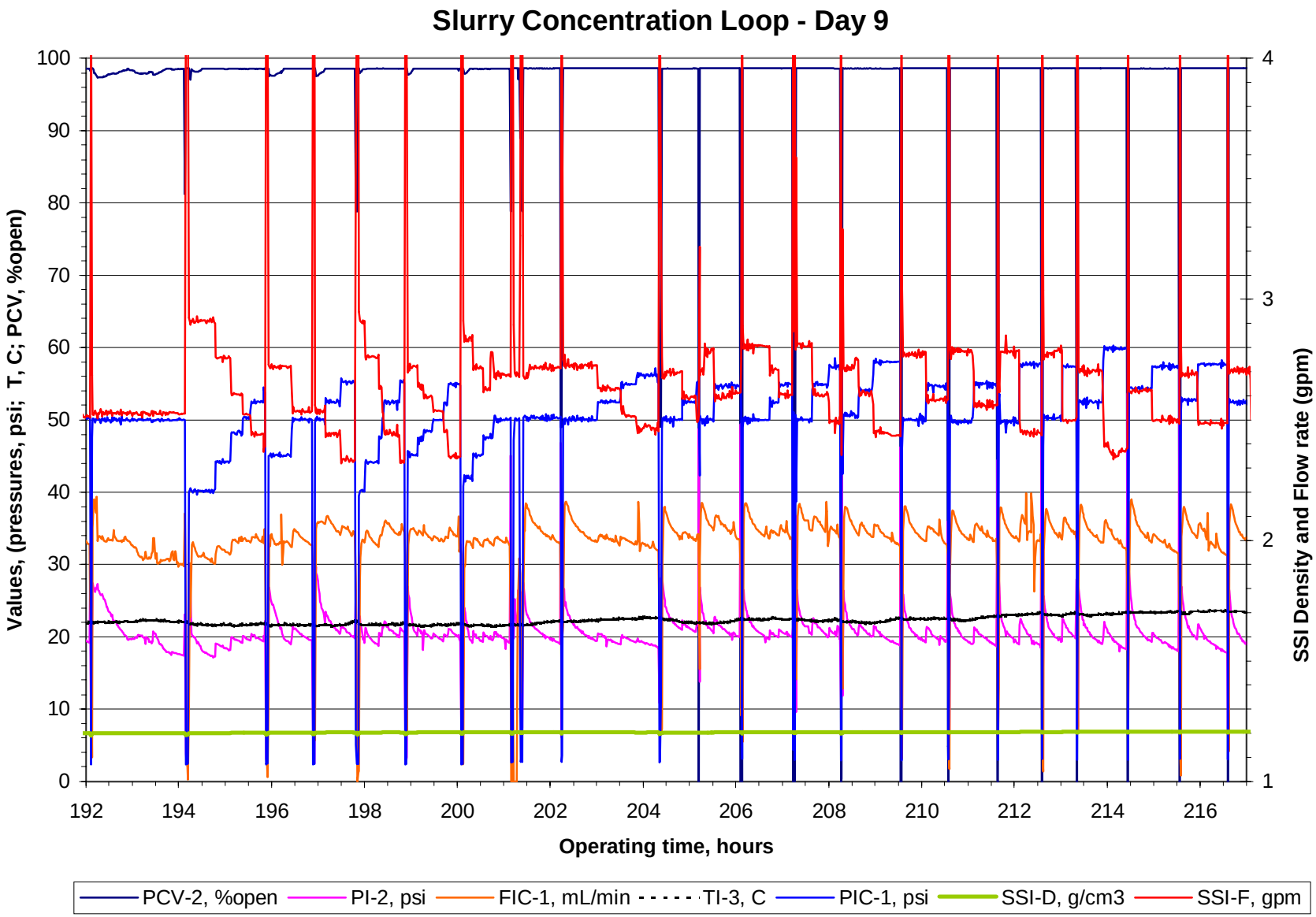

Slurry Concentration Loop - Day 4
Values, (pressures, psi; T, C; PCV, %open)
SSI Density and Flow rate (gpm)
Operating time, hours
0
10
20
30
40
50
60
70
80
90
100
1
2
3
4
72
74
76
78
80
82
84
86
88
90
92
94
96
PCV-2, %open
PI-2, psi
FIC-1, mL/min
TI-3, C
PIC-1, psi
SSI-D, g/cm3
SSI-F, gpm

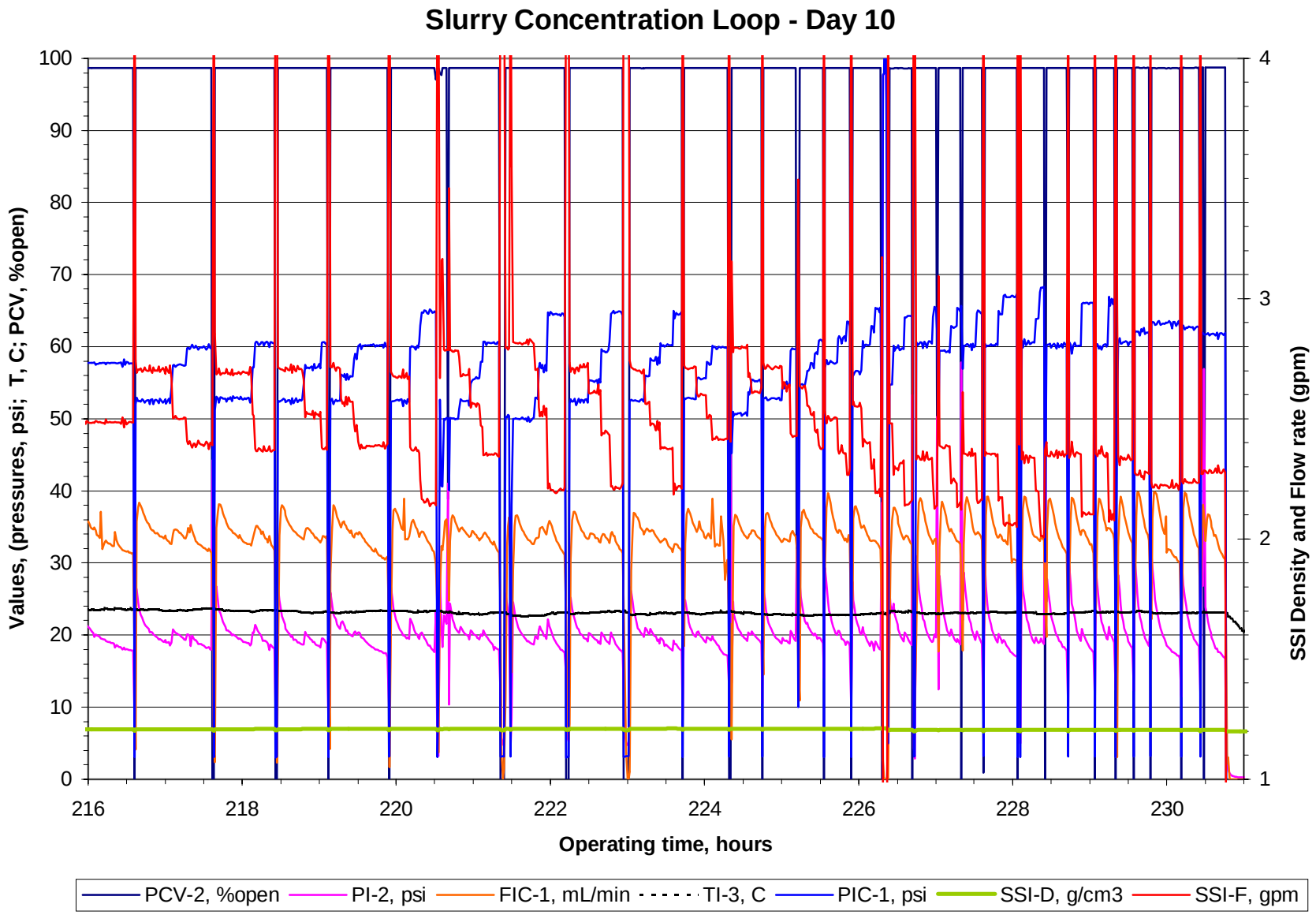

Slurry Concentration Loop - Day 5
Values, (pressures, psi; T, C; PCV, %open)
SSI Density and Flow rate (gpm)
Operating time, hours
0
10
20
30
40
50
60
70
80
90
100
1
2
3
4
96
98
100
102
104
106
108
110
112
114
116
118
120
PCV-2, %open
PI-2, psi
FIC-1, mL/min
TI-3, C
PIC-1, psi
SSI-D, g/cm3
SSI-F, gpm


Slurry Concentration Loop - Day 6
Values, (pressures, psi; T, C; PCV, %open)
SSI Density and Flow rate (gpm)
Operating time, hours
100
90
80
70
60
50
40
30
20
10
0
4
3
2
1
120
122
124
126
128
130
132
134
136
138
140
142
144
PCV-2, %open
PI-2, psi
FIC-1, mL/min
TI-3, C
PIC-1, psi
SSI-D, g/cm3
SSI-F, gpm


Slurry Concentration Loop - Day 7
Values, (pressures, psi; T, C; PCV, %open)
SSI Density and Flow rate (gpm)
Operating time, hours
100
90
80
70
60
50
40
30
20
10
0
4
3
2
1
144
146
148
150
152
154
156
158
160
162
164
166
168
PCV-2, %open
PI-2, psi
FIC-1, mL/min
TI-3, C
PIC-1, psi
SSI-D, g/cm3
SSI-F, gpm


Slurry Concentration Loop - Day 8
Values, (pressures, psi; T, C; PCV, %open)
SSI Density and Flow rate (gpm)
Operating time, hours
0
10
20
30
40
50
60
70
80
90
100
1
2
3
4
168
170
172
174
176
178
180
182
184
186
188
190
192
PCV-2, %open
PI-2, psi
FIC-1, mL/min
TI-3, C
PIC-1, psi
SSI-D, g/cm3
SSI-F, gpm


Slurry Concentration Loop - Day 9
Values, (pressures, psi; T, C; PCV, %open)
SSI Density and Flow rate (gpm)
Operating time, hours
0
10
20
30
40
50
60
70
80
90
100
1
2
3
4
192
194
196
198
200
202
204
206
208
210
212
214
216
PCV-2, %open
PI-2, psi
FIC-1, mL/min
TI-3, C
PIC-1, psi
SSI-D, g/cm3
SSI-F, gpm


Slurry Concentration Loop - Day 10
Values, (pressures, psi; T, C; PCV, %open)
SSI Density and Flow rate (gpm)
Operating time, hours
0
10
20
30
40
50
60
70
80
90
100
1
2
3
4
216
218
220
222
224
226
228
230
PCV-2, %open
PI-2, psi
FIC-1, mL/min
TI-3, C
PIC-1, psi
SSI-D, g/cm3
SSI-F, gpm

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