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Disclaimer:

Saltstone Third Quarter Calendar Year 2018 (3QCY18) Toxicity Characteristic Leaching Procedure (TCLP) Results

K. A. Hill

April 2019

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Savannah River National Laboratory™
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EXECUTIVE SUMMARY

The aqueous waste from Tank 50 (salt solution) is sampled quarterly for transfers to the Saltstone Production Facility (SPF). Salt solution is treated at SPF and disposed of in the Saltstone Disposal Facility (SDF). A SDF waste form (saltstone) was prepared in the Savannah River National Laboratory (SRNL) from the Tank 50 Waste Acceptance Criteria (WAC) sample[1] and Z-Area premix material for the third quarter of calendar year 2018 (3QCY18).[2,3] Results from this memorandum support Task 2: 'Grout Leaching Analyses' of the Task Technical Request (TTR)[3] prepared by Savannah River Remediation (SRR). After a 28 day cure, a sample of the SDF waste form was collected and shipped to a certified laboratory for analysis using the Toxicity Characteristic Leaching Procedure (TCLP).[4] The 3QCY18 saltstone sample met the South Carolina (SC) Code of Regulations for Hazardous Waste Management Regulations (HWMR) 61-79.261.24 and 61-79-268.48 requirements for a non-hazardous waste form with respect to Resource Conservation and Recovery Act (RCRA) metals and Underlying Hazardous Constituents (UHCs), and also met the SPF WAC.[5-7]

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LIST OF ABBREVIATIONS

D&S-FE	DWPF & Saltstone Facility Engineering
EC&ACP	Environmental Compliance & Area Completion Projects
EPA	Environmental Protection Agency
ES	Environmental Stewardship
ETF	Effluent Treatment Facility
LOD	Limit of Detection
LOQ	Limit of Quantitation
MRL	Minimum Reporting Limit
MS	Matrix Spike
MSD	Matrix Spike Duplicate
NRC	Nuclear Regulatory Commission
RCRA	Resource Conservation and Recovery Act
RL	Reporting Limit
SDF	Saltstone Disposal Facility
SPF	Saltstone Production Facility
SRNL	Savannah River National Laboratory
SRNS	Savannah River Nuclear Solutions
SRR	Savannah River Remediation
SwRI®	Southwest Research Institute
TCLP	Toxicity Characteristic Leaching Procedure
TTQAP	Task Technical and Quality Assurance Plan
TTR	Technical Task Request
UHC	Underlying Hazardous Constituents
WAC	Waste Acceptance Criteria

1.0 Introduction

The SPF receives waste from Tank 50 for treatment. The following dates were selected starting from the last quarterly sampling date to the current quarterly sampling date. Tank 50 accepted the following transfers from July 12 (when it was 51% full) to September 20, 2018 (when it was 49% full):[8] During this same time period there was a total of 65.4 kgal of Tank 50 material transferred out to Z Area.

- ~4.1 kgal from 211-H
- ~1.3 kgal from Effluent Treatment Facility (ETF)
- ~0.9 kgal from other
- ~52.0 kgal from the Actinide Removal Process / Modular Caustic Side Solvent Extraction Unit (ARP/MCU) Decontaminated Salt Solution Hold Tank (DSSHT)

On September 20, 2018, a salt solution sample was taken from Tank 50[1] and used to prepare a SDF waste form sample, referred to as a saltstone sample.[9] The 3QCY18 saltstone sample was prepared on October 29, 2018.[9] Once the 3QCY18 saltstone sample cured for 28 days, it was crushed, sieved, packaged, and deemed "collected".[10] The sample was then shipped to Southwest Research Institute (SwRI®) to analyze for toxicity per the TCLP method.[2,4] This saltstone sample determines whether the non-hazardous nature of the grout meets the requirements of the SC Code of Regulations 61-79.261.24[6] for RCRA metals and 61-79.268.48[5] for inorganic/organic UHCs (for informational purposes only[3]).

2.0 Experimental

Saltstone preparation was performed at SRNL. DWPF & Saltstone Facility Engineering (D&S-FE) provided SRNL with the saltstone grout recipe as well as the premix components.[3,9] Table 2-1 shows all premix components obtained to date for CY2018 samples with specific LOT numbers.[9]

Table 2-1. Premix Components for CY2018

Premix Component	Date SRNL Received	LOT #
Holcim Cement 3QCY18	10/22/2018	2018-IR-05-1644
Holcim Cement 2QCY18	8/15/2018	2018-IR-05-1545
Holcim Cement 1QCY18	3/27/2018	2018-IR-05-0371
LeHigh Slag 3QCY18	10/22/2018	2018-IR-05-1301
LeHigh Slag 2QCY18	8/15/2018	2018-IR-05-1299
LeHigh Slag 1QCY18	3/27/2018	2018-IR-05-0366
SE Fly Ash 3QCY18	10/22/2018	2018-IR-05-1297
SE Fly Ash 2QCY18	8/15/2018	2018-IR-05-0807
SE Fly Ashl 1QCY18	3/27/2018	2018-IR-05-0546

The saltstone sample was prepared using the mixing method outlined in SRNL Environmental Stewardship (ES) work instructions and the 3Q premix components in Table 2-1.[11] The sample was left to cure in a Ziploc® sealed plastic bag for at least 28 days. After curing, the sample was crushed and sieved using the method outlined in ES work instructions.[12] Material that passed through the 3/8-inch sieve was subsequently screened through a No. 4 sieve (4.76 mm). The material retained on the No. 4 sieve was packaged in a primary container (250 mL High Density Polyethylene (HDPE) bottle) and shipped on the same day that it was prepared to SwRI® by Environmental Compliance & Area Completion Projects (EC&ACP).[3]

3.0 Results

Table 3-1 summarizes the analytical results provided by the vendor, SwRI.[13] The first eight rows show data for the RCRA metals and the next four rows show data for the UHC metals from the TCLP leachates. The last four rows show results from solids analyses of the saltstone for benzene, phenol, total and amenable cyanide. The entire vendor report is documented and included as a reference.[13] For comparison, the previous quarter and four quarter average results are shown. The Regulatory Toxicity[6] values and the WAC Limits are from Table 6 of the WAC[7] and reflect the requirements in the applicable version of the document. Note that the vendor used a "modified" Method 1311 where sample mass was restricted due to the elevated activity of the sample.[13] This methodology is consistent with the joint guidance from the Nuclear Regulatory Commission (NRC) and Environmental Protection Agency (EPA) for mixed radioactive and hazardous waste.[14] The reported detection limits for both As and Se have increased relative to the past four quarters. The reported detection limit of <0.05 mg/L for As is 2.5X higher than all four previous quarters that reported an As detection limit of <0.02 mg/L. The reported detection limit of <0.10 mg/L for Se is 4X higher than the previous 3Q17 detection limit for Se of <0.025 mg/L. Also, in past quarters detectable values of Se have been reported at levels below the <0.1 mg/L such as 2Q17 at 0.0258 mg/L, 1Q18 at

0.0417 mg/L and 2Q18 at 0.052 mg/L. No indications were given in the SwRI® report[13] pertaining to any change in materials of testing or analytical methods that could account for these observations of increased detection limits for both As and Se. One explanation of the increased reported detection limits for As and Se for this quarter is that both analyses were determined from diluted samples whereas in the previous four quarters no indication was made that analyses for these two elements used diluted solutions.

The reported value for Pb of 0.0102 mg/L is above the minimum detection limit, but lower than the quantification limit. Lead values in the previous four quarters have all been less than detectable at levels in the range of <0.005 mg/L to <0.0075 mg/L. The analyzed value for Be of 0.017 mg/L is about 3X higher the previous quarters including three values of <0.005 mg/L and a detectable value of 0.00539 mg/L in the 1Q18 sample. There was no appreciable change going from the 2Q18[15] to 3Q18[1] Tank 50 supernate concentrations that could explain the increases in Pb and Be for this 3Q18 regulatory sample, assuming that the premix additives for grout production were similar in the two quarters. The Tank 50 average supernate values for Pb and Be for the 2Q18 and 3Q18 Tank 50 supernates were reported as Pb = 0.411 mg/L ± 0.0225 mg/L and Be as < 0.0989 mg/L, and Pb = 0.544 mg/l ± 0.302 mg/L and Be as <0.0194 mg/L, respectively. The ± values indicated are the one-sigma uncertainties calculated from triplicate analyses of the Tank 50 supernate. These uncertainties do not factor in any method/instrument uncertainties. Hence, the Pb supernate concentration values agree within the 95% confidence interval for the two measurements. One can estimate a maximum level of components expected in the TCLP leachate if one were to assume no retention in the waste form and use the solid mass to leachant mass of 1:20 in the TCLP. Using the supernate concentrations for Be of <0.0989 mg/L and <0.0194 mg/L and taking no credit for dilution of the salt solution into grout and assuming the premix additive to not contribute any Be to the matrix, the calculated maximum values for Be in the TCLP leachate are <5.00E-3 mg/L for the 2QCY18 sample and <1.00E-03 mg/L for the 3QCY18 sample. Thus, this calculation indicates that the reported TCLP leachate concentration for the 2QCY18 sample could be within the possible expected value, but the reported Be concentration in the 3QCY18 sample of 1.7E-02 mg/L is an order of magnitude greater than the maximum amount calculated of < 1.00E-3 mg/L. This calculation and comparison suggest either that the premix additives from the 3QCY18 contained a source of leachable Be or that the reported TCLP Be value is high biased. The reported value for Cr from a diluted sample analysis of 0.26 mg/L is ~ 5X higher than the previous 2QCY18 TCLP value of 0.0477 mg/L[15] and is ~ 8X higher than the previous four-quarter average of 0.0326 mg/L. This increase in Cr, which is known to be a redox sensitive cation, cannot be attributed to any changes in grout processing, as the cured grout is always stored in Ziploc® sealed plastic bags until preparation for shipment, nor to any significant increase in the Tank 50 supernate Cr concentration as the 3QCY18 Tank 50 average supernate Cr concentration is 54.6 mg/L ± 2.57 mg/L compared to the prior 2QCY18 Tank 50 average supernate Cr value of 51.2 mg/L ± 0.5 mg/L. The ± values indicated are the one-sigma uncertainties calculated from triplicate analyses of the Tank 50 supernate. These uncertainties do not factor in any method/instrument uncertainties.

Mercury TCLP values for this 3Q18 sample are similar to the past two quarters (2Q18 and 1Q18) in the range of 0.058 to 0.059 mg/L. Prior to the 2018 samples, the 2Q17 and 3Q17 Hg TCLP values were in the lower range of 0.0134 mg/L and 0.0051 mg/L, respectively. There was no data collected for the 4Q17 sample. Mercury speciation analyses for recent past 2Q17 through 3Q18 shows that the total mercury levels in the Tank 50 supernate have ranged from a low of 69.8 mg/L for 2Q18 to a high of 81.4 mg/L for the 3Q17 sample as shown in Table 3-2.[16] The corresponding methyl Hg values expressed as mg Hg/L ranged from 28.2 mg/L to 36.6 mg/L. The methyl Hg species is the dominant Hg species in the Tank 50 supernate (relative to other Hg species measured like elemental Hg(0) or ionic Hg(I) and/or Hg(II)) with methyl Hg to total Hg ratios shown in Table 3-2 that are in the range of 0.346 to 0.510.

Table 3-3 provides comparison between analytical results for each analyte to SwRI®'s Limit of Detection (LOD) and Limit of Quantitation (LOQ) for the TCLP leachates and to the Reporting Limits (RL) for the

solids analyses. Antimony, arsenic, cadmium, selenium, silver, thallium, benzene, amenable cyanide and phenol were all less than the detection limit or reporting limit. Appendix A includes summaries of results from blanks, laboratory control samples, matrix spikes, and matrix spike duplicates.

Table 3-1. 3QCY18 Saltstone Sample TCLP and Solids Analysis Results

Analyte	Result[13] (mg/L)	Regulatory Toxicity[6] (mg/L)	WAC Limit[7] (mg/L)	Results	
				Previous Quarter[17] (mg/L)	Previous Four Quarter Average[17-20] (mg/L)
RCRA Metals					
Arsenic (As)	<0.050[UD]	5.0	2.5	<0.020[U]	0.0200^
Barium (Ba)	1.84[D]	100.0	50	1.69[D]	1.16
Cadmium (Cd)	<0.005[U]	1.0	0.5	<0.005[U]	0.005^
Chromium (Cr)	0.26[D]	5.0	2.5	0.0477	0.0326*
Lead (Pb)	0.0102[B]	5.0	2.5	<0.0075[U]	0.00625[+]
Mercury (Hg)	0.0583	0.2	0.1	0.0588	0.0341
Selenium (Se)	<0.1[UD]	1.0	0.5	0.0521	0.0362*
Silver (Ag)	<0.010[U]	5.0	2.5	<0.010[U]	0.01[^]
Underlying Hazardous Constituents (UHCs)					
Antimony (Sb)	<0.025[U]	-	-	<0.025[U]	0.0225[+]
Beryllium (Be)	0.017	-	-	<0.005[U]	0.005*****
Nickel (Ni)	0.0717[D]	-	-	0.0568	0.0331*
Thallium (Tl)	<0.005[UD]	-	-	<0.005[UD]	0.005^
Select Solids Analyses of Regulatory Interest					
	(mg/kg)			(mg/kg)	(mg/kg)
Benzene	<0.00089[U]	-	-	<0.00095[U]	0.00094[+]
Amenable Cyanide	<0.232[UJ]	-	-	0.3[J]	2.47*
Total Cyanide	11.1	-	-	11.0[J]	10.7
Phenol	<0.771[UJ]	-	-	<0.876[UJ]	1.23*

-Indicates a location in the table for which an entry would not be appropriate.

[U] Non-detected analyte
[D] Results reported from a dilution.
[J] Matrix spike and/or matrix spike duplicate criteria was not met.
[B] Analyte was detected at the instrument at or above Limits of Detection (LOD), but less than Limit of Quantitation (LOQ).
* Contains qualifier of "U" in at least one quarter.
[+] Contains qualifier of "U" in all quarters with multiple Reporting Limits (RL) or Limits of Detection (LOD).
^ Contains qualifier of "U" in all quarters with same RL or LOD.

Table 3-2. Mercury Speciation Data from Past Tank 50 Salt Solutions[16]

Tank 50 Sample	Total Hg (mg/L)	Methyl Hg (mg/L)	Ratio Methyl Hg/Total Hg
2QCY17	72.2	32.2	0.446
3QCY17	81.4	28.2	0.346
1QCY18	71.8	36.6	0.510
2QCY18	69.8	28.5	0.408
3QCY18	70.4	30.7	0.436

Table 3-3. RCRA Metal TCLP Result Concentrations, Limit of Detection, and Limit of Quantitation[13]

Analyte	Methods	LOD	LOQ	Sample Results	Qualifiers
		(µg/L)	(µg/L)	(µg/L)	
Antimony (Sb)	6010D	25.0	50.0	<25.0	U
Arsenic (As)	6010D	50.0	100	<50.0	UD
Barium (Ba)	6010D	50.0	100	1840	D
Beryllium (Be)	6010D	5.00	10.0	17.0	-
Cadmium (Cd)	6010D	5.00	10.0	<5.00	U
Chromium (Cr)	6010D	50.0	100	260	D
Lead (Pb)	6010D	7.50	15.0	10.2	B
Mercury (Hg)	7470A	1.00	2.00	58.3	-
Nickel (Ni)	6010D	10.0	20.0	71.7	D
Selenium (Se)	6010D	100	200	<100	UD
Silver (Ag)	6010D	10.0	20.0	<10.0	U
Thallium (Tl)	6020B	5.00	10.0	<5.00	UD
-	-	-	**RL (mg/kg)**	**(mg/kg)**	-
Benzene	8260C	-	-	<0.00089	U
Amenable Cyanide	Amenable cyanide 9012B	-	0.232	<0.232	UJ
Total Cyanide	Cyanide 9012B	-	0.232	11.1	-
Phenol	Phenol 9065	-	0.771	<0.771	UJ

- Indicates a location in the table for which an entry would not be appropriate.
[U] Result is less than the Limit of Detection (LOD) and/or Reporting Limit (RL).
[D] Result is reported from a dilution.
[J] Matrix spike and/or matrix spike duplicate criteria was not met.
[B] Analyte was detected at the instrument at or above Limits of Detection (LOD), but less than Limit of Quantitation (LOQ).

4.0 Conclusions

Analyses of the SDF waste form prepared from the 3QCY18 Tank 50 salt solution sample and premix material resulted in the following findings.

- The RCRA metal TCLP result concentrations met the SC Code of Regulations 61-79.261.24 requirements for a nonhazardous waste form.[6]

- The measured concentrations of the TCLP RCRA metals and additional inorganic/organic UHCs met the SC Code of Regulations 61-79.268.48 non-wastewater standards.[5]

- The measured concentrations of the TCLP RCRA metals met the SPF WAC.[7]

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Appendix A. Quality Assurance

The following subsections include summaries of results from blanks, laboratory control samples, matrix spikes, and matrix spike duplicates. The data package also includes data for calibration verifications, interference checks and serial dilutions.[13]

Table A- 1 shows all TCLP extraction fluid blank concentrations and the solid matrix blank concentrations. In the solid matrix blank, total cyanide was present at levels above the reporting limit of 0.232 mg/kg. Antimony, arsenic, barium, beryllium, cadmium, chromium, lead, mercury, nickel, selenium, silver, thallium, benzene, amenable cyanide, and phenol were all less than detection limit or reporting limit.

Table A- 1. TCLP Extraction Fluid Blank and Solid Matrix Blank[13]

Analyte	TCLP Blank (µg/L)	Qualifiers
Antimony (Sb)	<25.0	U
Arsenic (As)	<25.0	U
Barium (Ba)	<5.0	U
Beryllium (Be)	<5.00	U
Cadmium (Cd)	<5.00	U
Chromium (Cr)	<5.00	U
Lead (Pb)	<7.50	U
Mercury (Hg)	<0.100	U
Nickel (Ni)	<5.00	U
Selenium (Se)	<50.0	U
Silver (Ag)	<10.0	U
Thallium (Tl)	<5.00	UD
Analyte	Solid Matrix Blank (mg/Kg)	Qualifiers
Benzene	<0.00050	U
Amenable Cyanide	<0.239	U
Total Cyanide	0.751	-
Phenol	<0.983	U

[U] Result is less than the Limit of Detection (LOD) and/or Reporting Limit (RL).
[D] Result is reported from a dilution.
- Indicates a location in the table for which an entry would not be appropriate.

Table A- 2 shows all LCS recoveries meet SwRI®'s acceptance limit in the range of 80% to 120% for metals and phenol, 70% to 130% for benzene and 105.2% for total cyanide, which was within the manufacturers acceptance limit.[13] The laboratory control samples are clean aqueous solutions analyzed to assure integrity of the analytical technique exclusive of matrix effects.

Table A- 2 Laboratory Control Sample[13]

Analyte	**Laboratory Control (µg/L)**		**Recovery (%)**
	True	**Found**	
Antimony (Sb)	500	448	89.6%
Arsenic (As)	2000	1720	86.0%
Barium (Ba)	2000	1880	94.0%
Beryllium (Be)	50.0	46.6	93.2%
Cadmium (Cd)	50.0	43.1	86.2%
Chromium (Cr)	200	177	88.5%
Lead (Pb)	500	449	89.8%
Mercury (Hg)	1	0.988	98.8%
Nickel (Ni)	500	423	84.6%
Selenium (Se)	2000	1600	80.0%
Silver (Ag)	50.0	46.3	92.6%
Thallium (Tl)	2000	1860	93.0%
Analyte	**Laboratory Control (mg/Kg)**		**Recovery (%)**
	True	**Found**	
Benzene	10	10	100%
Amenable Cyanide	-	-	-
Total Cyanide	42.2	44.4	105.2%
Phenol	24.9	25.3	101.6%

- Indicates a location in the table for which an entry would not be appropriate.

Results from analysis of the matrix spike (MS) and the matrix spike duplicate (MSD) are given in Table A-3 and Table A- **4**. These results shown in Table A- 3 all analytes met the recommended quality control acceptance criteria for MS and MSD percent recoveries (75-125%) and the Relative Percent Difference (RPD) acceptance limits (0-20%) except for mercury. In Table A- **4**, results show benzene and phenol met the recommended quality control acceptance criteria for MS, MSD and RPDs. In Table A- **4**, results show total cyanide did not meet the recommended quality control acceptance criteria for MS, MSD and RPDs.

Table A- 3 TCLP Leachates Matrix Spike and Duplicate Results[13]

Analyte	Initial Concentrations (μg/L)			Spiked Sample** (μg/L)		Recovery (%)		RPD (%)
	Parent Sample Result	Qualifiers	Spike Added	Spike	Spike Duplicate	Spike	Spike Duplicate	
Antimony (Sb)	<25.0	U	5000	4630	4700	92.6	94.0	1.5
Arsenic (As)	<50.0	UD	2500	2340	2460	93.6	98.4	5.0
Barium (Ba)	1840	D	5000	6830	6870	99.8	100.6	0.8
Beryllium (Be)	17.0	-	500	427	429	82.0	82.4	0.5
Cadmium (Cd)	<5.00	U	500	384	392	76.8	78.4	2.1
Chromium (Cr)	260	D	1000	1180	1180	92.0	92.0	0.0
Lead (Pb)	10.2	B	2500	2120	2160	84.4	86.0	1.9
Mercury (Hg)	58.3	-	5.00	64.0	64.7	114.0	128.0	12.0*
Nickel (Ni)	71.7	D	2500	2130	2170	82.3	83.9	1.9
Selenium (Se)	100	UD	2500	2580	2650	103.2	106.0	2.7
Silver (Ag)	<10.0	U	500	455	459	91.0	91.8	0.9
Thallium (Tl)	<5.00	UD	2500	2300	2350	92.0	94.0	2.2

U Result is less than the Limit of Detection (LOD) and/or Reporting Limit (RL).
D Result is reported from a dilution.
B Analyte was detected at the instrument at or above Limits of Detection (LOD), but less than Limit of Quantitation (LOQ).
* Parent value exceeded 4 times the spike added; therefore, MS/MSD %Recovery and %RPD are not required for evaluation.
- Indicates a location in the table for which an entry would not be appropriate.
** SwRI® Sample ID = W-18098-00001 MS/MSD

Table A- 4 **Organic UHCs Matrix Spike and Duplicate Results[13]**

Analyte	Initial Concentrations (mg/kg)				Spiked Sample (mg/kg)		Recovery (%)		RPD (%)
	Result	Qualifiers	MS-Spike Added	MSD-Spike Added	Spike	Spike Duplicate	Spike	Spike Duplicate	
Benzene**	0.0	U	0.018	0.019	0.018	0.019	100	100	0.0
Amenable Cyanide	-	-	-	-	-	-	-	-	-
Total Cyanide**	11.0	-	1.81	1.91	12.5	13.9	77.3	149.7	64.0*
Phenol**	0.771	UJ	20.2	21.2	0.807	0.846	0.0	0.0	0.0

[U] Result is less than the Limit of Detection (LOD) and/or Reporting Limit (RL).
[J] Matrix spike and/or matrix spike duplicate criteria was not met.
*Parent value exceeded 4 times the spike added; therefore, MS/MSD %Recovery and %RPD are not required for evaluation
- Indicates a location in the table for which an entry would not be appropriate.
** SwRI® Sample ID = W-18098-00001 MS/MSD

Distribution:

J. P. Arnold	C. A. Langton
T. R. Ball	K. R. Liner
C. J. Bannochie	M. J. Mahoney
M. J. Barnes	K. B. Martin
M. N. Borders	M. W. McCoy
J. M. Bricker	R. T. McNew
K. M. Brotherton	P. W. Norris
L. W. Brown	J. E. Occhipinti
T. B. Brown	F. M. Pennebaker
N. F. Chapman	R. C. Player
J. H. Christian	M. M. Potvin
W. A. Condon	J. W. Ray
A. D. Cozzi	C. Ridgeway
C. L. Crawford	L. B. Romanowski
C. C. DiPrete	K. H. Rosenberger
K. D. Dixon	A. Samadi-Dezfouli
R. E. Edwards	D. C. Sherburne
S. D. Fink	F. M. Smith
E. J. Freed	A. V. Staub
E. W. Harrison	J. Stevens
C. C. Herman	C. B. Sudduth
K. A. Hill	B. J. Wiedenman
P. J. Hill	T. L. White
J. F. Iaukea	A. W. Wiggins
V. Jain	L. A. Wooten
V. M. Kmiec	R. H. Young
	Records Administration (EDWS)