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Savannah River Technology Center

Monthly Report

April 1994

Westinghouse Savannah River Company
Savannah River Site
Aiken, SC 29808

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Savannah River Technology Center

Monthly Report

April 1994

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Executive Summary

Tritium

- The final report on the Acorn Cleaning Study was completed on schedule to successfully meet the Level 3 Award Fee milestone. Recommendations were made for an "optimized" procedure for cleaning of future Acorn systems and for tritium testing to prove-out the new procedure. (page 9)
- Nine tritium stripper experiments, which mock the use of a Pd/zeolite stripper for a larger (10,500 liter) glovebox, were completed. The Pd/zeolite stripper system produced a reduction in tritium activity of two to three orders of magnitude after 15-20 hours. (page 9)
- Discussions were held with Tritium personnel and plans were formulated to define SRS needs and timing to enable support for loading of an Acorn unit for replacing the Heather on the W-76 system for Los Alamos National Laboratory (LANL). (page 10)
- CHTS is tracking Replacement Tritium Facility (RTF) requests for technical assistance. Benefits to RTF and CHTS personnel include defining the problem, scope, timing, and resources required as well as less duplication of efforts for problems previously addressed. (page 10)
- The relationship between the accuracy of the in-bed accountability technique and the number of calibration points used in calibrating the bed was estimated. A report showing this correlation was issued. (page 11)
- After Tritium's Line 3 Operational Readiness Review (ORR), hot targets in various reactor basins will be processed. Tentatively, it is believed that some 44 spent melt crucibles will be moved over the next five to six years once processing begins. SRTC's Packaging and Transportation is providing Tritium with necessary design criteria for a package to transport processed spent melts to the ILT vault, and with documentation in the form of a Safety Assessment to support this move. (page 11)
- At the request of DOE, SRTC submitted cost estimates and delivery schedules for providing two kilograms of palladium deposited on kieselguhr by each of three options to the British Atomic Weapons Establishment. (page 11)

- An investigation of the RTF $\text{LaNi}_{4.25}\text{Al}_{0.75}$ (LANA.75) storage material in the "as received" or "virgin" state was completed to meet the Level 3 controlled milestone TCI36C. The results show the material is well behaved and allow the prediction of storage bed response in tritium processing applications. A complete description of this study is documented in WSRC-TR-94-0183. (page 12)
- Non-Nuclear Consolidation is proceeding at a rapid pace. SRS is scheduled to start receiving units that will be transferred from EG&G Mound Laboratory in July 1994. (page 12)

Separations

- Small-scale laboratory experiments were conducted to determine the reaction products of the runaway tributyl phosphate nitric acid reaction similar to the event at Tomsk, Russia. The main reaction products found were butanol and butene. This data is being used to refine the computer model used to simulate SRS conditions. (page 14)
- Studies on a larger scale showed the difficulty in getting the reaction to runaway. Ten attempts to obtain a runaway reaction in a stainless steel reaction vessel resulted in one runaway reaction because of excessive heat losses. Further venting experiments will be performed by a commercial vendor specializing in this area. Further SRTC tests will concentrate in the area of heats of reactions and reaction of products. (page 14)
- A program to quantify the mixing quality of two phases in the canyon tanks began. Previous studies have dealt with solutions, not two phases. This is important in detecting organic in the tanks as well as solids, which are more of a problem as operations are slowed and solutions are stored for long periods of time. (page 15)
- The americium-curium (Am/Cm) stored in F Canyon must be removed before shut down. Cm is a national asset for heavy element research. It is desirable to ship it to Oak Ridge National Laboratory instead of throwing it away. Ion exchange shows promise as a technique to separate the Am/Cm from rare earths and to reduce the shipping costs and reduce the radioactivity of the shipments. (page 15)
- The levels of hydrogen must be kept below the explosive limit in the B-Line dissolvers. A fast means of online measurement is under development using fiber optics and the Laser-Raman system. A successful test was completed in which a fully automated measurement of a 1% hydrogen sample in a mixture of nitrogen, CO_2 , and methane was performed in 30 seconds. (page 16)

Environmental

- Potentiometric maps of the water table and Barnwell/McBean aquifer for the general separations area indicate that the groundwater flow beneath the in-tank precipitation (ITP) processing tanks is towards McQueen Branch. Postulated leaked waste is expected to flow downward and northeast and reach the Congaree aquifer before discharging to McQueen Branch. Waste is then expected to flow northwest and discharge to Upper Three Runs. The groundwater travel-time from the ITP tanks to Upper Three Runs is estimated to be over 150 years. The relatively long groundwater transit time coupled with contaminant retardation means that only long-lived radionuclides will contribute to exposure. (page 17)
- All cells in the sCILS Facility were filled this month, and rototilling and fertilizing are proceeding well. (page 17)
- Uncertainties in the atmospheric tritium dose estimated for 1993 have been calculated for inclusion in the annual environmental report. (page 17)
- At the Environmental Protection Agency's request, Environmental Restoration is conducting a CERCLA-type risk assessment on a single unit of the tank farm (tank 16). A report summarizing historical exposures and current health physics practices at the tank farm has been finalized. (page 17)
- A natural regeneration survey of the Pen Branch delta and its tail has been completed. Good natural cypress regeneration is occurring on the exterior region of the delta and south of the Pen Branch boardwalk areas. (page 17)
- The Environmental Restoration Department has installed five new recovery wells as part of a system to remediate contaminated groundwater in the vicinity of Savannah River Technology Center (SRTC). The Environmental Sciences Section of SRTC has been commissioned to perform pumping tests on each of the new wells to determine hydraulic properties of the recovery well and contaminated aquifer. A step test was conducted on well RWM 14C to determine the specific capacity. Results of the test indicate that the well was properly installed and developed and can be optimally operated at 35-40 gpm. (page 18)
- Sampling and analytical procedures have been developed to assist Environmental Restoration during the restart of the horizontal air extraction at the Integrated Demonstration Site. SRTC personnel will monitor influent and effluent TCE, PCE, and will deduce HCl production from these data to comply with regulatory requirements. (page 18)
- The continuous water sampler for the cone penetrometer developed by SRTC personnel (patent disclosure) is being reconfigured to fit the Fugro, Inc. cone penetrometer. The water sampling system will be used in the western sector characterization activities planned for next month. (page 18)

- A sampling and analysis plan for the Metallurgical Laboratory Basin Carolina Bay was transmitted to the Environmental Restoration Department. This plan provides guidance for the collection and analyses of environmental samples at this Carolina Bay. A report, "Ecological Characterization of the Metallurgical Laboratory Basin Carolina Bay", was transmitted to the Environmental Restoration Department. This report describes the ecosystem potentially at risk, identifies potential ecological stressors, and discusses the baseline conditions. These reports support RCRA/CERCLA. (page 18)
- Sample collection for the March 1994 quarterly tritium survey of the F-Area and H-Area seep lines was completed on schedule. Data have been received and are being analyzed. Preliminary indications are that the tritium concentrations are reducing in the up-gradient part of the wetlands and remain about the same in the mid to lower part of the wetland areas. (page 18)
- Radionuclide concentration measurements in groundwater samples collected from six background ground water monitoring wells in the F-Area and H-Area seepage basins were statistically analyzed to characterize and tabulate the average concentrations of a suite of radionuclides as specified in the RCRA post closure care permit for these waste sites. In addition, the South Carolina Department of Health and Environmental Control (SCDHEC) requested a statistical evaluation of the water quality data for two of the background wells to determine if there was an impact on these wells from either the burial ground complex or the abandoned process sewer lines. The final report was submitted to Environmental Restoration in a format that could be submitted to SCDHEC in support of the RCRA Part B post closure care permit. (page 19)
- The ion trap/mass spectrometer utilization report was completed and submitted (SR141003). This report documents the use of the ion trap mass spectrometer (IT-MS) for analysis of groundwater from well-characterized wells. The goal of this task is to validate the use of IT-MS as a screening device for groundwater samples from well characterized wells. (page 19)
- Field support was also provided for Coleman Blackhawk personnel performing surface and borehole seismic and radar surveys in support of the Coleman Data Fusion Program funded by OTD's Characterization, Monitoring and Sensor Technology Program. The purpose of the research and development project is to improve non-invasive methods for detecting clay lenses because the percent of clay in sediments and soils influences contaminant migration. (page 20)
- A meeting was held on March 31, 1994, in Augusta, Georgia, with representatives from a major United States oil company and an electronics manufacturing firm. These organizations presented information on a site that they own that has groundwater contaminated with trichloroethylene. They are having principal investigators associated with the Integrated Demonstration (ID) review their characterization information so that technologies developed as part of the ID can be utilized at one of their sites. (page 20)

- In support of the barometric pumping project, continuous concentration and flow measurements with and without the baro-ball control valve were made to determine contaminant removal efficiency. The removal rate of the well monitored dropped by a factor of four without the control valve to eliminate flow back into the formation. (page 20)
- A cooperative research and development agreement (CRADA) was submitted for proposed work with a meteorological company. The scope of the work involves improved bi-directional vane (bi-vane) performance, prototype construction, and wind tunnel testing. (page 21)

Waste Management

- The equation for flammability of benzene, hydrogen, and alcohol mixtures in the ITP safety analysis report is being revised. The revised equation corrects weaknesses in selected regions of concentrations and is conservative relative to LeChatelier's rule and all existing experimental data. (page 22)
- Process requirements for Extended Sludge Processing are being developed. The first draft was sent for review on schedule on April 15, 1994. (page 22)
- Flammability calculations were performed for transferring 100,000 gallons of decontaminated spent salt solution waste from non-radioactive testing of the ITP process to saltstone. (page 22)
- SRTC was asked to assist in a program designed to develop repair strategies and techniques for fixing the waste tank cooling coil failures. (page 22)
- The University of Florida and SRTC are involved in a program to develop microwave technologies on destruction and vitrification of circuitry and reclaiming precious metals that exist in these components. (page 23)
- The March 18, 1994 Stop Work Order from DOE on the \$30 million Office of Technology Development funding is in effect. (page 23)
- The Office of Technology Development-supported Sol-Gel Indicator team was a corporate winner of the George Westinghouse Signature Award of Excellence. (page 23)
- A new program involves the development of SRTC sol-gel composites with special properties to improve separation and measurement capabilities of trace radioactive elements in environmental samples. (page 24)
- The mission of the Mixed Waste Integrated Program is to develop enhanced final wasteforms and treatment for delisting mixed waste. The technical objective is to develop and characterize glasses proposed for mixed wastes and to demonstrate the vitrification processing of these glass chemistries. (page 24)

- Sample analyses continue to be provided for Extended Sludge Processing (ESP) Baseline Tests. The results for the two-pump Tank 51H tests are documented as well as the results for the initial sample from Tank 42H testing. All sample results to date were provided within the 14-day target response envelope. (page 25)
- Assistance was provided in drafting the Process Requirements for ESP and supporting documentation. (page 25)
- An analysis of the effect of adding tri-n-butyl phosphate to Tank 50H as a result of the upcoming ITP benzene stripper tests was completed. The addition poses no safety concerns. This information is being used for the Test Authorization revision to allow transfers from Tank 50H to Saltstone. (page 25)
- As a move to improve cost-efficiency and technical collaboration of the two missions, a formal interaction structure was created for Defense Waste Processing Technology (DWPT) and Interim Waste Technology (IWT) personnel in SRTC who support comparable research areas for ITP and DWPF. This structure includes periodic technical review meetings. The first meeting was April 18, 1994. (page 25)

General

- Several planning meetings were held to begin work on the scope, methodology, and path forward for the WSRC program to support the required, "SRS Plutonium Vulnerability Study/Nuclear Materials Management EIS". A SRTC Technical Area Task Team was formed and work has begun. (page 27)
- To support the 5480.23 SRTC SAR Preliminary Hazard Assessment (PHA) program, the current SRTC data bank was sorted for safety-related initiators, ancillary support systems occurrences, chemical and radionuclide incidents involving personnel, and natural phenomenon events. (page 27)
- Draft addenda for the SRTC 5480.23 SAR Task Plan (SRT-TML-94-8002) were prepared for FY 94 work scopes for Chapters 2 and 3 SAR support work. (page 27)
- The Chemical Information and Inventory System "Area Report" was modified to include downloads of EPA Reportable Quantities, Threshold Planning Quantities, and the OSHA Threshold Quantity for chemical constituents. (page 27)
- SRTC OSR Revision Request 94-0001 was submitted to DOE-SR on March 30, 1994 and review and approval was requested by April 22, 1994. In several meetings with DOE-SR, no technical issues were identified, but several administrative comments were addressed. (page 27)
- The Oversight Committee gave SRTC the approval to hire ten PhDs for critical needs. (page 28)

- The Budget Module is being modified to conform with site changes expected for the 1995 AOP Budget Call. These modifications will be made for SRTC, ALD, and EDD budget development and input. (page 28)
- LSS (SSD) approached SIPCM to assist with developing a programmatic control tool for use in scheduling/controlling LSS operations. SIPCM generated an integrated schedule at the request of LSS (SSD) for its SED Tank Removal program. (page 29)
- SRTC/SIPCM is actively involved in a Westip effort to standardize the cost estimating practices across the site. This effort will involve issuing a Cost Estimating Manual as part of the overall Budget Formulation/Development Procedure required for cost/budget validation. (page 29)

Progress and Accomplishments

Tritium

Acorn Cleaning Studies

D. L. Fish, W. J. Rogier, and E. F. Dyer

The final report on the Acorn Cleaning Study was completed on schedule to successfully meet the Level 3 Award Fee milestone. This report summarizes the conclusions of the entire Acorn Cleaning study, answers several questions left pending after the initial report was completed in September 1993, and provides additional insights into the cleaning process. The study recommends a modified procedure to the AFR procedure utilized for cleaning Acorn reservoirs. The proposed modified cleaning procedure will result in a substantial time savings for the cleanings, due to reductions in the final pumpdown time, the time between the flushes, and the length of the flushes. The total number of flushes was increased to improve the cleaning, but the overall time for processing was still reduced to approximately one third of that required for an AFR cleaning. In addition to the time savings, the cleaning study showed that substantially reduced control requirements are necessary for the temperature and pressure conditions under which the cleaning is conducted. Each item results in simplification of the process and represents an easing of the level of difficulty for plant operations while maintaining the same quality of product cleanliness.

The basic understandings gained from this study will enable significant reductions, in the time, required to develop cleaning methodologies for advanced Acorn designs. Recommendations were made to the national laboratories for

tritium loading and storage testing of units cleaned with the "optimized" procedure to enable detection of any long-term differences from units cleaned under AFR conditions.

Function Test Facility Stripper Tests

J. E. Klein and J. R. Wermer

A small Metal Hydride Research inert glovebox in the Materials Test Facility was used for tritium stripper experiments designed to simulate the use of a Pd/zeolite stripper for a larger (10,500 liter) sealed glovebox. Nine tritium releases and clean-up tests are complete. The experiments consisted of releasing deuterium spiked with tritium and using Pd/zeolite columns to reduce the glovebox activity. The amount of deuterium was scaled to simulate tritium releases into the glovebox of 0.03 g, 0.1 g, 1.0 g, 10 g, and 30 g. The Pd/zeolite stripper system produced a reduction in tritium activity of two to three orders of magnitude after 15–20 hours with a flow rate scaled to simulate operations in the 10,500 liter glovebox.

The test results show that Pd/zeolite material does an excellent job of reducing the tritium activity in the glovebox. At the flow rate scaled to simulate operations in the 10,500 liter glovebox, initial stripper efficiencies typically range between 85 and 90% and decreased as clean-up operations continued. A slower stripper flow rate increased stripper efficiency, but increased the time needed for glovebox clean-up. During clean-up operations, the tritium concentration in the glovebox levels-out at a nominal activity of $10_4 \mu\text{I}/\text{m}_3$ and then decreases very slowly. Glovebox focussing tests (tests run with the stripper in by-pass mode) show that the glovebox activity increases with time. The increase in glovebox activity is assumed to be tritiated water

desorbing off the surfaces inside the glovebox. High flow nitrogen and air purges of the glovebox significantly reduce the activity in the glovebox.

A draft report summarizing the test results was issued. Based on feedback of this report, additional stripper tests may be made to aid in designing and operating the Function Test Facility stripper.

W-76 Acorn for Los Alamos National Laboratory

D. L. Fish and T. J. Warren

Several discussions were held with Tritium personnel to determine an acceptable method for loading Acorn units as a potential replacement for the Heather units on the W-76 system. Needs were discussed for loading of development units and for loading of actual units for field installation. Two documents were written to help address five items requested of SRS by Los Alamos National Laboratory (LANL) regarding our abilities to load Acorn development units for this program. The first document (SRT-HTS-94-031) defined the number and type of prototype development units needed to develop the cleaning, loading, and functioning methodologies for the W-76 Acorn system. The second document (SRT-HTS-94-0030, Rev. 1) provided a revised summary of the costs, type of funding, and schedule for implementing the W-76 Acorn testing program. The information in this second document was originally requested and provided to Kansas City for incorporation and integration in their master schedule for developing the W-76 Acorn units for DOE-AL and LANL.

Replacement Tritium Facility Technical Issues Tracking

A. S. Horen, T. Motyka, L. K. Heung, J. E. Klein, R. A. Malstrom, G. S. Nichols, J. H. Scogin, and J. R. Wermer

CHTS is tracking Replacement Tritium Facility (RTF) requests for technical assistance. SRTC/CHTS provides technical consultation and assistance for RTF startup activities. Benefits to RTF and CHTS personnel include defining the problem, scope, timing, and resources required as well as less duplication of efforts for problems previously addressed. Only specific RTF issues, typically requested by RTF personnel, are tracked in this manner; general RTF issues and technology topics are not included. The status of RTF issues generated and completed, as well as total manhours spent addressing RTF issues is reported in the *RTF Monthly Project Status Report*.

The total number of RTF issues generated and closed are cumulative since 1992 and will continue to be tracked. The cumulative number of manhours devoted to specific RTF issues was reset for the calendar year.

Key RTF Technical Issues for March 1994 include:

- providing Thermal Cycling Absorption Process (TCAP) training (classroom)
- providing support for the TCAP Tritium Separation Demonstration Test (SOP-AP-233-00395)
- developing interim TCAP operating procedures
- providing support for Z-bed recovery technical issues
- providing technical support for hydride bed inventory and accountability issues
- outlining TCAP ONLINE sequence modifications

Replacement Tritium Facility In-Bed Accountability Support

J. E. Klein

The Replacement Tritium Facility in-bed accountability (IBA) process will be used to determine the amount of tritium stored on the ^{233}H metal hydride beds by measuring the steady-state temperature rise (DT) of a gas flowing through the nitrogen jacket of a bed. To "calibrate" the beds using the IBA method, different amounts of tritium need to be loaded onto the bed to develop the DT versus tritium loading (grams T_2) correlation for the bed. The relationship between the accuracy of the IBA technique and the number of calibration points used in calibrating the bed (N) was estimated using the results of the SRTC IBA development program. A report (WSRC-TR-94-037) showing this correlation was issued.

The analysis shows that for N less than six, the IBA accuracy is a strongly decreasing function of N. For N equal six or more, no significant gains in IBA calibration accuracy is obtained. Requiring six or fewer calibration points for each bed calibration, instead of the original nine, will reduce the total number of IBA calibration points to be run in RTF by over 24. With each calibration point taking at least one day of process operations, this will significantly reduce the cost and time needed to implement IBA in RTF.

Tritium Crucible Package Transport Design and Safety Assessment

P. S. Blanton and A. G. Eggers

The Tritium Extraction Facility (Line 3) can temporarily store 12 processed crucibles before having to move the spent melts from their facility to the burial grounds. Previous plans for moving the spent melts involved having an external RAM packaging agency design and acquire a Department of Transportation Type B transportation cask that could transport the crucibles from the Tritium Facility to the ILT vault.

The present path forward will have Packaging and Transportation (P&T) act as the cognizant packaging design agency in determining necessary design criteria to transport the spent melt in an alternate Type B package, per the 19Q TSM to the ILT vault. P&T and Tritium are developing the necessary crucible transport design requirements. Upon completing these requirements, a preliminary package and shield/transport design, schedule and cost estimate proposal will be presented to Tritium. This proposal is expected to be completed by the July 1, 1994.

The proposed alternate Type B package design will be based on the present crucible overpack to provide containment and a transport shield similar to the existing coffin design to meet necessary shielding requirements.

Palladium Deposited on Kieselguhr for Atomic Weapons Establishment

W. C. Mosley

The British Atomic Weapons Establishment inquired about the possibility of purchasing two kilograms of palladium deposited on kieselguhr (Pd/k) via WSRC. At the request of DOE, SRTC submitted cost estimates and delivery schedules for each of the following options:

- Pd/k conforming to WSRC procurement specifications [$\geq 55\%$ Pd, $> 80\%$ within 30–50 mesh, < 250 ppm chlorine] manufactured by the palladium chloride deposition process
- existing Pd/k with 45–55% Pd
- Pd/k conforming to WSRC procurement specifications manufactured by the tetramine-palladium (II) nitrate deposition process.

More details on the above options and estimates at cost and delivery schedule for each option can be found in SRT-HTS-94-0119.

"Virgin" LANA.75 Characterized for Level 3 Milestone

J. S. Hölder and D. E. Moseley

As part of the Tritium Exposure Program in the Materials Test Facility (MTF), an investigation of the Replacement Tritium Facility storage material $\text{LaNi}_{4.25}\text{Al}_{0.75}$ (LANA.75) in the "as received" or "virgin" state was conducted. For this study, tritium desorption isotherms were measured at three temperatures to determine the thermodynamics of the metal tritide. The work was carried out on the Experimental Tritium Manifold (ETM) in the MTF.

Three tritium desorption isotherms were collected for the LANA.75 sample at 40°C, 80°C, and 115°C in such a manner as to minimize the degree of tritium aging acquired by the sample during the measurements. At all temperatures, smooth isotherms were observed with flat pressure plateaus and no tritium heel formation, indicating that the material was well behaved. The capacity of LANA.75 to absorb tritium was calculated in terms of the composition to be $T/M = 0.67$ at 40°C. This value is large and comparable to that for palladium.

The tritium delivery pressure is dictated by the pressure plateau as a function of temperature, whereas, the slope of the plateau affects the delivery pressure as a function of the composition of the storage bed. The slopes of the plateaus were found to be very small, which is desirable, and increased slightly with temperature. The thermodynamic parameters of the change in enthalpy (ΔH) and change in entropy (ΔS) for the formation of the LANA.75 tritide were determined at the compositions of $T/M = 0.14, 0.30, \text{ and } 0.46$. These values allow the tritium delivery pressure for a storage bed to be predicted as a function of temperature over a wide range of compositions.

A complete description of this study, including the isotherms, van't Hoff plots, and calculations of ΔH and ΔS , is documented in WSRC-TR-94-0183, *The Thermodynamic Properties of 'Virgin' LANA.75*. This work served to complete the Level 3 controlled milestone, TCI36C.

Non-Nuclear Consolidation—EG&G Mound Laboratory Unit Transfer (U)

K. A. Dunn and K. M. Keeler

Non-Nuclear Consolidation (NNC) is proceeding at a rapid pace. SRS is scheduled to start receiving units that will be transferred from EG&G Mound Laboratory in July 1994. SRS and EG&G Mound Laboratory personnel have been working together to support this transfer of units. This support includes issues such as the unit transfer schedule, unit contamination, and unit documentation requirements. Currently, the unit transfer schedule shows a completion date for shipping the W-87 Vireyards as July 1995 and a completion date for shipping the development units as June 1996. Several suggestions were provided to EG&G Mound Laboratory to reduce the time for shipping development units. However, SRS has no control over whether EG&G Mound Laboratory incorporates the suggestions into the schedule since these proposed changes impact operations only at EG&G Mound Laboratory.

SRS provided a memo to EG&G Mound Laboratory outlining the requirements for packaging secondary containers, including a discussion concerning the contamination limits of the units. Some units contain some transferable alpha contamination, which was obtained from the glove-box line. To accelerate the packaging of the units, SRS agreed to accept the units following one decontamination step at EG&G Mound Laboratory, even if the units still have some alpha on them. SRS will unpack the secondary containers and proceed to survey the units as they are received. If necessary, the units will be decontaminated at that time.

Paperwork requirements, primarily unit information, needed to accept the units from EG&G Mound Laboratory are outlined in a memo to EG&G Mound Laboratory personnel. Nominal information on the units was received so that the Unresolved Safety Question (USQ) process can proceed. However, a large portion of unit specific information is still required. This information is needed primarily for the historical data.

Two EG&G Mound Laboratory Unit Transfer Team meetings were held to outline the necessary requirements at SRS for receiving these units. Tritium personnel involved in these meetings included Packaging and Receiving, Calorimetry, MC&A, Radiological Control Operations, and Tritium Engineering. These meetings were extremely productive and will help ensure that SRS is prepared to receive the EG&G Mound Laboratory units.

**Progress and
Accomplishments**

Separations

Tributyl Phosphate Safety Studies

W. S. Cavin, G. S. Nichols, J. R. Smith,
T. B. Garbutt, E. F. Dyer, and M. C. Thompson

In April 1993, a radionuclide separation facility in Tomsik, Russia experienced an explosion in a processing tank and severely damaged the facility. As an outgrowth of a DOE task team investigation, several DOE sites are modeling the chemistry to understand the conditions leading to the explosion. In addition, SRTC is trying to assure conditions for safe operation in F and H Canyons.

The decomposition products from a runaway reaction of tributyl phosphate (TBP) saturated with concentrated nitric acid (HNO_3) were measured to support computer modeling of the "red oil" reaction. The runaway reaction was created by heating TBP/ HNO_3 until self-heating rapidly increased the solution temperature. Experiments were performed by saturating TBP with concentrated nitric acid and placing 0.5 g in a test tube. Temperatures in excess of 200°C were obtained by wrapping heating tape around the test tube. The reaction tube assembly was placed in a large polypropylene bottle to capture the decomposition gases. A pump transferred the product gases to various gas scrubbers (to measure CO_2 , NO_2 , NO , etc.). The system was semi-closed to maintain ambient pressure and measure the net product volume of water insoluble gases.

Five experimental runs were performed. In each case, TBP was shaken with concentrated nitric acid; 0.5 g of TBP/ HNO_3 was removed and placed in the reaction tube, which was purged with nitrogen to remove the oxygen. The heat control was set for 250°C, but the heat of the reaction drove the temperature to a maximum of 265°C. The major organic compounds found in the gas phase (in decreasing amounts) were n-butanol, butene, butyl-nitrate, di-n-butyl ether, butyl acetate, and methyl acetate. Data from three runs were used to calculate a butene production equal to $17 \pm 6\%$ (95% confidence limit) of the theoretical maximum. The large error is probably due to the autocatalytic nature of the oxidation reaction.

A major defense against a Tomsik-like incident is the provision of adequate venting on tanks and vessels containing TBP and HNO_3 . Equipment to demonstrate safe venting was set-up in Cell 2 or the Fracture Toughness Test Facility in Building 774-A. A 450 ml stainless steel pressure reactor was modified for the tests. The modifications included connections to two pressure transducers, three thermocouples, an automatic feed valve, and a vent. (The 0.025-inch vent size for 100 ml of TBP/ HNO_3 was based on experimental work performed by Fauske and Associate, a company that routinely specifies venting requirements for the chemical process industry.) All temperatures and pressures were recorded on a computer for subsequent analysis.

Ten venting experiments were performed. In each case, the adequacy of the vent was not demonstrated because of problems associated with the stainless steel reactor. Rapid self-heating was seen in experiments using 50 and 100 ml of TBP/ HNO_3 ; however, heat losses from the reactor vessel offset the heat generated by chemical reactions preventing the runaway in all but one case. A runaway reaction was observed in one 100 ml experiment using rapid heating (230°C setpoint) and a glass liner as an insulator in the reactor. The runaway reaction was observed when the temperature of the liquid reached 165°C; however, the adequacy of the vent could not be assessed because of excessive splattering (from the rapid heating) and plugging of the vent line. After cooling, the reactor contained a viscous, black liquid in the glass liner with indications of the splattering on the walls and top of the reactor. It is apparent that (adiabatic) conditions necessary for the runaway reaction cannot be achieved with this low volume, high mass system. Fauske and Associates will demonstrate adequate vent sizing using equipment that provides the adiabatic conditions necessary for this scale.

Further experiments at SRTC will be aimed at measuring the heat of reaction of TBP/nitric acid at various temperatures and determining the decomposition temperatures.

Organic in Aqueous Analysis for Evaporators

R. R. Livingston

Since the tributyl phosphate/runaway reaction, is a result of the degradation of the organic TBP in nitric acid at high temperatures, limiting the quantity of organic material fed to the evaporators is one method of defense. To help provide assurance that minimal quantities of organic (<0.5%) are being fed to the evaporator, the Analytical Development Section is developing a rapid screening method for TBP/n-paraffin in nitric acid solutions. The method should allow the department to provide quicker turnaround times on organic in aqueous (O/A) samples and allow canyon personnel to pull additional samples. The method involves adding an indicator dye, congo red, to the sample and mixing. The organic phase, when dealing with a two-phase system, is highlighted by the indicator. This permits visual inspection and semi-quantitative measurement of O/A.

Mixing in Canyon Tanks

N. M. Hassan, E. A. Kyser, and Y. J. Simpkins

Separations Engineering requested Chemical Technology to develop the documentation to quantify the mixing quality in the canyon tanks. The tanks contain up to 0.5% (volume) organic in aqueous phase and the solutions are mixed well before introduction into the process evaporators. Flat-blade agitators with sufficient power input are used to assure adequate mixing of the two-phase immiscible liquids. No documentation of the mixing quality in the canyon tanks is available in the SRS/SRL archives.

Several experimental runs were performed as part of the canyon mixing study using a cylindrical, flat-bottomed plexiglass tank. The agitator was a fabricated scale model, plant style flat-blade agitator, with the shaft centrally located in the tank. The tank contained a 5/16-inch organic layer (n-paraffin) floating on water of various heights ranging from 4 to 12 inches. The two-phase immiscible liquids were agitated at different speeds ranging from 200 to 420 rpm. After

agitation was fully established, the liquid mixture was sampled from the bottom of the tank with a pump to simulate the canyon samples.

The mixing pattern of the two-phase immiscible liquids was videotaped. Visual observation of the mixing characteristics in the tank showed that two separate phases initially existed. As the mixing began, the organic layer broke into the aqueous phase resulting in small organic bubbles/droplets rotating with the bulk of the liquid. When the agitator speed was increased, the liquid mixture became severely turbid with a whitish color. Samples taken from the bottom of the tank at different agitator speeds showed a general consistency in the trend of measured organic (n-paraffin) in the aqueous samples. Increasing the agitator speed or decreasing the liquid level (aqueous phase) resulted in the increase of organic content in the samples; however, the reproducibility of the organic measurement in the samples taken at the same conditions was poor. Work is continuing to refine technique and analytical error.

Curium-Ion Exchange Studies

R. A. Pierce, D. G. Karraker, and J. D. Clark

Ion exchange is being evaluated as one aspect of a program to disposition the curium-amerium (Cm-Am) stored in F-Canyon Tank 17.1. If recovered, the Cm-Am will be shipped to Oak Ridge National Laboratory. Ion exchange will separate the Cm-Am from various lanthanides because of their impact on shipping container capacity (lanthanum, cerium, praseodymium, neodymium) and on shielding (europium).

Earlier results using a nitrate-based system showed that lanthanum, cerium, praseodymium, and neodymium could be separated from samarium, europium, and gadolinium using a TevaSpec resin and a 4 M LiNO₃ column rinse solution. Additional tests using Am in a solution of lanthanides revealed that the Am (and probably the Cm) would elute with the europium, thus providing a separation of the Cm-Am from the bulk of the lanthanides.

Tests were also conducted with a landscape resin to separate Cm-Am from europium in a dilute nitric acid stream. This would be instrumental in separating the Cm-Am from Europium-154 (a heavy gamma emitter); however, further investigation of the chemistry involved revealed that the low-acid conditions necessary to effect this separation will be unattainable in F Canyon.

Fiber Optic Raman Hydrogen Sensor for HB-Line

S. E. Nave and W. R. Toole

ADS is developing a fiber optic Raman system to detect hydrogen in the offgas of the HB-Line dis-solvers. A successful test, in which a fully automated measurement of a sample containing 1% hydrogen in a mixture of nitrogen, CO₂, and methane, was performed. With the total time to complete a measurement set at 30 seconds and the sample pressurized to 200 psi, the minimum detectable hydrogen concentration was 0.1%. Since these measurements were made, a method has been developed in construction of the fiber optic probes that reduces the background noise due to fluorescence in materials used in the fiber construction. This development involves removing a polyamide buffer coating from the probe tip and may result in either reducing the minimum detectable hydrogen concentration or reducing the measurement time.

Environmental

Subsurface Contaminant Transport Analyses to Support In-Tank Precipitation/ H-Area Safety Basis

G. P. Flach

Potentiometric maps of the water table and Barnwell/McBean aquifer for the general separations area indicate that the groundwater flow beneath the in-tank precipitation (ITP) processing tanks is towards McQueen Branch. Coupled one-dimensional flow models of the water table and Barnwell/McBean aquifers with surface recharge and leakage between the water table and Barnwell/McBean and Congaree aquifers were created and calibrated. The resulting hydraulic conductivity estimates are consistent with measured data. Because the ITP tanks are located near the groundwater divide for the water table and Barnwell/McBean aquifers, there is a significant downward flow component. Postulated leaked waste is expected to flow downward and northeast and reach the Congaree aquifer before discharging to McQueen Branch. Waste is then expected to flow northwest and discharge to Upper Three Runs. The groundwater travel-time from the ITP tanks to Upper Three Runs is estimated to be over 150 years. The relatively long groundwater transit time coupled with contaminant retardation means that only long-lived radionuclides will contribute to exposure.

sOILS Facility

K. H. Lombard and T. C. Hazen

All cells in the sOILS Facility were filled this month, and rototilling and fertilizing are proceeding well. Several potential customers, including the SRS Fire Department, have requested that their material be remediated. The waste is being analyzed to determine the most appropriate method for processing. The SRS Fire Department has 400 gallons of oily mousse that has low benzene and will not ignite. The Fire

Department submitted samples for TCLP analysis. The possibility of a batch bioreactor system to remediate this waste in their present storage tanks will be investigated.

PREDICT Calculations for 1993 Atmospheric Tritium Releases

D. M. Hamby

Uncertainties in the atmospheric tritium dose estimated for 1993 have been calculated by PREDICT for inclusion in the annual environmental report for the second consecutive year. These estimates will have a short narrative description of the methodology along with the dose probability distributions.

Worker Exposure Assessment for Waste Tank 16

D. M. Hamby

At the Environmental Protection Agency's request, Environmental Restoration is conducting a CERCLA-type risk assessment on a single unit of the tank farm (tank 16). Because of its location, however, only a worker exposure assessment is possible under current use conditions. Radiological control surveys over the past eight months and employee exposure records were utilized for the assessment. Radiological access requirements for the tank farm were also examined. A report (WSRC-TR-94-0141) summarizing historical exposures and current health physics practices at the tank farm has been finalized.

Pen Branch Mitigation Action Plan

E. A. Nelson

A natural regeneration survey of the Pen Branch delta and its tail has been completed. Good natural cypress regeneration is occurring on the exterior region of the delta and south of the Pen Branch boardwalk areas. Some small cypress seedling regeneration appears to have been lost because of the extreme flooding that occurred

two winters ago, but will not require intervention to satisfy the mitigation. The survey crew has completed the topographical survey of the swamp and has provided the data for the transects in the Savannah River swamp. The data has been forwarded to the Waterways Experiment Station to be included in the model preparation for the system.

A/M Area Northern Sector Aquifer Tests

R. L. Nichols, C. May, B. Pemberton, D. Jackson, and J. Haselow—ESS

The Environmental Restoration Department installed five new recovery wells as part of a system to remediate contaminated groundwater in the vicinity of Savannah River Technology Center (SRTC). The Environmental Sciences Section of SRTC has been commissioned to perform pumping tests on each of the new wells to determine hydraulic properties of the recovery well and contaminated aquifer. A step test was conducted on well RWM 14C to determine the specific capacity of the well. The specific capacity is the gallon per minute (gpm) yield of the well per foot of drawdown in the well and is used to establish the operating conditions for a recovery well. Results of the test indicate that the well was properly installed and developed and can be optimally operated at 35-40 gpm. Data from a nearby monitoring well were also collected during the step test. Data from the observation well were used to estimate the hydraulic properties of the aquifer and results indicate that the aquifer has properties similar to the rest of the A/M Area aquifer.

A/M Area Groundwater Remediation

J. Rossabi, M. Keenan, S. Burdick, R. Raymond, and B. Riha—ESS

Sampling and analytical procedures have been developed to assist Environmental Restoration during the restart of the horizontal air extraction at the Integrated Demonstration Site. When the system is restarted, SRTC personnel will monitor influent and effluent TCE, PCE, and will deduce HCl production from these data to comply with regulatory requirements.

A/M Area Western Sector Characterization

J. Simmons, J. King, M. Keenan, C. May, J. Rossabi, and C. Eddy-Dilek—ESS

Volatile organic constituent head space analyses are complete on soil cores that were collected in the A/M Area western sector. The analyses aided in characterizing TCE groundwater contamination in that area.

The continuous water sampler for the cone penetrometer developed by SRTC personnel (patent disclosure) is being reconfigured to fit the Fugro, Inc. cone penetrometer. Fugro, Inc. recently won the cone penetrometer contract. The water sampling system will be used in the western sector characterization activities planned for next month.

Metallurgical Laboratory Basin Carolina Bay Ecological Support

Gary P. Friday—ESS

A sampling and analysis plan for the Metallurgical Laboratory Basin Carolina Bay was transmitted to the Environmental Restoration Department. This plan provides guidance for the collection and analyses of environmental samples at this Carolina Bay. These data are used to characterize the environment that is potentially at risk as required under RCRA/CERCLA.

A report, "Ecological Characterization of the Metallurgical Laboratory Basin Carolina Bay", was transmitted to the Environmental Restoration Department. This report describes the ecosystem potentially at risk, identifies potential ecological stressors, and discusses the baseline conditions. This report supports RCRA/CERCLA.

March 1994 Quarterly Tritium Survey of F-Area and H-Area Seep Lines

V. Rogers—ESS

Sample collection for the March 1994 quarterly tritium survey of the F-Area and H-Area seep lines was completed on schedule. Data have

been received and are being analyzed. Preliminary indications are that the tritium concentrations are reducing in the up-gradient part of the wetlands and remain about the same in the mid to lower part of the wetland areas. Concentrations at some of the mid points are the same or slightly elevated indicating a shift in flow net pattern that resulted from dewatering (closing) the basins.

Analysis of Background Radionuclide Concentrations for the F Area and H Area Seepage Basins

E. W. Jenkins, R. C. Tuckfield, J. H. Weber,
and M. D. Wise—ATS

Groundwater samples collected from six background wells in the F-Area and H-Area seepage basin from 1988 to 1993 were statistically analyzed to determine the average concentration for a required list of radionuclides in each of three aquifers in the two areas. Plots of the results over time, by laboratory, and method were done to identify trends, outlier values, large detection limits, and other problems with the data. After qualification of the data, the average concentration was estimated in each of the six wells and over all three aquifers in both F Area and H Area for 25 radionuclides.

The average concentration was compared with the 90th percentile of the radionuclide detection limit. All constituents were less than the comparison detection limits except for americium-243 in the Barnwell/McBean aquifer in H Area, plutonium-239 and plutonium-240 for the Barnwell/McBean aquifer in H Area, and radium-226 in the Congaree aquifer in F Area and the water table in H Area. The americium-243 detection limit was 0.25 pCi/L with the average concentration in the Barnwell/McBean aquifer in H Area of 0.61 pCi/L. However, few americium-243 analyses were done for any of the wells. The detection limit for plutonium-239/240 was 1.0 pCi/L with the H-Area Barnwell/McBean average of 1.05 pCi/L. The radium-226 detection

limit was 1.0 pCi/L with the F-Area Congaree average of 1.02 pCi/L and the H-Area water table average of 1.16 pCi/L. In addition, SCDHEC requested a statistical evaluation of the water quality data from two of the background wells in F Area to ensure these wells did not indicate an impact from either the burial ground complex or the abandoned process sewer line.

The evaluation included comparing the historical concentrations of these radionuclides with those reported in 1993 by means of statistical process control charts. The charts showed that nearly all quarterly concentration measurements were within the usual $+3\sigma$ control limits for all constituents in both wells. In addition, between well comparisons were made for these two wells with corresponding background wells in H Area in the same aquifer by statistically comparing the proportion of the results above detection limits. There were no statistically significant differences between the two F-Area background wells and the H-Area background wells in the proportion of results reported above detection. Since there was no significant increase in proportion of results above detection and the 1993 results were not significantly greater than would be expected, there is no evidence of past or present contamination to the two F-Area background wells in question.

Ion Trap/Mass Spectrometer

J. Rossabi and C. Eddy-Dilek—ESS

The ion trap/mass spectrometer utilization report was completed and submitted (SR141003). This report documents the use of the ion trap mass spectrometer (IT-MS) for analysis of groundwater from well-characterized wells. Analytical results obtained with the IT-MS are compared to results obtained using baseline methods. The goal of this task is to validate the use of IT-MS as a screening device for groundwater samples from well characterized wells.

Geophysical Demonstration Field Support

B. Pemberton and C. Eddy-Dilek—ESS

Site support is currently being provided for two field demonstrations of geophysical systems developed under DOE funding. Coleman Research visited SRS for a pre-site survey for demonstration of the earth penetration radar imaging system (EPRIS) on March 10, 1994. Arrangements were made to host the field demonstration of this equipment on March 29, 30, and 31. Field testing was completed on March 31, 1994.

Field support was also provided for Coleman Blackhawk personnel performing surface and borehole seismic and radar surveys in support of the Coleman Data Fusion Program funded by OTD's Characterization, Monitoring and Sensor Technology Program. The purpose of the research and development project is to improve non-invasive methods for detecting clay layers because the percent of clay in sediments and soils influences contaminant migration. Two types of geophysical information, seismic and electrical resistivity, will be combined to form a better subsurface image of clay layers. Two field sites were selected for the surveys: a traverse through M Area targeted at identification of the clay units that control DNAPL movement and a traverse across the proposed site for the New Sanitary Landfill at SRS. Coleman Blackhawk was provided with available borehole geologic and geophysical data, maps, and other technical information. Groundwater pumps were removed from seven wells to facilitate the field survey. The field work was initiated March 29 and was completed April 13 of this year.

Technology Transfer

**B. Looney, R. Nichols, C. Eddy-Dilek,
and J. Haselow—ESS**

A meeting was held on March 31, 1994, in Augusta, GA, with representatives from a major United States oil company and an electronics manufacturing firm. These organizations presented information on a site that they own that has groundwater contaminated with trichloroethylene. They are having principal investigators associated with the Integrated Demonstration (ID) review their characterization information so that technologies developed as part of the ID can be utilized at one of their sites.

Based on initial review, it appears that there are some technologies that will provide cost-effective solutions to their problem. This effort is being conducted as part of the technology transfer initiative associated with the ID close-out.

Barometric Pumping Test

B. Riha, J. Rossabi, and B. Pemberton—ESS

In support of the barometric pumping project, continuous concentration and flow measurements with and without the baro-ball control valve were made to determine contaminant removal efficiency. Allowing air to flow into the well from the surface effectively dilutes the concentrations in the subsurface. The removal rate of the well monitored dropped by a factor of four without the control valve to eliminate flow back into the formation. PCE, TCE, TCA, and CO₂ gas concentrations were measured with the Bruel and Kjaer multi-gas monitor. The control valve has been reinstalled to determine the length of time and total flow volume required to reestablish maximum removal efficiency. The cumulative data from the test will also enable the estimation of the mass transfer rates in the zone of influence of this well.

Potential Cooperative Research and Research Development Agreement

M. J. Parker—ETS

A cooperative research and development agreement (CRADA) was submitted for proposed work with a meteorological company. The scope of the work involves improved bi-directional vane (bi-vane) performance, prototype construction, and wind tunnel testing. The proposed work would be to determine the characteristics of an improved bi-vane, fabricate a prototype, and test the prototype in the 735-7A wind tunnel. A report that documents the wind tunnel testing results would be written. The cooperative work would lead to improved wind direction measurements for emergency response and meteorological databases.

**Progress and
Accomplishments**

Waste Management

Flammability Equation for the In-Tank Precipitation Safety Analysis Report

D. D. Walker

A new flammability equation for benzene, hydrogen, and alcohol mixtures was developed. The equation used in the Safety Analysis Report (SAR) was based on a statistical fit of limited experimental data. A review of this equation indicated that it predicted that certain vapor mixtures with compositions intermediate between the data points would not be flammable even though one component was in excess of its individual lower flammability limit. The new equation is based on LeChatelier's rule and correctly predicts the flammability of all of the experimental data and is at least as conservative as LeChatelier's rule for intermediate compositions. The change is not expected to impact other analyses in the SAR. A technical report was drafted and is under review.

Extended Sludge Processing Process Requirements

M. S. Hay

Process requirements for Extended Sludge Processing (ESP) are being developed. The first draft was sent for review, on schedule, on April 15, 1994. The target values for feed material sent from ESP to the Defense Waste Processing Facility (DWPF) were calculated based on the feed design bases of DWPF (SRT-LWP-94-031). A previous document summarized target values for ESP based on an early draft of the design bases. The limits for ESP were calculated after proportioning the design basis value between in-tank precipitation (ITP) and ESP. A separate document will be issued to document the rationale used to divide the feed limits between ITP and ESP. The target values for ESP may be exceeded without affecting the acceptability of the combined ITP and ESP feed if ITP has not used their full allotment for targets apportioned between ITP and ESP. Mechanisms for handling deviations from the ESP target values that

interface with ITP and DWPF must be established. This document was generated in support of the development of ESP Process Requirements.

Effect of Tri-n-Butyl Phosphate on Flammability Potential of Salt Solution Transfers from In-Tank Precipitation Facility to Saltstone

M. J. Barnes

Flammability calculations were performed regarding transferring 100,000 gallons of decontaminated spent salt solution waste from non-radioactive testing of the in-tank precipitation (ITP) process. Transferring 100 kgal of ITP spent salt solution (containing 25 gallons of tri-n-butyl phosphate, and ITP laboratory waste with an annual hexane waste amount of 960 g) is intrinsically safe in Tank 50H, the saltstone hold tank, and the low-point drain tank. Under the most conservative conditions, the composite lower flammability limit will not be reached in any of the three tanks. These calculations were performed to support the test authorization document addressing transferring the solution from Tank 50H to saltstone. To perform the calculations, the solubility of 1-butanol in simulated, decontaminated 4.8 molar sodium salt solution at 50°C was experimentally measured and found to be 4.9 g/L of solution. This value was used with the theoretical vapor pressure of 1-butanol (at 50°C) to calculate a Henry's Law constant of 0.026 for 1-butanol for these conditions. It is recommended (but not necessary if the above limit on hexane waste is accepted) that the volume of Tank 50H be at least 115 kgal, or greater, prior to a transfer to the saltstone holding tank. Acceptance of the 960 mg/L hexane limit and the 115 kgal fill limit would result in an added degree of safety. A document is being prepared.

Waste Tank Cooling Coil Study

G. G. Wicks and J. R. May, II

Loop tests to evaluate internal fixes are continuing. Hanford asked SRTC to assist them by defining potential repair products for a new device

they developed. We saw their new delivery system and received photographs of the device for delivering repair products to cracks in metal piping.

Microwave Vitrification Study

G. G. Wicks

The University of Florida and SRTC are involved in a program to develop microwave technologies. The effort focuses on minimizing new waste currently generated, reducing and immobilizing existing specialty wastes (defense, industrial, medical, and domestic wastes), and recycling waste into useful products. Our primary interest is focused on destroying and vitrifying circuitry and reclaiming precious metals that exist in these components.

A joint paper with University of Florida entitled, "Microwave Energy for Waste Remediation Applications", was presented at the 1994 Spring MRS meeting. The paper was well received and Dow Chemical representatives expressed special interest in the circuitry work and suggested a future presentation at their Midland, MI facility.

The SRS legal staff drafted a joint patent with the University of Florida on portions of this technology.

Economic Development Funding Program

J. L. Steele, J. T. Coleman, B. G. Kitchen, and W. M. Powell

The March 18, 1994 Stop Work Order from DOE is still in effect. No additional procurements are being awarded and no charges can be made against EDX activity codes until the review process is complete and authorization to proceed is given. The current focus is to provide DOE with requested information needed for decisions on TTP authorizations. This includes preparing project documentation and cost estimates that clarify local community impact, both short-term and long-term, for each potential TTP. In addition, Vendor Forum proposals were reviewed to assure that those appropriate to this program are considered for funding.

Sol-Gel Indicators

G. G. Wicks

This effort involves developing a new class of materials, sol-gel indicators (SGI), that can be deposited on substrates and used to conduct *in situ* environmental measurements on parameters like pH and determine the presence of metal ions like uranium or other species of interest. Team members and program PIs include R. Livingston (AAG), L. Baylor (ERAT), M. Whitaker (ERAT) and coordinator, G. Wicks (WERP), along with resources including P. O'Rourke and G. Hooker.

The Office of Technology (OTD) supported SGI team was a corporate winner of the George Westinghouse Signature Award of Excellence.

SGI team member, L. Baylor, represented the SRTC team. He presented an update of SGI activities during the OTD mid-year program review on April 18-19, 1994 in Denver, CO.

SGI-uranyl indicator compositions (three indicators, two sol-gel formulations) for uranyl response were formulated and tested.

Additional SGI-uranyl indicator compositions were prepared with modified sol-gel glass matrices with larger pore sizes (two indicators, 24 sol-gel formulations) by varying pH, alcohol/water ratio, and drying agent content.

To assess the ability of chemometrics to discriminate between response to uranyl and response to various interfering metals, arsenazo III was tested in an aqueous solution with seven different interfering metals. The response of arsenazo III to uranyl extended to longer wavelengths than the other metals and could be distinguished from these other components. If the sol-gel can be formulated to allow uranyl ions to interact with the arsenazo indicator more effectively, this system could provide a means for determining uranyl concentration, even in the presence of interfering metals.

Windows for flow cells were fabricated of SGI-arsenazo III for a more detailed assessment in a flow injection apparatus.

Sol-Gel Composites for Radiochemical Detection and Separations

G. G. Wicks

A new program involves developing SRTC sol-gel composites with special properties to improve separation and measurement capabilities of trace radioactive elements found in environmental samples. Team members and program Principal Investigators (PIs) include R. Livingston (AAG), L. Baylor (ERAT), M. Whitaker (ERAT), R. Sigg (AAG), and coordinator, G. Wicks (WERP).

The following summarizes current events.

- Subcontract with the University of Florida was approved and should begin next month.
- Final signatures are being obtained for the SCUREF subcontract with South Carolina State University.
- Negotiations and paperwork for a CRADA with W. R. Grace were completed and are awaiting DOE-SR approval.
- Capital projects paperwork was prepared and routed for authorization signatures.
- Our scope of work and agreed upon deliverables will be reviewed in light of a cut of \$150,000 from this project.
- This month, new experiments were initiated assessing impregnation and migration of various metal ions of different sizes in sol-gel matrices.

Mixed Waste Integrated Program

B. D. Helton and G. S. Bumgarner

The mission of the Mixed Waste Integrated Program (MWIP) is to develop enhanced final wasteforms and treatment for delisting mixed waste. The technical objective is to develop and characterize glasses proposed for mixed wastes and to demonstrate the vitrification processing of these glass chemistries. Program PIs include D. Bickford, C. Cicero, C. Jantzen, A. Kielpinski,

G. Ramsey, and R. Schumacher of Glass Technology; J. Marra and K. Imrich of Material Technology; and J. Whitehouse of Thermal Hydraulics. Activities supporting the MWIP at SRS are provided below.

Los Alamos National Laboratory (LANL) Simulated Sludge Crucible Studies

Product Consistency Test (PCT) results were returned for five of the LANL simulated sludge glasses. Using the theoretical composition of these glasses, they were all well below the normalized PCT results for the high level waste Environmental Assessment (EA) glass. Resource Conservation and Recovery Act metal releases were at detection limits. Glass characterization and toxic characteristic leaching procedure analyses of these glasses are ongoing.

Mixed Waste Integrated Program Mid-Year Review

A Mixed Waste Integrate Program (MWIP) mid-year review was held in Gaithersburg, MD, April 19-21, 1994. Participants from the SRS included D. Bickford, G. Ramsey, J. Marra, K. Imrich, C. Cicero, R. Schumacher, J. Whitehouse, J. Corey (EED), and D. Helton. MWIP PIs throughout the DOE complex were invited to attend. Discussions focused on the FY 94 program accomplishments and requirements for FY 95.

Molten Metal Meeting

A meeting was held on April 29, 1994, at SRTC with Molten Metal Technology, Inc. to discuss SRS's needs and related issues. Molten Metal Technology received a \$10 million Programmatic Research and Development Announcement contract to expand work on catalytic extraction processing (CEP) for DOE. CEP remediates hazardous and low-level radioactive waste by processing scrap metal containing radioactive contaminants. The company expects CEP will be used in other applications such as recovering valuable metals and remediating hard-to-treat mixed wastes.

Extended Sludge Processing Baselining Test Support

M. S. Hay and N. E. Bibler

Twelve Extended Sludge Processing (ESP) samples were received at SRTC for analyses during the recent Tank 51H two-pump test. During the first half of this test, two slurry pumps were operated for approximately 100 hours with both pump turntables rotating. Six dip samples were obtained and analyzed at SRTC. The anion concentrations in the supernate remained constant throughout the test indicating that no further mixing of the wash water with the more concentrated interstitial supernate in the sludge layer occurred. The total solids in the final sample reached 17.3 wt% (vs. 19.8 wt% in the initial four-pump test). In the second half of the test, the pumps were operated for an additional 100 hours with one of the pumps directionally fixed at a riser where sludge level soundings are taken. Six more dip samples were obtained and analyzed at SRTC. Again, the anion concentrations remained constant while the total solids reached 17.3 wt%. The analytical results of all twelve samples were transmitted to the customer within the 14 day time response target. The first ESP Baselining test sample from Tank 42 was received on April 21, 1994 with results being reported on May 3, 1994.

Some of the analytical results on solids from Tank 51H samples ESP-111A and ESP-112 have been received from ADS. The analysis of these samples is necessary because a sample of the sludge layer in Tank 51H was not obtained prior to start-up of ESP Baselining tests. The results of these samples will be used to verify the composition of Tank 51H solids used in modeling studies for predicting the amount of washing required for Batch 1.

Extended Sludge Processing Process Requirements

M. S. Hay

Process requirement for Extended Sludge Processing (ESP) are currently being developed. The first draft was sent out for review on schedule on April 15, 1994.

The target values for feed material sent from ESP to Defense Waste Processing Facility (DWPF) were calculated based on the feed design bases of DWPF (SRT-LWP-94-031). A previous document summarized target values for ESP based on an early draft of the design bases. The limits for ESP were calculated after proportioning the design basis value between in-tank precipitation (ITP) and ESP. A separate document will be issued that will document the rationale used to divide the feed limits between ITP and ESP. The target values for ESP may be exceeded without affecting the acceptability of the combined ITP and ESP feed if ITP has not used their full allotment for those targets, which were apportioned between ITP and ESP. Mechanisms for handling deviations from the ESP target values must be established that interface with ITP and DWPF. This document was generated in support of the development of ESP Process Requirements.

Tri-n-Butyl Phosphate Combustibility Studies

R. F. Swingle

At the request of High Level Waste Engineering, a study was initiated to study the combustibility of tri-n-butyl phosphate (TBP) and to determine under what, if any, conditions TBP might be expected to burn in Savannah River Site (SRS) waste tanks. Initial tests indicate that TBP is difficult to ignite, as its flash point of 181°C would indicate. Attempts to ignite TBP heated to 70°C were made under the following conditions with little or no success:

- pure TBP with a candle wick
- pure TBP in a pool
- various quantities of TBP over salt solutions
- TBP over salt solution with a burning potassium tetraphenylborate (KTPB) wick
- TBP over salt solution with n-butanol (TBP reduction of nitrate and nitrite in a fluidized-bed and packed-bed reactors. In a packed-bed reactor, the rate of nitrate and nitrite reduction decreases as the particle size increases because the specific surface area of the particles decreases. For a fluidized-bed electrode, there are two competing factors, specific surface area and effective specific resistivity of the discontinuous metal phase. The later factor dominates as the flowrate increases.

The memorandum Purchase Order for organics destruction testing was approved by DOE-SR and sent to DOE-Richland for concurrence this month. Work is scheduled to begin at PNL next month. Dr. J. Surma, Pacific Northwest Laboratory, is the Principal Investigator for this work.

Candidate waste streams were identified from the Hanford, SRS, Idaho Falls, and Oak Ridge sites. Compositions for these streams were obtained from contacts at each site. Information on nitrate destruction was provided to J. White of the U.S. Bureau of Mines and R. S. Richards of Associated Technical Consultants. Pretreatment of Hanford low-level waste to remove nitrate and nitrite is being considered for Phase II development to simplify the offgas treatment required for vitrification of the waste.

The simplified model of the electrochemical cell written in SPEEDUP is complete and compared with the more rigorous FORTRAN model. Both models give similar results and are being validated with experimental data. The evaporator model was linked to Properties Plus. Current tasks include:

- adding solids in the electrochemical cell and evaporator models
- adding a migration term in the SPEEDUP cell model
- performing an economic analysis and design of the evaporator system
- linking the more rigorous FORTRAN cell model to SPEEDUP

A flow-through porous electrode was assembled at the University of South Carolina. Bulk electrolysis experiments were initiated to evaluate different porous electrode materials and determine kinetic parameters for nitrate reduction.

General

SRTC Safety Support

S. P. Tinnes, R. C. Edwards, H. A. Ford,
P. K. Baumgarten, et al.

SRS Plutonium Vulnerability Study/Nuclear Materials Management EIS

Several planning meetings were held to begin work on the scope, methodology, and path forward for the WSRC program to support the required "SRS Plutonium Vulnerability Study/Nuclear Materials Management EIS". Site task teams are being formed for each key facility. The SRTC Technical Area team will consist of representatives from Laboratory Services (the custodian), the SRTC research organizations, and STD-PST.

The SRTC Technical Area Task Team began work to address/answer the draft "Facilities and Materials Question Sets" (i.e., Question Sets 1 and 2). Scoping work was performed to determine the program for addressing/answering the draft of "Vulnerability Question Set" (i.e., Question Set 3). It is estimated that the "SRS Plutonium Vulnerability Study/Nuclear Materials Management EIS" work will delay all SRTC 5480.23 SAR safety documentation activities by about two months.

5480.23 SRTC Safety Analysis Report Preliminary Hazard Assessment Program

To support the 5480.23 SRTC safety analysis report (SAR) Preliminary Hazard Assessment (PHA) program, the current SRTC Data Bank was sorted for safety-related initiators, ancillary support systems occurrences, chemical and radionuclide incidents involving personnel, and natural phenomenon events. Forty-five separate sorts were performed for each key SRTC Technical Area facility (such as 773-A, 735-A, 736-A, 776-A, etc.). In addition, sorts were performed for SRTC facilities outside the Technical Area (such as TNX, 777-10M, etc.). These sorts will be used to identify occurrences in specific facilities and individual laboratories, which will be a key input for the SRTC PHA.

SRTC 5480.23 SAR Task Plan (SRT-TML-94-8002)

Draft addenda for the SRTC 5480.23 SAR Task Plan (SRT-TML-94-8002; Task Leader—S. P. Tinnes) were prepared and are being reviewed for the following FY 94 work scopes:

- preparation of Chapter 2 ("Facility Description") for the 5480.23 SAR (Assistant Task Leader—R. A. Moyer, SAIC).
- preparation of the Preliminary Hazard Assessment (PHA) to support Chapter 3 ("Hazard and Accident Analysis") of the 5480.23 SAR. The original PHA was completed during the 1986-1987 period. The PHA must now be updated to reflect current and future missions for SRTC (Assistant Task Leader—R. A. Moyer, SAIC).
- preparation of a performance and systems analysis for the SRTC Ventilation System to support Chapter 3 of the 5480.23 SAR (Assistant Task Leader—H. A. Ford).

Chemical Information and Inventory System Download

The Chemical Information and Inventory System (CIIS) "Area Report" was modified to include downloads of Environmental Protection Agency Reportable Quantities, Threshold Planning Quantities, and the Occupational Safety and Health Act Threshold Quantity for chemical constituents. Inclusion of these quantities in the inventory reports will facilitate the review of chemical inventories during building walk-downs as to their importance in possible releases. The download to personal computer Filemaker Pro® is being tested and will be applied to Buildings 773-A, 773-2A, and 773-45A.

SRTC Operational Safety Requirements

C. L. Smith

SRTC Operational Safety Requirements (OSR) Revision Request 94-0001 was approved by WSRC on March 30, 1994 and transmitted to DOE-SR the same day for review and approval. DOE review and approval was requested by April 22, 1994. The proposed OSR revision

involves eight Limiting Conditions for Operation (LCOs) and associated bases concerning the ventilation exhaust systems and diesel generator fuel oil sampling. A meeting was held on April 14, 1994 with DOE-SR, Halliburton NUS, the facility and PSTS personnel to discuss the review scope and basis. HNUS is preparing the Validation Package for DOE. Another meeting was held on April 21, 1994 with DOE and the facility to discuss DOE comments. The comments were administrative; no technical issues were identified. Documentation of the information discussed in the meeting was prepared and submitted to DOE on April 22, 1994. Although DOE approval was not provided by April 22, 1994 efforts continue to obtain this approval to minimize impact on facility operation.

The SED LCO and associated bases are ready to be issued as Revision 1 upon facility notification. The SED LCO may be incorporated with the LCOs in OSR Revision Request 94-0001 for issuance.

PhD Staffing

J. Hammett

The Oversight Committee gave SRTC the approval to hire ten PhDs for critical needs. As of April 29, 1994, SRTC received four acceptances: a radiological chemist and a mass spectroscopist for ETS, a senior chemist for ADS, and a chemist for IWTS. Presently, SRTC is waiting for responses to four open offers and are interviewing for two other positions. A status report was issued and reviewed by C. Thiessen.

PMICS Implementation within SRTC

P. W. Doolittle

Budget Module

The Budget Module is being modified to conform with site changes expected for the 1995 AOP Budge Call. These modifications will be

made for SRTC, ALD, and EDD budget development and input. A hard dollar cost savings proposal is being generated to substantiate considerable savings in the overall budget development process by using the PMICS Budget Module.

SIPCM represented SRTC on restructuring initiatives and task teams dealing with site standardization of the AOP development process.

AOP Baseline Variance Module

Monthly cost variance reports are being generated from the PMICS AOP Baseline Variance Module to aid the programs analysts/financial liaisons in determining cost schedule variances for a given section, organization, WBS, or activity code. A hard collar cost savings proposal is being generated to substantiate a considerable savings in effort currently expended in requesting, compiling, and analyzing financial data by using the PMICS AOP Baseline Variance Module.

Discrepancies in the monthly tritium (WBS 1.03) IBARS financial statements, as a result of the tritium clearing rules for GB and GE, undermine the actual performance of SRTC on affected programs. This problem is being addressed within PMICS to ensure that all costs associated with tritium programs are accurately reflected against the AOP spendout plan for GB. GE distributions will also be summarized and reported.

Project Management Integration and Costing System (PMICS Module)

Training of each section's programs analyst/financial liaison on the fundamental use of PMICS's scheduling utilities and overall project management concepts was completed. Proficiency tests for use of this utility are scheduled to be completed during the week of April 25, 1994. Advanced training in using the resource allocation and costing/budget utilities is planned for the first week of May 1994.

PMICS orientation presentations were held for all groups in the Environmental Sciences Section.

PMICS—Site Interface

P. W. Doolittle

LSS (SSD) approached SIPCM to assist in developing a programmatic control tool for use in scheduling and controlling LSS operations. Subsequent meetings with F. Grimm and J. Hendrix indicated that Primavera was obtained for use in scheduling WMS activities as part of an ongoing site initiative. Further evaluation of the WMS interface requirements will be conducted to determine the applicability of PMICS for this effort on a sitewide basis.

SIPCM generated an integrated schedule at the request of LSS (SSD) for its SED Tank Removal program. The schedule is being reviewed and modified before LSS formally issues it.

With the introduction of the Primavera scheduling software, a decision should be made as to whether or not to transfer the PMICS Tank Removal Program to the LSS Primavera schedulers.

Westip Cost Estimating Task Team

P. W. Doolittle

SRTC/SIPCM is actively involved in a Westip effort to standardize the cost estimating practices across the site. This effort will involve issuing a Cost Estimating Manual as part of the overall Budget Formulation/Development Procedure required for cost/budget validation. A draft procedure was issued for site review and approval.

This task team is also addressing the various disciplines involved with programs control management (WBS structure, AOP development, integrated planning and scheduling, cost estimating and budget development, performance variance analyses, and reporting concerns), as they are inter-related. Some issues are being addressed as part of the site restructuring initiatives and efforts from this task team should carry over into more specific committees.

Items of Interest

On March 8–11, 1994, M. R. Buckner attended the International Policy Forum: Management and Disposition of Nuclear Weapons Materials Conference in Leesburg, VA.

On March 24, 1994, R. E. Pevey participated in PhD orals for J. Malo at the Georgia Institute of Technology, Atlanta, GA.

On March 24, 1994, M. R. Buckner led the EMAC SPC/SPM User's Group meeting in Atlanta, GA. He was recently appointed Chairman. The group includes members from each Westinghouse Business Unit.

On March 27–30, 1994, R. L. Reed participated in the Plutonium Vulnerability Assessment Working Group Kick-Off Meeting in Gaithersburg, MD, as part of the Nuclear Material Disposition Project.

M. D. Wise received the Rothamsted Award for Excellence in Statistical Research from the Brigham Young University Department of Statistics. The Rothamsted Award is given "to graduate students who exemplify the work of [R. A.] Fisher at Rothamsted [Experimental Station]".

A technical review of support program for the in-tank precipitation and Late Wash facilities was held on April 18, 1994.

Presentations

M. S. Hay and N. E. Bibler, of Defense Waste Processing Technology, attended a DOE-sponsored High-Level Waste Sludge Washing Workshop in Richland, WA where they presented data on sludge characterization, aluminum dissolution, and washing experiments conducted at SRTC. The purpose of the workshop was to bring research staff from various DOE sites together to discuss the current level of knowledge on sludge processing, identify gaps or overlaps in current research, identify common needs and problems, and develop recommendations for program managers on future directions and funding of such work.

"Criticality Assessment of Initial Operations at the Defense Waste Processing Facility" by B. C. Ha and T. G. Williamson, was presented at Waste Management '94 Waste Management Symposia in Tucson, AZ on March 3, 1994.

Publications

M. J. Barnes, J. P. Bibler, and M. L. Meyer published, *Trip Report for the Underground Storage Tank—Integrated Demonstration (UST-ID) Mid-Year Technical Review in Oak Ridge, TN (U)*, SRT-LWP-94-043, on April 11, 1994.

M. J. Barnes published, *ITP Salt Solution Calculations (U)*, SRT-LWP-94-047, on April 22, 1994.

R. F. Swingle published, *Task Quality Assurance Plan to Evaluate the Effects of TBP and its Byproducts on Benzene Removal from ITP Salt Solutions (U)*, WSRC-RP-94-311, on March 21, 1994.

M. S. Hay and N. E. Bibler published the following reports:

Analytical Report for ESP Support Sample SS3-ESP-114 and 114A (U), SRT-LWP-94-051, on March 22, 1994.

Analytical Report for ESP Support Sample SS3-ESP-115 and 115A (U), SRT-LWP-94-044, on April 11, 1994.

Analytical Report for ESP Support Sample SS3-ESP-116 and 116A (U), SRT-LWP-94-045, on April 11, 1994.

Analytical Report for ESP Support Sample SS3-ESP-117 and 117A (U), SRT-LWP-94-057, on April 19, 1994.

Analytical Report for ESP Support Sample SS3-ESP-118 and 118A (U), SRT-LWP-94-058, on April 19, 1994.

Analytical Report for ESP Support Sample SS3-ESP-119 and 119A (U), SRT-LWP-94-059, on April 19, 1994.

M. S. Hay published, *Target for Extended Sludge Processing Feed to DWPF Based on the DWPF Feed Design Bases (U)*, SRT-LWP-94-031, on March 22, 1994.

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