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Technical Direction and Laboratories

Fiscal Year 1999 Annual Report

Prepared for the U.S. Department of Energy
Assistant Secretary for Environmental Management

Project Hanford Management Contractor for the
U.S. Department of Energy under Contract DE-AC06-96RL13200

Fluor Hanford
P.O. Box 1000
Richland, Washington

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Technical Direction and Laboratories

Fiscal Year 1999 Annual Report

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J. Y. Bourges
B. A. Crawford
D. L. Herting
J. R. Jewett
L. L. Lockrem
E. Tchemitcheff
G. L. Troyer
R. S. Viswanath
W. I. Winters
Numatec Hanford Company

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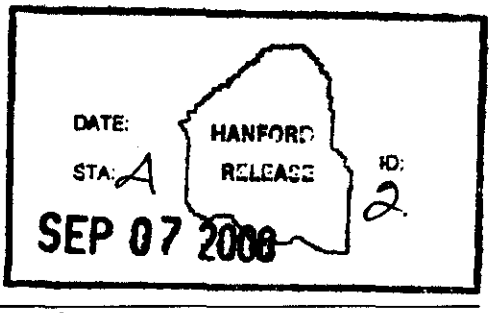
Fluor Hanford
P.O. Box 1000
Richland, Washington



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NHC
Numatec Hanford Corporation
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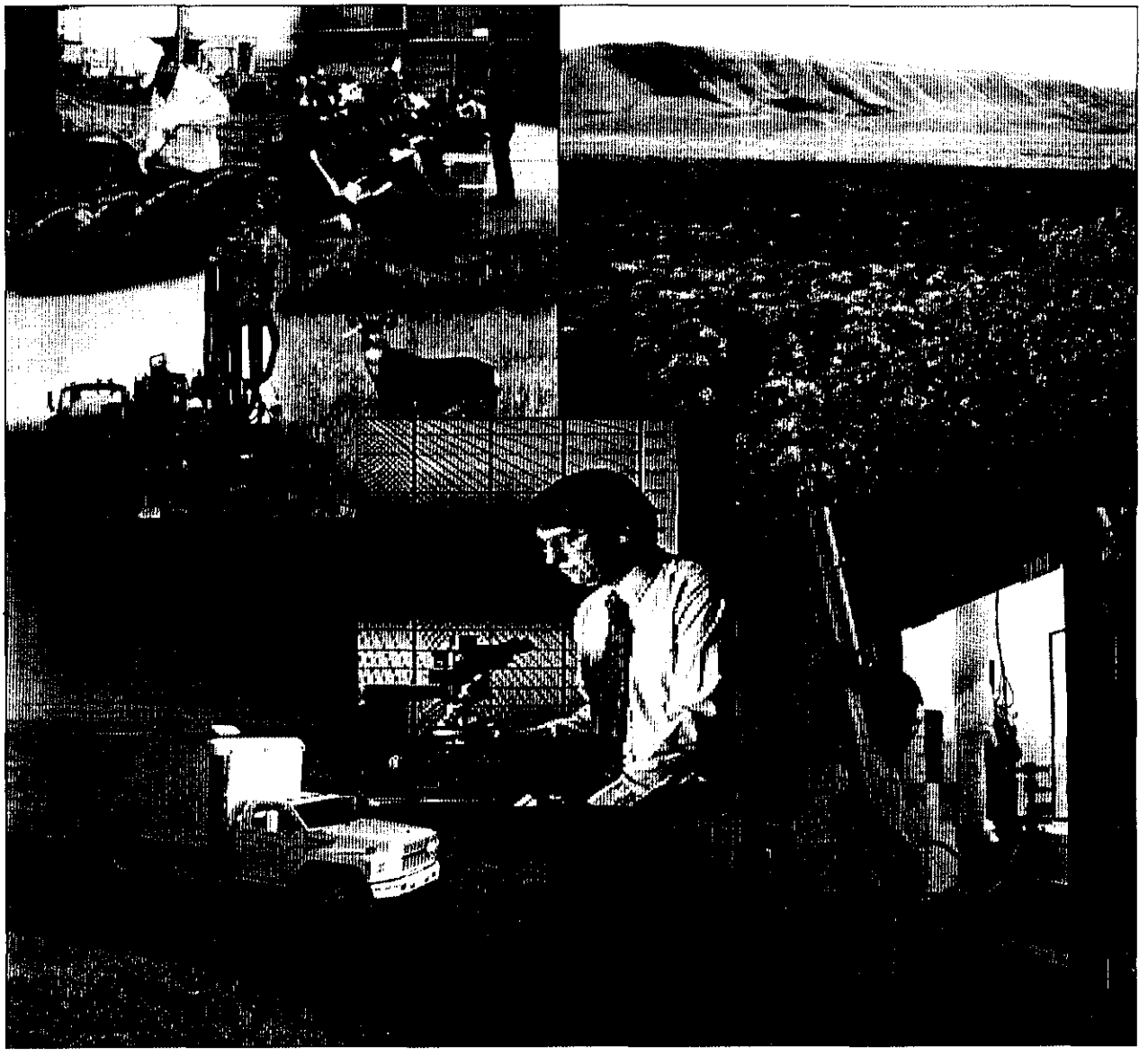
1999 ANNUAL REPORT

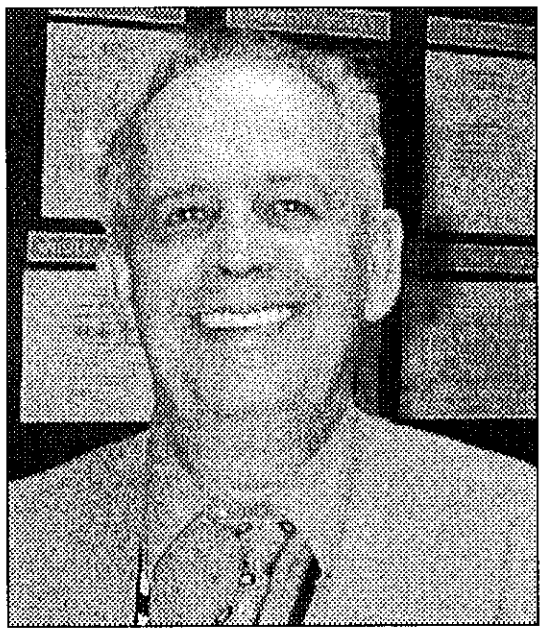
NUMATEC HANFORD CORPORATION

TECHNOLOGY, OPERATIONS AND PROCESS SCIENCE



Dr. Jacques Bourges Contribution to Hanford:

Technology, Operations and Process Science would like to acknowledge Dr. Jacques Bourges' contribution to the Hanford Site for his technical leadership and unconditional support. Dr. Bourges has 30 years at the French Atomic Energy Commission in Actinide Chemistry and Separation Process in high Radioactive Medium and Spent Fuel Processing. He has served in various management positions at (Institute of Atomic Energy) CEA Fontenay-aux- Roses, CEA/Marcoule. He was honored in 1997 by a special award for outstanding accomplishments in converting weapons-grade plutonium to civilian use.



Dr. Jacques Y. Bourges

Dr. Bourges joined the Project Hanford Management Team (PHMC) in December 1996 and managed the Numatec Hanford Corporation's analytical organization until his retirement in September 1999. Dr. Bourges continuous support was an invaluable asset to the success of Analytical Services and Process Chemistry.

EXECUTIVE SUMMARY

In calendar year 1999, Numatec Hanford Corporation (NHC) was responsible for introducing and implementing new technologies and providing technical assistance to the Project Management Hanford Team (PHMC), composed of the integration contractor and its subcontractors. Within NHC, the Technology, Direction and Laboratories (TD&L) group was matrixed with Waste Management Federal Services of Hanford, Inc., (WMH), operate and provide program direction to the analytical laboratories through the management structure of Hanford Analytical Services (HAS). The TD&L group was responsible for the following: introducing technologies to improve the cost efficiency in the laboratories, providing technical support to laboratory operations, interfacing with laboratory customers on technical issues, providing analytical services not available through the production laboratory operations, and providing bench scale process chemistry tests and demonstrations. NHC personnel were co-located in the radiological laboratory facility and are available to provide process chemistry and analytical services on highly radioactive samples from the programs on site. In addition, one component of the TD&L group provided in-situ and at-line chemical analyses and sampling services.

The TD&L group was composed of three operating units: Process Chemistry, Analytical Technology, and Special Analytical Support (SAS). Each had unique and complementary capabilities.

The Process Chemistry team provided bench scale testing and consultation to support chemical process development and trouble shooting. The Analytical Technology team provided technical assistance for routine analytical data generation for WMH laboratory operation, designated facility engineering functions, and new analytical chemistry technology development and implementation. The SAS team provided and maintained portable analytical measurement systems. Measuring capabilities were normally installed in trucks and trailers for easy mobility to the sampling or process location.

In April 1999, TD&L supported consolidation and transfer of field laboratory capabilities to minimize the cost impact of services to the Hanford Site. The SAS organization was transferred to Waste Management Laboratories. SAS personnel and workscope were reassigned to the Waste Sampling Characterization Facility (WSCF) within the Hanford Analytical Services (HAS). The TD&L group worked with program managers and project engineers to effectively provide support to this transition.

Also in April 1999, TD&L consolidated Process Chemistry and Analytical Technology into a new organization, Technology, Analysis and Process Science (TAPS). WMH further consolidated activities in October 1999 by adding project coordination to the TAPS organization. The organization is now named Technology, Operations and Process Science (TOPS).

The text of this annual report summarizes the technologies supported during FY 1999. The names of individual contributors are included so that technical questions can be directed to them. Primary achievements and issues are summarized below.

Technical Direction and Laboratories Group Fiscal Year 1999 Achievements:

- Organized teams and established lines of communication and responsibilities with primary laboratory customers.

- Assisted WMH/HAS to integrate analytical services with site programs by conducting technical workshops to identify new capabilities and by interfacing routinely with these laboratory customers on technical issues.

- Introduced technologies to improve laboratory cost-efficiency and test methodologies to apply to in-situ measurements. Technologies that have been successfully tested include the gamma detector and x-ray fluorescence system for use in cone penetrometer deployment into the vadose zone.

- Met cost and schedule commitments supporting program performance agreements. The HAS team completed 56 vapor sampling and analysis events for tank C-106 tank headspace analyses below estimated cost and ahead of estimated schedule. SAS established the analytical baseline for transuranic headspace program.

  The Process Chemistry team completed studies for retrieval, salt dissolution, interim stabilization and Waste Integration Team (WIT) programs. The Analytical Technology team completed the demonstration of a neutron activation sodium assay method and organic support for the low-activity waste (LAW)data quality objective (DQO).

Technical Direction and Laboratories Vision for Fiscal Year 1999

In FY 1999, the TD&L group addressed the challenge of incorporating commercial practices into the laboratories. Competition for funding from the reduced site budget increased. At the same time, the organization adapted technologies to meet new program needs. The team undertook the following improvement actions:

- Lead in implementing new technologies required to meet new customer measurement requirements for HAS. The primary laboratory customer programs changed dramatically during FY 1998 and 1999 with the restructure of the River Protection

Project (RPP). Other program needs were identified and supported by the PHMC laboratories. A proactive effort will be necessary to continue improvement of communications with project personnel of the new programs. The HAS laboratories demonstrated greater flexibility and capabilities in line with the direction of laboratory customers and minimized workscope leaks to competitor laboratories.

- Actions for improvement required reorganizing the HAS laboratories with involvement from personnel at all levels of the organization, clear accountability at all levels, and increased professional development of personnel.

- Consolidated fixed and field laboratory capabilities to minimize the cost impact of services to the site and maintained sufficient core competencies to meet customer program needs in new technologies and services. Laboratory personnel worked with program managers and project engineers to effectively provide appropriate service.

- Collaborated with the Pacific Northwest National Laboratory (PNNL) to effectively transfer developed technologies from research to routine operation.

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LIST OF TERMS

ASP	Analytical Services Project
CENTRC	capital equipment not related to construction
CBC	Columbia Basin College
DWPF	Defense Waste Processing Facility
DOE-RL	U.S. Department of Energy –Richland Operations
DQO	data quality objective
EPA	U.S. Environmental Protection Agency
FY	fiscal year
GC/MS	gas chromatograph/mass spectrometer
HAS	Hanford Analytical Services
HLAN	Hanford Local Area Network
HLW	high-level waste
HEDTA	N-hydroxyethlene diaminetriacetic acid
ICP/AES	inductively coupled plasma atomic emission spectrometry
ICP/MS	inductively coupled plasma mass spectrometry
LA/MS	laser ablation mass spectrometer
LAW	low-activity waste
LIMS	Laboratory Information Management System
LMSI	Lockheed Martin Services, Inc.
LOI	Letter of Instruction
ORNL	Oak Ridge National Laboratory
PCB	polychlorinated biphenyl
PFP	Plutonium Finishing Plant
PHMC	Project Hanford Management Team
PNNL	Pacific Northwest National Laboratory
QA	quality assurance
PRSST	Propagating Reactive System Screening Tool
PUREX	Plutonium-Uranium Reduction Extraction (facility)
RPP	River Protection Project
ROI	return on investment
RSST	Reactive System Screening Tool
SESC	SGN Eurisys Services Corp.
SRL	Savannah River Laboratories
TAPS	Technology, Analysis and Process Science
TTP	Technical Task Plan
TCR	Tank Characterization Reports
TD&L	Technical Direction and Laboratories
TFA	Tanks Focus Area
TIC	tentatively identified compounds

TOC	total organic carbon
TOPS	Technology, Operations and Process Science
TRU	transuranic waste
TTP	Technical Task Plan
TWRS	Tank Waste Remediation Systems
VOA	volatile organic analysis
VOC	volatile organic compounds
WIPP	Waste Isolation Pilot Plant
WSCF	Waste Sampling and Characterization Facility
WIT	Waste Integration Team
WSDOE	Washington State Department of Ecology
WSU	Washington State University

1.0 INTRODUCTION

This annual report summarize achievements and list reports issued by members of TD&L, NHC group during Fiscal Year (FY) 1999, (October 1, 1998 through September 30, 1999). This report, issued by this organization, describes work in support of the Hanford Site and other U.S. Department of Energy, Richland Operations Office (DOE-RL) programs. It includes information on the organization make-up, interfaces, and mission of the group.

The TD&L is a group of highly qualified personnel with diverse disciplines (primarily chemistry specialties) that provide process, analytical, and in-situ chemistry services to engineering customers. This year of operation and interfaces with other contract organizations consumed considerable administrative efforts. Attention was directed to the technical challenges presented by the changing roles, responsibilities, and priorities of Hanford programs.

2.0 ORGANIZATION AND MISSION

NHC is responsible for introducing and implementing new technologies and providing technical assistance to the PHMC, composed of the integration contractor and its subcontractors. Within NHC, the TD&L group is matrixed with WMH to operate and provide laboratory services. The TD&L group is responsible for the following: introducing technologies to improve cost efficiency in laboratories, providing technical support to laboratory operations, interfacing with laboratory customers on technical issues, providing analytical services not available through production laboratory operations, and providing bench scale process chemistry tests and demonstrations. NHC personnel are co-located with WMH, in the radiological laboratory facility and are available to provide process chemistry and analytical services on highly radioactive samples from programs on site. In addition, one component of the TD&L group provides in-situ and at-line chemical analyses and sampling services.

Technical Directions and Laboratories Mission

After the consolidation of SAS with WSCF and the merger of Process Chemistry, Analytical Technology of NHC with Laboratory Information System (LIMS) and Analytical Production of WMH, the new organization, Technology Operations and Process Science (TOPS), established the following charter.

Charter

The 222-S Laboratory - TOPS organization provides expertise in 1) process chemistry, 2) analytical chemistry and 3) data and project management in support of the Analytical Service Project (ASP) customers. The mission of the organization is to investigate new chemical processes, analytical technologies and data/project management systems and to provide direct support to ASP in the implementation and performance of these areas of expertise.

Responsibilities

Technology, Operations and Process Science

1) Manage assigned laboratory activities in a manner that ensures excellent technical results, timely completion of commitments, safe and effective use of resources, environmental compliance and customer satisfaction.

2) Actively participate in program technical planning to ensure alignment between customer chemistry and production requirements and ASP capabilities.

3) Provide chemistry expertise in support of program planning, technical studies, and laboratory operations.

4) Comply with all DOE-RL directives, federal and state regulations and Project Hanford requirements while meeting customer and public expectations.

5) Provide technical assistance to ASP in achieving higher laboratory production, safety and quality goals.

6) Lead and coordinate technology interface programs with other DOE-RL sites, university programs and international communities.

Process Chemistry

1) Design and conduct studies to understand chemistry and chemical behavior of waste materials.

2) Perform and document process engineering (bench-scale) studies in the evaluation of existing and new processes.

3) Develop and perform special physical and characterization measurements for customer requirements.

4) Interface with programs and provide chemistry expertise in the design of studies and the evaluation of results.

5) Develop and implement special sample handling and testing systems for hot cells and other laboratory sample handling configurations.

Analytical Chemistry

1) Develop, test and implement new and/or improved measurement technologies for radiochemical, inorganic and organic constituents to support ASP laboratories and other Hanford programs.

2) Perform special analyses of samples that are not amenable to existing ASP procedures or operations.

3) Develop hot cell procedures and provide direct analytical chemistry support to laboratory operations groups (hot cells, radiochemistry, inorganic chemistry and organic chemistry).

4) Provide analytical technical consulting, document reviews and technical support to Hanford Site Programs including the ASP laboratory operations.

Washington State University and Columbia Basin College Liaison

1) Develop mutually acceptable technical courses that can be offered to Hanford Site's technical staff.

2) Promote interactions between Hanford staff and university personnel.

3) Promote development of innovative chemical methodologies supporting Hanford Site needs.

4) Develop material examination technology program for community college vocational program.

Data and Project Management

1) Implement an analytical project management business strategy for a diverse set of customer requirements utilizing Project Coordinators to ensure that customer schedule, and data quality requirements are achieved.
2) Generate high quality and timely data reports for customers and data users.
3) Develop performance metrics for each laboratory method as the basis for fully integrating analytical work scheduling with the ability to track and trend analytical production.

4) Develop an integrated schedule that incorporates the resources of Operations, Radiological Control, Maintenance, Engineering, Generator Services, Analytical Services and support functions.

Provide database administration and software development for the LIMS with a software platform that operates efficiently with minimal down time.

5) Develop analytical data report format generation for a variety of customers and data users.

6) Provide computer software and hardware expertise to the ASP organization and act as the primary interface to LMSI on Hanford computer related programs.

8) Utilize data and project management tools to implement continuous improvements to increase quality, timeliness and cost effectiveness of the organization.

In the matrixed role of technology development and implementation contractor for the Hanford Analytical Laboratories, TD&L identifies the capabilities required by Hanford Site programs that are not available in the laboratories; and it evaluates commercial, foreign, and research contractor offerings. Technologies are evaluated for applicability to program need and for practicality of implementation in a production laboratory. Selected technologies are purchased; tested on nonradioactive materials for precision, accuracy, and operability with radioactive samples; and documented for performance. Laboratory personnel are trained if a capability is to be transferred to WMH, and to be used for routine analyses on program samples. The personnel of TD&L may continue to perform tasks if samples are infrequent, require excessive time to perform, or require specialized expertise.

The TD&L organization, managed by Terry A. Flament, was composed of three operating units: Process Chemistry, Analytical Technology, and HAS. Each group has unique and complementary capabilities and a mission.

3.0 TECHNICAL DIRECTION AND LABORATORIES BASES OF OPERATIONS

3.1 TD&L Operating Units

3.1.1 Process Chemistry

The Process Chemistry team provides bench scale engineering and consultation assistance. The team is composed primarily of physical, inorganic, and general chemists and chemical engineers. This mix of disciplines provides process flowsheet evaluations and laboratory scale testing for diverse program needs. Evaluations frequently require experimental data generated by simulating operating conditions and using actual samples of radioactive materials. Laboratory technicians, highly skilled in laboratory techniques and building operations, perform much of the information gathering. The chemist/engineer in charge of each project provides instructions in the form of test plans. These plans, which are formalized documents, allow changes when

testing dictates. Letter or formal document to the program requesting the services documents test results and evaluations.

3.1.2 Analytical Technology

The Analytical Technology team provides technical assistance for routine analytical data generation for WMH laboratory operations, designated facility engineering functions, and new analytical chemistry technology development and implementation. This diverse team provides nonroutine analytical services and consultation for programs when special requests are received before a capability is transferred to the production laboratory operation. The formal education of staff members includes degrees in inorganic, analytical, and general chemistry and computer science. A laboratory technician, with expert skills in analytical laboratory operations and techniques, assists project leads in developing new technologies and provides nonroutine services. Facility engineers support the maintenance of the laboratory systems and the safety envelope. The team makes significant contributions during the preparation of analytical requirements in program planning documents.

3.1.3 Special Analytical Support

The SAS team provides and maintains portable analytical measurement systems. Measuring capabilities are normally installed in trucks and trailers for easy mobility to the sampling or in-situ measurement site. These systems are largely self-contained, but they also can be connected to distribute electrical services, if available. Capabilities exist for performing organic and inorganic constituent concentration analyses of gases, inductively coupled plasma atomic emission spectroscopy, radiochemistry and nuclear spectroscopy measurement systems and can be deployed to program-specified locations. Scanning electron spectroscopy with X-ray fluorescence and Mossbauer spectroscopic systems are available in a fixed laboratory configuration although plans have been made to install these systems in a portable configuration for greater accessibility.

The personnel who perform this analytical support are contracted from SGN Eurisys Services Corp., an NHC sister company under the PHMC contract. Although a diverse mix of chemistry and geochemistry disciplines is routinely used, other disciplines can be contracted to respond to specific program sampling and measurement requirements. The work of this team is coordinated and contracted by the manager who reports to the TD&L group.

The teams, aligned according to scientific disciplines, participate in nationally recognized standards and performance assessment programs including the following:

- Proficiency Environmental Testing Program to meet the requirements of ISO/REMCO N263, the protocol for Proficiency Testing of Analytical Laboratories.

- Washington State Department of Ecology (WSDOE) Environmental Laboratory Accreditation for field analysis at the Hanford Site. Only two other mobile laboratories have this accreditation; they are located in Seattle and specialize in organic analysis.

3.2 WORKSCOPE

Program requirements for analytical or process chemistry work are usually defined in planning documents. Commitments for extended service are developed by programs using the seven-step data quality objectives (DQO) process. In this process, a program defines the processes to be used, develops a listing of measurements believed to be necessary to monitor the process, and evaluates the listing by testing the consequences of erroneous decisions. Specific measurement criteria are established, and the product is documented. Personnel from TD&L have been active in negotiating certain program DQOs. Although laboratory capabilities are not of primary importance while establishing measurement criteria, the laboratories must recognize the program requirement for additional capabilities early enough so that time is available for acquiring the capability before laboratory support is requested.

Other means used to direct activities of the laboratory include the following: 1) a letter of instruction, 2) a test plan usually negotiated by TD&L and program personnel, 3) a program plan that defines the objectives and criteria to be met, and 4) a task order that delineates the required level of service and where the technical criteria will follow. Each of these means requires statements describing the work to be done. The work is usually for short term, single-use type activities. When the statements are received, TD&L management assigns the work to the team with expertise in the discipline required.

3.3 ADMINISTRATIVE SUMMARY

The TD&L group liquidates by charging Hanford Analytical Services customers for performing analytical, process chemistry, field sampling, and analyses. Most cost recovery is by direct charges for labor and materials. This year of operation, TD&L overhead costs represented approximately 14% of the total TD&L budget. The remainder of the approximately $6 million is charged directly to supported programs for services provided.

Approximately 70 percent of the direct program funding was received from Tank Waste Remediation System (TWRS) projects. The major contributors were Tank Characterization, Retrieval and the Transuranic Project. All TD&L teams provided non-overlapping support for these customers. The laboratory support for staging, pretreating, and characterization of feed material for privatized vitrification contractor(s) increased in FY 1999, and it will be a major portion of the FY 1999 and FY 2000 RPP project funding.

Limited development of instrumentation and measurement technologies occurred during the year. Although there are many areas, which can benefit from improvement, funding has been reduced so that only the highest priority areas can be pursued. These are generally new capabilities necessary to provide an immediate service to the program customer. Staff reductions across the site also have reduced the personnel available for development activities.

Development Area for Fiscal Year 1999

The development activities to support of the River Protection Project (RPP) in FY 1999 included the following: 1) continued evaluation of three methods for performing sodium assay analyses on LAW in support of waste transfers to the Privatization contractor, 2) development of further inductively coupled plasma/mass spectrometry (ICP/MS) applications for elemental and isotopic analysis to support the LAW/HLW DQO requirements, 3) development of organic analysis methods for analysis of highly radioactive wastes and 4) more rigorous sludge dissolution procedures. Work on implementing hot cell rheology and sample preparation procedures were also performed.

4.0 TECHNICAL DIRECTION AND LABORATORIES TECHNICAL ACTIVITIES

4.1 INTRODUCTION

Members of all TD&L teams performed additional tasks as a joint effort. Although these are not solely developmental, they represent part of the overall mission of the NHC. The task of identification of technology needs and support to WMH was supported in various alignment meetings.

4.1.1 Identification of Technology Needs

A series of alignment meetings were held with technical and management representatives of WMH's analytical services. The capabilities of the TD&L teams were presented, program needs were identified, timeframes for capabilities were established, and a determination was made as to whether development funding was included in baseline

determination was made as to whether development funding was included in baseline funding documents. The alignment meetings identified the need for new laboratory capabilities, funding capital equipment not related to construction (CENTRC) vision and mission within analytical services.

4.2 PROCESS CHEMISTRY

The following summaries report the major activities performed by members of the Process Chemistry team of TD&L. This team was managed by J. R. (Jim) Jewett, PhD; one chemical engineer, J.F. O'Rourke; five chemists, D.B. Bechtold, PhD; M.A. Beck, D.L. Herting, PhD; B.E. Hey, and J.C. Person, PhD; and four technicians, J.W. Chenault, D.W. Edmonson, L.A. Guerra, J.R. Smith and one secretary, F.J. Hurley.

4.2.1 Physical Property Measurement

Developed and qualified analytical procedure (LA-519-105) for performing viscosity and shear strength measurements in the 11-A/3 Hot Cell. This procedure was used for rheological analyses on samples from tanks 241-S-111, 241-U-109, and 241-SY-102.

Eighty-seven viscosity, nineteen-shear strength, and thirteen particle size distribution measurements were performed at the 222-S Laboratory.

Performed Polarized Light Microscopy analysis on samples from tanks BY-102, AX-104, and AW-101.

Wrote Percent Solids Analysis procedure for WIT DQO.

4.2.3 Retrieval Program

Completed a laboratory study on samples of waste from tank 241-AN-107. This study evaluated the amount of dilution required for efficient removal and transfer by pipeline of the waste from the underground waste tank for immobilization. Measurements included viscosity, density, and percent solids at 10 °C and 30 °C.

Completed laboratory testing associated with the Tank 241-C-104 Retrieval Study. This study provides the data required to develop engineering plans for sluicing and pumping operations for Tank 241-C-104.

Laboratory dilution testing of waste from tank 241-AW-101 was completed, supporting the RPP Retrieval program. Test plans and procedures were also issued for retrieval testing of waste from tanks 241-C-104, 241-AN-107 and 241-AZ-102.

4.2.4 Tanks Focus Area (Saltcake Dissolution)

The Saltcake Dissolution task funded by the Tanks Focus Area (TFA), DOE EM-50, completed its second year of funding in CY 1999, and entered its third year. Technical Task Plan (TTP) Number RL0-8-WT-41 was revised and re-issued for FY 2000. Approximately one-half of the funding supports analytical work by WMH.

The TFA Work scope during CY 1999 included five sub-tasks. Saltcake dissolution tests were completed for tanks A-101 and S-102 (sub-tasks 1 and 2). Solids characterization was completed on BY-102 saltcake (sub-task 3). Sequential dissolution tests designed to mimic in-situ retrieval operations were completed on BY-102 saltcake (sub-task 4). Feed precipitation tests (sub-task 5) were completed on supernatant liquid mixtures from a variety of tanks.

A work/study position through Columbia Basin College was utilized to support Process Chemistry laboratory testing. This support was provided for six months.

4.2.5 Interim Stabilization

Performed a mixing study on samples from Tanks 241-U-105 and 241-SY-101. This study determined these two waste types could be mixed without causing deleterious reactions.

Laboratory tests were done to support saltwell pumping from tanks 241-SX-104 and 241-AX-101. Tank farm operations had experienced pipe plugging during attempted transfers from SX-104. The reason for the plugging was identified, and the amount of dilution required to prevent the plugging was established. Pumping and transfer of waste from SX-104 to SY-102 was completed without further incident. Pumping and transfer of waste from AX-101 is scheduled for spring 2000.

Dr. D.L. Herting was a participant in the Tank SY-101 Risk Assessment Workshop. The purpose of the workshop was to plan a path forward for mitigating the waste level growth in the tank. A letter of appreciation was received after the workshop from R.E. Raymond, SY Farm Project Lead. A "white paper" on SY-101 dissolution kinetics was issued May 10, 1999. The first campaign to transfer waste from SY-101 to SY-102 and back dilute the waste remaining in SY-101 was completed in December 1999.

Performed AW-102 boildown tests to support 242-A Evaporator Operations.
Performed boildown tests on a composite of SY-101 and SY-102 liquids to support planned mitigation of SY-101 crust growth.

Performed waste feed boildown tests to support Effluent Treatment Facility.

The boildown procedure was revised and updated.

4.2.6 Waste Integration Team Program

Worked with customer to develop a plan for the dilution testing of tank 241-SY-101 waste. There were a large number of tests on a very aggressive schedule, with the schedule especially tight for the report of preliminary results (the February 8, 1999 Letter of Instruction (LOI) required this report by April 22, 1999). This deadline was met, including preparing the Test Procedure, performing the tests, extracting useful information from the laboratory analyses, and preparing the report.

Performed solubility-screening tests on waste samples from tanks AN-107 and AW-101. These tests are in support of the requirements of the Privatization DQO.

Presented an invited paper at the Gas Generation in Solid-Phase Nuclear Materials and Matrices Symposium at the 1999 Winter Meeting of the American Nuclear Society, with more than 50 attendees. The session chairman thanked Dr. J.C. Person for the quality of his presentation. Published an extended abstract of this paper and was co-author on a second paper at the same symposium.

4.2.7 324 B-Cell Facility

Input was provided for writing the Letter of Instruction (LOI) for 324 B-Cell Waste Characterization. A test plan was formulated and executed to determine contamination levels on waste metal coupons from B cell. Technical consultation was provided for seven meetings between WMH and the customer for recovery from issues associated with the B-Cell campaign. This support included performing sample preparation and evaluating the curvature of the contaminated metal. Designed and executed a study evaluating the relative effectiveness of eleven methods for removing a fixed contamination layer from contaminated metal. The results of the study were used to design the treatment method for the contaminated metal.

4.2.8 Spent Nuclear Fuels (SNF)

A task supporting Spent Nuclear Fuels Processing was completed in FY 1999 by issuing one supporting document report to guide process testing of sludge residue leaching, and by presenting a co-authored paper on this topic at the 23rd Annual Actinide Separations Conference.

Two revisions of a sample-processing plan were accomplished to support a large campaign of SNF K-Basin sludge characterization. Thirty-three large samples were successfully received into a hot cell, consolidated, and composited into water and sludge samples. The sludges were subjected to video-recorded settling studies and then aliquotted for further lab analysis. The RSST (Reactive System Screening Tool) method was resurrected with a new Laboratory Technology procedure in order to subject several sludge aliquots to self-heat calorimetry.

4.2.9 Miscellaneous

X-ray diffraction instrumentation was brought on-line at 222-S for the first time in several years. Samples of BY-102 saltcake were analyzed to support TFA program testing.

A test plan for Insoluble Anions Analytical Development was drafted and approved to conduct caustic fusion experiments leading to improved RPP waste tank solids analytical capability to support the privatization DQO.

Several laboratory thermogravimetric analysis method results were technically reviewed in support of RPP Tank Characterization Reports.

4.3 ANALYTICAL TECHNOLOGY

A summary of general support services provided by Analytical Technology and specific development activities are provided. The Analytical Technology Team was managed by W.I. (Bill) Winters and included eight chemists, P.B. Bachelor, S.A. Catlow, B.A. Crawford, PhD; D.R. Hansen, S.G. Metcalf, R.W. Schroeder, G.L. Troyer, M.A. Purcell and K.B. Wehner; and two chemical technicians, J.M. Kunkel and J.K. Watts, and one secretary, F.J. Hurley.

The following summarizes the progress and status of analytical development activities for the RPP characterization program in FY 1999. The work on the following activities is presented in greater detail in the following documents.

- Implementation of Na Assay Method (CACN-102318)
- Development of ICP/MS Support for LAW/HLW DQO (CACN-102320)

- Implementation of Organic Support for the LAW DQO (CACN-102321)
- Improve HLW DQO Analytical Methods (CACN-102326)

Due to limited scope, the work performed on HLW Hot Cell Preparation Methods (CACN-102325) is only described below.

4.3.1 Sodium Assay

The Implementation of Na Assay Method focused on the evaluation and implementation of three analytical techniques: Neutron Activation, Flame Emission and Gravimetric Analysis.

4.3.1.1 Sodium Assay by Neutron Activation

For neutron activation several goals were achieved. Preparation for replenishment of the 222-S ^{252}Cf neutron sources was launched in FY 1999. The feasibility of this method for high precision sodium assay was demonstrated in FY 1998. Primary tasks to accomplish system upgrade and method development were identified. Procurement path, funds transfer, and authorization to procure with FY 1999 funds were completed by end of FY 1999. Actual preparations by Oak Ridge National Laboratory (ORNL) onsite installation are expected by mid-FY 2000. A procedure for operation of the sample handling system associated with the irradiation sources was also completed and approved.

4.3.1.2 Gravimetric Measurements for Sodium Analysis

A method of sodium analysis provided a foundation for implementation of highly precise weighing methods, sample handling measures and application of statistics to improve the analytical measurements. These measurements were proven to result in precision in measurement of sodium of < 1% at three sigma if performed carefully. Two technicians were trained on the methods that were applied. Subsequent discussions of the method with TD&L'S operations support personnel concluded that the method would be too difficult to run with real tank samples and operations personnel that may not be dedicated to the analysis. However, it was deemed worthy of use as a backup if the other methods (neutron activation analysis and flame emission spectroscopy) encountered problems.

The gravimetric procedure based on the precipitation of sodium magnesium uranyl acetate was transferred to a hood operation and tested on synthetic waste matrices. Hood conditions introduced new variables that impacted weighing and sample handling requiring development of new handling techniques. The gravimetric method produced very precise results; however, these results were several percent different than those obtained by inductively coupled plasma/atomic

emission spectrometry (ICP/AES) and earlier neutron activation results. The method is time consuming and technique sensitive; work was stopped at the end of the year on this method to concentrate on other activities. A synthetic standard with a reputable well established sodium concentration and significantly more testing would be necessary to obtain a better understanding of the small differences between methods. The other instrumental methods for sodium should provide nearly as good of precision, with more reliability and less effort. The option for further testing and using the gravimetric method should be maintained if operational or technical issues arise in the other techniques.

4.3.1.3 Sodium Assay by Flame Emission Spectrometry

Synthetic waste tank standards were provided to flame emission vendors for assessment of their capability to make highly accurate and precise sodium measurements. These tests indicated that this technology is capable of measuring sodium with an accuracy of about 0.5%. Different flame emission systems were evaluated for their adaptability to hood operations and a system ordered and received at the end of the year. Further work is needed in engineering and installation of the system in a hood before the final method can be developed.

Highly accurate measurements by all these techniques will require gravimetric aliquots and dilutions to be made. Accurate density measurements will then be required to convert these gravimetric values to volumetric values used by the process. Highly accurate digital density measurement systems were ordered for hot cell and hood operations. These systems are applicable to all the techniques being studied. Further work is needed to complete the installation and testing of these systems (FY 2000).

4.3.2 Development of ICP/MS Support for LAW/HLW DQO

ICP/MS procedures are in place for the key radionuclide isotopes; however, limited progress was made this year on those isotopes that require separations to remove isobaric interferences. A potential method for separating Ni-59 and Ni-63 from copper and cobalt interferences has been identified. A problem associated with differences between ICP/MS and radiochemical methods for Tc-99 analysis on tank C-104 sludge was evaluated further and found to be an alpha contamination problem with the radiochemical method. The alpha contamination is expected to be from Pu, which includes Pu-241, a low energy beta emitter that is measured with Tc-99 on the liquid scintillation counter. The use of ICP/MS for total elemental analysis of elements in the mass regions of high fission yields presents a challenging problem because natural isotopic abundance cannot be used to quantify the data. Efforts were made in evaluating this potential fission product interference on the analytes of interest and evaluating a software program

developed by Frank Kinard for Savannah River Laboratories (SRL). The new HLW/LAW DQO has added some additional ICP/MS elements because of lower detection limit requirements. Further work is needed on developing a systematic process for analyzing samples for these analytes and documenting the limitations of the analyses. (FY 2000).

TD&L supported the development of direct analysis of Tc-99 using ICP/MS methods from an indirect method in FY 1999, along with laboratory support for routine operations. This improvement promoted the use of Tc-99 standards for calibration on the ICP/MS instrument in the laboratory rather than a non-radioactive isobaric substitute, Ru-99. Once established, the ICP/MS Tc-99 method was used for comparison to the radiochemical method being used for Tc-99 analysis. Improvements were made to sample preparation based on results from these comparisons that resulted in improvement of the radiochemical method

Additional methods were also developed for ICP/MS analysis of I-129 and Sn-126. The development of an ICP/MS I-129 analysis allowed for an alternative method for analysis from the radiochemical method that can be used to advantage in sample analysis comparisons in a similar fashion as the Tc-99 work described previously. Development of the Sn-126 method provided a new method for analysis of this radionuclide where no other methods existed. Use of the tin ICP/MS method not only satisfied a fundamental analytical need for the laboratory to perform tin analysis (including Sn-113) but also indicated a larger need for separation methods to support ICP/MS analysis.

The need for separations to improve detection of analytes was apparent when running Sn standards on ICP/MS. Subsequent analyses indicated problems with isobaric interferences for analysis of Ni and Co, Pd and Ag, and Zr and Nb, as well. A path forward was discussed among TD&L and WMH scientists with experience in separations and ICP/MS analysis. The net result of the meeting provided the experimental design for a proposal that was sent to RPP for funding. Funding was provided by RPP for FY 2000 work. The foundation that was determined in FY 1999 to define this work included determination of best separations methods from the literature and historical site records, development of sample preparation methods that are suitable for separations techniques and assessment of improvements observed in ICP/MS data.

4.3.3 Implementation of Organic Support for the LAW DQO

The scope of work for the organic support for the LAW DQO changed throughout the year. The effort to develop a method for picric acid was successfully completed and is ready for testing on actual samples. The method includes picric acid concentration by solid phase extraction and measurement by ion-pairing or ion exchange liquid chromatography. A considerable amount of testing was completed on the development of a purge and trap thermal desorption procedure for

measuring volatile organics in high-level waste. The method needs installation and performance testing at the 222-S laboratory and WSCF for implementation. Draft procedures for this manual volatile organic analysis (VOA) method have been prepared. An automated VOA system was identified and purchased at the end of the year. When developed this system should improve reliability, turn around times and reduce personnel exposure. Equipment has been purchased and is staged to improve semi-volatile organic analysis methods for high-level waste. A large volume injection system for the gas chromatograph was purchased at the end of the year. This system is expected to improve the sensitivity of the analysis and partially compensate for the small samples of high-level waste that can be analyzed because of radiological concerns. A large number of activities remain to develop reliable organic methods for the complex high-level waste.

4.3.4 Improve HLW DQO Analytical Methods

Work was not initiated on the development of hot cell analytical methods until the end of the year. This activity focused on the development of more rigorous sludge dissolution methods like those used at SRL or West Valley vitrification operations. Another task was to evaluate methods for measuring water insoluble anions in sludges. Test procedures using oven heated sealed Teflon digestion vessels and aqua- regia and HNO_3/HCl/HF acid mixtures were prepared for sludge dissolution. The aqua-regia system was tested on ERA solid standards to evaluate its performance against EPA standard methods. Further work is needed on establishing the performance of the technique with these and other acid mixtures. Because the limitations of the digestion procedures are poorly documented for Hanford sludges, a comparison of the three methods on several classes of sludges should be performed and evaluated so that the best procedures can be applied. A test procedure for evaluating the effectiveness of KOH fusions followed by water digestion in dissolving insoluble anion compounds such as $SrSO_4$, LaF and AgCl has been written and approved. Test materials of insoluble compounds that may be found in the waste have been purchased for testing. Testing and evaluation remain to be completed.

4.3.5 Hot Cell Preparation Method Development

The hot cell preparation activity was aimed at completing the development and implementation of the hot cell rheology procedures. Work initiated on the rheology system in FY 1998 was completed and a procedure is in place for performing these analyses. The other activities in this task were aimed at developing the necessary techniques and procedures to perform the solubility testing required for the HLW/LAW DQO. A test plan has been implemented for performing these WIT directed solubility tests and they are being performed in the TD&L organization. In addition custom solubility tests for the retrieval program are being implemented by test plans. Changes in the HLW/LAW DQO solubility test may require some further work to meet the new requirements.

4.4 LabCore Support

TD&L provided support to develop the Y2K testing of data-handling capabilities on the LabCore system in FY 1999. Data input was streamlined by use of specific test parameters for all known sample and QC types. In this work a generic test set was developed that allowed for testing all variables resident on LabCore at the time of testing. In addition to Y2K development, methods were established for incorporation of post and pre-digestion spike data into the system. This capability was a particularly important parameter in improvement of reports of Tc-99 results. Addition of pre-digestion spike data allowed for Tc-99 tracking during sample preparation, providing the chemist a means to monitor changes in complex Tc-99 chemistry through the sample preparation process.

4.5 Waste Minimization and Pollution Prevention Activities

Technology, Operations and Process Science (TOPS) assisted in leadership of the Pollution Prevention committee for the 222-S facility. A TOPS scientist worked in a co-chair role to promote development of analytical methods that lead to waste stream reduction from the facility while remaining sensitive to the quality of analytical performance.

4.5.1 PCB Screening

Involvement on the part of TOPS in Pollution Prevention objectives allowed for funding of a polychlorinated biphenyl (PCB) Screening proposal for FY 2000. The work described in the PCB screening proposal takes advantage of the experience base at WSU that is present in the Analytical Chemistry group in TOPS. The proposal is specifically aimed at placing an operational procedure in the laboratory for timely and accurate analysis of PCBs in tank samples at detection limits that are currently being requested by the RPP customer (i.e. 2 ppb). Other work driven from the P2 committee lead involvement included development of potential paths for use of hazardous materials that had use elsewhere on site and support of P2OA training and return on investment (ROI) development with other scientists at the laboratory.

4.5.2 Laser Ablation

One activity that has been funded by the P2 Waste Minimization program that is being lead by TD&L is the development of the Laser Ablation ICP/MS as a tank waste-screening tool. The instrument was not operational at the 222-S facility last year but analytical comparisons of sample preparation methods were carried out at PNNL to support some of the waste minimization objectives for the use of this instrument at the 222-S facility. Tests included analyses of dry

powder tank simulants and simulant incorporated in glass matrices. Samples were run in batches that were designed to include solid blanks, standards and samples. Statistical sampling was performed on simulant samples run in batches as triplicates. The results of these limited tests indicated that the implementation of automated fusion for making glass disks is the best path for improvement of homogeneity in LA/MS samples. To that end, an automated fusion system was procured in FY 1999 that is also capable of use in generating liquid samples in a remote (i.e. hot cell) environment. Implementation of the auto-fusion instrument is scheduled for FY 2000 for the laboratory.

During FY 1999 two systems were procured to support reduction of detection limits for organic sample analyses of tank farm samples by semi-volatile and volatile methods. These instruments: a large volume injector and a purging auto-sampler; were designed to improve data quality and push detection limits lower than the US-EPA regulatory limits. In addition to improvements in data quality, the sampler allows for reduction in sample handling and increased productivity by performing automated sample preparation, purging and introduction to the GC/MSD.

In support of analysis of alpha bearing samples from Z-361 for semi volatile organic compounds (SVOCs) and volatile organic compounds (VOCs), TD&L and Cogema scientists worked to implement and draft procedures for SW-846 methods using purge and trap GC/MSD and extraction gas chromatograph/ GC/MSD methods for target compounds of concern in samples removed from Plutonium Finishing Plant (PFP) tank Z-361. These methods are currently undergoing required validation through the laboratory quality assurance (QA) group.
During much of FY 1999, TD&L scientists also supported implementation of organic analyses at the WSCF laboratory. In this work methods for analysis of gasoline and diesel fuel were implemented and quality objectives for support of the Waste Isolation Pilot Plant (WIPP) project were met that satisfied the WIPP program audits. TD&L scientists have both learned and provided regulatory guidance through validation of methods for this program. In addition, these scientists have assured that they remain current on program required reading assignments and method qualification requirements.

The primary focus of TD&L support to the 222-S Hot Cell support continued to be routine operations for tank farm sample break down and initial sample preparation. This effort included the extrusion of 166 tank farm core samples and the associated documentation and sample segmentation.

In order to improve analytical performance related to this type of sample, several initial analyses were brought online within the hot cells. These included preparation methods using acid digest for metal analysis and water leach for anion analysis. Performing these directly in the hot cells significantly reduces personnel radiation dosimetry in these and subsequent processes external to the hot cells. Direct pH measurements were also implemented in the hot cell such that both the analytical result is provided and subsequent sample conditioning can be planned prior to finalization of sample dilutions and transfers out of the hot cells. All three of these procedures were fully qualified and brought on line during FY 1999.

The cost of hot cell operation and cost accounting was addressed by reconstructing all costed activities. This evaluation led to defining defensible hot cell unit costs and overhead apart from the general 222-S overhead. This approach was used in FY 2000 budget planning, effectively making the hot cell activities a unique cost center. Coupled with this approach was the definition of 10 new analytical operation tests in the laboratory information system to assure proper cost tracking of the new approach.

4.6 Radiochemistry Development

4.6.1 Technetium

Efforts to evaluate and compare analytical methods for ^{99}Tc in Hanford tank sludges such as C-104 were completed during FY 1999. The radiochemistry method was diagnosed to have a problem with ^{241}Pu interference. This was successfully diagnosed and a correction put into use by the end of FY 1999. A white paper identifying historical development of analytical methods for ^{99}Tc at Hanford, HNF-5309, was published as a result. The evaluation and improvements will be used in determining best approach for waste tank remediation and waste processing relative to ^{99}Tc inventories.

4.6.2 Tin and Nickel Isotope Analysis

A method to separate and analyze for various isotopes of tin was investigated. The isotopes ^{121m}Sn and ^{126}Sn are of interest due to long half-lives and expected but uncertain quantity in Hanford wastes. A quantity of the latter isotope, ^{126}Sn, was successfully isolated demonstrating the utility of the method. Sufficient quantity of this isotope is now available at 222-S for estimating its half-life, a not well established value. Further work to prepare a formal procedure and report the half-life estimate was identified. Similarly, ^{63}Ni is of interest as a long-lived component in Hanford wastes. In general, the separation of measurable quantities of this isotope

has historically been a challenge. Investigation lead to the recommendation for use of a special ion exchange material. Material source was identified and procurement started at the end of FY 1999.

4.7 Laboratory Automation

Current computer technology and the Hanford Local Area Network (HLAN) provided a basis to demonstrate low cost video access to laboratory processes. Consumer video cameras were attached to several laboratory HLAN computers and video conferencing demonstrated. This feature would allow non-facility customers to view sample processing such as hot cell extrusions without being in the radiation zones. Equipment procurement of off-the-shelf equipment was recommended and started by the end of FY 1999.

4.8 University Programs

Activities of the WSU Tri-Cities chemistry program are centered on meeting both the academic needs of WSU students as well as the strategic objectives of the U.S. Department of Energy, Richland Operations (RL) and its' contractors. Chemistry courses at the junior, senior, and graduate level are provided for students in Agriculture, Biology, Chemistry, Chemical Engineering, Environmental Engineering, Environmental Science, General Studies, and Physical Sciences. Retraining and specialty training of the Hanford work force is a major feature of the program. DOE-RL and its' contractor staff comprise approximately 50 percent of the annual student enrollment in Chemistry.

TD&L staff was funded by DOE-RL to serve as the Washington State University Tri-Cities Chemistry Program Coordinator on a half time basis. The Program Coordinator is responsible for all-academic, administrative, community outreach, financial, and research activities of the program. The Program Coordinator reports administratively to the Campus Dean and academically to the Chemistry Department Chair in Pullman. In addition, as an adjunct faculty his duties included: teaching, guiding student research, serving on graduate student committees, and chairing graduate student committees. It should be noted that all teaching costs were borne by the university.

Chemistry course enrollment for the year was at an all time high. Special courses were developed and offered at times and locations selected to best serve the needs of the Hanford workforce. It is noteworthy that six students from the Nuclear Regulatory Commission Headquarters attended a special class on Nuclear Chemistry at Hanford. A special course titled Nuclear Waste Vitrification course was offered twice and had especially high enrollment.

Formal approval from the Washington State University Faculty Senate was obtained to create a new Chemistry course dedicated to staff at the Hanford site. The course title is Special Topics in the Chemistry of Nuclear Processes and Radioactive Waste Management. Beginning January 2000, a special entitled Chemistry of Hanford Processes will be offered.

TD&L staff taught many Chemistry courses during the year including: A.M. Choho, Nuclear Waste Vitrification (twice), B.A. Crawford, PhD, Trace Inorganic Analysis, and S.G. Metcalf and B.A. Crawford, Applied Inorganic Chemistry for Agriculture Students, Chemistry in Contemporary Society (four times), Quantitative Instrumental Analysis, and Trace Inorganic Analysis. In addition, M.A. Purcell gave invited lectures on neutron activation analysis.

A special course was offered during the summer at WSU Tri-Cities campus by the Environmental Science department. The title was Hanford in Context and was given in partnership with PNNL and WSU Tri-Cities. It was intended for students who were summer interns at Hanford but will be valuable new staff for Hanford contractors and DOE-RL in the future. TD&L staff served on the steering committee that developed the course and lectured on the Chemistry of Hanford.

A special seminar was arranged for presentation at Washington State University Tri-Cities. It was titled The Defense Waste Processing Facility (DWPF), An Introduction and Description and presented by P. J. Brackenbury of NHC. The seminar had unusually large attendance, approximately 61. In addition, plans for WSU courses in nuclear waste vitrification were briefly discussed.

TD&L staff (L.L. Lockrem and S.G. Metcalf) made a presentation entitled Enhanced WSU/CBC Partnership to the WSU Tri-Cities Dean, President of CBC, and Director of Training and Education for DOE-RL. This will facilitate closer coordination between the two institutions. On-going meetings with Columbia Basin College (CBC) have been initiated to develop Chemistry courses targeted at Native American students. Additional efforts have been initiated to have WSU's Chemistry program assist CBC to set up a new microscopy laboratory and instrument automation laboratory.

S. G. Metcalf participated in a short-term faculty exchange with the Universidad de Chile (in Santiago, Chile) and Universidad de Austral (in Valdivia, Chile). While in Chile, he presented a short course and invited lectures on Sampling and Analysis of Chemical Pollutants for Compliance with United States Environmental Laws. In addition, the National Center for Environmental Research was visited. At both universities and the National Center for Environmental Research numerous potential collaborations in both research and education were identified. Selected projects will be pursued. Specific information is available upon request. He departed March 31 and returned April 16. Washington State University International Programs provided all funding.

Dr. Asopuru Okemgbo is forming a Pacific Northwest chapter of the National Organization of Black Chemists and Chemical Engineers. This effort is underway in partnership with NHC, DOE-RL and WSU.

4.9 SPECIAL ANALYTICAL SUPPORT

The Special Analytical Studies, managed by L. L. Lockrem, is a unique organization of chemists, scientists, engineers, and technicians that specialize in developmental projects and field analytical services. Therefore, a variety of support activities are reported that are performed in laboratory facilities, at the sampling site, or in situ. The SAS group, managed by L.L. Lockrem and included 11 scientists; D.J. Alexander, D.B. Bonfoey, W.S. Calloway,PhD; M.J. Duchsherer, G.A. Fies, L.D. Lockard, L.A. Pingel, K.O. Pennock, M. Stauffer, and R.S. Viswanath, PhD; three technicians; C.M. Marquez, J.Y. Smith, C.S McCllelan; and one secretary, S.I. Ryburn.

4.9.1 Hanford Transuranic Waste Program

SAS played a significant role in establishing Hanford Transuranic (TRU) Program since 1995. TRU Program activities were very limited from 1995 to May 1998 due to funding constraints. In September of 1997, SAS staff accompanied by Waste Management Hanford Company staff visited Idaho National Engineering & Environmental Laboratory (INEEL) to understand their TRU Program and learn from their experience. Following this visit, SAS initiated to establish the infrastructure to perform TRU Drum Headspace gas sampling and analysis. SAS designed the sampling equipment (four 250 ml SUMMA canister assembly) to collect headspace gas samples from TRU waste Drums. In addition, SAS set up a Gas Chromatograph (GC) and a Gas Chromatograph/Mass Spectrometer (GC/MS) for the analysis of headspace gas samples for Volatile Organic Compounds (VOC) as well as Hydrogen and Methane. SAS performed the analysis of Cycle 11 Performance Demonstration Program (PDP) samples on an informal basis and met the criteria for a Pass. Following the Cycle 11, SAS also scored a "Pass" with 100 out of 100 in Cycle 12 PDP analysis. By scoring a Pass in two consecutive PDP Cycles, SAS met all the criteria for performing TRU Drum headspace gas analysis on a routine basis. The infrastructure set up also included the preparation of about dozen TRU specific sampling and analytical procedures. All this was done with the least amount of time and expense to the TRU Program as compared to the cost incurred at the other Department of Energy sites for the same set up.

SAS received the first batch of 16 drum samples on December 1, 1998, and completed all the analyses and data packages in 28 days of the sample receipt. The second batch of drum headspace gas samples was also analyzed and data packages prepared with in 28 days of the sample receipt. In both cases, SAS met the timeline requirement of 28 days. After the successful

completion of first two batches of drum samples, SAS also completed the analysis of Cycle 13 PDP and met all the criteria for a "Pass".

4.9.2 Tank Headspace Vapor Program (TWRS)

Tank headspace vapor program required the use of multiple sample collection techniques namely: SUMMA canisters, Triple Sorbent Traps (TST), Sorbent Tube Trains (STT) and Polyurethane foam traps (PUF); and multiple analytical techniques: GC, GC/MS and Ion Chromatography (IC). SAS provided extensive sampling and analysis support to TWRS in the resolution of a safety issue, which arouse during waste retrieval from tank C-106. This activity consisted of Phases II III and III. The phase I involved sampling and analysis of 24 SUMMA canisters (6.0 Liters), 19 TSTs, 18 STTs, 9 PUFs. SUMMA canisters and TSTs were analyzed for more than 60 target organic compounds and about 200 or more tentatively identified compounds (TICs). Phase II and III consisted of a smaller set of samples as compared to Phase I. The normal sample turn around time of three weeks was shortened to about a week to help TWRS resolve safety issue as quickly as possible.

In addition to Phase I, II and II of C-106 tank, SAS also provided sampling and analytical support for two other tank samples namely; AZ-102 and AY 102. A special study was also performed to evaluate the type of organic compounds that would break through a breathing filter. Samples of breathing filters used by tank farm personnel during tank waste retrieval were obtained and analyzed for a series of organic compounds. Analytical data was provided to the TWRS Program. TWRS management was very happy with SAS exemplary response to their request for support and complimented SAS staff and management for this timely and dedicated support.

SAS organization was then officially transferred from Numatec Hanford Corporation to Waste Management Hanford Company in April 1999. This resulted in a merger of SAS and WSCF Laboratories.

APPENDIX A

External Publications

Trans. Am. Nucl. Soc, Vol. 81, pp. 96-97, Gas Generation in Heated Waste from Two Hanford Tanks, 1999.

Trans. Am. Nucl. Soc, Vol. 81, pp. 93-95, Empirical Rate Equation Model of Hydrogen Generation for Hanford Tank Waste, 1999 (with T. A. Hu and K. M. Hodgson).

"Reverse Polarity Capillary Zone Electrophoresis of Natural Waters for Nitrate and Nitrite," A. A. Okemgbo, H. H. Hill, and S. G. Metcalf, Anal. Chem. 1999, 71, 2725.

"Metal Ion Interferences in Reverse Polarity Capillary Zone Electrophoretic Analysis of Hanford Defense Waste for Ethylenediaminetetraacetic Acid and N-Hydroxyethylethylenediaminetriacetic Acid," A. A. Okemgbo, H. H. Hill, and S. G. Metcalf, Anal. Chim. Acta, 396, (1999) 105.

"Analysis of Hanford Defense Waste for Nitrate and Nitrite by Reverse Polarity Capillary Zone Electrophoresis," A.A. Okemgbo, H.H. Hill and S.G. Metcalf, J. Chromatogr. *A*., 844, (1999) 387-394.

"Analysis of Hanford Defense Waste for Ethylenediaminetetraacetic Acid and N-Hydroxyethylethylenetriacetic Acid by Reverse Polarity Capillary Zone Electrophoresis," A.A. Okemgbo, H.H. Hill, and S.G. Metcalf, J. Microcol. Sep. 12(1), (2000) 48-56.

Supporting Documents

HNF-2917, Study of Radionuclide Leaching From The Residues Of K Basin Sludge Dissolution, July 30, 1998.

HNF-3495, Rev. 0, Implementation Plan for the Hanford Cone Penetrometer Multi-sensor and Soil Sampler Probe System, February 1999.

HNF-3551, Rev. 0, Test Plan for the Hanford Cone Penetrometer Multi-Sensor and Soil Sampler Probe Systems, December 1998.

HNF-3713, Compositing K Basin Sludge Samples, January 7, 1999.

HNF-4045, Rev. 0, WRAP Isolated Facilities Inventory Spreadsheet, May 1999.

HNF-4050, Rev 1, Total Measurement Uncertainty for Nondestructive Assay of Transuranic Waste at the Waste Receiving and Processing Facility, September 1999.

HNF-4051, Rev. 2, Quality Assurance Objectives for Nondestructive Assay at the Waste Receiving and Processing Facility, September 1999.

HNF-4094, Test Plan for Tank 241-C-104 Retrieval Testing, June 9, 1999.

HNF-4151, Rev. 0, Acceptance/Status of Construction At Project Suspension of the Hanford Cone Penetrometer Multi-Sensor and Multi-Sample Soil Sampler Probe Systems Procedures, July 1999.
HNF-4287, Rev. 0, In-Situ Air Permeability Measurements Using the Cone Permeameter at the 200 East Area of the Hanford Site, March 1999.
HNF-4298, Rev. 0, Final Report on Film Removal Study Test on 324 Facility B- Cell Samples April 1, 1999.
HNF-4298, Rev. 0, Film Removal Study Test Report Revision, May 3, 1999.
HNF-4751, Test Plan for Tank 241-AZ-102 Solubility Screening Tests, November 22, 1999.
HNF-4752, Test Plan for Tank 241-AZ-101 Solubility Screening Tests, December 3, 1999.
HNF-1761, Rev. 0, W-026, WRAP Facility Boxed Waste Assay System (BWAS) Acceptance Test Report, February 1999.
HNF-4964, Results of Dilution Studies with Waste from Tank 241-AW-101, September 24, 1999 Technical Task Plan RL0-8-WT-41, PHMC Pretreatment – Saltcake Dissolution, September 1999.
HNF-5193, Saltcake Dissolution FY 1999 Status Report, September 1999
HNF-5309, Rev. 0, Survey of Technetium Analytical Production Methods Supporting Hanford Nuclear Processing, October 20, 1999.
HNF-5391, Test Plan for Retrieval Testing of Sludge from Tanks 241-AZ-101, 241-AZ-102, and 241-AY-102, November 17, 1999
HNF-5443, rev. 0, Retained Gas Sampler Calibration and Simulant Tests, December 20, 1999.

HNF-SD-WM-DTR-046, Rev. 0A, Results of Dilution Studies with Waste from Tank 241-AN-105, October 14, 1999.

RPP-5456, Rev. 0, Final Report on Tank 241-AN-107 Retrieval Testing, December 9, 1999.
RPP-5230, Plan for Laboratory Tests to Support Retrieval of Waste from Tank 241-AN-103, October 27, 1999.

Internal Memos

"Solubility Screening Tests for Tank 241-AN-107," January 11, 1999.
"Shear Strength and Particle Size Distribution Measurements on Tank S-111 Samples," January 29, 1999.
"Shear Strength and Particle Size Distribution Measurements on Tank U-109 Samples," January 29, 1999.
"Solubility Screening Tests for Tank 241-AW-101," February 1, 1999.
"Particle Size Distribution Measurement on 291-Z Sump Pit Sample," March 15, 1999.
"101SY/102SY Composite Boildown," April 9, 1999.

82800-99-011, Test Plan for 1E, hood 1, April 12, 1999.
82800-99-017A, Test Plan for Technetium Analysis of C-104 Sludge Waste, April 4, 1999
82800-99-020, Summary of Rm 1D Hood 4 Waste Disposition, April 17, 1999
"Effluent Treatment Facility pH vs Nitrite Concentration," April 20, 1999.
"Dilution Studies of Tank 241-SY-101 Waste. Preliminary Results," April 22, 1999.
"Preliminary Results of Viscosity Measurements of Tank 241-SY-101 Samples," April 22, 1999.
82800-99-021, Justification for Non-Competitive Procurement-Digital Density Meters Material Request 00014838, April 26, 1999.
"Results of Viscosity Measurements of Tank 241-SY-101 Samples," May 3, 1999.
"Request for Permission to Start Up X-Ray Diffractometer, and Appointment of Custodian and Qualified Expert," May 4, 1999.
"Double-Shell Slurry Dissolution Kinetics," May 10, 1999.
"Additional Viscosity Measurements on Core 257/Segment 7 from Tank 241-SY-101 Samples," June 2, 1999.
"Test Procedure, C-104 Sludge Retrieval and Dilution Test," June 14, 1999.
"X-Ray Diffractometer Results for Batch Saltcake Dissolutions of BY-102, June 21, 1999.
82800-99-023, Pu Isotopics Discussion for Z-361, June 22, 1999.
"X-Ray Analysis of B-Cell Solids Sample," June 29, 1999.
"Preliminary Results of Dilution Studies on Samples from Tanks 241-U-103 and 241-U-102," July 21, 1999.
82800-99-027, Chemical Oxidation Processing of ESI-solutions, July 27, 1999.
"Viscosity Measurements for Tank 241-AN-105 Dilution Study," August 2, 1999.
"Physical Measurements for Tank 241-AW-101 Dilution Study," August 4, 1999.
"Results of Dilution Tests on Supernatant Liquid Samples from Tank 241-AN-105, August 5, 1999 with D.W. Edmonson).
"Curvature of Titanium Samples from the 324 Facility B-Cell," September 8, 1999.
"U-Farm Cooling Test Results, Final Report," October 1, 1999.
"Viscosity Measurements for Tank 241-SY-102," October 7, 1999.
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