WSRC-IM--90-29

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GUIDE TO SAVANNAH RIVER LABORATORY ANALYTICAL SERVICES GROUP (U)

APRIL 1990

Westinghouse Savannah River Company
Savannah River Site
Aiken, SC 29808

SAFETY • RESPONSIBILITY • SECURITY
SRS
SAVANNAH RIVER SITE

PREPARED FOR THE U.S. DEPARTMENT OF ENERGY UNDER CONTRACT NO. DE-AC09-88SR18035

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Section A

Analytical Services

MISSION OF ANALYTICAL SERVICES

The mission of the Analytical Services Group (ASG) is to provide analytical support for SRL R&D Programs using onsite and offsite analytical labs as resources. A second mission is to provide SRS operations with analytical support for nonroutine material characterization or special chemical analyses. The ASG provides backup support for the SRS process control labs as necessary.

HOW TO ESTABLISH SUPPORT

Authorize an SRL Service Order and establish a Laboratory Information Management System (LIMS) account.

Assistance with obtaining analytical services may be obtained from any of the following:

Contacts for help	Location	Phone
Group Manager	773-A,B144	X5-2198
LIMS Manager	773-A,B141	X5-3306
Supervisor	773-A,B161	X5-2352
Supervisor	773-A,C101	X5-2034
QA Clerk	773-A,B157	X5-3559

ASG uses the SRL Service Order System to authorize work. ***A SRL Service Order Form, OSR-24-C213, must be completed to initiate the process (see Figure 1).*** For most SRL programs a Service Order is automatically established on an annual basis and a new customer should ask if he already has a Service Order in place for his work requests.

The Service Order establishes cost controls, records the submitter and authorizers of the request, establishes a "Customer Datagroup" (a customer database) and creates a "Study" for storing sample and result records. The service order may include additional information from the customer. ASG will add statements indicating who is authorized to use the Study.

The Service Order is valid for the fiscal year and must be reauthorized during September for continuing work. A Service Order may be initiated at any time during the year. It can be revised upon request to change cost codes or cost code allocations.

The Service Order requires the signature of the customer's manager for approval. The manager needs to have authority for the amount being authorized. The Service Order will be signed by the ASG manager who agreed to support the work requested.

All ASG Service Orders will be reviewed during September with the authorizing managers to see if they wish to continue to authorize analytical support and if they want to alter the Datagroups and Study name assignments.

With the establishment of a Service Order and Study, ASG will establish a database in the ASG Laboratory Information Management System (LIMS) to store the sample records and results. **Customer accounts and LIMS user manuals may be obtained from the ASG LIMS Manager. A customer must have an ALL-IN-ONE account and a completed CPC-16 form on file with the Computer Protection Committee before a LIMS account can be created.**

Analytical data is stored in an ORACLE database. Data access is restricted to members of the same Customer Datagroup and is clustered by Study names and other sample information. The entire database may be queried using Structured Query Language (SQL) statements and special downloading of data for scheduling and result reporting is supported. For these services the customer should contact the LIMS manager. Prior planning facilitates effective use of the database.

TEST CONTROLS (Sampling, QA, and QC)

ASG Planner - Sample Plans and Test Controls

After a Service Order is established, the customer should contact ASG to obtain assistance with planning his analytical work. ASG will assign a "Planner" to assist the customer in establishing analytical support. The planner programs the ASG LIMS (computer sample tracking system) to support the customer. He is familiar with the ASG QA Action Plan (WSRC-RP-89-390), the routine analytical methods and instruments offered by ASG, and the use of offsite labs to supplement ASG services.

Working with the customer, the planner sets default instructions for ASG to safely handle samples and notes specific instructions for assigned methods. If timing is critical, the planner will negotiate ASG timing commitments with the customer and seek approval of ASG Task Supervisors in charge of the individual methods. The planner can assist with designing a Quality Control (QC) sampling plan.

A written Test Control plan may be required by a customer's QA program. For example, Environmental Protection Agency (EPA) hazardous waste analysis by RCRA SW-846 rules requires a written QA project plan in addition to the QA Program Plan provided by ASG. ASG will not write the plan but can provide information to support customer's plans. The commitments of ASG are defined in the ASG QA Action Plan (WSRC-RP-89-390); a copy of which may be obtained from the ASG QA Records office in 773A, B157, or via the Information Services Section. Copies of routine analytical method and instrument procedures and the QC data verifying the calibration of the methods and instruments may be obtained from the ASG QA Records offices.

The ASG Planner can, on request, print reports of the LIMS data and analytical sample plans being used to support a customer.

QA Levels

ASG provides two levels of QA controls for samples and will assist the customer with a third level which requires customer with a third level which requires customer actions to be effective. These are designated in the ASG QA Action Plan as Exploratory, Routine, and Customer-Assisted.

Exploratory level is for R&D samples of an exploratory or qualitative nature. The Exploratory level requires no controls above those required for all SRL research. Sample records are kept in research notebooks and samples are not logged or tracked in the LIMS. The Exploratory level is obtained by verbal agreement between the customer and the ASG Task Supervisor directing a particular analytical method. Costs are covered by direct development funds and may bypass the routine Service Order system described above. This level is not recommended for work other than scouting activities and method development.

Routine Level is for general R&D support and provides a solid base for customers who need a more rigorous QA program. All samples logged on the ASG LIMS receive this level of QA. ASG agrees to maintain a written method or instrument procedure for all Routine level samples, a QC program for testing each method and instrument, and a complete sample record system using the ASG LIMS.

The Customer-Assisted Level is used for samples requiring special QA. This level provides the same controls provided for routine level samples and additional controls which are negotiated with the customer. The additional controls enable

control of sampling, sample shelf life, and matrix effects on analytical methods which cannot be controlled by internal ASG operations.
The Customer-Assisted Level will include chain-of-custody options which ensure and document that the right sample is analyzed. A chain-of-custody record may be established (by the customer) which shows all persons handling a sample and any sample identity or labeling changes that occur during its path to ASG. ASG personnel may require customer-assisted controls, such as chain-of-custody, if they recognize that the importance of the sample requires them. An example of a chain-of-custody form is shown in Figure 2. A customer may design his own form.

Additional customer-assisted controls include the use of blanks, spikes, duplicates, and standards (blind and double blind) to track ASG performance. These controls can only be effective if the customer provides rapid feedback to ASG personnel for corrective actions. The total process is illustrated in Figure 3.

Establishing a Sample Record

Essential Information

For each sample logged into the ASG LIMS, a sample record is established which enables ASG to rapidly identify the sample, determine hazards, the analyses requested, and disposal requirements. Required responses to sample parameter prompts are programmed as defaults into the LIMS by the ASG planner when the sample plan for a customer is established. The customer may adjust these defaults, with each submission of a group of samples or on a case by case basis for each sample. The planner may change the defaults for the sample plan at the customer's request.

Security and Classification. The ASG LIMS is on an unclassified network and classified sample information must be handled by separate procedures. Please contact ASG/ADS management and the customers security officers for arranging special procedures which must be approved by management before submitting classified samples or information. As noted below special procedures may be required for physical protection of a classified material. The ASG LIMS does have substantial protection from unauthorized access to information but may not be appropriate for a given sample.

Sample Record Information. The following information is needed:

CUSTOMER DATA GROUP (e.g., Actinide Tech, DWPT TNX, REACTORS, M AREA etc.)

STUDY (e.g., Pu Scrap, IDMS, J. Bibler)

MATERIAL (Glass, Sludge, Pu Scrap, etc.)

PROFILE (lists of analytical methods, e.g., SEM/XRD)

SAMPLE PARAMETERS

ASG (Receiver Initials) ... used to activate ASG
Description ..... brief description
Radioactivity ..... how much and what
Fissionable..... no or yes, what and how much
Chemical Hazards ..... warn ASG personnel
Submitter ..... who gets the results
Disposal .....return or dispose of sample
Sample Size ..... helps identify sample
Heterogeneous ...if multiphase give instruction
Requester ..... technician or other contact
Analyses .....instructions for specific analysis within methods (e.g., IC: chloride, nitrate)
Comments..... special messages to ASG

Paper Logging of Samples - Establishing a LIMS Sample Record

Customers may establish samples records via form OSR 24-E33 or by directly logging samples on the ASG LIMS via the SRL ethernet network. Except for Exploratory samples, the LIMS record must be established prior to submitting sample for analyses.

OSR 24-E33 shown in Figure 4 captures the essential information. The form should be given to the planner to log the samples in the LIMS. The planner will return to the customer a list of ASG sample numbers for his samples. The designated submitter will also receive a summary of the logging process from the ASG LIMS.

The ASG sample numbers must be placed on the samples as described below prior to submission of samples to ASG for analyses. These numbers provide a unique identifier for the sample. ASG will assist paper loggers with the correct labeling of samples as necessary.

Computer Logging of Samples

A customer with an account on the ASG LIMS may directly log samples. The instructions for doing this are contained in the customer LIMS user manual obtained from the ASG LIMS manager. ASG will tailor your "pclink" terminal emulator for LIMS use. Special softkeys facilitate use. ASG provides keyboard stickers or keyboard templates to help identify database special keys. ASG Sample Log numbers must be placed on the samples according to the requirements below prior to submitting samples to ASG.

PHYSICAL SAMPLE HANDLING

Sample Labels

All samples must be individually labeled to show identification, date submitted, the submitter, and hazards. The identification must agree with that shown on the Analysis Request. If the Analysis Request is submitted via the LIMS, the computer-generated "Sample ID" must be shown on the label. Pressure sensitive labels can be obtained at the Sample Receiving Station in Building 773-A, B-150 and in the Analytical Records Office. Labels are available in two sizes - OSR-24-125B (large) and OSR-24-126C (small) - and three colors (white, yellow, and red). The colors are to be used as follows:

White. Indicates that the sample is non-radioactive.

Yellow. Indicates that the sample contains only activity associated with natural uranium or thorium.

Red. Indicates a danger signal. A red label indicates that the sample must be handled in a glovebox or with special attention to handling or shielding. Red labels are to be used on samples that contain radioactive isotopes other than those associated with natural uranium and thorium. Red labels are to be used with all samples that contain tritium.

The sample labels, OSR-24-125B or OSR-24-126C (Figure 5) should be filled out by the submitter to show:

LAB NO. Leave this space blank if submitting an Analysis Request Form. If submitting a Request for Analysis via the LIMS, put the "ASG Sample No." here.

ORIG (Submitter)
DATE (Date Submitted)
USER SAMPLE ID (I.D. or Description)
HAZARDS (List Chemical Hazards)

In addition, red and yellow labeled samples must be surveyed and tagged by Occupational Health Protection at the point of origin. Any sample package containing more than 0.25 g of fissionable isotopes must have a 3 inch x 3 inch "Fissile Material" label, OSR-24-C234A (Figure 6), or a "Fissile Material" symbol, OSR-24-C234B, visibly attached to it and be accompanied by an IINMTR, OSR16-A (Figure 7). The sample package must be labeled to specify the fissionable isotopes, their form and quantity. Customers should attach to each sample a chain-of-custody card if required by the customer's QA plan or if required by ASG planner personnel.

Samples that are not clearly and properly labeled will not be accepted by Analytical Sample Receiving personnel.

Classified Sample Pieces

Samples that are classified for security reasons require approved repositories and security plans. These plans must be approved by ASG and customer management prior to submission of samples requests. Special security handling must be noted on the service order requesting ASG support. Special training of ASG personnel may be necessary prior to doing the work.

Sample Containers

Containers for Normal Samples

Most liquid samples can be handled conveniently if they are submitted in small screw-cap specimen vials that are available from Chemical Stores in sizes 4 to 40 ml (Caption 23, Item 2470.00 through 2520.00). Gas samples for mass spectrometric analysis should be submitted at a pressure of one atmosphere or less. Gas samples at pressures greater than one atmosphere should be submitted in an approved pressure tested metal container with the pressure clearly marked and carrying a "Caution Tag". Solid samples should normally be submitted in wide-mouth screw-cap bottles.

Shielding and Containment

All samples which read >50 mr/hr at contact must be shielded. Samples containing large amounts of activity, i.e., >10E5 d/m, must be submitted in secondary containers. Small ice cream cartons are satisfactory for this purpose. Most samples reading >50 mr/hr will be generated in the High Level Caves and handled in High Level Caves Analytical cells. All other samples reading 0.50 mr/hr at contact cannot be sent to ASG without prior consultation with ASG supervision.

Samples which do not conform with all requirements will not be accepted by ASG unless special arrangements are made between supervisors in the submitting and receiving groups. Contact OHP for assistance with unusual or especially hazardous samples.

Sample Receiving and Storage

Savannah River Laboratory Samples

ASG Sample Receiving, Building 773A, B-150, is open only when an ASG sample receiving representative is present and is otherwise locked with no outside facilities for receiving radioactive samples.

If samples are known to present a storage hazard such as pressure buildup due to radiolytic gas evolution, chemical reaction, or if a sample reads 0.50 mr/hr through its carrier, special arrangements must be made with the responsible chemist or supervisor for safe immediate handling by

ASG because **THESE SAMPLES CANNOT BE PLACED IN NORMAL SAMPLE STORAGE.**

All radioactive and non-radioactive samples, except those described in the preceding paragraph, are to be delivered directly to the interior door of Building 773-A, B-150 via the central hall corridor. Alternatively, samples may be delivered directly to an ASG technician who agrees to receive the samples. Under no circumstances are samples to be left in or taken to the Analytical Offices.

Personnel delivering samples will need to wear shoe covers, lab coats, and safety glasses, and they must monitor the sample packaging and themselves at both the monitor located in Sample Receiving and the monitor located in the hall exit. If turnover is to an ASG technician, they should monitor at that point.

NOTE: Tell the ASG receiving technician that you have brought the samples, so they will be acknowledged as being received and therefore entered into the LIMS.

Fissionable Samples

All analytical requests or experimental samples involving fissionable isotopes in quantities greater than 0.25 g of Group 1 and Group 2 (DPST 68-108) materials must be approved by ASG supervision and the ASG Fissionable Material Coordinator before the sample is delivered to the ADS laboratories. All Group 3 (DPST 68-108) materials must be approved by ASG supervision and the ASG Fissionable Material Coordinator and must be transferred through SRL Nuclear Accountability (see Figure 8 for classifications of Groups 1, 2, and 3.) The Occupational Health Protection Engineer responsible for fissionable material control must approve the proposed route of the shipment and its subsequent storage (SRL Criticality Control Procedures, DPST-68-108).

All samples containing Accountable Nuclear Material must be approved by ASG supervision and the ASG Accountability Coordinator before the sample is delivered to ASG. Regulations for handling Accountable Nuclear Material are detailed in DPSTQM-58, Accountability Manual, Savannah River Laboratory, Part I.

Precious Metal Samples

Samples consisting of or containing material defined as precious metals must be accompanied by a Precious Metal Transfer Advice, OSR-22-7 (Figure 9), unless the sample is to be returned to the submitter or is to be considered as consumed during analysis and is thus no longer accountable. In all cases, the comment line on the analysis request must indicate that the sample does contain precious metals and must state the accountability status and final disposition of the sample.

Samples for Offsite Analysis

Samples requiring offsite analysis must be accompanied by the normal Service Order and analytical request to facilitate handling charges and reporting of results. Offsite regulatory samples may require special handling such as being kept iced. Customers should negotiate these requirements with ASG prior to submitting samples.

Samples from Savannah River Operations

Samples originating in the various SRS operating departments for analyses by ASG should be transferred to ASG through the Analytical Laboratories Department and must conform with all requirements of this procedure and with DPSTP-R-130, "On-Plant Shipments". Samples should enter Building 773-A via the E-wing receiving station in the back of Building 773-A. ASG may directly receive nonradioactive samples upon direct agreement, but **in all cases the process control lab in support of the operation should be notified of work sent to ASG.**

The Analytical Laboratories assists in coordinating the shipping of samples. The Separations Department controls the sample truck for shipment of hazardous and radioactive material from 200 Area. All plant shipments should be reviewed by the RHYTHM (Remember How You Treat Hazardous Material) representative before shipment and must conform to the appropriate DOT packaging requirements. Basically, samples must be kept in containers less than 1-quart in size and be properly labeled.

Sample Analysis (Completion Time)

In general, routine samples are analyzed according to date of receipt, and are completed within two to four weeks of the date of receipt. Immediate service or more rapid service may be obtained by authorization of overtime. Customers who split samples for parallel analyses as opposed to sequential analyses will receive faster turnaround. Direct discussion with method task supervisors and analysts is recommended to expedite analyses.

Sample Disposal

Unless a hazardous storage condition exists, samples will be retained for two weeks after all the analyses have been reported to allow time for the submitter to inspect the results and to request additional work on the sample if he so desires. Unless such a request is received within two weeks, or the submitter requests that the sample be returned or held, analyzed samples will be discarded. The maximum time for holding a sample in ASG facilities is 30 days. If additional hold time is needed by the submitter, it is the responsibility of the submitter to arrange for storage in his facility. Samples containing fissionable materials or organics must be returned to the submitter.

IV. RETRIEVING SAMPLE RESULTS

The ASG LIMS will automatically provide a report to the designated submitter of a sample when all analyses requested for the sample are completed. The report gives the sample results or points to them. Classified data is restricted from the LIMS and therefore the LIMS can only point to those sample results. Other data such as photographs are not easily handled at this time in the LLIMS database and must be pointed to. The customer should receive data directly from the Task Supervisor in these cases.

Customers having a LIMS account may easily retrieve results for their samples via direct query using the forms and reports supported by the LIMS. The customer may see partially complete results in this fashion.

On request, ASG provides sample status reports and result reports for a customer by running a special query. A report is for a given submission of samples or by each user sample number. The report will generally be restricted to a Customer Datagroup and Study. The customer should contact the ASG LIMS manager for these special reports. Special queries to the database are possible and results may be directly downloaded or uploaded to other computer systems as ASCII text files.

Because of limited data storage in the LIMS database, results which are greater than 1 year old may be moved to another database. The customer should consult with the ASG LIMS manager for information on retrieving archived results and the status of his older samples.

In cases where results are needed in a critical time, the customer must contact the individual Task Supervisors of the methods to arrange for a fast report or have the sample planner do this for him. Large programs can be provided with timely sample status reports to facilitate tracking sample results.

OSR 24-C213
(Rev 4-88)

SRL SERVICE ORDER

(See reverse side of pink copy for instructions on how to complete form.)

SECTION A — TO BE COMPLETED BY REQUESTOR

- ☐ CHEMICAL ANALYSIS
- ☐ DEVELOPMENT SERVICES, LSD
- ☐ ENGINEERING SERVICES, LSD
- ☐ PROJECT ENGINEERING, TOD
- ☐ HIGH LEVEL CAVES
- ☐ STANDARDS LABORATORY
- ☐ EQUIPMENT ENGINEERING
- ☐ GLASSBLOWING SHOP
- ☐ MINICOMPUTER MAINTENANCE SERVICES
- ☐ SCIENTIFIC COMPUTATIONS
- ☐ _____
- ☐ _____

REQUESTED BY | DATE | DATE REQUIRED

LOCATION | PHONE NUMBER

JOB NUMBER | DEPARTMENT / DIVISION

SECTION C — TO BE COMPLETED BY SERVICE GROUP

GAINE | GROUP | ORDER NUMBER

JOB TITLE

ESTIMATE — UNITS / HOURS — DOLLARS

☐ MONTHLY ☐ LUMP SUM

WORK REQUEST (OVER $500)

COST PURCHASE ORDER (OVER $500)

TOTAL COST (THIS ORDER)

ESTIMATED BY | LEAD ENGINEER

WORK REQUESTED / PROJECT OBJECTIVES

REFERENCES (PRIOR DISCUSSIONS, CORRESPONDENCE, PERSONS CONTACTED)

SECTION B — TO BE COMPLETED BY REQUESTOR FOR DESIGN OR ENGINEERING SERVICES ONLY

FIRE PROTECTION REVIEW ☐ YES ☐ NO

CRITICALITY REVIEW ☐ YES ☐ NO

ENVIRONMENTAL EVALUATION ☐ YES ☐ NO

PRELIMINARY PROCESS HAZARDS REVIEW

☐ REQUIRED — IF REQUIRED, ARE PROCESS HAZARD ITEMS IDENTIFIED? ☐ YES ☐ NO

☐ NOT REQUIRED — IF NOT REQUIRED, RESEARCH / CHIEF SUPERVISOR CONCURRENCE _____

DESIGN CATEGORY

☐ NUCLEAR PROCESS ☐ NON-NUCLEAR PROCESS ☐ GENERAL SERVICES

LIAISON | PHONE NUMBER | IF PROCESS HAZARDS ARE IDENTIFIED OR NP CATEGORY ASSIGNED, QUALITY REPRESENTATIVES CONCURRENCE IS REQUIRED _____

BASIC DATA

☐ ATTACHED ☐ TO BE DEVELOPED AND SUBMITTED ☐ INCLUDED ABOVE

SECTION D - AUTHORIZATION

EST COMPLETION DATE | ACTUAL COMPLETION DATE | SERVICE GROUP APPROVAL

COST ACCOUNT CODE | IDP NUMBER | REQUESTING DEPARTMENT / DIVISION AUTHORIZATION | DATE

DISTRIBUTION WHITE - SERVICE GROUP BLUE - REQUESTOR
YELLOW - BUDGET DIV PINK - SERVICE GROUP

Figure 1. SRL Service Orders, OSR 24-C213

Figure 2

Savannah River Site
Chain of Custody Record

Submitter _____ Date _____

Address _____

Radioactivity _____ Disposal _____

Fissionable _____ Sample Site _____

Chemical Hazards _____ Material _____

Heterogeneous _____

Submission ID: _____

Item #	User ID	ASG LIMS Sample ID	Sampled by/Date

Typical

Page 1 of 2

M90mar032.01

Figure 2. Savannah River Site Chain-of-Custody Record

Figure 2
(Continued)

Savannah River Site
Chain of Custody : Prefill Names and Organizations; Receivers and Releasers Will Sign Upon Routing

	Date/Time	Released By	Received By	Purpose
Submitter:		Organization:	Organization:	Sampler to Sample Packaging
		Name:	Name:	
Date		Signature	Signature	
Submission ID		Organization:	ASG, 773-A, B150	Analytical Service Group (ASG) acknowledges receipt of samples and routes to analyses locations
		Name:	ASG Receiving Technician	
No. of Item		Signature	Signature	

Method	Analyses Requested (Operational)	Analyst's Signature	Date Received	Date Completed

	ASG, 773-A	ASG, 773-A, B150	ASG collects samples from analyses locations and returns to customer
	Name (Last Analyst):	ASG Disposing Technician	
	Signature	Signature	
	ASG, 773-A, B150	Organization:	Return Chain of Custody Record to designated submitter for record keeping
	ASG Disposing Technician	Submitter:	
	Signature	Signature	

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M90mar032.01

Figure 2. Savannah River Site Chain of Custody Record (Continued)

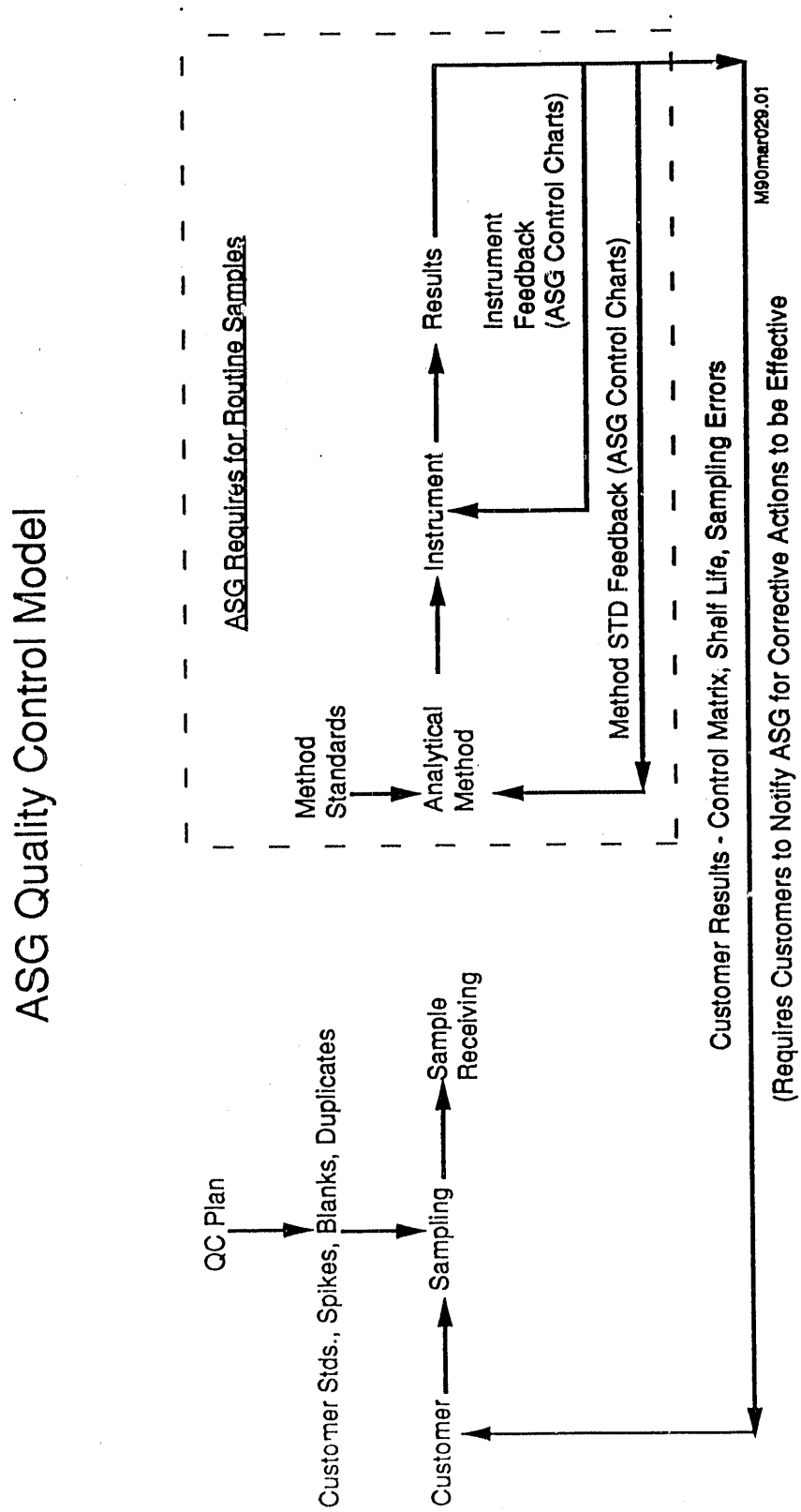


Figure 3. ASG Audit/Control Model

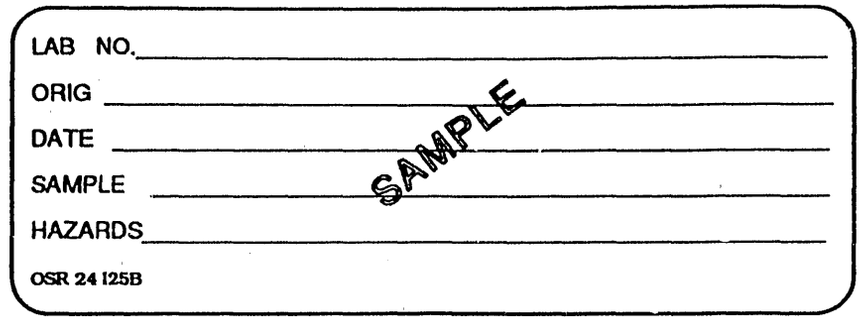
OSR 21-E33 (Rev 2-83)

ANALYSIS REQUEST
SAVANNAH RIVER LABORATORY
ANALYTICAL DIVISION

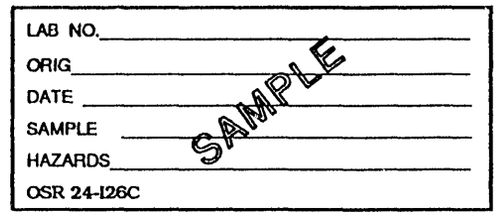
SERVICE ORDER NO.	BADGE NO.	REQUESTOR	DATE
RADIOACTIVE MATERIAL (YES OR NO) ___ ALPHA ___ BETA/GAMMA ___ MAJOR NUCLIDE ___		CRITICALITY MATERIAL (YES OR NO) ___ MAJOR ISOTOPE ___ WEIGHT ___	
HAZARDOUS MATERIAL (YES OR NO) ___ CARCINOGEN ___ TOXIC ___ EXPLOSIVE ___ OTHER ___		SAMPLE DELIVERED TO ADD REC ___ ADD CELL ___ CHEMIST NAME ___	
RETURN SAMPLE (YES OR NO)	PRINT THIS REQUEST (YES OR NO)	ANALYSIS REVIEWED WITH	COMMENT

ADD SAMPLE NUMBER	SAMPLE ID	SAMPLE TYPE	LABEL COLOR	ANALYSIS							
		SOLID ___ g LIQUID ___ o/a VOLUME ___ ml GAS ___ yes/no	RED YELLOW WHITE	EST ANAL							
		SOLID ___ g LIQUID ___ o/a VOLUME ___ ml GAS ___ yes/no	RED YELLOW WHITE	EST ANAL							
		SOLID ___ g LIQUID ___ o/a VOLUME ___ ml GAS ___ yes/no	RED YELLOW WHITE	EST ANAL							
		SOLID ___ g LIQUID ___ o/a VOLUME ___ ml GAS ___ yes/no	RED YELLOW WHITE	EST ANAL							
		SOLID ___ g LIQUID ___ o/a VOLUME ___ ml GAS ___ yes/no	RED YELLOW WHITE	EST ANAL							
		SOLID ___ g LIQUID ___ o/a VOLUME ___ ml GAS ___ yes/no	RED YELLOW WHITE	EST ANAL							
		SOLID ___ g LIQUID ___ o/a VOLUME ___ ml GAS ___ yes/no	RED YELLOW WHITE	EST ANAL							
		SOLID ___ g LIQUID ___ o/a VOLUME ___ ml GAS ___ yes/no	RED YELLOW WHITE	EST ANAL							
		SOLID ___ g LIQUID ___ o/a VOLUME ___ ml GAS ___ yes/no	RED YELLOW WHITE	EST ANAL							
		SOLID ___ g LIQUID ___ o/a VOLUME ___ ml GAS ___ yes/no	RED YELLOW WHITE	EST ANAL							
		SOLID ___ g LIQUID ___ o/a VOLUME ___ ml GAS ___ yes/no	RED YELLOW WHITE	EST ANAL							
		SOLID ___ g LIQUID ___ o/a VOLUME ___ ml GAS ___ yes/no	RED YELLOW WHITE	EST ANAL							
		SOLID ___ g LIQUID ___ o/a VOLUME ___ ml GAS ___ yes/no	RED YELLOW WHITE	EST ANAL							

Figure 4. Analysis Request, Savannah River Laboratory Analytical Division, OSR 21 E33

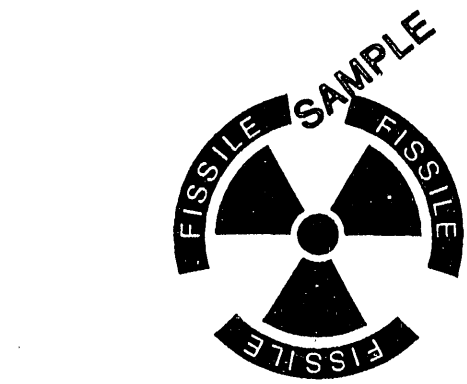


OSR 24-125B



OSR 24-126C

Figure 5. Sample of Pressure Sensitive Labels



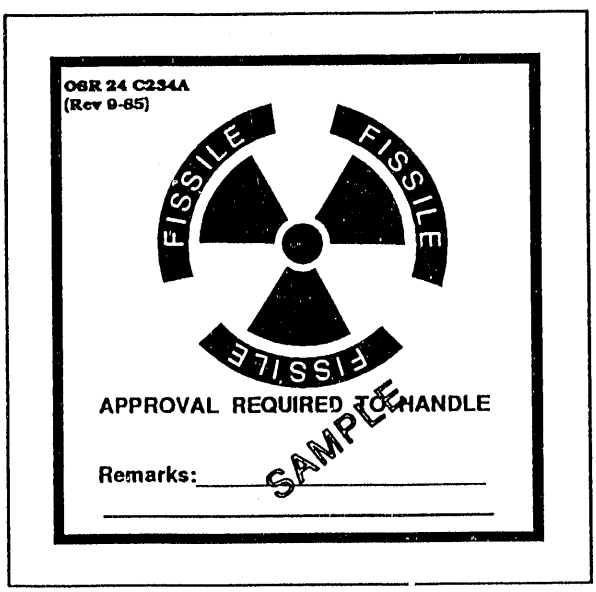


Figure 6. Fissile Material Labels

BM - 07/24/89 - 11:30 - #16-1A - (36-B)(3rd)

OSR 16-1A (Rev. 7-89)

INTERCUSTODIAL & INTERDIVISIONAL NUCLEAR MATERIAL (NM) TRANSFER REPORT SAVANNAH RIVER LABORATORY

CHECK APPLICABLE BOXES	DISTRIBUTION
☐ Custodian Change ☐ Location Change ☐ Requires new S/N ☐ Requires IDTR (see remarks) ☐ Receipt into Existing S/N ☐ Other*	White - Shipper to receiver to SRL Accountability Pink - Shipper Goldenrod - Receiver Blue - OHP

SECTION A (Complete for all accountable NM, DPSTOM-58, Fig 1.1) See reverse side for instructions and reportable weight units.

FROM			TO: "Q" CLEARANCE REQUIRED	
1. TRANSFERRED BY	BADGE NO.		7. RECEIVED BY	BADGE NO.
2. NM CODE	SERIAL NUMBER		8. NEW SERIAL NO.	
3. NM QUANTITY	% NM %	% TOT %	9. SECTION	BLDG
4. NM PHYSICAL DESCRIPTION			10. LAB (ROOM NO.)	LOCATION
5. SECTION				
6. LAB (ROOM)	LOCATION			

SECTION B (Complete for materials in Table 1 & 2, General rules, DPSTP-68-108)
NOTE: COMPLETE SECTION B PER DPST-68-108, GENERAL RULES, SECTION C.2, "CRITICALITY SAFETY MASS LIMITS."

11. DPST-68-108		17. DPST-68-108	
12. TRANSFERRING AREA		18. RECEIVING AREA	
13. ADMINISTRATIVE MASS LIMITS	BOUNDARY ZONE MASS LIMIT (220g)	19. AREA ADMINISTRATIVE MASS LIMITS	BOUNDARY ZONE MASS LIMIT (220g)
14. AREA PRE-TRANSFER NM INVENTORY	BOUNDARY ZONE PRE-TRANSFER NM INVENTORY	20. AREA PRE-TRANSFER NM INVENTORY	BOUNDARY ZONE PRE-TRANSFER NM INVENTORY
15. AREA POST-TRANSFER NM INVENTORY	BOUNDARY ZONE POST-TRANSFER NM INVENTORY	21. AREA POST-TRANSFER NM INVENTORY	BOUNDARY ZONE POST-TRANSFER NM INVENTORY
16. AREA NET NM INVENTORY CHANGE	BOUNDARY ZONE NET NM INVENTORY CHANGE	22. AREA NET NM INVENTORY CHANGE	BOUNDARY ZONE NET NM INVENTORY CHANGE

SECTION C (Authorization required on all NM transfers)

23. NM CUSTODIAN	DATE	PHONE	26. NM RECEIVING CUSTODIAN	DATE	PHONE
24. SECTION MANAGER OR DESIGNEE		DATE	27. SECTION MANAGER OR DESIGNEE		DATE
25. HP		DATE	28. RECEIVER		DATE

REMARKS

For SRS or off-plant shipments, please supply: RECEIVER'S NAME _____.

PHONE NO. _____, MATERIAL BALANCE AREA (MBA) _____, CONTROL BALANCE ACCOUNT (CBA) _____.

Copy of request for off-plant shipment - Radioactive material, OSR 24-C214, required for off-plant shipments only.

*OTHER _____

*U.S. GOVERNMENT PRINTING OFFICE: 1989-642-067/40760

Figure 7. Intercustodial and Interdivisional Nuclear Material (NM) Transfer Report, Savannah River Laboratory, OSR 16-A

GROUP 1: COUNTED TOWARD METHOD "A" TOTAL.

Protactinium, any isotopic mixture.
Enriched uranium, not counting any U-238 which is present.
Neptunium, any isotopic mixture.
Plutonium, any isotopic mixture with more Pu-240 than Pu-241.
Americium containing less than 10% (AM-242 + Am-242M).
Curium containing less than 5% (Cm-243 + Cm-245).

GROUP 2: EXCLUDED FROM METHOD "A" TOTAL

Depleted uranium in any amount.
Normal uranium, if total weight is one metric ton or less.
U-238 present in enriched uranium.
Any isotopes of atomic number 90 (thorium) or below.
Group 3 materials totalling ≥0.1 grams (collectively).

GROUP 3: METHOD "A" INAPPLICABLE WITH THE PRESENCE OF MORE THAN A TOTAL OF 0.1 GRAMS (COLLECTIVELY) OF THE FOLLOWING MATERIALS.

Plutonium with as much Pu-241 as Pu-240, or more.
Americium containing 10% (Am-242 + Am-242m), or more.
Curium containing 5% (Cm-243 + Cm-245), or more.
All isotopes of atomic number 97 or above (Bk, Cf, etc.).

Figure 8. Isotope Groups of Method "A"

OSR 22-7 (Rev. 6-89)

PRECIOUS METALS TRANSFER ADVICE
Savannah River Site

DISTRIBUTION:
ORIGINAL TO TRANSFERRING CUSTODIAN
DUPLICATE TO PROPERTY ACCTG, PRECIOUS METALS, BLDG 742-A
TRIPLICATE TO RECEIVING CUSTODIAN

Date _____

FULL DESCRIPTION OF ITEMS TRANSFERRED	PRECIOUS METAL WEIGHT	FOR ACCTG. DEPT. USE	
		VALUE	VOUCHER

FROM

LOCATION _____

ACCOUNT _____

TO

LOCATION _____

ACCOUNT _____

RETIREMENTS

REASON: EXCESS ☐ DAMAGED ☐

SCRAP ☐ BURIED ☐

CHARGE
COST CODE: _____

(DESCRIPTION ABOVE SHOULD INCLUDE ESTIMATED PRECIOUS METAL WEIGHT REMAINING IN EACH ITEM.)

RECEIVED BY _____
CUSTODIAN

TRANSFERRED BY _____
CUSTODIAN

APPROVAL OF CONSUMPTION _____
DIVISION MANAGER

Figure 9. Precious Metals Transfer Advice, OSR 22-7

Personnel

ANALYTICAL DEVELOPMENT SECTION

Manager		
C. E. Coffey		5-3711
Quality Assurance		
E. F. Sturcken	Manager	5-2790
H. B. Aiken	QA Records	5-5255
Solid Waste and NDA Aassay		
R. C. Hochel	Manager	5-1344
Liquid Waste Analytical Support		
K. Andringa	Manager	5-5314
Process Control and Analyzer Development		
C. W. Jenkins	Manager	5-3049
Analytical Services Group		
W. A. Spencer	Manager	5-2198

ANALYTICAL SERVICES GROUP ORGANIZATIONAL CONTRACTS

Management Support Personnel		
Bill Kerrigan	LIMS Manager	5-3306
George Bizub	Computers	5-3306
Lynda Weatherford	Sample Receiving	5-3953
Shirley McDaniel	Property	5-1211
Dot Matthews	Secretary	5-2605
Patty Thomas	Clerk	5-3559
Sptectroscopy		
Mel Bryant	Task Manager	5-8176
Bob Bean	Task Manager	5-1211
Labs		
AA, B143		
Rosemary Ingram	Analyst	5-1368
ICPES, B151		
Miriam Cooley	Analyst	5-5523
ICPMS, B147		
Rodell Harris	Analyst	5-5523
DISS, B101		
Beverly Burch	Analyst	5-1285

Material Characterization

Ed Sturcken	Task Manager	5-2790

Labs

SEM/CSEM, C108/C059

Dan Steedly	Task Manager	5-2167
Jack Durden	Analyst	5-2324

XRD/XRF/Particle Size, C110

Art Jurgensen	Task Manager	5-5318
Roy Howell	Analyst	5-1784
Joyce Hunter	LANL Analyst	5-1784

Microprobe, C146

Laura Feezel	Task Manager	5-8177
	Analyst	5-2924

Porosity/BET Surface Area, C143

Bob Malstrom	Task Manager	5-3140
Debbie Marsh	Analyst	5-8273

Radiation Chemistry

Bob Hochel	Task Manager	5-1344

Labs

Rad Chem Prep, B138

Roy Rhinehart	Task Manager	5-2034
Arlene Ray	Analyst	5-1340

Counting Room, B145

Roy Rhinehart	Task Manager	5-2034
Glenda Fulmer	Analyst	5-1228
Charlie Parkman	Analyst	5-1228

Neutron Activation, B003

Glenda Fulmer	Analyst	5-2549

Ions in Solution

Amy Almon	Task Manager	5-1236
Liz Baumann	Task Manager	5-1421

Labs

Ion Chromatography, B134

Peggy Widener	Analyst	5-1359

Ions in Solution (Continued)

Labs

Electrochemistry, B159		
Sharon Fulton	Analyst	5-4136
Titrations (acids, bases, CL, F, A1, C03), B154		
Roy Rhinehart	Task Manager	5-2034
Maxine Williams	Analyst	5-1367
Nancy Wallace	Analyst	5-1367

Wet Chemistry and Misc. Analyses

Mike Polochko	Task Manager	5-8178
Roy Rhinehart	Task Manager	5-2034
Mike Whitaker	Task Manager	5-6288

Trace Uranium
Leco Analyzers (carbon, nitrogen, hydrogen)
Wet Carbon Analyzers
TSD, TSS, Density
Organic/Aqueous
Oil and Grease
Flashpoint
Fe2/Fe3
Flow Injection Techniques
Misc. Analyses

Maxine Williams	Analyst	5-1367
Nancy Wallace	Analyst	5-1367
Mira Gray	Analyst	5-1295

Offsite Analyses, B161

Edna DeWeese	Task Manager	5-2352
Mike Polochko	Task Manager	5-8178

Organics (GC, IR), B046

Bruce Buchanan	Task Manager	5-1963
Miriam Cooley	Analyst	5-5611
Sharon Fulton	Analyst	5-5611

TNX Labs

Tim Policke	Task Manager	5-6006
Chuck Nold	Task Manager	5-6242
Mike Whitaker	Task Manager	5-6288

IONS IN SOLUTION

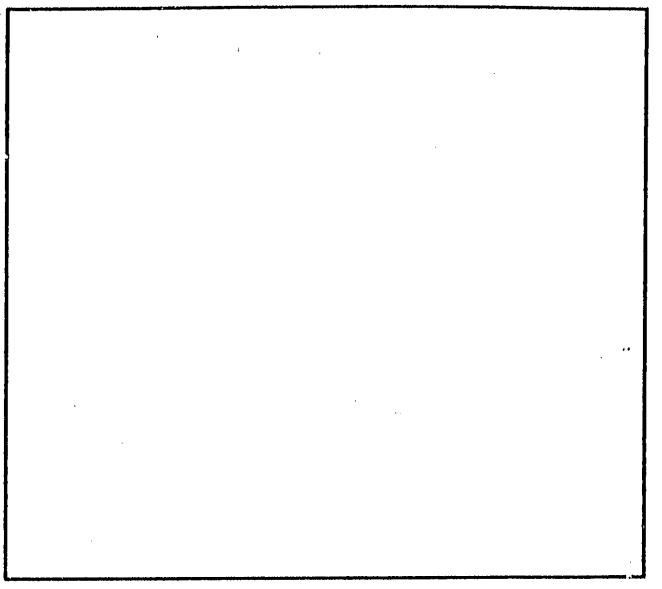
Conductivity of Solutions

Application
Determines the electrical conductivity and resistivity of solutions.

Sample
State: Aqueous solutions
Size: 25 - 100 mL
Radioactivity Level: None

Determination Range
Conductivity range is 0 - 999,000 micromhos/cm at 25°C.

Basic Principle
Conductivity is numerical expression used to describe the ability of an aqueous solution to carry an electric current. This ability is dependant upon the concentration, mobility, valence, and relative concentrations of ions present as well as the temperature of the solution. The conductivity measurement involves the determination of resistance of a segment of solution between two parallel electrodes by means of Ohms law.



Instrumentation
Amber Science Model 1052A Digital Conductivity Meter

Examples
- Assess degree of mineralization of distilled and deionized water.
- Evaluation of dissolved mineral concentrations in wastewaters.
- Estimates of total dissolved solids in samples.

Electrochemical Methods

Applications

- Quantitative and qualitative elemental analysis on more than 90% of all elements in the periodic table in aqueous and organic solutions.
- Separation and speciation of component mixtures.
- Synthesis.
- Development of online analysis methods and instrumentation which perform the above applications.

Sample

State: Solid, liquid, or solutions

Amount: Dependent upon analysis, usually 50 mg minimum.

Radioactivity: Radioactive samples can be analyzed depending upon the specific analysis method. Analyses done in the hood can have an activity of no more than 10^6 d/m/mL.

Determination Range

Dependent upon sample type and analysis method. Sub part per billion detection limits are available

Principle

A wide variety of sophisticated electroanalytical techniques, capable of yielding precise results at very low concentrations of material, are available. The mass or concentration of a component is generally measured by its effect upon impressed voltage, by the change in its chemical state with the passage of current, or by its effect on an electrode.

Electroanalytical techniques can be used for both quantitative and qualitative work. These methods are ideally suited for trace species analysis; many online sensors based on simple electrochemical principles have been developed. In quantitative work, species with concentrations from 10^{-1} to 10^{-9}M have been determined with accuracies from a few tenths of one percent to ten percent relative.

Instrumentation

BAS 100A Electrochemical Analyzer (with high current and ultra low current capabilities)

PAR 384 Polarographic Analyzer

PAR 173 Potentiostat/Galvanostat

BAS LCEC Analysis System

Examples

Electrochemical analysis of alkali metals

Electrochemical analysis of transition metals

Trace analysis of actinide metals in waste streams

Electrolytic oxidation of SRL organic waste

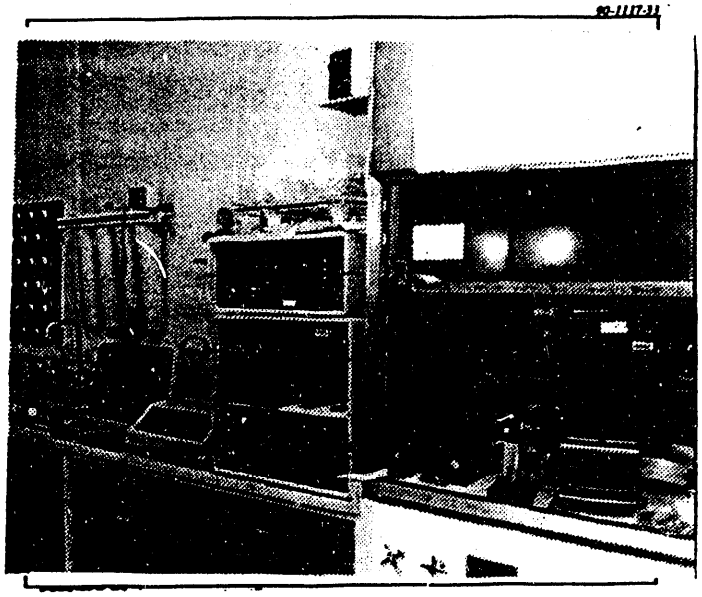
Determination of Pu(III)/Pu(IV) ratio in F-Canyon

Trace analysis of cesium in aqueous waste samples

Voltammetric determination of oxidation state of melter glass.

Ion Chromatography Methods

Task Supervisor: A.C. Almon
Technical Analyst: P.R. Widener



Applications

- Quantitative and qualitative elemental analysis on more than 90% of all elements in the periodic table in aqueous and organic solutions.
- Separation and speciation of component mixtures.
- Development of online analysis methods and instrumentation which perform the above applications.

Sample

State: Solid, liquid, or solutions
Amount: Dependent upon analysis, usually 50 mg minimum.
Preparation: Solids must be dissolved into appropriate solution. Liquids will be diluted into appropriate solution.

Determination Range

Dependent upon sample type and analysis method. Sub part per million detection limits are available. Results are generally given in micrograms per mL.

Time Required

Variable, depending on number and type of species being analyzed and type of analysis being performed. Once the analysis has been started, analysis time is no more than 20 minutes.

Principle

Ion chromatography is the separation of substances by their differential migration on an ion exchange column or on a sheet impregnated with an ion exchanger. The sample ions are moved down or eluted from the column with an eluent solution. This is accomplished through the competition of eluent ions and sample ions that react with functional groups on the ion exchanger. Since these reactions are reversible, the ions will pass between the stationary and mobile phases several times as it travels down the column. The ability of a sample to compete with the eluent ions depends on the characteristics of the ion exchanger and the sample ion. The affinity of the sample ion for the ion exchanger is unique, thus providing the basis for performing separations of ion mixtures.

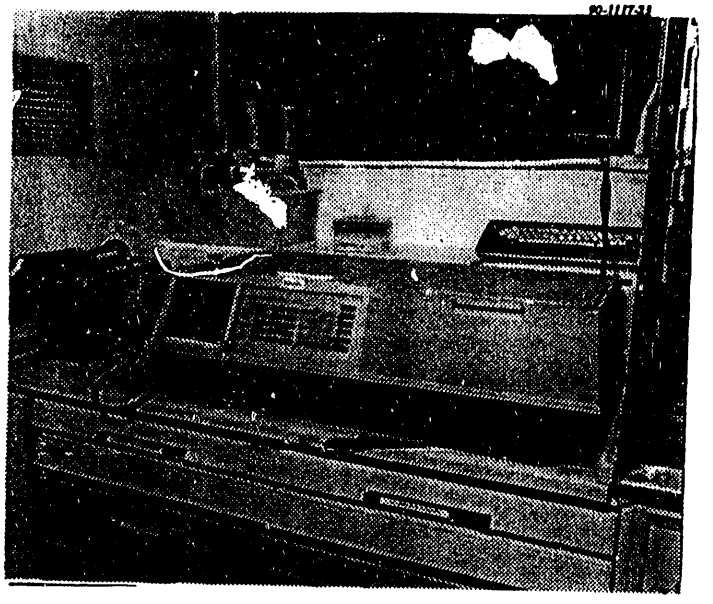
Instrumentation

- Dionex Model 2020i Chromatography System (3 units) with conductivity and spectrophotometric detection capabilities and gradient pump capacity.

Examples

- Analysis of alkali metals
- Analysis of transition metals
- Analysis of Lanthanide metals
- Anion analysis: Cl^-, Fl^-, NO_2^-, NO_3^-, SO_4^{2-}, PO_4^{3-}, $C_2O_4^{2-}$, CHO^{2-}
- Cation analysis: NH_4^+

Spectrophotometric Methods



Application

Colorimetry is used to determine the concentration of a wide variety of substances in solution.

Sample

State: Samples must be in solution. Liquid samples should be homogeneous and contain no solids or precipitates.

Size: Minimum sample size varies with the specific absorptivity and the concentration of measured and interfering ions.

Radioactivity: Radioactive samples can be analyzed depending upon the specific analysis. Samples must have less than 10^6 d/m/ml alpha or 10^8 d/m/ml beta-gamma.

Determination Range

Varies with type of analysis. Ranges from 1 ppm level to parts per thousand.

Basic Principle

When an electromagnetic wave passes through a medium, (e.g., liquid or gas) some loss of intensity occurs. If the losses caused by reflections and scattering in the medium are ignored, then the absorption of radiation by the substances in the medium is a function of their concentration. The absorbance depends upon the composition of the medium, the length of the radiation-absorbing path, and the concentration of absorbing species in the medium. This relationship is expressed by Beers law and is the basis for determining concentrations by colorimetry.

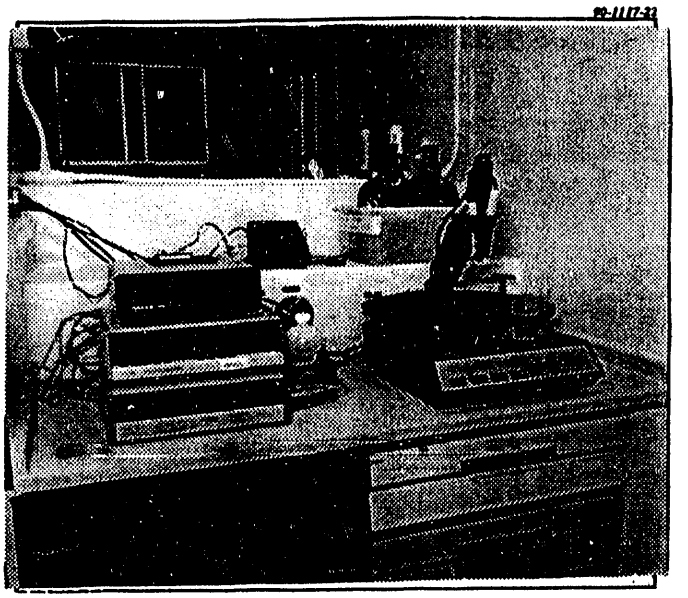
Instrumentation

Brinkman PC 800 Probe Colorimeter
Hewlett-Packard Model 8451-A Photodiode Array Spectrophotometer

Examples

Determination of Fe(II)/Fe(III) ratio in glass
Determination of Copper(I) in precipitate hydrolysis samples

Titration Methods



Application

Quantitative and qualitative analysis of aqueous and organic solutions.

Sample

State: Liquid or solutions

Amount: Dependent upon analysis, usually 50 mg minimum.

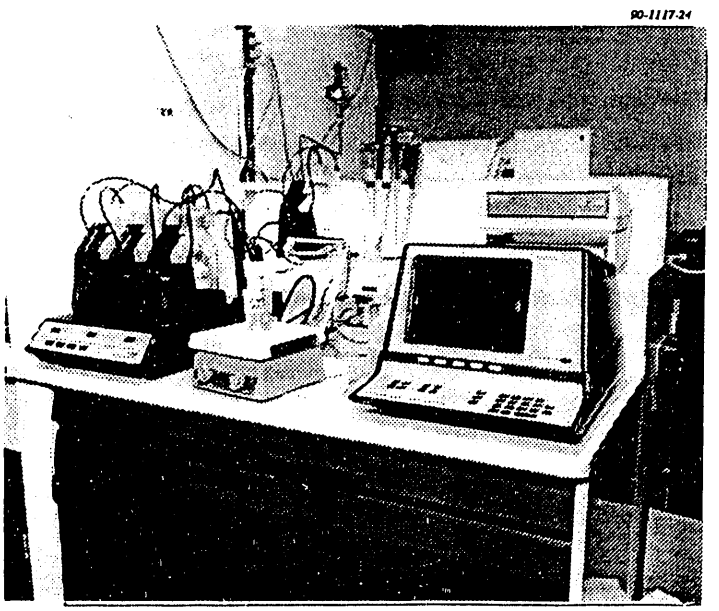
Radioactivity: Radioactive samples can be analyzed depending upon the specific analysis method. Analyses done in the hood can have an activity of no more than 10^6 d/m/mL.

Determination Range

Dependent upon sample type and analysis method. Part per billion detection limits are available.

Principle

In a titration, the amount of substance present in a sample is determined by measurement of the quantity of reagent - called the titrant - required to react stoichiometrically with that substance.



The titrant is added to the sample until the reaction with the species of interest is complete. This endpoint is measures by an abrupt change in some characteristic of the solution such as color, refractive index, conductivity, temperature, or potential difference between reference and indicator electrodes immersed in solution.

Instrumentation

VIT-90 Video Titrator

Mettler DL40RC Memotitrator

Examples

Water determination using Karl-Fischer reagent

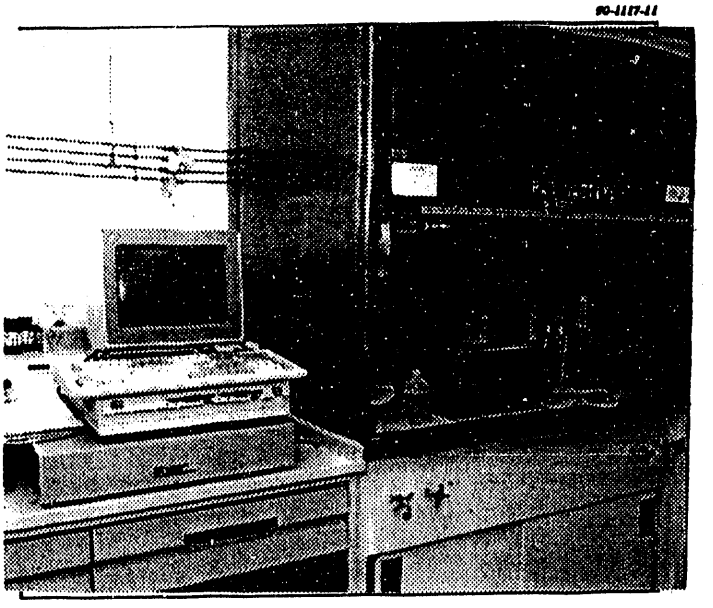
Determination of Tetraphenylboron using silver nitrate

Determination of Tetraphenylborate

Determination of Hydroxide, Aluminate, and Carbonate

Determination of total acid

Uranium Analysis



Application

This technique is applicable to the measurement of ultra trace levels of uranium in aqueous and organic solutions, including radioactive solutions.

Sample

State: Aqueous or organic liquids
Size: 1 mL or more
Radioactivity level: Samples must have less than 10^6 d/m/mL alpha or 10^8 d/m/mL beta-gamma.

Determination Range

1 ppb to 100 ppm. Samples higher than 100 ppm should be diluted before submitting.

Basic Principle

This technique, Pulsed Laser Fluorometry, is based on the fluorescence exhibited by uranium in phosphoric acid when exposed to ultraviolet light provided by a UV laser. The uranyl ions emit a characteristic blue-green luminescence which is quantitively measured by a photodetector. The fluorescence spectrum consists of three regularly spaced peaks at approximately 494, 516, and 540 nm.

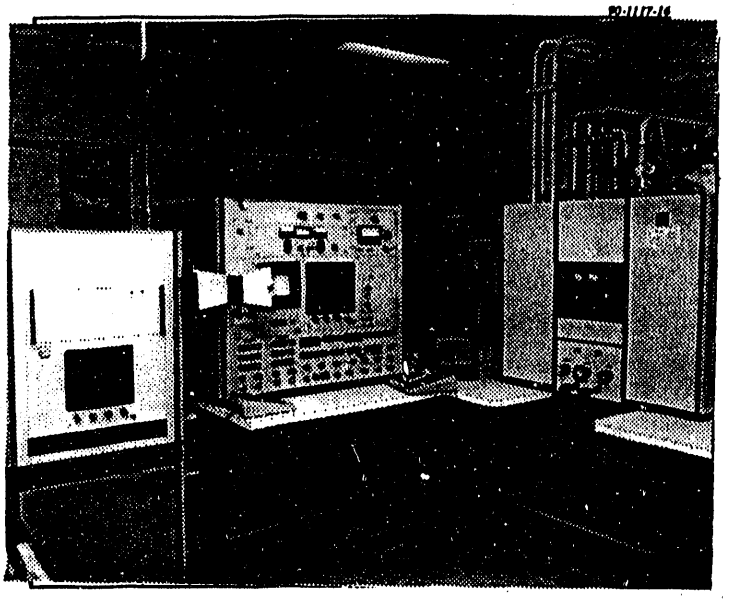
Instrumentation

Scintrex UA-3 Uranium Analyzer
Hewlett Packard HP-85b Microcomputer

Examples

Uranium content determined in wells and streams, sludge dissolutions, Pu solutions, and organic solutions.

Electron Microprobe Analysis



Application

Qualitative and quantitative microscopic analyses of samples to determine elemental composition and distribution. Elements with atomic numbers greater than four can be analyzed.

Sample Characteristics

State: Solid

Size: Samples must fit on specimen mounts 1 inch or 1 1/4 inches in diameter or on a 1 x 2 inch rectangular slide. Sample areas as small as 1 μm^2 can be analyzed.

Preparation: Quantitative analysis requires that the sample have a highly polished surface. Nonconductive or semi-conductive samples will be coated with a thin conductive carbon film.

Time Required

Sample Preparation: Variable, depending on nature of sample.

Instrument Time: Variable, depending on complexity of sample and number of elements exam. Nominal times range from two hours to two days.

Detection Limits

50 ppm to 500 ppm depending on element, matrix material, and counting time.

Basic Principle

A finely focused electron beam is used to generate characteristic x-rays from the elements in a sample which are then detected by a wavelength or energy dispersive spectrometer. The x-ray intensity is proportional to the concentration of the corresponding element. In addition, detection of backscattered electrons, absorbed electrons, and secondary electrons provide information about the atomic number of the elements in the sample and the surface topography. The use of well characterized standards is required for quantitative analysis.

Instrumentation

Applied Research Laboratories Scanning Electron Microprobe Quantometer

Tracor Northern TN 5500 Energy Dispersive X-Ray System

Examples

Determination of the distribution and composition of phases in lathanum-nickel-aluminum alloys

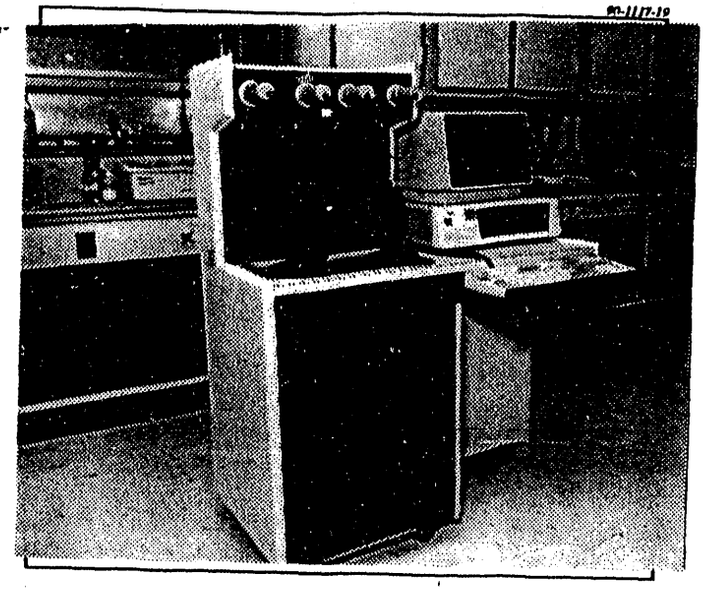
Flow lines and microscopic elemental inhomogeneities in stainless steel

Mineral identification in DWPF waste glasses

Identification of foreign inclusions and second phase material in U-Al alloy fuel tubes

Identification of corrosion products

Mercury Intrusion Porosimetry



Application
Determination of pore volume, pore distribution, and pore surface area.

Sample
State: Solids-powders, pellets, or chunks
Size: 0.1 to 25 g
Radioactivity Level: None

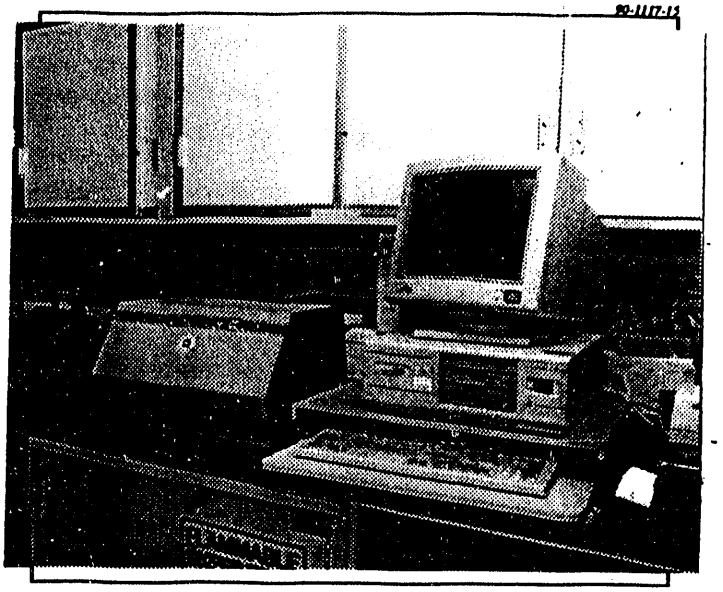
Determination Range
The instrument is capable of measuring pore sizes from 0.003 to 200 micrometers.

Basic Principle
Pore structure analysis is performed by measuring the intrusion of mercury into the pores of a sample as pressure is increased. Intrusion and extrusion of mercury is measured by a change in electrical capacitance. These capacitance changes are proportional to the sample pore size.

Instrumentation
Micromeritics Autopore II 9220 Mercury Porosimeter.

Examples
Analysis of glass, metal powders, cement, and pigments.

Particle Size Analysis



Application
Particle size and distribution of powders.

Sample
State: Solid (powders)
Size: Particles must be less than 1000 microns when received unless ultrasonic dispersal of an agglomerated material to a smaller size is possible.
Amount: 0.05 to 2 g for small volume recirculator (0.5 g is typical)
0.5 to 20 g for large volume recirculator
0.02 to 0.5 g for small sample cell
Radioactivity Level: None

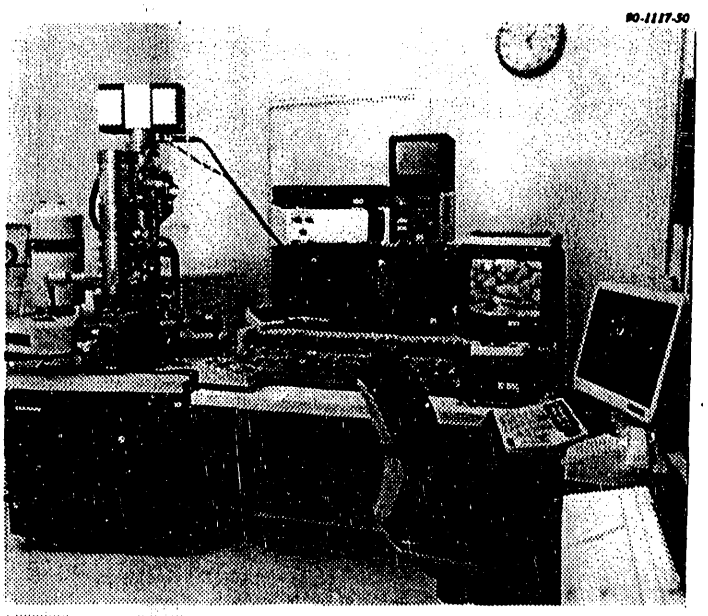
Determination Range
0.7 to 700 microns in diameter

Basic Principle
The sample is suspended in liquid and agitated with a circulating pump. A laser beam is projected through the liquid and is scattered by the particles by the phenomenon of low angle forward scattering. Detectors measure the scattering to get the particle size distribution.

Instrumentation
Leeds and Northrup Microtrac Standard Range Analyzer

Examples
- ceramic powders
- metal powders

Scanning Electron Microscopy/ Contained Scanning Electron Microscope (SEM/CSEM)



Application

Examination and analysis of the microstructural characteristics of solid objects.

Sample

State: Solid

Size: Powders and small objects are mounted on 15 mm diameter pedestals. Polished specimens must fit into 1 1/4 inch metallography mounts. Bulk samples may be studied in a 1 inch vise and should be no more than 1 inch high.

Preparation: All nonconducting samples will be coated with a carbon film for samples requiring elemental x-ray analyses or highly conductive metal film for high resolution images.

Determination Range

Highly dependent upon the matrix composition. Manufacturer's specifications for image resolution is 6 nm. Practical resolution on most samples is 20 nm. Nominal sensitivity of EDispersive X-ray analysis is is is 1000 ppm to 1/2%. Elements greater than atomic number 10 may be detected on the EDS and boron, carbon, and oxygen on the Peak WDS while all elements greater than atomic No. 4 may be detected on the Microspec WDS.

Time Required

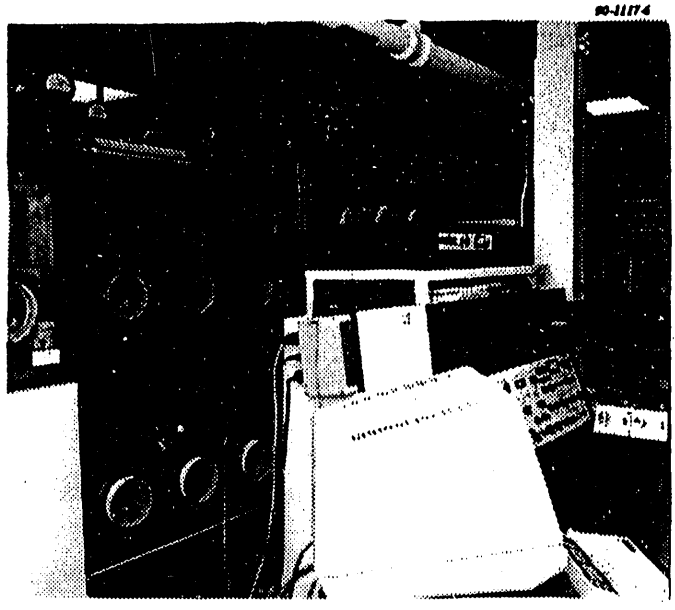
Sample preparation: Variable, depending on nature of sample and type of analysis.

Instrument time: Variable, depending upon complexity of sample and type or types of information required by customer. Nominal times range from two hours to two days.

Principle

The SEM uses an electron beam up to 40 kV which it scans across the surface of the sample under vacuum, producing an image on a CRT display on the console. The SEM has both a secondary electron detector and a backscattered electron detector. Images detected by the secondary detector are sensitive to topography while images from the backscatter detector are sensitive to atomic number. X-rays are generated when the electron beam impinges on the sample. X-rays of element No. 11 or greater may be detected on the Energy Dispersive System (EDS) and X-rays of Boron, Carbon, Oxygen, Sulphur, and Molydenum can be detected on the Peak Wavelength Dispersive System and elements greater than atomic No. 4 may be detected on the Microspec WDS.

Other capabilities of the system include particle size measurement, x-ray mapping and linescan profile, and percent phase distribution. Replicas may be made of surfaces of samples too large to fit into the SEM if they may not be cut. This allows studies of fractures, cracks, surface roughness, and texture.



Scanning Electron Microscopy/ Contained Scanning Electron Microscope (SEM/CSEM)

(Continued)

Instrumentation

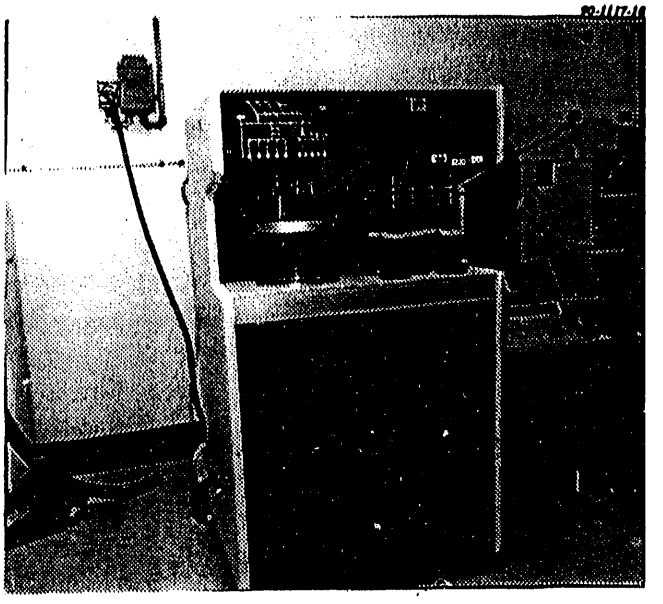
ISI DS-130 Scanning Electron Microscope
Tracor Northern TN-5500 Energy Dispersive X-Ray System
Peak Focus Wavelength Dispersive Spectrometer

In containment: (for examination of radioactive samples)
Cambridge S250 Scanning Electron Microscope
Tracor Northern TN-5500 Energy Dispersive X-ray System
Microspec Wavelength Dispersive X-ray System

Examples

Fracture and failure analysis
Analysis of corrosion scale
Reactions of glass melter
Examination of welds
Analysis of plugged filters
Leached glass studies
Examination of fuels
Dissolution of scrap Pu

Surface Area Analysis



Application
Determines the BET surface area and pore distribution of samples.

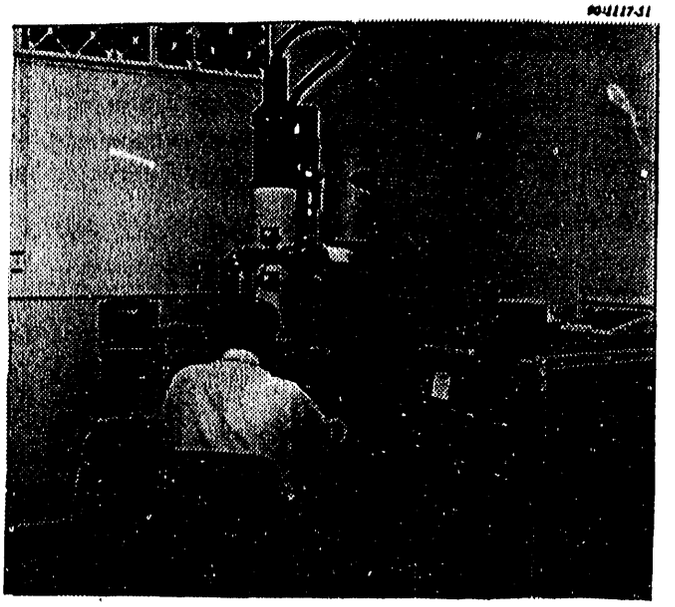
Sample
State: Solid
Size: 1 to 5 g of sample
Radioactivity Level: None

Determination Range
Surface areas can be measured from less than 1 to 2000 sq meters/g and pore sizes from 0.99 to 0.001 *u*m.

Basic Principle
The quantity of nitrogen gas adsorbed onto or desorbed from sample at an equilibrium pressure is measured. The adsorption occurs at liquid nitrogen temperature and is proportional to the specific surface area of the sample. Desorption isotherms are used to relate the pore size to volume of adsorbate lost as then pressure decreases.

Instrumentation
Micromeritics Digisorb 2600

Examples
Surface area and pore size of polymer beads, glass beads, and metal powders.



Transmission Electron Microscopy (TEM/STEM)

Application

Analysis of materials using microstructural information from images and crystallographic information using diffraction patterns. Elemental analyses of atomic numbers greater than 10 are obtained using Energy Dispersive X-rays.

Sample

State: Solid

Size: Sample diameters are 3 mm. Metal samples are ground, dimpled, and thinned to perforation by electropolishing. Glasses and ceramics are ground, polished, and thinned to ~20 nm using an Ion Mill. Fines of particulates suspended on a carbon film may have edges thin enough for the electron beam to penetrate.

Determination Range

Images up to 480,000x magnification, lattice images to 2 nm, and EDX above atomic No. 10 may be obtained on materials thin enough for penetration of a 120 kV electron beam.

Time Required

Sample Preparation: Variable, depending on sample (two hours to two weeks).

Instrument Time: Variable, depending on complexity of sample and information needed (two to five days).

Principle

The TEM uses a static beam of up to 120 kV electrons to illuminate a desired region (~2-70 μm diameter) of an electron-transparent specimen which is immersed in the objective lens of the microscope. Using intermediate lenses, either the image or the diffraction pattern is projected onto a fluorescent screen for observation or a photographic surface for recording. By use of selected area diffraction (SAD) techniques, it is possible to obtain crystallographic information from regions as small as 0.5 μm diameter, and by use of minilenses, it is possible to reduce this further by perhaps a factor or 2X-5X. Use of the STEM mode can reduce the beam probe size down to less than 10 nm. The STEM mode may be used in conjunction with detectors to produce secondary, backscatter, and transmitted images. X-rays are generated when the electron beam impinges on the sample. X-rays of element atomic No. 11 or greater may be detected on the Energy Dispersive (EDS) system.

Instrumentation

Philips EM 400T Transmission and Scanning Transmission Electron Microscope, Kevex Micro-x 7000 Energy Dispersive X-ray Analyzer

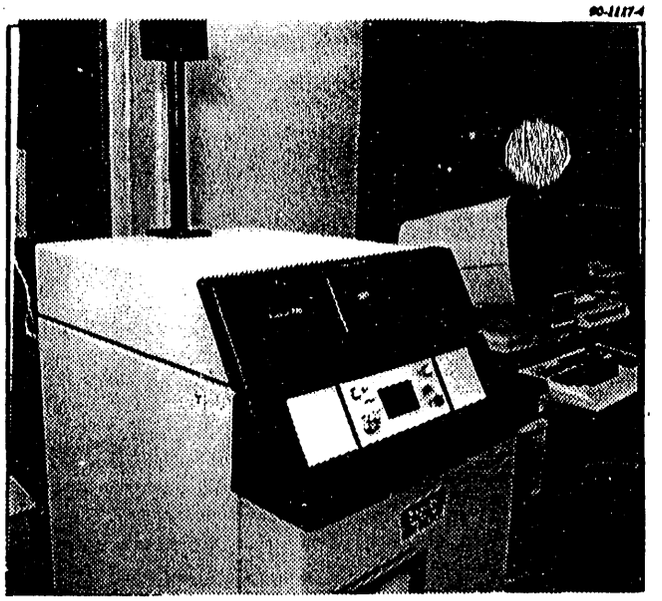
Examples

Images and diffraction patterns of microstructures

Lattice imaging of oriented planes

Weak beam imaging showing phases in other orientations

Observation of stacking faults, dislocations, and other structural factors



X-Ray Powder Fluorescence (XRF)

Application

Identification and quantitation of major and trace elements, C through U in solids and liquids.

Sample

State: Solid or liquid

Size: 5 to 10 g optimum. Smaller samples can be used in special applications.

Preparation: Powders, fused pellets prepared from powders, or polished surfaces 3/4 to 1 3/8 inches in diameter are the preferred solid sample form. Liquids are usually analyzed as received.

Determination Range

Most elements can be determined at the 5- to 100-ppm level up to 100%. Elements can usually be determined within ± 5% relative if present in an amount at least 10 times the detection limit concentration and suitable standards are available.

Time Required

Instrument Time: 1 to 10 minutes depending on the type of sample and precision desired.

Analysis Time: Variable according to the standard and sample preparations required for the analysis and the number of samples. Average time is approximately 20 minutes.

Principle

The sample is exposed to characteristic and Bremsstrahlung x-radiation from a high-intensity x-ray tube. The energy of the x-rays absorbed by the sample is partially reemitted as fluorescent x-rays characteristic of the elements in the sample. The fluorescent x-rays can be dispersed by energy using an Si (Li) detector. The intensities of the separated x-rays for the elements of interest are measured and compared to the intensities derived from a set of calibration standards.

Equipment

Kevex Model 0700/7077
Energy Dispersive X-Ray System
Kevex Quantum Detector

Examples

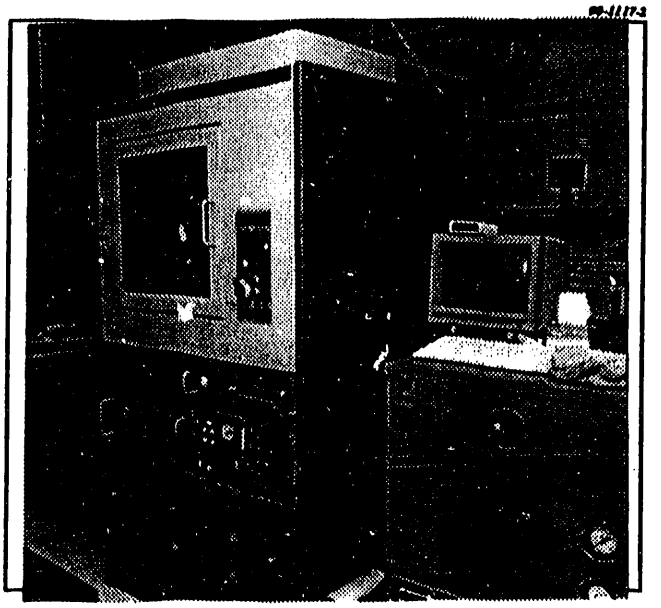
Rapid qualitative analysis for elements present, C through U.

Determination of S, Cl, Br, in water and organic liquids.

Determination of the major rock-forming elements in silicate minerals and glasses.

Determination of major components in cement and fly ash.

X-Ray Powder Diffraction (XRD)



Application

Identification of crystalline phases and measurement of average crystallite size.

Sample

State: Solid (preferably) or paste
Size: 2 g desirable with a minimum of 0.1 mg
Preparation: Sample should be ground to 1-10 mm particle size.

Determination Range

The most intense reflections from some compounds can be detected at 0.5 weight percent. For most compounds, the limit for a positive, qualitative identification is 5 weight percent or greater. Semiquantitative results can be obtained on some types of samples at ±20 percent of the amount present. Average crystallite size determinations are limited to values between 10 and 500 A on single-component materials.

Time Required

Instrument Time: 1-2 hours
Analysis Time: 2-3 hours

Principle

A randomly oriented powder sample is irradiated with a monochromatic beam of x-rays. These x-rays are diffracted from the crystalline layers at angles dependent on the distance between the crystalline planes. The intensities and the angles of the diffraction reflections are recorded and matched with the more than 50,000 compounds indexed in the JCDPS reference file.

Average crystallite size determination is made from the measured width of the diffraction reflection at half maximum.

Equipment

2-Siemens Model D500 Automated Scanning Diffractometers - one in containment
2-Philips Automated Scanning Diffractometers

Examples

Identification of chemical compounds, corrosion products, and precipitates.
Identification and semiquantitative analysis of minerals in rock samples.
Identification of crystals formed in DWPF waste glasses.
Determination of average crystallite size of actinide powders.

ORGANICS

Fourier Transform Infrared Spectroscopy (FTIR)

Application

FTIR analysis has been used to confirm the presence of water, alcohols, quantitatively measure an organic species, and monitor off-gases from a test set-up.

Sample

State: Solid, liquid, or gas
Size: Minimum of 100 *ug*
Radioactivity Level: None

Sampling

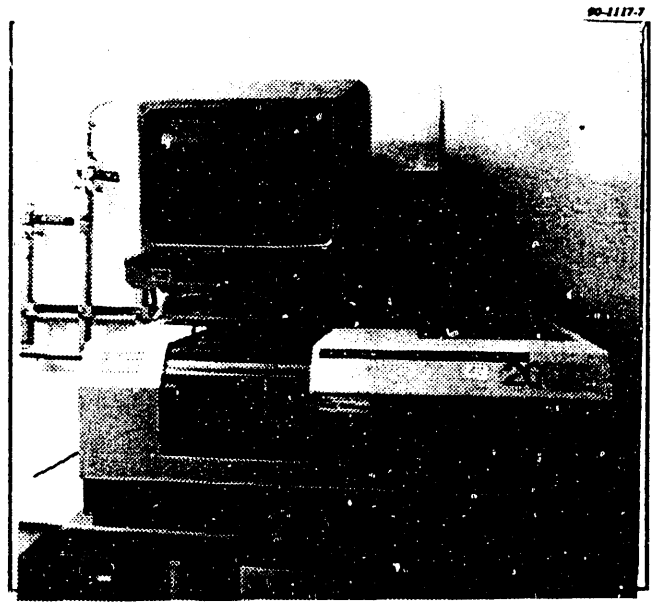
The simplest sampling method is transmission measurements made on organic liquids. This is done by placing a drop of the sample on NaCl, KBr, or AgCl windows. Only organics can be measured using NaCl, KBr or AgCl windows, because aqueous samples will attack these windows. For aqueous samples, CaF_2 or BaF_2 windows must be used. These windows have a shorter useful range but are impervious to water.

If the sample is a solid, then things become more complicated. If the sample is soluble in organic solvents, then a nujol mull can be used or a KBr pellet can be pressed. The final method of measuring solids is to use Diffuse Reflectance Infrared Fourier Transform (DRIFT) spectroscopy.

Aqueous samples are difficult to measure because of the high absorptivities of the O-H vibration in the mid-infrared region and because water attacks most window materials. A Cylindrical Internal Reflection (CIRCLE) cell can be used to measure aqueous samples.

Determination Range

All wavelengths ranging from 4000 to 400 cm^{-1} are detected with a 2 cm^{-1} resolution.



Basic Principle

The FTIR spectrometer consists of a broad band infrared source, interferometer, and a detector. Light from the source is directed into the interferometer, where a beamsplitter transmits 50% of the light to a fixed mirror and reflects 50% of the light to a moving mirror. The light then recombines at the beamsplitter, is directed through the sample and impinges on the detector. The resulting signal is then output to a computer for processing. This instrument differs from a dispersive instrument in the replacement of the grating with an interferometer and the signal processing necessary to obtain a spectrum. The interferogram consists of a power versus time spectrum. Because the detector only measures total light, the value at each time is the sum of the energy from all wavelengths. The Fourier transform maps this time domain onto the wavelength domain.

Instrumentation

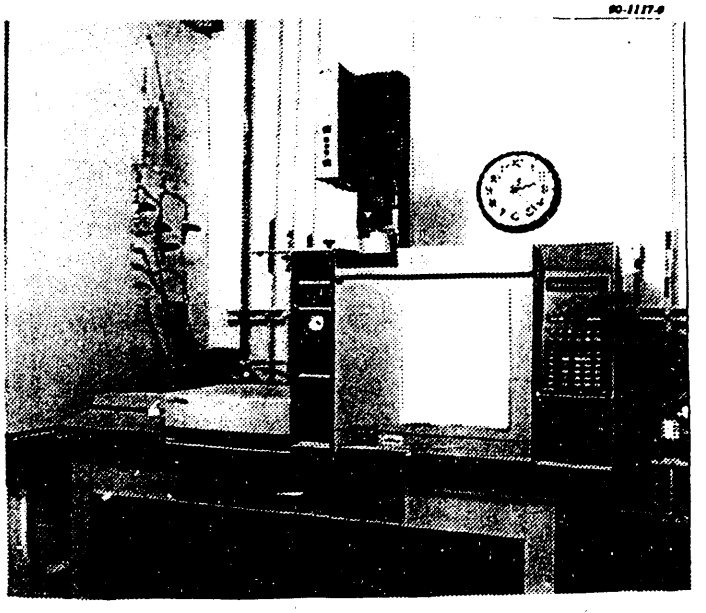
Nicolet 20-DX Spectrometer with the following accessories:

- CICRLE cell
- Drift attachment
- Gas cells

Examples

Material characterization and identification.

Gas Chromatography (GC)



Application

GC analysis is used for quantification of organics in samples. Columns exist for the analysis of both polar and non-polar species.

Sample

State: Liquid or gas
Size: 0.1 mL liquid, 3 cc gas
Radioactivity Level: Samples must have less than 10^6 d/m/mL alpha or 10^8 beta-gamma.

Determination Range

Low ppb to percentages depending on detector and column used.

Basic Principle

The method is quite simple. A sample is introduced in a flowing and inert carrier. The column contains either a solid support or a thin film of a high boiling oil coated on a solid support. Separation occurs because species have different solubilities in the liquid phase or a different affinity for the solid phase. The carrier and the separated species pass into a detector that is sensitive to the desired components.

Instrumentation

- (2) Hewlett-Packard Model 5890 Gas Chromatographs with autosamplers. One is in containment for low level radioactive samples.
- Hewlett-Packard Model 5840 Gas Chromatograph with purge-and-trap attachment.

Examples

- Percentage ethylene glycol
- Percentage Tri-butyl Phosphate
- Percentage sorbed organic
- Identification of ethyl, methyl alcohols
- Identification of diesel and gasoline samples
- Percentage hydrogen evolved

RADIATION CHEMISTRY

Alpha Pulse Height Analysis (APHA)

Application

APHA is used when it is necessary to distinguish between several alpha emitting isotopes. Examples are samples containing two or more isotopes such as: ^{235}U, ^{241}Am, ^{238}Pu, ^{239}Pu, ^{252}Cf.

Sample

Radioactivity Level: Trace levels to 10^5 d/m/mL
State: Sample which are or can be converted to liquid
Size: 1 mL or more
Preparation: The liquid sample must be evaporated or electroplated onto a planchette for counting.

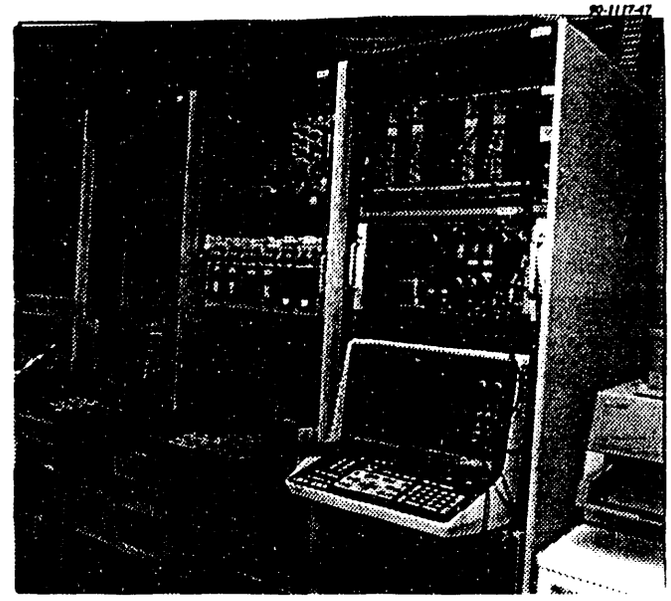
The thickness and evenness of the deposited source affect the quality of the analysis. For best results dilute or salt-free samples should be used.

Detection Range

Detection limit of 10^{-15} Ci/sample is possible for long counting times (16 to 24 hours). Typical range is 10^{-11} to 10^{-7} Ci/sample.

Basic Principle

Like gamma-rays, alpha particle emission from a nucleus is quantized. An alpha detector together with pulse processing electronics and a multichannel analyzer is used to record the number and energy of alpha particles emitted from the source. The resultant spectrum can be analyzed for energy to determine the emitting isotope, and the peak areas are proportional to the isotope's concentration. However, because of the very short range of the alpha particle in matter and limited resolution of most detectors, results are easily compromised by the nature and quality of the sample source.

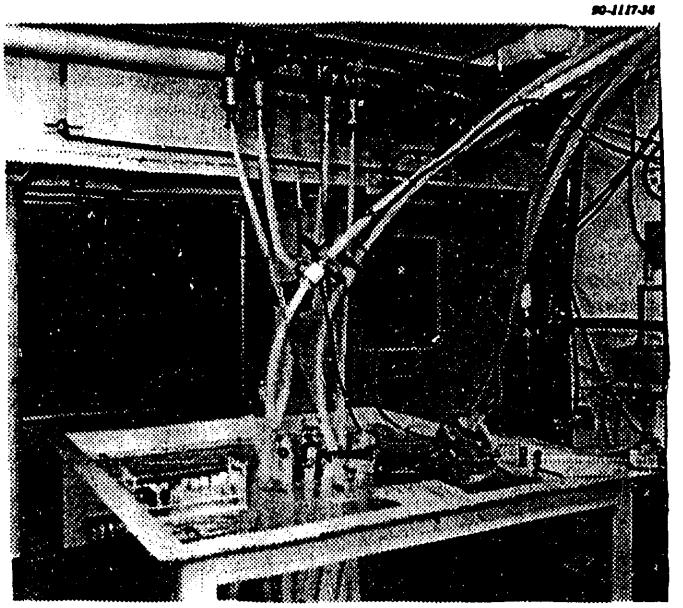


Instrumentation

The ADS counting room uses six Tennelec TC 256 alpha spectrometers coupled to a Canberra Series-80 multichannel analyzer. The spectrometers operate under vacuum to reduce spectral degradation due to air absorption of the alpha particles. Data analysis is performed on the counting room's DEC micro-VAX computer using an ADS developed program.

Examples

APHA using various types of detectors is a well established practice for analysis of actinide elements. Both OHP and Laboratories departments use APHA similarly to ADS for various actinide containing samples.



Californium Neutron Activation Analysis (CNAA)

Application

CNAA finds application in nondestructive multi-element assays of many solid and liquid samples. Typical types of samples include ion exchange resins, soils, cements, coal, fly ash, freeze dried vegetation, and biological samples as well as aqueous samples. Solutions may contain most acids except HCl and HBr which will strongly activate and interfere with some trace analyses. Elemental analyses are determined based on natural isotopic abundances. Samples generally should not be radioactive except for very long-lived or fissile elements such as ^{129}I ^{235}U ^{239}Pu.

Sample

Radioactivity Level: Trace or none
State: Most liquids or solids
Size: About 10 ml or 10 g (solids must fit into 2-inch x 0.7-inch diameter vial called a rabbit)
Preparation: Little or none

Detection Range

1-100 ppm for about 50-60 different elements for activation with 100 mg of ^{252}Cf. (On 9/30/89, Cf source is about 11 mg.)

Basic Principle

Many stable elements, under neutron irradiation, become radioactive. If a gamma-ray spectrum of a sample is taken after irradiation, the identities of stable ^{23}Na upon neutron capture becomes 15-hour ^{24}Na which emits several easily measured gamma-rays. From a knowledge of capture cross sections, neutron flux, and irradiation time, gamma-ray peak intensities can be used to compute elemental concentrations. Sometimes it is more accurate and convenient to simultaneously irradiate a suitable standard along with the sample, and compare the sample activity directly to that of the standards.

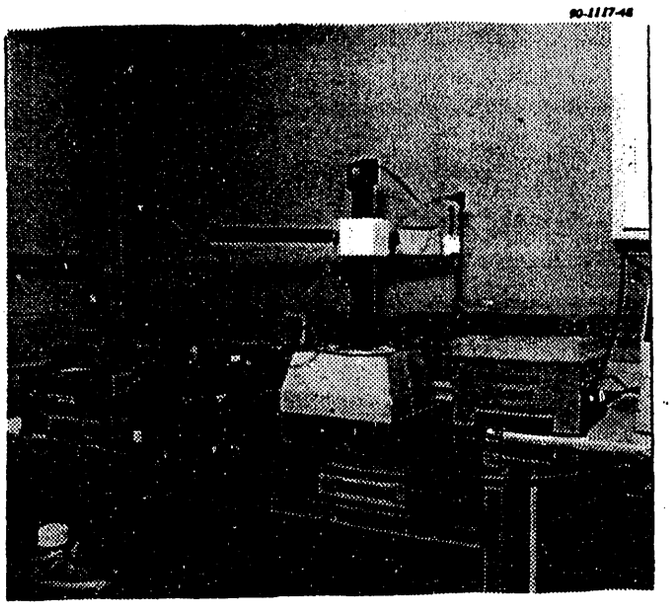
Instrumentation

The ADS CNAA facility (Building 773-A, B-003) offers both manual and automatic pneumatic transport irradiation capabilities. A Canberra Series-85 multichannel analyzer coupled to a large coaxial germanium detector provide gamma counting and analysis, and delayed neutron counting is also available for analysis of uranium or other fissionable materials.

Examples

A significant advantage of CNAA is that it is nondestructive and requires little or no sample preparation. As a result it has been used on everything from moon rocks to bioassay samples.

Gamma Pulse Height Analysis (GPHA)



Application

GPHA is one of the most useful and flexible nondestructive analysis techniques available. It is rapid, accurate, and can be used for almost any type of gamma-ray emitting sample. It is used to routinely measure fission or activation products, as well as many of the actinides.

Sample

Radioactivity Level: 10^{-7} to 10^{-4} Ci

State: Almost any liquid that emits gamma-rays

Size: 3 mL or more

Preparation: None for most liquids. Solids or gases can also be counted qualitatively, but usually require custom-made standards for quantitative analysis.

Detection Range

100 nCi/mL to 0.1 mCi/mL or suitable dilution.

Basic Principle

A detector [NaI(T1), Ge(Li), or high purity Ge], pulse processing electronics, and a multichannel analyzer measure the energy and intensity of gamma-rays emitted from the sample. The number of events recorded by the detector and their energies are plotted as a spectrum. Energy analysis usually uniquely identifies the isotopes present in the sample, while the areas of gamma-ray peaks in the spectrum are proportional to the amount of the isotopes present.

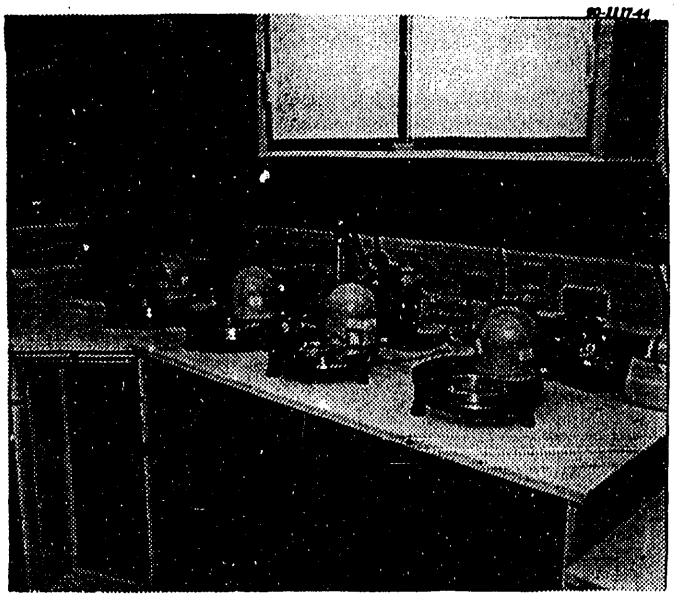
Instrumentation

The ADS counting room uses a Canberra Series-85 multichannel analyzer to record spectra from any of several high purity germanium detectors. Both low energy photon spectrometers (LEPS) and coaxial detectors for >60 keV energies are available. Spectrum analysis is done by Canberra's APOGEE program which runs on a DEC micro-VAX computer.

Examples

GPHA not only is used for ADS counting room analyses, but also is the basis of many monitors and analyzers across the site. For these applications, both NaI and Ge systems are used. If only one or two sample components need to be measured, a NaI based system is often simpler and less expensive than Ge systems.

Gross Alpha, Beta, Gamma Counting



Application

Gross counting is primarily used to measure the activity of a single isotope in a sample. Gross alpha or gross beta/gamma counts are widely used for process control samples pulled in canyon operations. Gross counting is also useful for comparisons of total activities in sets of similar samples.

Sample

Radioactivity Level: $10^{(2)}$ to $10^{(6)}$ d/m/mL

State: Aqueous or organic liquid (Dilute HNO_3 preferred)

Size: 1 mL or more

Preparation: Usually <1 mL of sample is evaporated on a planchette. Sample pretreatment can be used for separation of interferences, matrix selection, and activity adjustment.

Detection Range

10 nCi/mL to 0.1 micro-Ci/mL without pretreatment. With longer than normal count times, detection limits of <1 pCi are possible.

Basic Principle

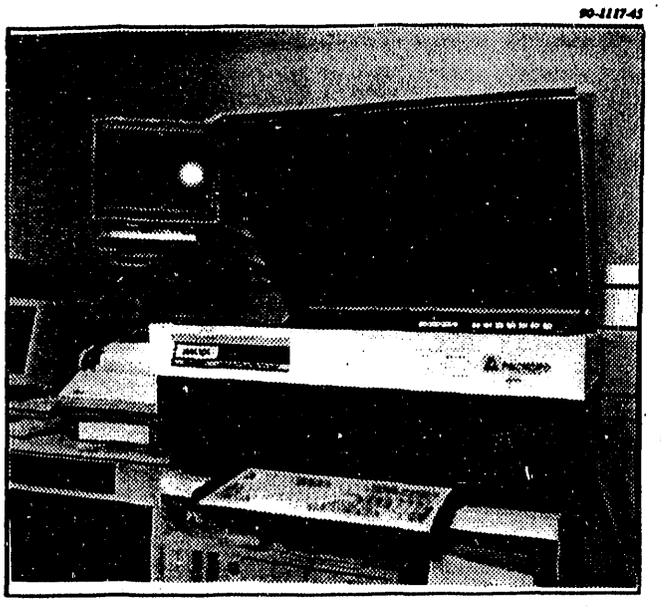
Most radioactive materials emit alpha or beta particles, and often gamma-rays as they decay. These particles and gamma-rays are all forms of ionizing radiation which are easily detected and measured with appropriate instruments. In the ADS counting room, gross alpha rates are measured with gas flow proportional counters. Gross beta/gamma rates are determined by Geiger-Mueller tube counters. A portion of the sample is evaporated on a metal disk, and counted in a fixed position relative to the detector. Standards, measured in the same way, are used to determine the detector efficiency. Knowing the detector efficiency, a count-rate for a measured sample is converted to a decay-rate.

Instrumentation

Most gross alpha counts are made on four Eberline MS-2 gas flow proportional counters systems interfaced to the ADS LIMS computer. One Eberline Model SAC-4 scintillation alpha counter is also available. Gross beta/gamma counts are normally run on two Eberline Model BC-4 beta counters.

Examples

Gross counting methods are widely used across the site for analyses of various process samples. In well characterized process samples or samples containing only one radionuclide, gross counts are fast, simple, and inexpensive methods of analysis.



Liquid Scintillation Counting

Application

The determination of alpha and beta activity in aqueous and organic liquid samples. Offers very high counting efficiency, and is the standard method for H-3, C-14, and other low-energy beta emitters.

Sample

Radioactivity Level: $10^{(2)}$ to $10^{(6)}$ d/m/mL
State: Aqueous or organic liquid (dilute HNO_3 preferred)
Size: 1 mL or more
Preparation: Usually none if sample contains only one, or sometimes two, radioactive species. Sample pretreatment can be used for separation of interferences, matrix selection, and activity adjustment. For radioactive mixtures, the analysis gives a gross count unless a separation is done to isolate a particular component.

Detection Range

10 pCi/mL to 0.1 micro-Ci/mL without pretreatment. With longer than normal count times, detection limits of <1 pCi are possible.

Basic Principle

When a radioactive material is mixed with certain organic compounds (scintillators), ionizing radiation (alpha, beta, or gamma) can produce excited electronic states in the organic which then de-excite by emitting light. The number of light quanta emitted is proportional to the energy deposited by the radiation. By mixing the sample and scintillator together in an organic solvent (scintillation cocktail) and photo-multiplying the light output, an energy spectrum can be obtained. From the spectrum, both energy and decay-rate information are available for analysis.

Instrumentation

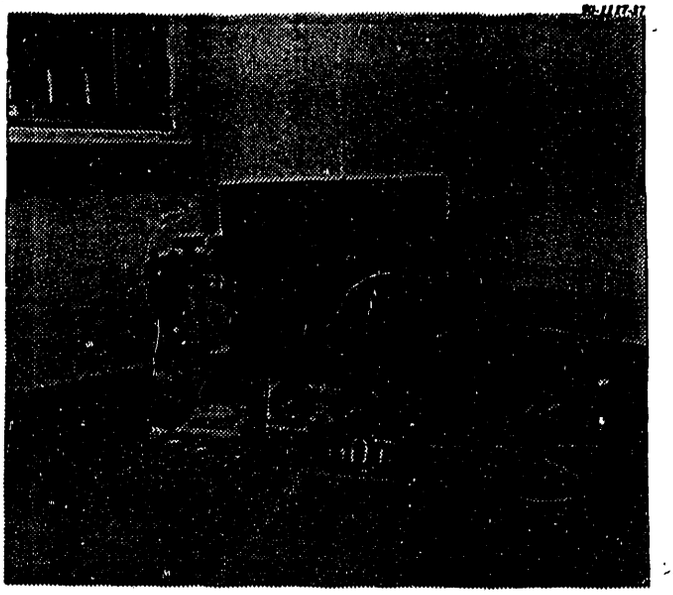
Two Liquid Scintillation Analyzers are available in the ADS Counting Room. A Packard 4000 Tri-Carb instrument is used for most routine analyses while the newer Packard 2250 Tri-Carb, with color spectral display and multiple analysis features, is available for R&D work.

Examples

Liquid Scintillation counting is used for determining H-3 in oils and other organics, C-14 and I-129 chemically removed from stack gases, ion exchange separations of Bk-249 and Eu-152, and S-35 in aqueous samples removed from fuel storage basins.

SPECTROSCOPY

Dissolution Methods



Application

Preparation of samples by conversion of solids into solutions. Solids filtered from solution and solids obtained by evaporation of the volatile liquid are converted into solutions.

Sample

State: Solids, liquids with suspended solids, and slurries.

Size: 1.0 g of ground solids (per required analyses)

Preparation: Powders should be ground and mixed. Suspended solids are filtered and the material transferred to a container and converted to a solution by combinations of ashing, acid digestion, fusion, and dilution. Slurry materials are dried and a portion of the material treated as suspended solids.

Radioactivity Level: Samples must have less than 10^6 d/m/g alpha or 10^8 d/m/g beta-gamma.

Principle

Some solid materials can be dissolved using a combination of acids with furnace heat employing metal crucibles. Microwave heating can be employed using closed plastic containers for some dissolutions. Fusions which employ sodium peroxide or potassium carbonate can be used for some material followed by uptake in acid or water.

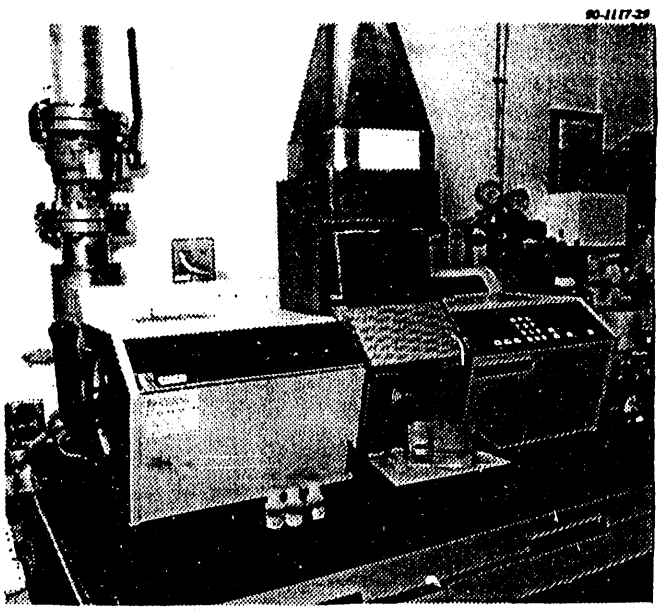
Equipment

CEM Microwave Digestion
Muffle furnaces
Ni, Zr, and Pt crucibles
Teflon® bottles
ACS grade acids

Examples

Defense Waste Processing (DWP) materials: frit, sludge, slurry, and glass.

Atomic Absorption Spectroscopy (AA)



Application

Quantitative determination of metal elements in aqueous solutions (Cs, Cu, Fe, Hg, K, Li, Mg, Mn, Mo, Na, Ni, Pb, Si, Sr, Zn, Ag).

Sample Characteristics

State: Solid, liquid, or solutions

Size: 1.0 g ground solid
10 to 50 mL liquids
10 to 50 mL solutions

Preparation: Solids will be transformed into appropriate solutions by combinations of ashing, acid digestions, fusions, and dilution. Solutions are diluted when required to per form the anlysis.

Radioactivity Level: Solutions should be less than 10^6 d/m/mL alpha or 10^8 d/m/mL beta gamma.

Principle

Source radiation generated by a hollow cathode lamp for the element of interest is passed through the vapor of the element. Radiation absorbed by the vapor is proportional to the concentration of the element in the vapor. A vapor of the element is produced by nebulizing a solution containing the element into a well controlled flame. Sample quantitation is performed by comparing the absorption of the unknown solution with absorption values obtained from calibration solutions.

Instrumentation

Varian Model AA-475 Atomic Absorption/Flame Emission Spectrophotometer

Shimadzu Model AA-670 Atomic Absorption/ Flame Emission Spectrophotometer

Examples

Determination of Na and K in Defense Waste Procession Technology Glass

Determination of metal content of natural and effluent waters

Cold Vapor Atomic Absorption Spectroscopy (Mercury)

Application

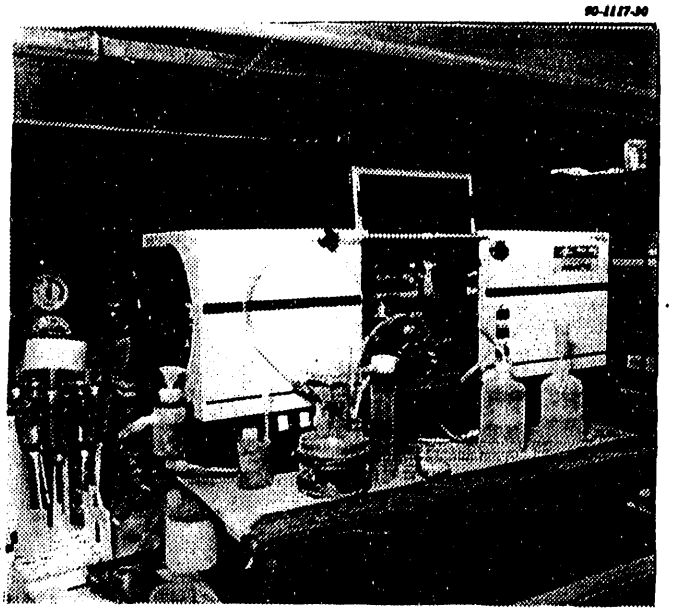
Quantitative determination of mercury (Hg) in aqueous solutions.

Sample

State: Liquid aqueous solutions
Size: 10 to 50 mL solutions
Preparation: Filter and dilute to perform the analysis. Sulfuric and nitric acid are added to oxide the mercury.
Radioactivity Level: Solutions should be less than 10^6 d/m/mL alpha or 10^8 d/m/mL beta-gamma.

Principle

The Hg in an aliquot of the sample is oxidized to the +2 state. The aliquot of the sample is introduced into a reaction cell. Stannous chloride is added to the sample to reduce the Hg to its elemental form. The cell is sealed to contain the Hg which is released into the vapor state. The vapor is then swept with an air flow into the instrument. The vapor enters a quartz flowcell. Source radiation generated from a hollow cathode is directed through the flowcell. The absorption by the Hg vapor as it flows through the cell is measured and recorded.



Sample analyses are completed by comparing unknown solutions with analytical calibration solutions.

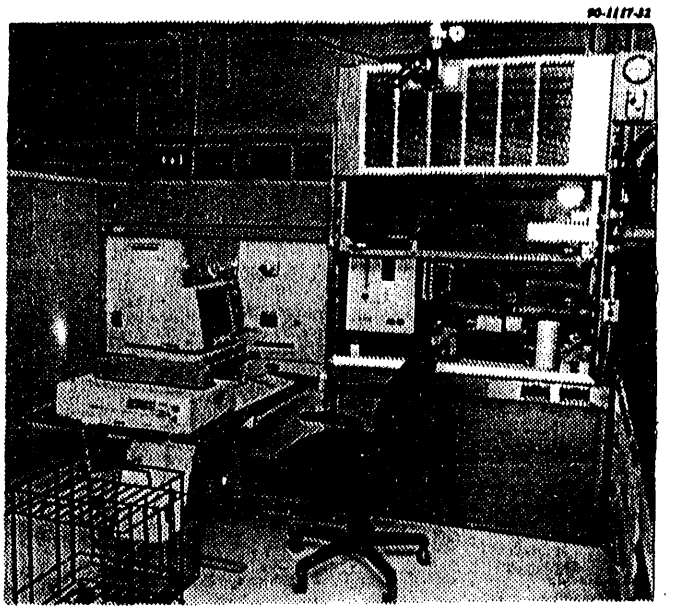
Instrumentation

Shimadzu Model AA-670 Atomic Absorption/ Flame Emission Spectrophotometer with Mercury Hollow Cathode lamp

Examples

Determination of Hg in waste tanks contents.
Determination of Hg in sumps solutions.
Determination of Hg in natural and effluent waters.

Inductively Coupled Plasma Emission Spectroscopy (ICP)



Application

Quantitative determination of metal elements in aqueous solutions. All metals and some nonmetals can be analyzed by this technique.

Sample

State: Solid, liquid, or solutions

Size: 1.0 g ground solids
10 to 50 mL solutions

Preparation: Solids and liquids are converted into appropriate solutions by combinations of ashing, acid digestions, fusions, and dilutions. Solutions will be diluted as necessary to properly analyzed the sample material.

Radioactivity Level: Solutions should be less than 10^6 d/min/mL alpha or 10^8 d/m/mL beta-gamma.

Principle

Atomic emission spectra of the elements are generated by nebulizing a solution containing the sample into an argon plasma which operates at 8000 to 10,000 degrees Kelvin. A monochromator/polychromator disperses the radiation onto a slit and phototube/phototubes. The polychromator/phototubes measure emission radiation for several elements simultaneously. The intensity of the measured spectrochemical emission radiation is proportional to the concentration of the elements introduced into the argaon plasma. Quantitative sample analyses are performed by comparing unknown solutions with analytical calibration solutions.

Instrumentation

Applied Research Laboratories (ARL) Model 3580 Inductively Coupled Plasma Emission Spectrometer (Scanning/Sequential Monochromator and Simultaneous Polychromator)

Examples

Determination of the trace metal content in reactor moderator and coolant solutions.

Determination of the elemental content of Defense Waste Processing (DWP) glass digests and fusions.

Determination of the elemental content of waste solutions.

Determination of the elemental content of DWP frit digests and fusions.

Determination of the elemental content of waste tank contents.

Determination of the elemental content of natural and effluent waters, well water, and sump samples.

Inductively Coupled Plasma/Mass Spectroscopy (ICP/MS)

Application

Quantitative elemental analysis of solutions for most all elements in the periodic table. Isotope ratio determinations can be performed for some elements depending on the matrix of the sample.

Sample

State: Aqueous solutions

Size: 10 to 50 mL solutions

Preparation: Samples must be free of particulate. Solids must be prepared by converted into solutions by combinations of ashing, acid digestions, fusions, and dilutions. Solutions will be diluted as required to perform the required analyses.

Radioactivity Level: Samples must be less than than 10^6 d/m/mL alpha or 10^8 d/m/mL beta-gamma.

Principle

Solutions to be analyzed are diluted to perform preliminary scans. Solutions are nebulized into an argon plasma which maintains temperatures from 8000 to 10,000 degrees Kelvin. The sample is atomized in this environment to produce predominantly atoms of the elements along with formation of oxides and molecular species. A minute portion of the plasma is drawn into the vacuum chamber of a quadruple mass spectrometer. A system of ion lenses transmits the ions to a quadrupole mass spectrometer while blocking the emission radiation from the plasma. The mass spectrometer scans the mass region in seconds to record the elemental information. Ions are detected by an electron multiplier after passing through the quadrupole. Electrical pulses are produces by the impact of each ion. The magnitude of the integrated pulses for a fixed time is proportional to the concentration of the elements ions in the plasma. Sample analyses are performed by comparing unknown solutions with analytical calibrations. Semiquantitative analyses are performed by using internal standards of isotopes not present in the original sample. Molecular and isobaric (atomic species which have the same atomic weight with different Z numbers) interferences must be identified and may prevent quantitative measurements. These interferences make the instrument most useful for measurements above mass 80.

Instrumentation

VG Plasmaquad I Inductively Coupled Plasma Mass Spectrometer

Examples

Determination of the elemental content of reactor moderator and coolant.

Determination of the elemental content of natural, effluent, and well waters.

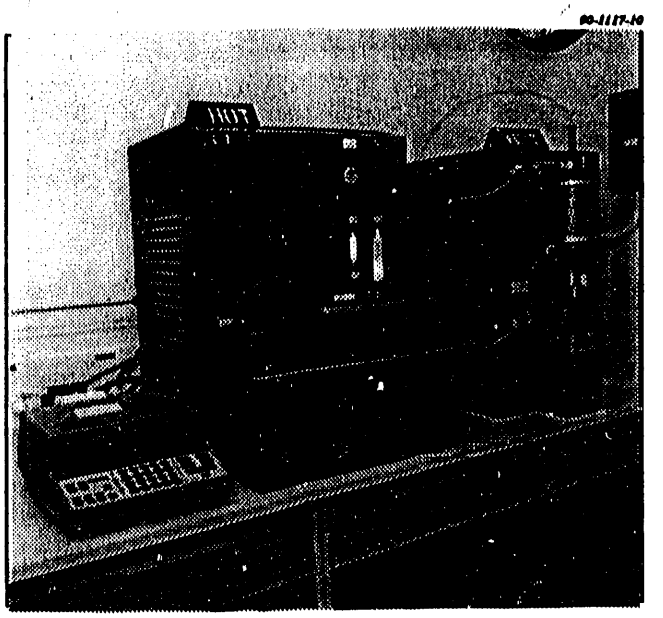
Determination of the isotopic ratio of Boron in reactor solutions.

Determination of stable isotopes in aqueous solutions.

Determination of long half-life radioisotopes.

WET CHEMISTRY AND MISCELLANEOUS ANALYSIS

Carbon/Sulfur Determination (LECO Method)



Application

The simultaneous determination of total carbon and sulfur in solids.

Sample

State: Solids
Size: Sample weight 0.1 to 5.0 g and maximum diameter and length of 0.75 inch
Radioactivity Level: None

Determination Range

Carbon: 0.0001 to 6.0%
Sulfur: 0.0001 to 0.35%

Basoc Principle

The sample is combusted in a high-frequency induction furnace at temperatures of up to 1700°C. The combustion is carried out in an oxygen atmosphere and the carbon is converted to CO_2 and the sulfur to SO_2. The gases are transported by an oxygen carrier-gas stream to infrared sources. By the absorption of infrared energy the quantity of CO_2 and SO_2 are independently determined and the percents of total carbon and sulfur are calculated.

Instrumentation

LECO C S-244 Carbon/Sulfur Determinator

Examples

Carbon and sulfur content of ferrous and nonferrous metals, ores, limestone, glass, and cement.

Flow Injection Analysis

Applications

FIA utilizes a wide variety of detection devices to analyze for several different types of chemical species. Typical detection instrumentation interfaced to FIA systems are:

- Spectrometric Method — UV/VIS spectrophotometry, chemiluminescence, fluorimetry, and infrared spectroscopy
- Electrochemical Methods — amperometry, conductance voltametry and ion selective electrodes, to mention a few. Likewise an extensive array of analytical techniques are utilized with FIA technology to automated solution chemistry systems. A few example techniques are: complexiметric, colorimetric, redox, standard addition, stopped flow, titration, extraction, gas diffusion, and ion exchange. An extensive assortment of analysis have been employed through the creative combination of detection devices and analytical techniques. Many of these laboratory methods are also applicable to online use.

Sample

State: Typically aqueous solutions, organic solutions in special situations, solids after dissolutions

Size: 2 to 20 mL

Determination Range

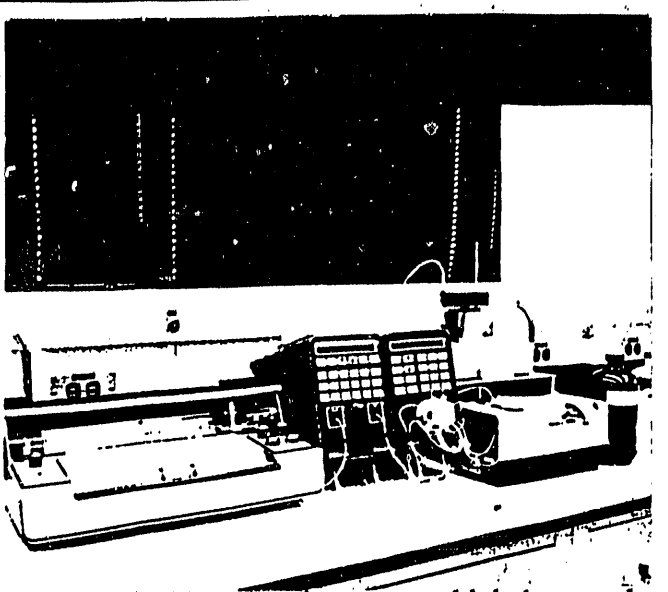
Typically 1 to 200 parts per million, some procedures are capably of low parts per billion depending upon the species being analyzed.

Time Required

After sample preparation completion, if required, most procedures can analyze samples at a rate of thirty to sixty per hour.

Principle

Flow injection analysis is an automated system that is based on the introduction of a well define sample volume into a continuous flowing stream.



Generally this stream is reagent which the sample complexes with to form a measurable reaction product. However, FIA is a very flexible analytical tool and it is possible to arrange the system to do additional task such as introduction of multiple reagents, dilutions, preconcentrations, column separations, multiple sample, and injections per sample, to mention a few.

Equipment

Tecator 5020

Fiatron modular system

Examples

The determination of:

Nitrate/ Nitrite

pH

Chloride

Sulfate

Iron II/Iron III/Total Iron

Chromium II/Chromium IV/Total Chromium

Silicates

Phosphates

Water Hardness

Alkalinity

Ammonia

Surfactants

Polysaccharides

Polyvinyl Alcohol

Formaldehyde

Phenol

Flash Point Determination

Application
The determination of flash point by Pensky-Martins closed cup method.

Sample
State: Liquids, liquids with suspensions of solids, and liquids that form surface films
Size: 75 mL
Radioactivity level: Samples must have less than 10^6 d/m/mL alpha or 10^8 d/m/mL beta-gamma.

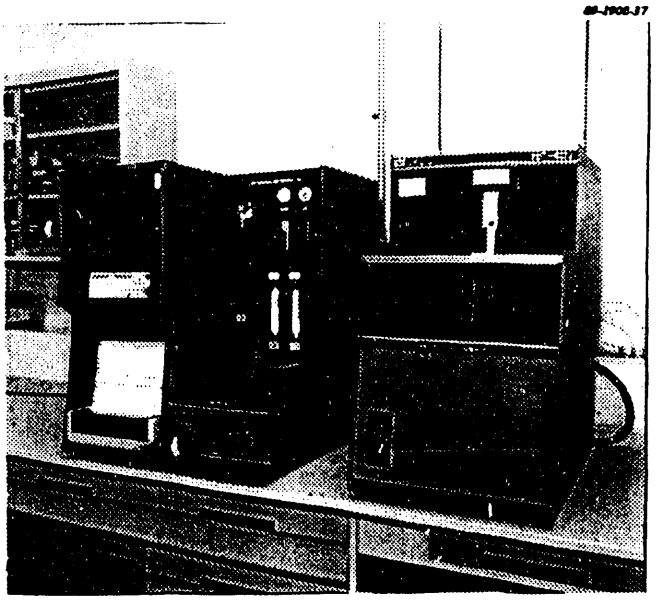
Determination Range
From 0°C to 400°C (32°F to 752°F).

Basic Principle
The sample is contained in a closed cup and heated at a slow, constant rate with continual stirring. An ignition source is directed into the cup at regular intervals with simultaneous interruption of stirring. The flash point is defined as the lowest temperature at which the ignition source causes the vapor of the sample to ignite and instantaneously propagate itself over the surface of the sample.

Instrumentation
Herzog 327 Automatic Flash point Determinator

Examples
Flash point determination of waste oils, machine coolants, and spent scintillation solutions.

Hydrogen Determination (LECO Method)



Application

Hydrogen determination in ferrous and nonferrous metals by either inert gas fusion or hot extraction.

Sample

State: Solids - pins, chips or powders
Size: Sample weight range is 0.001 to 20 g with 1.0 to 5.0 g nominal
Maximum physical dimensions are:
diameter - 0.375 inch
length - 2.1 inches
Radioactivity Level: None

Determination Range

From 0.001 to 2000 ppm at 1 g

Basic Principle

Hydrogen is extracted from the sample in a high-temperature induction furnace by either sample fusion or hot extraction at temperatures below then sample melting point. The released hydrogen gas is carried away from the combustion chamber by a nitrogen carrier gas and is processed and cleaned by several filters. The hydrogen content is the measured by a thermal conductivity cell.

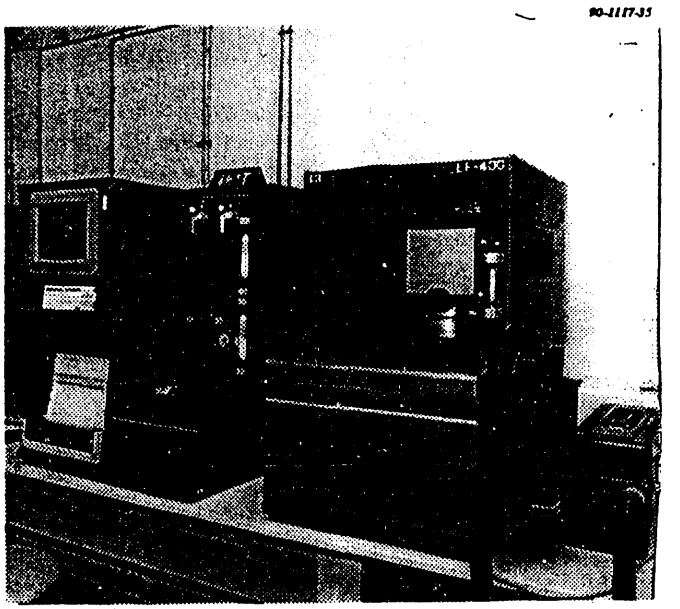
INSTRUMENTATION

LECO RH-402 Hydrogen Determinator

EXAMPLES

Hydroden determination in copper, steel, and transition metal hydrides.

Nitrogen/Oxygen Determination (LECO Method)



Application

Determination of total nitrogen and oxygen in both metal and inorganic materials by the inert gas fusion technique. Specific nitrides and oxides may also be separated and identified by this method.

Sample

State: Solids - pins, chips, or powders
Size: Sample weight range of instrument is 0.010 to 5.000 g with 1.000 g nominal
Maximum physical dimensions are:
diameter - 7.5 mm
length - 18 mm
Radioactivity Level: None

Determination Range

Oxygen: 0.00001(0.1 ppm) to 0.1% at 1 g
Nitrogen: 0.00001(0.1 ppm) to 0.5% at 1 g

Basic Principle

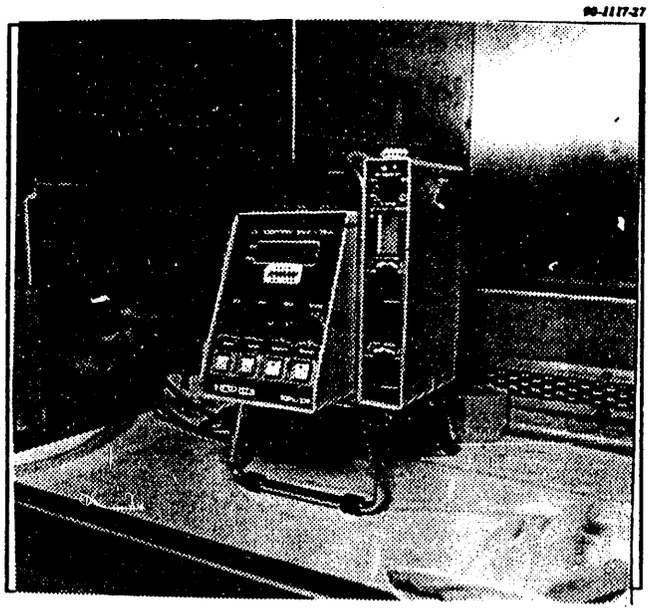
The sample is fused in a high temperature electrode furnace at temperatures up to 3000°C. Nitrogen and oxygen are released from the sample as N_2 and CO and are carried through the system by helium. The CO is oxidized to CO_2 and is selectively measured by an infrared cell. The N_2 is measured by thermal conductivity. The instrument can also use gradual temperature increase to separate nitrides and oxides.

Instrumentation

LECO TC-436 Nitrogen and Oxygen Determinator

Examples

Nitrogen/Oxygen determination in metals, ores, and ceramics.



Oil and Grease in Aqueous Suspension

Application
Determination of oil and grease in aqueous suspensions.

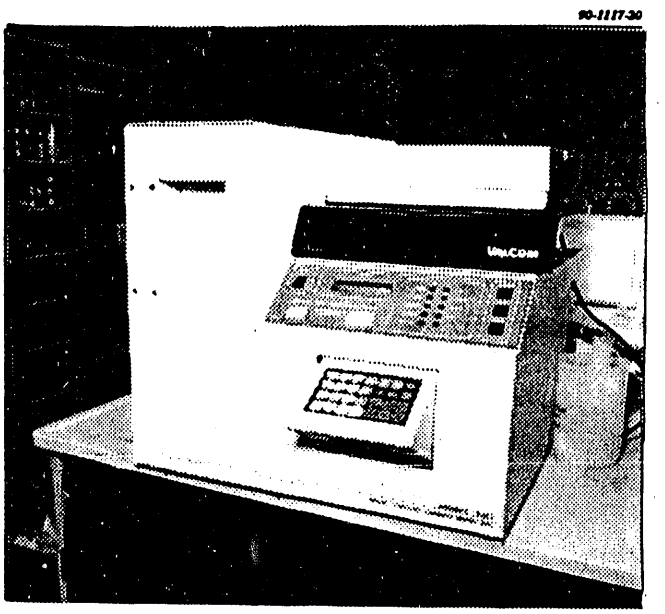
Sample
State: Aqueous solutions
Size: 1.0 to 100 mL depending on concentration
Radioactivity Level: None

Determination Range
Concentrations of 0.5 ppm and above can be determined with 2% RSD.

Basic Principle
Dissolved or emulsified oil and grease is extracted from water by trichlorotrifluoroethane. The infrared absorption of the extracted hydrocarbons is then measured and used to calculate the quantity of oil and grease. Other materials such as sulfur compounds, certain organic dyes, and chlorophyll may also be extracted by the solvent and be included in the determined quantity.

Instrumentation
OCMA-220 Oil Content Analyzer

Examples
Oil and grease determination in wastewaters or treated effluents.



Total Carbon Analysis (TC, TOC, TIC)

Application

The determination of Total Organic Carbon (TOC) and Total Inorganic Carbon (TIC) in aqueous samples.

Sample

State: Liquids only. Capable of processing samples with suspended particles less than 0.5 mm with a maximum concentration of 0.5% by weight.

Size: 0.3 mL to 10.0 mL

Radioactivity level: Samples must have less than 10^6 d/m/mL alpha or 10^8 d/m/mL beta-gamma.

Determination Range

The working concentration range of the analyzer is 0.05 µg/ml to 150 µg/ml. Samples with higher carbon concentrations must be diluted prior to analysis.

Basic Principle

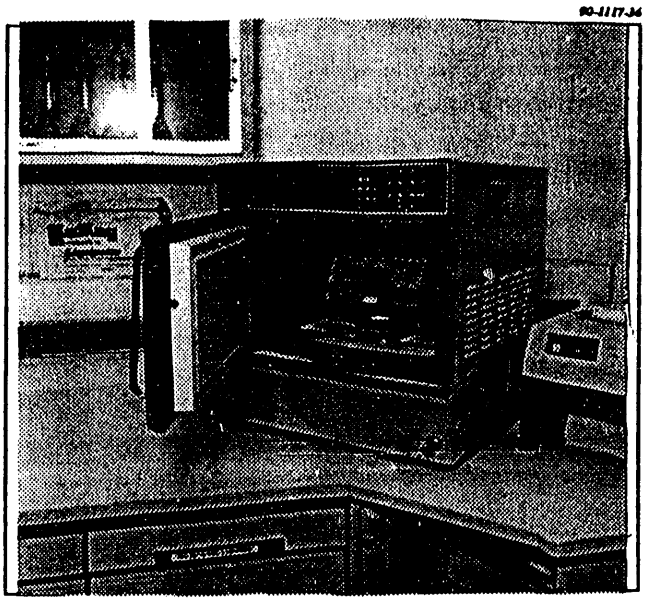
The inorganic carbon is converted to CO_2 by acidification with phosphoric acid. The CO_2 is then purged, trapped, and quantified by an infrared detector. After this step, the organic carbon is oxidized with sodium persulfate and the CO_2 produced quantified as above.

Instrumentation

OIC-700 Total Carbon Analyzer

Examples

TOC and TIC determinations on condensates, waste streams, and waste tanks.



Total Solids by Microwave Drying

Application
To directly measure the percent total solids in nonflammable samples.

Sample
State: Liquids or solids
Size: Typical is 5.0 mL
Radioactivity Level: None

Determination Range
From 0.0 to 100% with a sensitivity of 0.1%

Basic Principle
Total solids is defined as the material residue remaining in a vessel after evaporation of a sample and subsequent drying in an oven at a defined temperature. Total solids includes both total suspended solids and total dissolved solids. In this method, samples are dried to completion in a microwave oven and the percent total solids calculated and displayed.

Instrumentation
CEM AVC 80 Analyzer - Remote model and bench model.

Examples
- Determination of percent solids in sludges.
- Determination of feed ratios for DWPF processes.

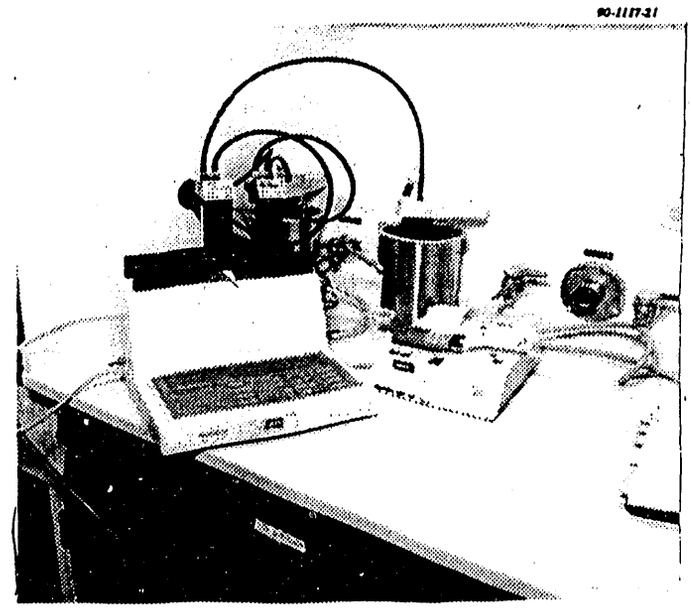
Water Determination With Karl Fisher Reagent

APPLICATION
Quantitative water determination with Karl Fisher Reagent.

SAMPLE
State: Solid or liquid
Size: 1 to 20 mL depending on water concentration
Radioactivity Level: Samples must have less than 10^6 d/m/g alpha or 10^8 d/m/g beta-gamma.

DETERMINATION RANGE
From 0.1 to 100% moisture can be determined with a precision of ±1% relative.

BASIC PRINCIPLE
The dead-stop endpoint amperometric titration method is utilized in this procedure. Two polarized electrodes are immersed in the solution and water is titrated with Karl Fisher reagent the current flow between the electrodes is monitored.



At the endpoint of the titration, the current increases beyond the established baseline and the titration ceases. The resulting titrant volume is compared to volumes obtained from standards and the results calculated.

INSTRUMENTATION
Mettler DL40RC Memotitrator

EXAMPLES
- Water content of organic solvents

Analytical Development

PROCESS CONTROL AND ANALYZER DEVELOPMENT

SCOPE

The major function of the Process Control and Analyzer Development Group (PC&AD) is development of inline/online measurement systems integrated with automated control systems. The general goal of the group's work is enhancement of SRS processes by providing more timely analytical information, reducing sample handling and the subsequent chemical and radioactive exposures to people, and improving process control.

CAPABILITIES

The full service provided by the group involves investigation of the problem to validate that what is requested is actually what is needed, identification of existing means to provide the need or development of a new system, laboratory testing, prototype assembly and field testing, revisions as necessary, turnover to the customer with documentation. Once a system has been developed, additional installations are normally performed by other site engineering and project groups with assistance from PC&AD. Custom hardware is transferred to private vendors for future fabrication needs as much as possible.

PROCEDURE

Work is initiated by customer request, such as development of an online oxygen analyzer for a tritium process line, and by the group through anticipating future SRS needs, such as work presently under way to develop in situ sensors for environmental monitoring. Customers may contact any of the personnel on the attached list to discuss potential work. New work can be funded by operations departments through a Request for Technical Assistance (RTA) and by SRL departments through direct funding. Ongoing R&D programs are agreed upon annually with operating departments. Customer requests for work may possibly be included in an ongoing program.

EXAMPLES

Activities in which the group is involved to support its objective are reflected in the attached list of contacts and cover development of methods, hardware, software, and new technology. The following is a partial list of work performed or in progress to give examples of these activities:

- Developed a simple modification to the canyon air-lift samplers that allowed installation of fiber-optic interfaces for remote online analysis by spectrophotometry; developed needed fiber-optic/sampler interface hardware.
- Automated canyon samplers to allow operation from the control room and possible control by DCS.
- Developed diode array spectrophotometer for use with fiber-optics.
- Developed chemometric models for determination of uranium and nitrate concentration room H-Canyon online spectrophotometer spectra; developed model for online plutonium determination in SRL resin test system; developed model to determine PCB concentration in oils.
- Performed systematic assessment of F-Canyon processes to determine where process control improvements could be most beneficial.
- Installed NO_x, oxygen and acid analyzers on F-Canyon NO_x absorber column and developed a computer model to determine the optimum control scheme to maximize acid production while limiting emissions.
- Developed an improved, low maintenance alpha detector for use in low activity online monitoring applications (liquid and off-gas).

- Developed a system of gamma monitors interfaced with process functions to interlock systems upon detection of plutonium accumulation.
- Improved the reliability of operation and testing of F-Canyon neutron monitors through the use of improved detectors and in situ pulse height analysis.
- Developed a method of in situ measurement of plutonium content of TRU waste culverts in the burial ground.
- Developed a custom configuration for an active well coincidence counter to NDA reactor fuel rod billets.
- Working with LANL to develop chemical sensors with indicators trapped in polymer coatings on fiber-optic cables.
- Developed quantitative at-line mercury analyzer for tritium process lines.

ROBOTICS DEVELOPMENT

SCOPE

Development of remote radiochemical and cold wet chemical analysis methods.

PRINCIPLE

The robotics laboratory combines robotics and laboratory stations to automate procedures used in sample preparation and chemical analyses. The feasibility of methods automation is determined by the following characteristics:

- Elimination of human error
- Routine preparation of replicate samples, standards, and controls
- Computer storage of customized procedures
- Increased productivity through unattended operation of multi-task methods
- Reduced human exposure to radiation and chemical hazards

INSTRUMENTATION

- Zymar Robotics Systems (2 systems)
- CRS Industrial Robot in contained hood

EXAMPLES - METHODS CURRENTLY UNDER DEVELOPMENT

- Automation of Davies-Gray Uranium analysis
- Automation of alpha and beta plate making
- Automation of Plutonium TTA extraction and alpha analysis
- Automation of gamma sample preparation and analysis
- Filling and capping reagent dilution vials

REQUEST FOR TECHNICAL ASSISTANCE (RTA)

SCOPE

Organizations outside SRL obtain specific technical assistance for non-routine, non-baseline budgeted tasks from SRL and transfer funds to cover the costs via the RTA procedure. Routine analyses to be performed by ASG may be obtained by the SRL Service Order System as defined in MRP 5.05 of WSRC-1.03.

RESPONSIBILITIES

The requester is responsible for providing information in the RTA form in a manner that clearly identifies the work objectives and any special requirements. Requests for Analytical Development should be sent to the the Manager of the Analytical Development Section (ADS).

The Budget Section of SRL will provide SRL subcodes and keep records of expenditures.

PROCEDURE

The complete procedure for RTA's is located in the WSRC MANAGEMENT REQUIREMENTS AND PROCEDURES MANUAL (WSRC-1.03) in Section MRP 5.04.

Supplemental Laboratories

PLANT PROCESS LABORATORIES (ANALYTICAL LABORATORIES)

Analytical Laboratories (Building 772-3F)

Manager	W. R. Jacobsen	557-3420
	Staff	

Special Laboratories (Building 707-H)

Manager	M. B. Hughes	557-8807

Central Laboratories (Building 772-F)

Manager	P. T. Deason	557-4331

Within this department are several specialized laboratories which are further described below. Some are limited in the amount of radioactivity that can be handled or have procedures in place for only one material form. The current laboratories are:

Materials Laboratory (Building 320-M)

Manager	Lori M. Chandler	725-2106
Supervisor	Sandra Hightower	725-2140
Chemists	Debbie Bryson-Lewis	725-1143
	Bernard Nora	725-3895
	Ron Livingston	725-3639

Materials Handled

Enriched Uranium, U-Al, Al, environmental samples, production essential materials for specification analyses.

Analyses Performed

Uranium assays by classic titration of solutions, Al composition verification by emission spectrometer, other solid materials such as stainless steel components by emission spectrometer, volatile organics in water by gas chromatography, essential material specification analyses such as pH, conductivity, flash point, resin capacity, and other wet chemistry procedures.

Comments, Limitations, Special Instrumentation

This area and laboratory are not equipped to handle any radioactive samples except non-irradiated uranium.

This lab has a liquid chromatograph (LC) as well as the gas chromatographs, but most of the analyses possible on the LC are in the development stage. There are two emission spectrometers for determining composition and trace elements in metallic samples and one direct current argon plasma spectrometer (DCAP) for liquid samples. An inductively coupled plasma spectrometer (ICP) is expected to be purchased to replace the DCAP within the next year. This will improve the sensitivity of trace element analyses over that of the DCAP.

Water Quality Laboratory (Building 772-D)

Manager	Lori Chandler	725-2106
Supervisor	Kathy Johns	725-6945
Chemists	Dick Thomasson	725-6991
	Gerry Levi	725-6820
	Robert Reed	725-6854

Materials Handled

Heavy water, light water, and environmental samples.

Analyses Performed

Deuterium oxide content by infra-red analysis, tritium content by liquid scintillation counting, pH, conductivity, chloride, sulfate, other anions by ion chromatograph, peroxides and turbidity by spectrophotometer, neutron absorption, alpha and beta-gamma counting, coliform analyses in environmental samples.

Comments, Limitations, Special Equipment

This laboratory is not equipped to routinely handle any sample which cannot be easily dissolved. It is essentially a water laboratory. Mercury analyses can be done here but all other metallic trace element analyses are performed in the Central

*(All telephones are in area code 803)

Laboratory. Purchase of an atomic absorption instrument within the next year will allow metallic analyses to take place in the Water Quality Laboratory.

Central Laboratory (Building 772-F)

Managers	Stephen Lee	557-4343
	Paul Deason	557-4331
Supervisor	Gary Blessing	557-4391
Chemists	Cloyd Denard	557-4217
	Mike Boerste	557-3010

Materials Handled

Moderator, basin water. Special samples of sludges, solids, etc., can usually be arranged.

Analyses Performed

Radionuclides by gamma spectroscopy. Radiochemical separation, beta counting, and alpha spectroscopy.

Comments, Limitations, Special Equipment

The Central Laboratory will provide limited service until late 1990 due to extensive renovation work. Some of the work is being handled in Analytical Laboratories expansion Building 772-1F, such as the basin PHAs and DCAP work. Plans are being made currently to ensure analytical continuity for the remainder of the samples needed by Reactor.

Tritium Facilities Laboratories (Building 232-H)

Manager	Dave Fauth	557-8715

This laboratory handles only gaseous samples for determination of weight percent composition of oxygen, nitrogen, carbon dioxide, etc. **This is a process control laboratory for tritium production and specialized to that purpose.**

Environmental Laboratories (Building 735-A)

Manager	Dennis Stevenson	725-2778

*(All telephones are in area code 803)

This laboratory handles only very low activity environmental samples of soils, vegetation, and water. This laboratory also has expertise in bioassay procedures.

APPENDIX

ABBREVIATED LIST OF ROUTINE ANALYSES

INSTRUMENT/ TECHNIQUE	ANALYTE	LABORATORY
ION Chromatograph	Anions	WQL
UV-Vis		
Spectrophotometer	Peroxide	
	Turbidity	WQL
Alpha, Beta-Gamma	Radioactivity	WQL, CL
Gamma Spectrometry Alpha Spectrometry Beta Separations	Radionuclides	CL
Gas Chromatography	Volatile Organics	ML, CL
Mass Spectrometry	Gas Components	TFL, CL
Liquid Scintillation	Tritium	WQL
Infra-Red Analyzers	D_2O	WQL
pH, Conductivity Probes	pH, Conductivity	WQL
Oil & Grease	Oil & Grease	WQL
Material Specifications		ML
Environmental Field Samples		EL

WQL = Water Quality Laboratory
ML = Materials Laboratory
SRL = Savannah River Laboratory
EL = Environmental Laboratory
CL = Central Laboratory
TFL = Tritium Facilities Laboratories

INTRODUCTION TO DWPT ANALYTICAL LABORATORY

INTRODUCTION

The laboratory at TNX is called the DWPT Analytical Laboratory. Personnel include 12 technicians, 1 first-line supervisor, and 3 chemists. The instrumentation currently available include:

- 2 gas chromatographs
- 2 high pressure liquid chromatographs
- 2 ion chromatographs
- 1 diode array spectrometer
- 1 atomic absorption spectrophotometers
- various wet chemistry items such as titrators, pH meters, ovens, etc.

This laboratory's function is to support as much as possible the various DWPT (Defense Waste Processing Technology) research processes: IDMS (Integrated DWP Melter System), PHEF (Precipitate Hydrolysis Evaporator Facility), and SRAT/SME (Sludge Receipt and Adjustment Tank/Slurry Mix Evaporator), and laboratory bench top experimentation.

Other processes, ORF (Organic Refluxing Facility), ETP (Effluent Treatment Facility), IWT (Intermediate Waste Technology), and ATS (Actinide Technology Section) have solicited or are soliciting support from the DWPT Analytical Laboratory. This laboratory provides back-up to support non-radioactive analyses received by ADS (Analytical Development Section) when their instrumentation needs repair.

Current endeavors include investigating slurry analyses, low level benzene determinations, accurate mercury (Hg) measurements by cold vapor atomic absorption, and installation of a laboratory management information system.

DWPT ANALYTICAL LABORATORY

Group Manager: Chris T. Randall

Supervisor: Calvin L. Cooks

Technicians: Janet P. Cockrell
Henry H. Franks
Alphis (Al) Grubbs
Buren (Bob) L. Jolley
Sandra J. Keel
Sammie O. King
Dorothy (Dot) T. Tipton

Eric M. Frickey
Lee K. Price
Kenneth (Ken) E. Ready

Clerk: M. (Mary Lou) L. Brown

Technical Advisors: Charles (Chuck) R. Nold
Timothy (Tim) A. Policke
Michael (Mike) J. Whitaker

Sample Preparation

Pre-preparation:

Method: **Vitrification**
Sample Type(s): Glass
Glass Frit
Sludge

Method: **Filtration**
Sample Type(s): Liquid
Slurry
Sludge

Dissolution:

Analyte(s): **Most elements, including the noble metals (Ag, Au, Ir, Pd, Pt, Rh) except As, Cd, Cs, Hg, K, Na, Ni, Os, P, Rb, Re, Te, Se**

Method: **Na_2O_2 fusion/HCl uptake**
Sample Type(s): Glass
Glass Frit
Melter Feed
Sludge
Purpose(s): AA, ICP

Analyte(s): **Most elements, including the noble metals (Ag, Au, Ir, Pd, Pt, Rh) except As, Cd, Cs, Hg, K, Na, Ni, Os, P, Rb, Re, Te, Se**
Method: **Na_2O_2 fusion/H_2O uptake**
Sample Type(s): Glass
Glass Frit
Sludge
Purpose(s): IC, ISE

Analyte(s): As, Cd, Cs, Hg, K, Na, Ni, Os, P, Rb, Re, Te, Se
Method: **HF/HCl Bomb**
Sample Type(s): Glass
Glass Frit
Sludge
Purpose(s): AA/ICP

Carbon

Analyte(s): **Total Carbon** (> 100 ppb)
Total Inorganic Carbon (> 100 ppb)
Total Organic Carbon (> 100 ppb)
Method(s): CO_2 IR detection
Instrument(s): O I Corp Model 700
Beckman Model 915B
Sample Introduction: Aspiration
Sample Type(s): Liquid (no particulates)
Slurry (aqueous phase, filtered if necessary)
Sludge (diluted)
Units: ppm (ug/ml, mg/l)

Elements

Analyte(s): **Aluminum (Al)**
Antimony (Sb)
Barium (Ba)
Boron (B)
Calcium (Ca)
Cerium (Ce)
Cesium (Cs)
Chromium (Cr)
Cobalt (Co)
Copper (Cu)
Iron (Fe)
Lithium (Li)
Magnesium (Mg)
Manganese (Mn)
Nickel (Ni)
Potassium (K)
Silicon (Si)
Sodium (Na)
Strontium (Sr)
Titanium (Ti)
Zinc (Zn)
Zirconium (Zr)
Method(s): Flame Atomic Absorption
Instrument(s): Varian AA-10
Varian AA-400
Sample Introduction: Aspiration
Sample Type(s): Liquid (filtered, trace organics only)
Slurry (dissolved)
Sludge (dissolved)
Solids (dissolved)
Units: ppm (ug/ml, mg/l)

Analyte(s): **Mercury (Hg)**
Method(s): Cold Vapor Atomic Absorption
Instrument(s): Varian AA-400
Sample Introduction: Aspiration
Sample Type(s): Liquid (filtered, trace organics only)

Slurry (dissolved by Na_2O_2 fusion, HFHCl bomb, or Aqua Regia)
Sludge (dissolved by Na_2O_2 fusion, HFHCl bomb, or Aqua Regia)
Solids (dissolved by Na_2O_2 fusion, HFHCl bomb, or Aqua Regia)

Units: ppm (ug/mL, mg/L)

Analyte(s): **Boron (B)**
Titanium (Ti)

Method(s): Graphite Tube Atomizer (GTA) Atomic Absorption
Instrument(s): Varian AA-400
Sample Introduction: Deposit
Sample Type(s): Liquid (filtered)
Slurry (dissolved by Na_2O_2 fusion, HFHCl bomb, or Aqua Regia)
Sludge (dissolved by Na_2O_2 fusion, HFHCl bomb, or Aqua Regia)
Solids (dissolved by Na_2O_2 fusion, HFHCl bomb, or Aqua Regia)
Units: ppm (ug/mL, mg/L)
Status: NOT APPROVED

Fe(II)/Fe(III) Ratio

Analyte(s): **Fe(II)**
Method(s): Colorimetric (562 nm) measure Fe(II), convert Fe(III) to Fe(II), measure Fe(II), calculate ratio
Instrument(s): Hewlett Packard 8451A Diode Array Spectrometer
Sample Introduction: Liquid (no particulates)
Sample Type(s): Glass
Units: ppm (ug/mL, mg/L)

Ions

Analyte(s): **Chloride (Cl^-)**
Fluoride (F^-)
Formate (CHO_2^-)
Nitrate (NO_3^-)
Nitrite (NO_2^-)
Oxalate ($C_2O_4^{2-}$)
Method(s): Ion Chromatography
Instrument(s): Dionex 2110i
Dionex 4500i
Sample Introduction: Injection onto Column
Sample Type(s): Liquid (filtered, no particulates)
Slurry (dissolved by Na_2O_2 fusion, HFHCl bomb, or Aqua Regia)
Sludge (dissolved by Na_2O_2 fusion, HFHCl bomb, or Aqua Regia)
Units: ppm (ug/mL mg/L)

Analyte(s): **Ammonium (NH_4^+)**
Chloride (Cl^-)
Fluoride (F^-)
Method(s): Ion Selective Electrodes/ Ion Analyzer
Instrument(s): Radiometer Ion 85
Sample Introduction: Liquid
Sample Type(s): Liquid (no particulates)
Slurry (dissolved by Na_2O_2 fusion, HFHCl bomb, or Aqua Regia)
Sludge (dissolved by Na_2O_2 fusion, HFHCl bomb, or Aqua Regia)
Units: ppm (ug/mL mg/L)

Analyte(s): **Aluminate (AlO_2^-)**
Carbonate (CO_3^{2-})
Hydroxide (OH^-)
Method(s): Titration
Instrument(s): Mettler DL40GP Memotitrator
Sample Introduction: Liquid (basic solution pH > 7)

Sample Type(s):	Liquid (no particulates) Slurry (dissolved by Na_2O_2 fusion, HFHCI bomb, or Aqua Regia) Sludge (dissolved by Na_2O_2 fusion, HFHCI bomb, or Aqua Regia)
Units:	ppm (ug/mL, mg/L)

Organics

Analyte(s):	**Benzene** (> 0.5 ppb) **Chlorobenzene** (> 0.5 ppb)
Method(s):	Gas Chromatography Gas Chromatography, Purge and Trap (ppb levels)
Instrument(s):	Hewlett Packard 5890
Sample Introduction:	Injection into Capillary Tube onto Column
Sample Type(s):	Liquid (filtered, no particulates, no headspace) Slurry (filtered, no particulates, no headspace) Sludge (filtered, no particulates, no headspace)
Units:	ppm (ug/mL, mg/L)
Analyte(s):	**Biphenyl** (> 0.5 ppb) **Diphenyl** (> 0.5 ppb) **Terphenyl** (> 0.5 ppb) **Phenol** (> 0.5 ppb) **Phenylboric Acid** (> 0.5 ppb)
Method(s):	High Pressure Liquid Chromatography
Instrument(s):	Hewlett Packard 1090
Sample Introduction:	Injection onto Column
Sample Type(s):	Liquid (filtered, no particulates) Slurry (filtered, no particulates, no headspace) Sludge (filtered, no particulates, no headspace)
Units:	ppm (ub/mL, mg/L)

pH

Analyte(s):	**Hydronium Ion** (H_3O^+, H^+)
Method(s):	Glass Electrode/Ion Analyzer
Instrument(s):	Orion Research Model 701A Ionalyzer
Sample Introduction:	Liquid
Sample Type(s):	Liquid (no particulates) Slurry (dissolved by Na_2O_2 fusion, HFHCI bomb, or Aqua Regia) Sludge (dissolved by Na_2O_2 fusion, HFHCI bomb, or Aqua Regia)
Units:	ppm (ug/mL, mg/L)

Solids

Analyte(s):	**Total Solids**
Method(s):	Microwave Drying
Instrument(s):	CEM AVC-80
Sample Introduction:	Sandwiched between 2 Glass Plates
Sample Type(s):	Slurry (if solids > 5%) Sludge
Units:	

OFFSITE LABORATORIES

SCOPE

The Analytical Services Group (ASG) will assist SRL customers in obtaining offsite analysis. The extent of ASG involvement is based on the customers needs.

CAPABILITIES

The laboratories offer a wide range of services in analytical chemistry and environmental analysis. Examples of categories of testing include:

- General Chemistry
- Organics
- Waste Analysis (RCRA) and Waste Characterization
- USEPA Priority Pollutant Analyses

Only pre-qualified vendors who have met the QA requirements and have the proper certifications and licensing will be utilized. Some of these labs include:

- Normandeau and Associates
- Enwright Environmental Consulting Lab
- General Engineering Laboratories
- Envirodyne Engineering
- Galbraith Laboratories, Inc.
- IT Laboratory
- Other DOE Labs

RESPONSIBILITIES

The customer is responsible for submitting the samples to ASG. The samples must be accompanied by the normal service order and analytical request to facilitate the handling charges and reporting of results. When special sample handling is required the customer should negotiate these requirements with ASG prior to submitting samples.

ASG assumes the responsibility for establishing service contracts with qualified laboratories for routine analyses. If special studies or extensive testing is required, the customer may be required to negotiate the service contract.

ASG will also be responsible for contracting the vendor to make arrangements for testing, packaging of samples, preparation of shipping papers, obtaining authorization for shipment, shipping the samples, getting results from vendor, supplying customer with hard copy of results, and entering results into ASG LIMS.

PROCEDURE

The samples must be submitted to ASG Sample Receiving, Building 773-A, B-150, by the procedure outlined in Section A. (ANALYTICAL SERVICES) of this manual.

The following information is required from the originator of the samples:

- Material description
- Chemical form (solid, liquid, or gas)
- Material hazard (organic, radioactive, carcino gen, flammable)
- Amount submitted (gms, mls, etc.)
- Analyses requested

If the sample is radioactive, the following additional information is required:

- Radioisotope to be shipped (Am, Pu, U, etc.)
- Radioactive content by radioisotope (curies, grams, etc.)
- Wet chemical analyses and counting

The complete procedure for sample preparation for offsite analysis, ADS-0101 (PREPARATION FOR OFFSITE ANALYSES), is located in the ADS Procedure Manual.

Edna DeWeese, 5-2352, is the Task Supervisor responsible for coordinating offsite analysis.

HOW TO USE ADSLIMS

A step-by-step guide for the IBM PC and Macintosh user

This document was produced by
Susan Lance, SCD

This document was produced at the
Savannah River Laboratory,
Building 773-A, room B-141
Aiken, SC 29808

Phone 725-3306

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CONTENTS

INTRODUCTION

WHO SHOULD USE THIS MANUAL?

This manual is written for ADSLIMS user who has an IBM personal computer or Apple Macintosh that is used as a terminal and is using the terminal emulation package pcLink, Pacer Software, Inc..

Users of other terminal emulation packages should contact W. J. Kerrigan, 5-3306.

WHAT TYPE OF EQUIPMENT DO I NEED?

This manual will cover the use of the following equipment:

- IBM P/S 2
- IBM AT
- IBM XT

- Macintosh II
- Macintosh SE

HOW DO I USE THIS MANUAL?

This manual is designed to give you step-by-step instructions.

- Select the type of computer that you are using
- Follow the step-by-step instructions
- You type commands that are in *italic*
- Keys are enclosed in < >
- An Appendix contains additional information

WHAT IS ADSLIMS?

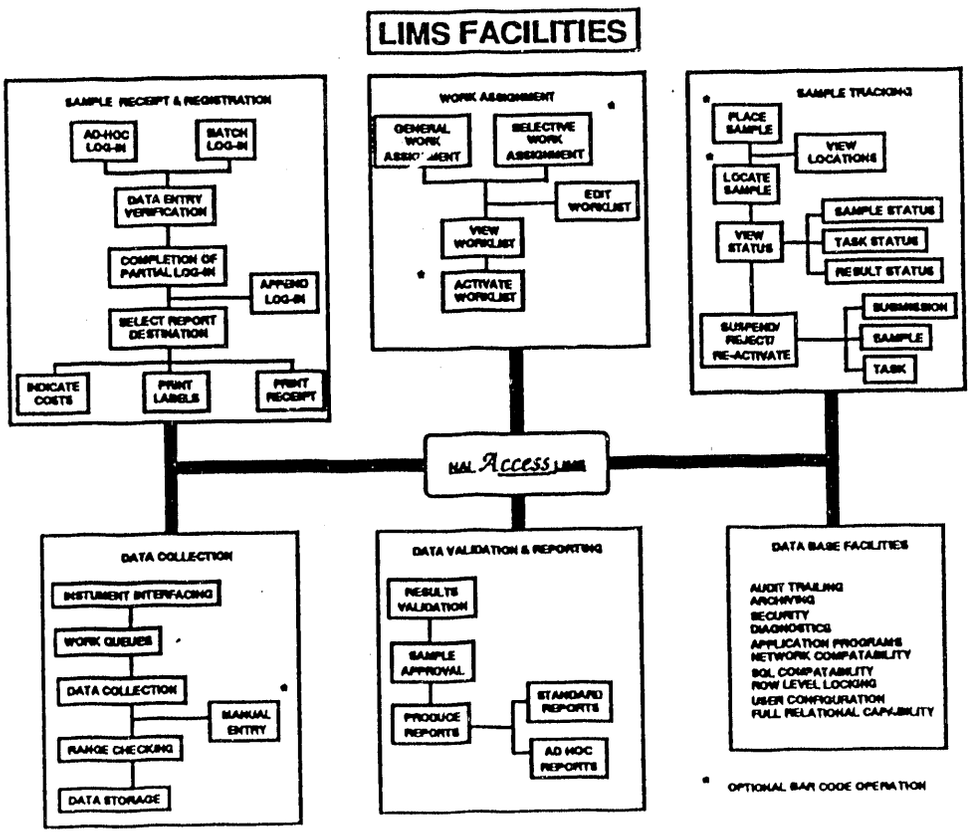
WHAT IS ACCESS*LIMS™ - A BRIEF DESCRIPTION

ACCESS*LIMS™ is an interactive - online Sample Management System and Laboratory Information Management System (LIMS) purchased from PE Nelson Systems, Inc., Cupertino, CA.

ADSLIMS allows you to store data about samples and the tests performed, to retrieve the data quickly and easily and to track the progress of the samples from the time they are logged into ADSLIMS until their testing is finished. This information remains online for about a year.

The ACCESS* LIMS product is supported by Oracle relational database management system (RDBMS) on a dedicated Digital Equipment Corporation (DEC) VAX/VMS computer that is a node on the local (SRL) area network (LAN). ADSLIMS users are captured accounts.

A SCHEMATIC OF ACCESS*LIMS™



LOGGING ON TO ADSLIMS

IBM PC

Access to ADSLIMS

- To have access to ADSLIMS, you must have an account. To request an account, contact **W. J. Kerrigan, 5-3306.**
- This manual is written for users of pcLink, the current site terminal emulation package for All-in-1.

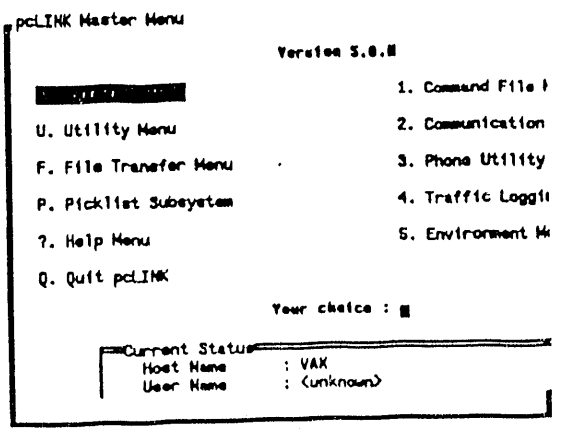
Instructions:

1. Turn on your computer.
2. You should see the DOS prompt which will look like this:

   C:\>

3. Type the following commands after the DOS prompt: The command you type appears in *italic*.
   - C:\> ***cd\ pclink***
   - Press the <Enter> key
   - C:\PCLINK> ***pclink***
   - Press the <Enter> key

The pcLINK Master Menu screen will appear and look like this:



Macintosh

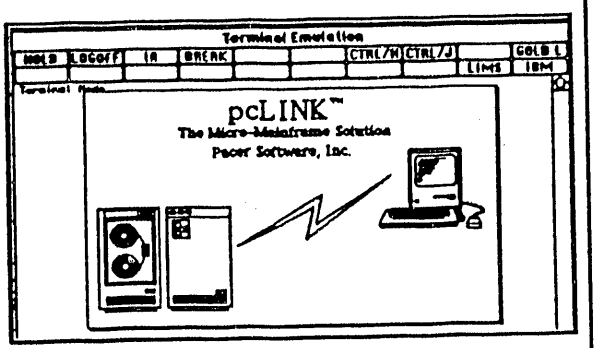
Access to ADSLIMS

- To have access to ADSLIMS, you must have an account. To request an account, contact **W. J. Kerrigan, 5-3306.**
- This manual is written for users of pcLink, the current site terminal emulation package for All-in1.

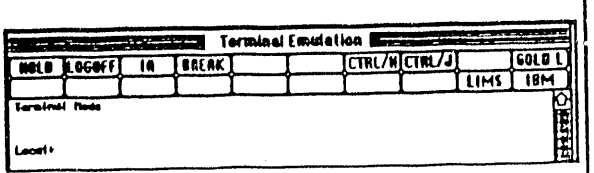
Instructions:

1. Turn on your computer.
2. Locate and double-click on the All-in-1 icon. The icon looks like this:

3. The Terminal Emulation screen will appear and look like this:



4. Press the <Return> key twice or until you see the Local> prompt:



LOGGING ON TO ADSLIMS continued

IBM PC

4. Select the option - 1. Terminal Mode by using the directional arrows to highlight it, if necessary.
   Press the **<Enter>** key.

```
Entering VT220 emulation...
```

5. Press **<Enter>** at the pcLINK Terminal Mode screen.

```
pcLINK Terminal Mode

  (there is no traffic logging being performed)

  Press <ALT> M to invoke Main Terminal Control Menu.
```

6. At the **Local >** prompt:
   Type ***C ADSLIMS***
   Press the **<Enter>** key.
   This will connect you to ADSLIMS.

```
pcLINK Terminal Mode

  (there is no traffic logging being performed)

  Press <ALT> M to invoke Main Terminal Control Menu.

Local> C ADSLIMS
```

7. Type in your **Username** at the prompt on the Analytical Development Section screen. Your username will be given to you when you receive your account.

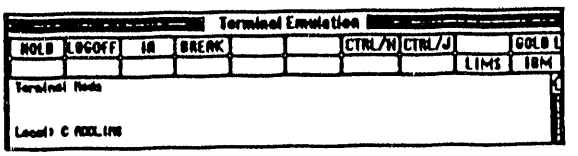
```
  Analytical Development Di

                    VAX/VMS Version V5.1
          DECNet Node: SLADMS     LAT Service:
Username: LANCE_SE
```

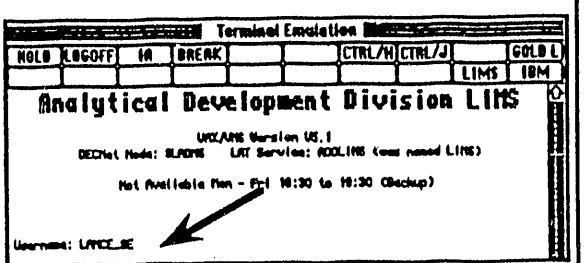
8. Type in your **Password** at the prompt and press the **<Enter>** key. Your password will not appear on the screen when you type.

Macintosh

5. At the **Local >** prompt
   Type ***C ADSLIMS***
   Press the **<Return>** key. This will connect you to ADSLIMS.

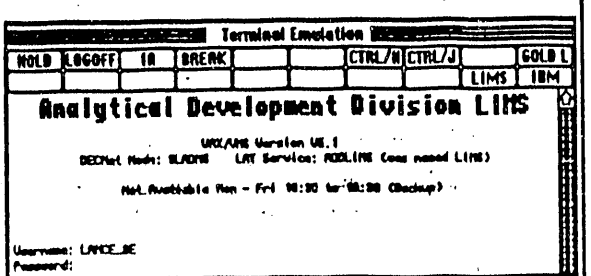


6. Type in your **Username** at the prompt on the Analytical Development Section screen. Your username will be given to you when you receive your account.



7. Type in your **Password** at the prompt and press the **<Return>** key.

   Your password will not appear on the screen when you type.



CHANGING YOUR VMS PASSWORD

IBM PC

Macintosh

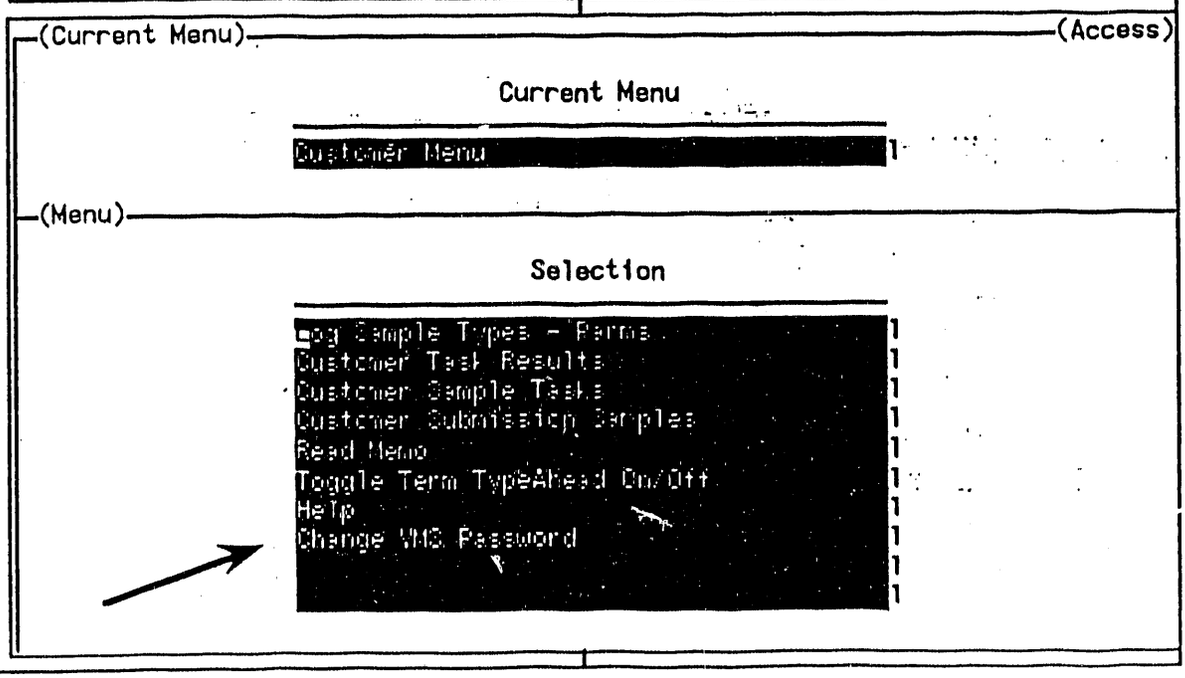
TO CHANGE YOUR VMS PASSWORD:

- Select ***Change VMS Password*** from the Current Menu by pressing the down directional arrow until the cursor is on the selection.

Press <Enter>	Press <Return>
• Type in your old password Press <Enter>	• Type in your old password Press <Return>
• Type in your new password* Press <Enter>	• Type in your new password* Press <Return>
• Verify your new password by typing it again. Press <Enter>	• Verify your new password by typing it again. Press <Return>

*Your password must contain six characters or more and may not have spaces or some special characters. Use a password that you will remember. Only you know your password.

You should change your password periodically to provide security of the system. Never write down your password and leave it near your terminal where it might be used by others.



SPECIAL FUNCTION KEYS

IBM PC

Macintosh

TO OBTAIN YOUR SPECIAL FUNCTION KEYS OR SOFTKEYS:

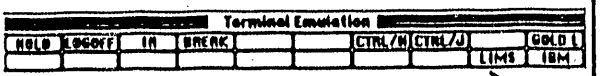
- Both the IBM PC and the Macintosh have special keys which can be used with pcLINK and ADSLIMS.
- You should receive a diskette when you get your ADSLIMS account. This diskette will allow you to install the special keys on your personal computer.
- For instruction on how to install your special keys, see the Appendix in the back of this manual.
- For more information or assistance, call **W. J. Kerrigan, 5-3306.**

USING FUNCTION KEYS - IBM PC:

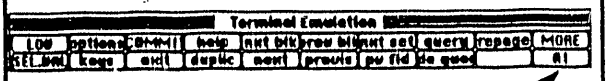
- Function keys are those keys that appear
  - On the top row of your keyboard if you have an IBM enhanced keyboard. There will be 12 keys labeled F1 - F12.
  - On a pad to the left of your regular typing keys on the IBM XT or AT keyboard. There will be 10 keys labeled F1 - F10.
  - These keys also may be referred to as PF keys but are the same as F keys.
- To load your set of functions keys for use with pcLINK and ADSLIMS, follow the instructions given in the Appendix of this manual.
- More information on key mapping is available in the Appendix.
- <Ctrl Z> will back you out of any screen while you are in ADSLIMS.

USING SOFTKEYS ON THE MAC:

- After you have installed your softkeys, you will notice a new item ADSLIMS under Terminal Emulation at the top of your screen.
- Click on the ADSLIMS button with the mouse to activate the ADSLIMS softkeys.



- To use the softkeys in ADSLIMS, click on the button you want. See the Appendix for definition of the functions.



- To toggle back to the All-in-1 softkeys, click on *A1*.
- To load your set of softkeys for use with pcLINK and ADSLIMS, follow the instructions given in the Appendix of this manual.

CUSTOMER MENU - LOGGING SAMPLES

IBM PC	Macintosh

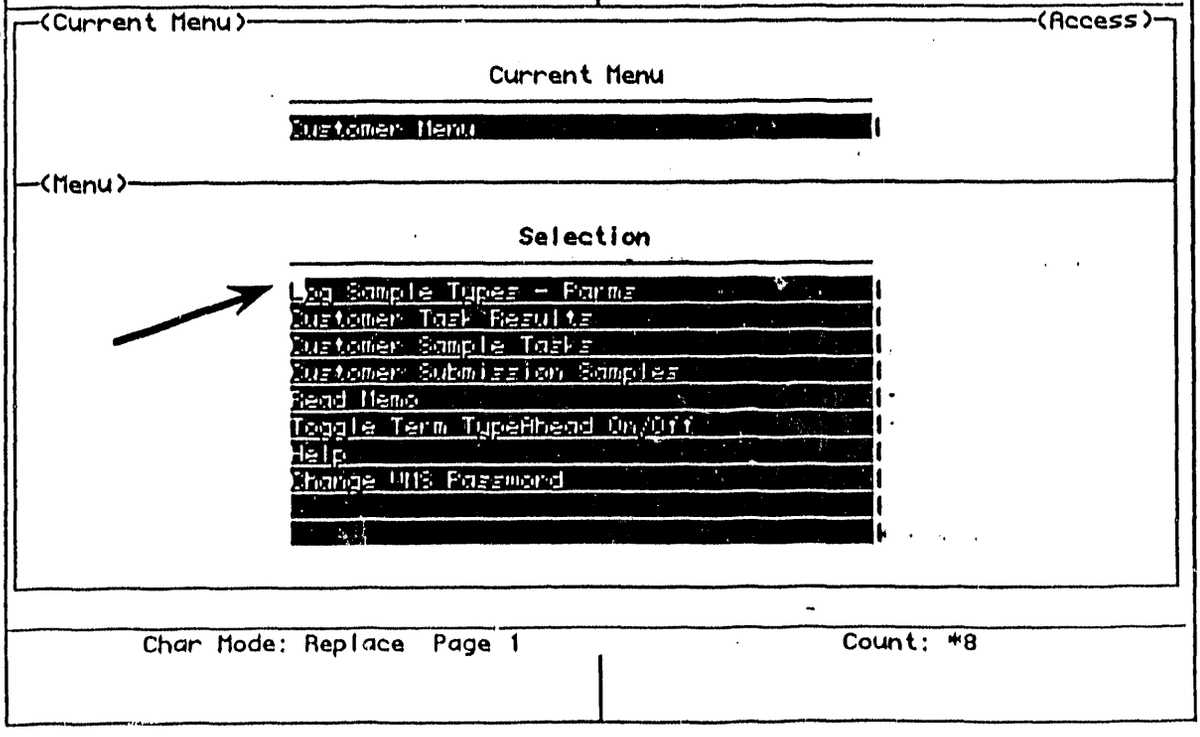
TO ENTER YOUR SAMPLES:

- Select ***Log Sample Types Parms*** by placing the cursor on that line.

Press <Enter>	Press <Return>

- Move the cursor by using the up and down directional arrows* on your keyboard.
- The cursor is the small blinking box-shaped object on your screen. It identifies your location on the screen.

* If you are using an IBM XT style keyboard, turn off your NUM LOCK before using the directional arrows. If you have the NUM LOCK activated, you will type in numbers.



SAMPLE TYPES - LIST OF VALUES

IBM PC	Macintosh

TO ENTER REQUIRED INFORMATION:

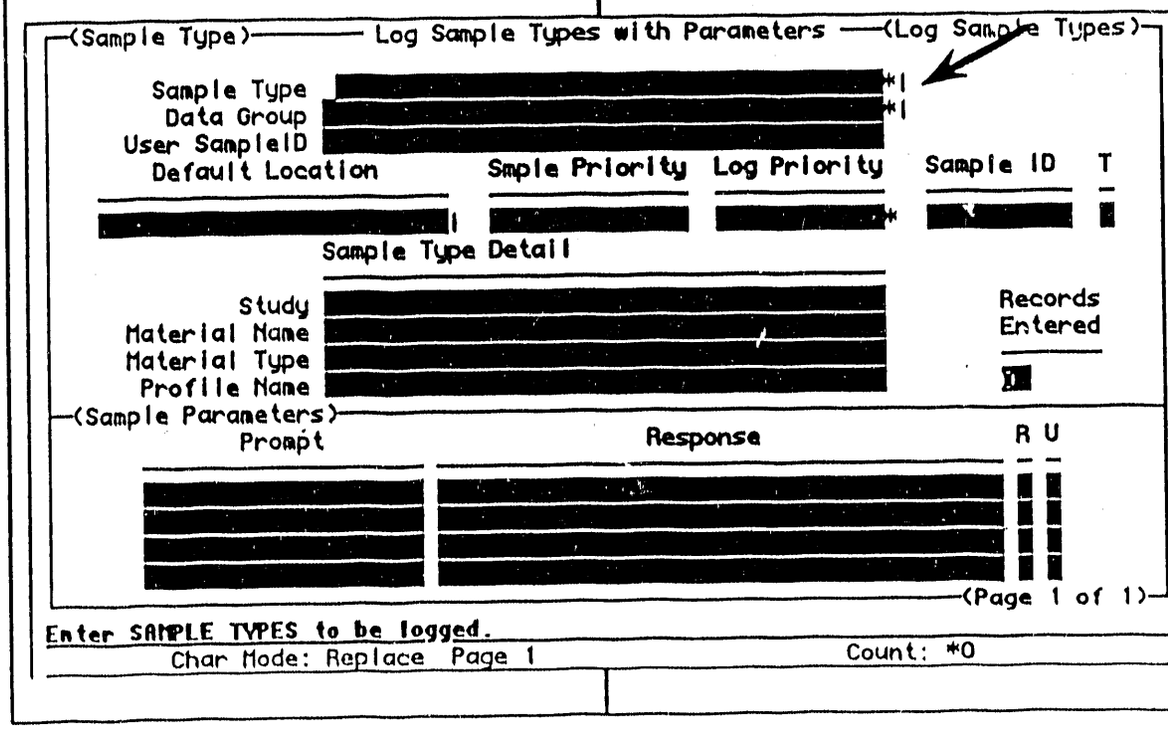
- An "*" indicates required information. This information must be entered before you can continue to the next field.
- An "I" indicates a list of values (LOV) help is available for a field.

TO OBTAIN A LIST OF VALUES FOR THE SAMPLE TYPE FIELD:

- Use the LOV key

*Press LOV <Ctrl F3>	Click on LOV in the softkeys

* Hold down <Ctrl> key and strike <F3>



SAMPLES TYPES - QUERY

IBM PC	Macintosh

TO QUERY SAMPLE TYPES FOR YOUR DATA GROUP:

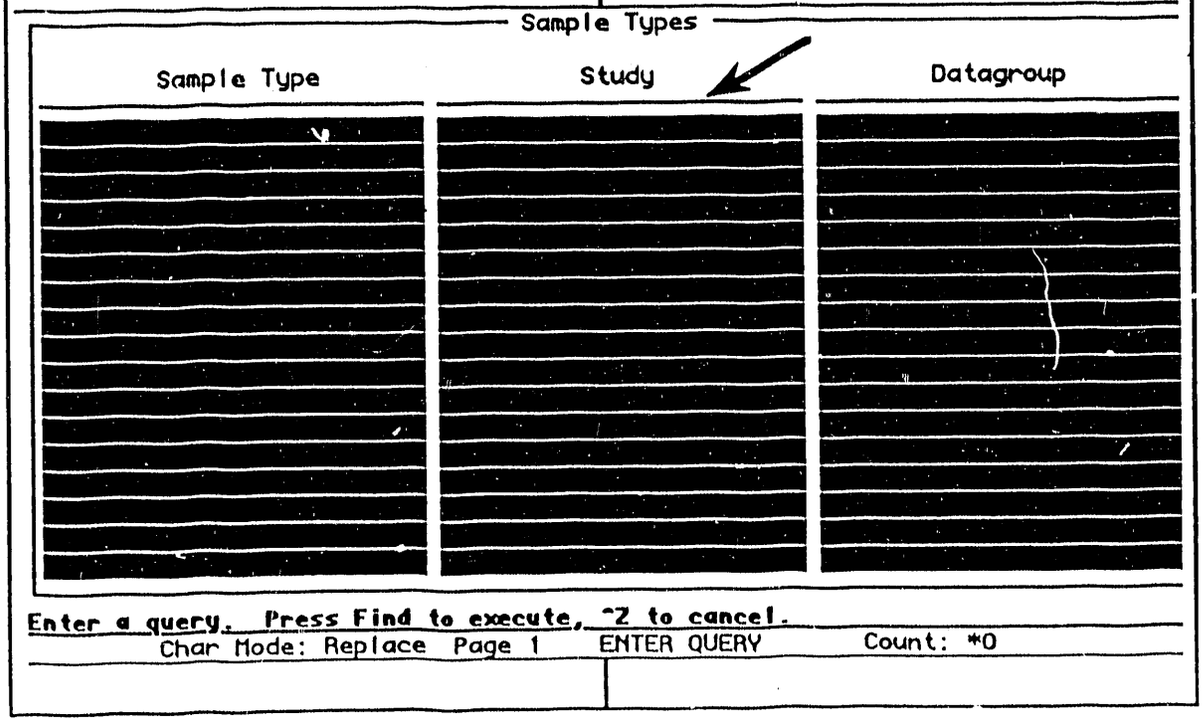
- You have access only to the Sample Types of your Data Group.
- To get a listing of all Sample Types for your Data Group:
- Use Execute Query key.

*Press Execute Query <Alt F1>	Click on **do query** in softkeys

*Hold down the <Alt> key and strike <F1>

- This fills in a list of 17 lines of Sample Types on the screen.
- More efficient queries can be made using your Study to limit your search to particular sample types.

  For example, type in ***%ICP%*** to limit the search to ICP Study. The % is a wildcard which allows all ICP studies to be found.



SAMPLES TYPE - SELECTION

IBM PC	Macintosh

TO SELECT A SAMPLE TYPE:

- A list of 17 Sample Types can be shown on the screen at one time. Check for additional types by using the down directional arrow or Next Set key.

Press Next Set <F10>	Click on Nxt Set in softkeys

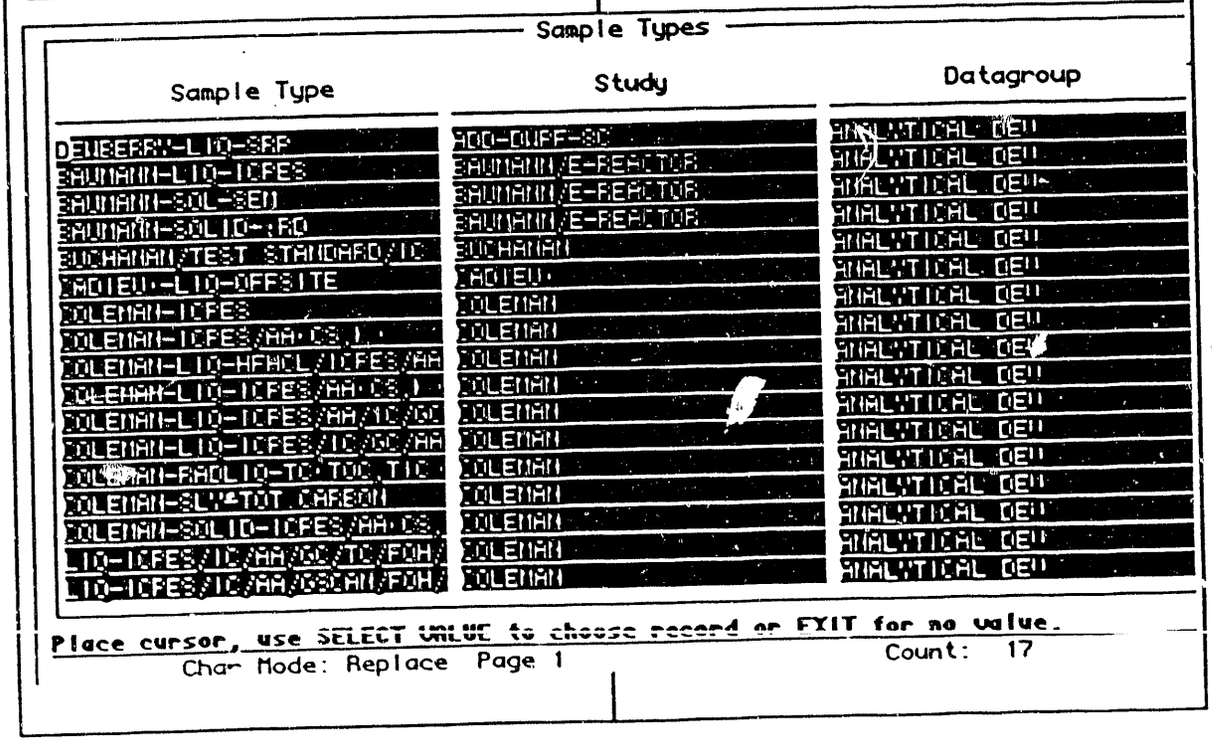
- To select a Sample Type, place the cursor on the correct line by using the directional arrows. Use the Select Value key.

Press Select Value <Ctrl F1>	Click on SEL.VAL in softkeys

- The system will redisplay the Log Sample Type form.
  PLEASE WAIT! The system will retrieve all information including parameters based on study and sample type.

- To exit the screen without selecting a Sample Type, use the Exit key.

Press <Ctrl Z> or <F4>	Click on exit in softkeys



USER SAMPLE ID NAME

IBM PC	Macintosh

TO ENTER THE USER SAMPLE ID:

- The screen will return to the Sample entry form. The cursor should be on the User Sample ID line. If it is not, use the <Tab> key to move the cursor to that position.
- Enter the name that you will use to identify the sample on this line. Cluster tests using unique names like "WELL" # or "TANK" # to facilitate retrieval of clustered results.

TO EDIT THE USER SAMPLE ID:

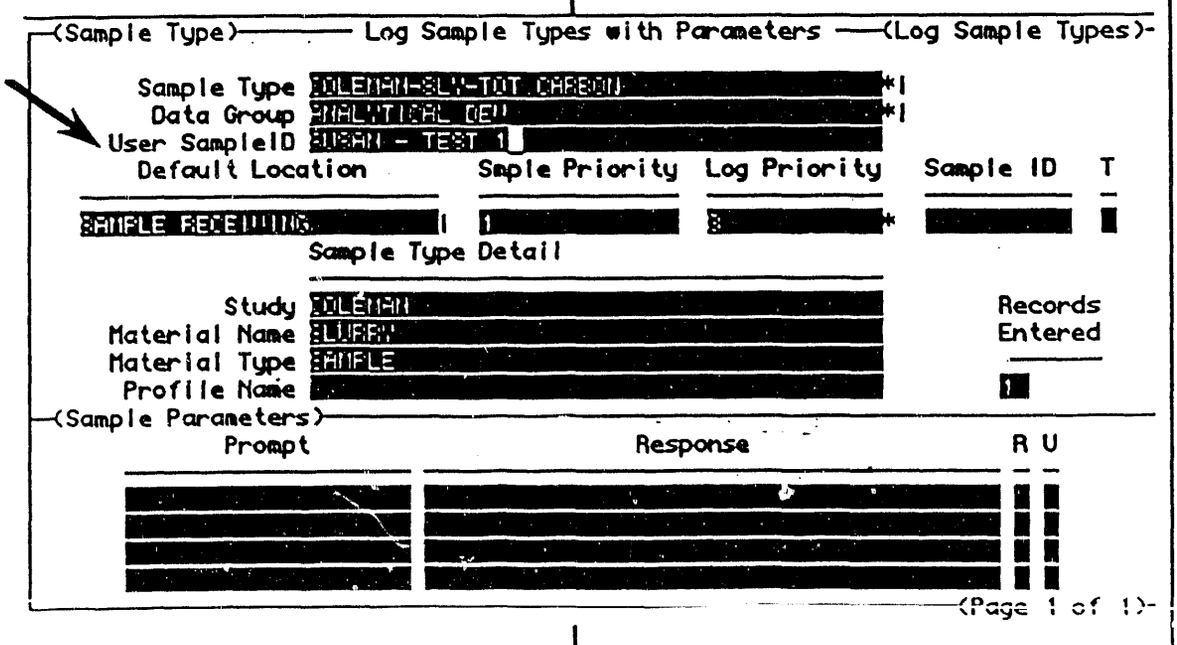
- Use the <delete> or <Backspace> key to make corrections on the User Sample ID line.

TO EXIT THE SCREEN WITHOUT SAVING THE USER SAMPLE ID:

Press <Ctrl Z>	Click on **exit** in softkeys

- Anwer *N* for No. You are returned to the Customer Menu.

Do you want to commit the changes you have made?



SAMPLE ID NUMBER

IBM PC	Macintosh

TO ASSIGN A UNIQUE SAMPLE ID:

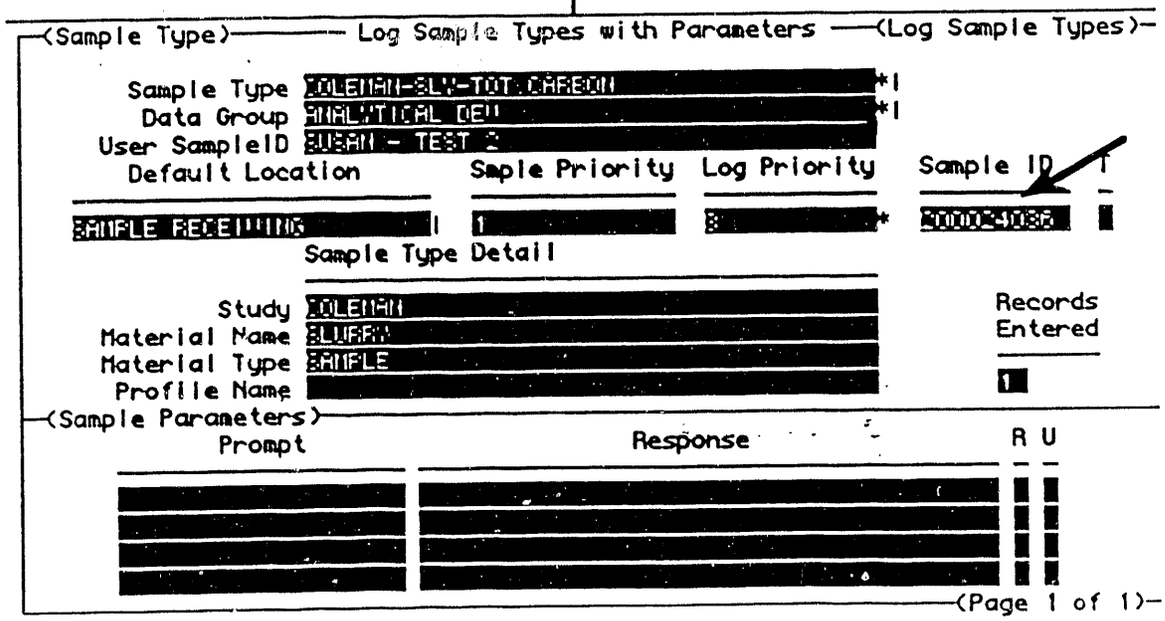
- ADSLIMS assigns a unique Sample ID for each sample you log.
- Use the Next Block key to move to the next block.

Press Nxt Block <F6>	Click on **nxt blk** in softkeys

- The Sample ID will be assigned automatically by the system.
- **Write down this number.** The Sample ID number will be used to track your sample.
- There will be message at the bottom of the screen "*Press any function key to acknowledge message*". Acknowledge the message.

Press <Enter>	Press <Return>

- Complete the parameters.



SAMPLE PARAMETERS

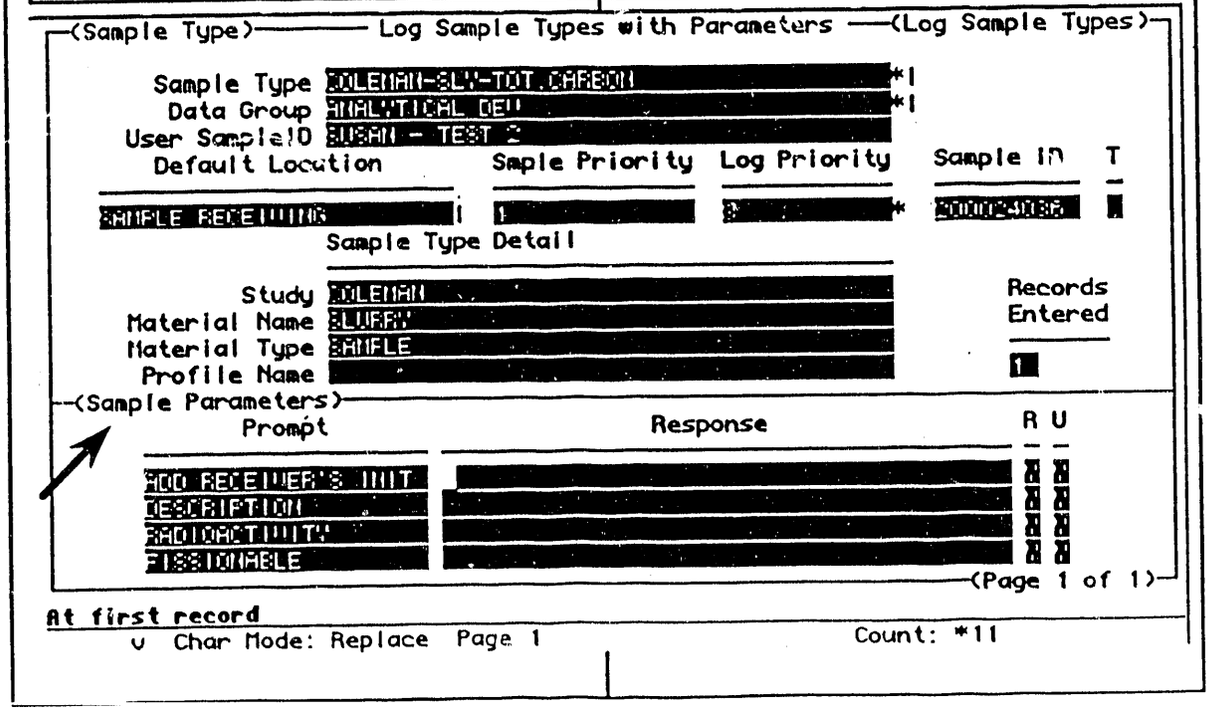
IBM PC	Macintosh

TO RESPOND TO SAMPLE PARAMETERS:

- The Sample Parameter block brings up information associated with the sample type.
- If the parameter requires an answer, it must be answered or the samples will be logged as incomplete.

TO DENOTE A RECEIVED SAMPLE:

- If you do not have the sample yet and only want a Sample ID number for reference, do not respond to the prompt for initials.
- When you deliver the sample, have ADS respond to the prompt to put the sample online. You will receive a Received Sample Report.
- All other required parameters must be entered for the sample to be complete and online.
- Use the directional arrows to move within the Sample Parameters block. Use the <delete> or <Backspace> key to make corrections.

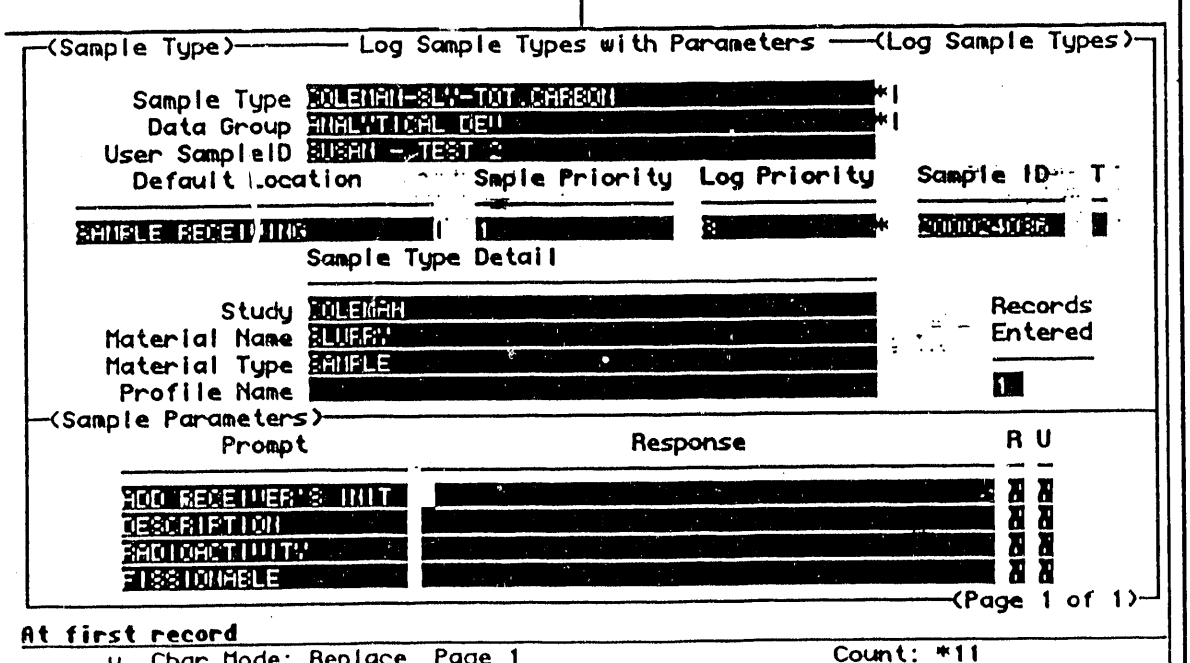


SAMPLE PARAMETER continued

IBM PC	Macintosh

TO EDIT THE SAMPLE PARAMETER BLOCK:

- Use the directional arrows to move within the Sample Parameter block.
  Use the <delete> key or <backspace> key to make corrections.
- Use the <Return> or <Enter> key to move within the Sample Type block. The Up Directional Arrow retrieves the previous record and the Down Directional Arrow retrieves the next record.



ENTER SAMPLES OF SAME TYPE

IBM PC	Macintosh

TO ENTER MORE SAMPLES OF THE SAME TYPE:

- Return the cursor to the top half of the screen

IBM PC	Macintosh
*Press Create Record<Alt F2> **Press Duplicate Record <F7>	**Click on DUPLIC in softkeys

*The screen will clear.
**Wait until the old Sample ID number has cleared.

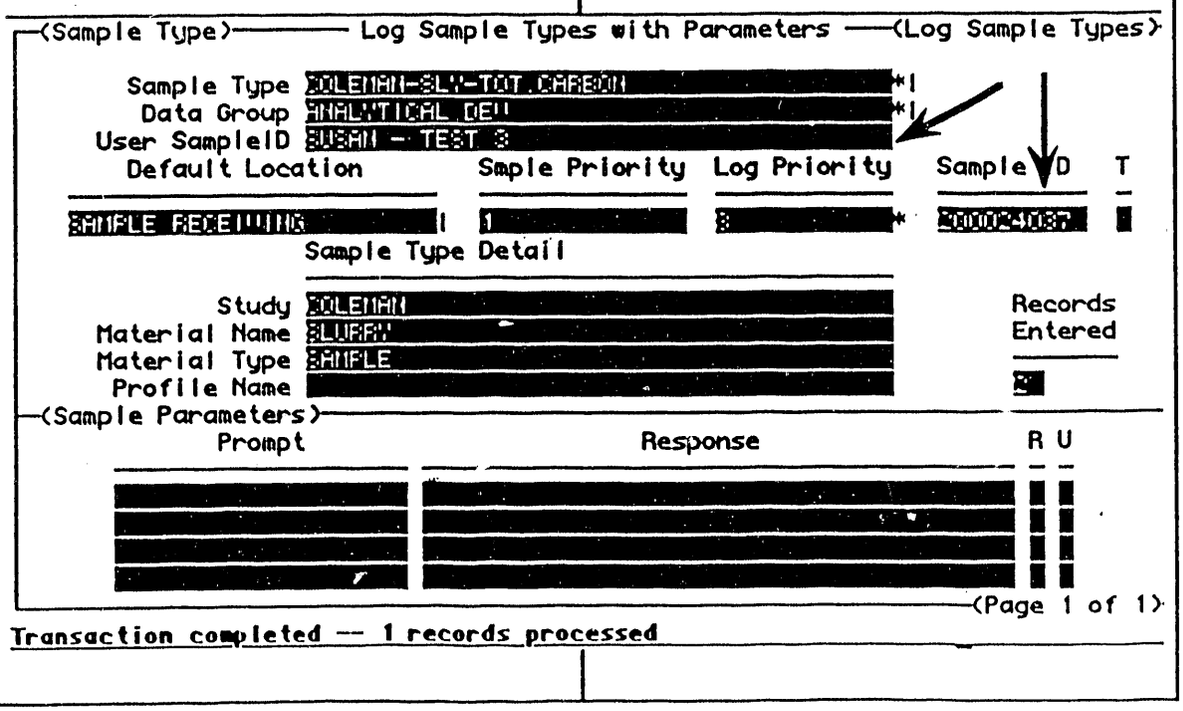
- Move to the User Sample ID line. Change the sample name if you want a different name for the next sample.
- If there are no changes, commit the new sample.

IBM PC	Macintosh
Press <Ctrl F6>	Click on COMMIT in softkeys

- **Write down the Sample ID.**
- Acknowledge the the message *Press any function key to acknowledge message.*

IBM PC	Macintosh
Press <Enter>	Press <Return>

- For additional samples, follow the same steps.



SUBMIT SAMPLE

IBM PC	Macintosh

TO SUBMIT A SAMPLE SET:

- Samples may be edited before leaving the submission session by using the directional arrows to page through the samples.
- Once editing is complete, exiting the Sample Submission Form (Log Sample screen) submits the sample(s) to the batch logger.

Press <Ctrl Z> or <F4>	Press <Return>

- Samples may be viewed online after being processed (generally about 30 mins.)
- System messages are logged in the Read Memo option on the Customer Menu and complete submissions are noted here with the submission ID.

CUSTOMER TRACKING:

- You can track the progress of the samples through the Customer tracking forms (shown below) from the time they are logged into ADSLIMS until their testing is finished. This information remains online for about a year.

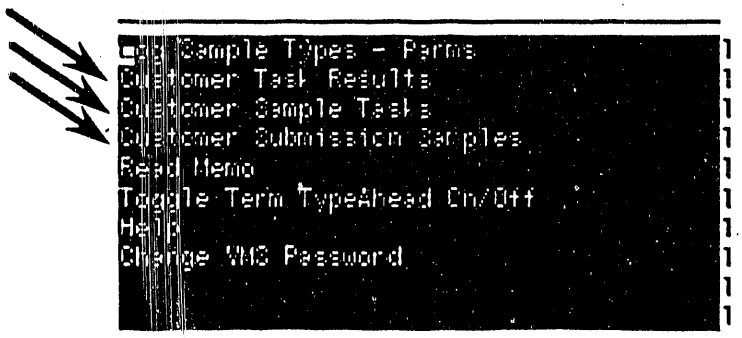
(Current Menu)——————————(Access

Current Menu

Customer Menu

(Menu)

Selection



TRACK YOUR SAMPLE - Online

IBM PC	Macintosh

CUSTOMER TASK RESULTS:

- Summarizes detail of the task done by ADS and is the most detailed of all of the tracking forms.

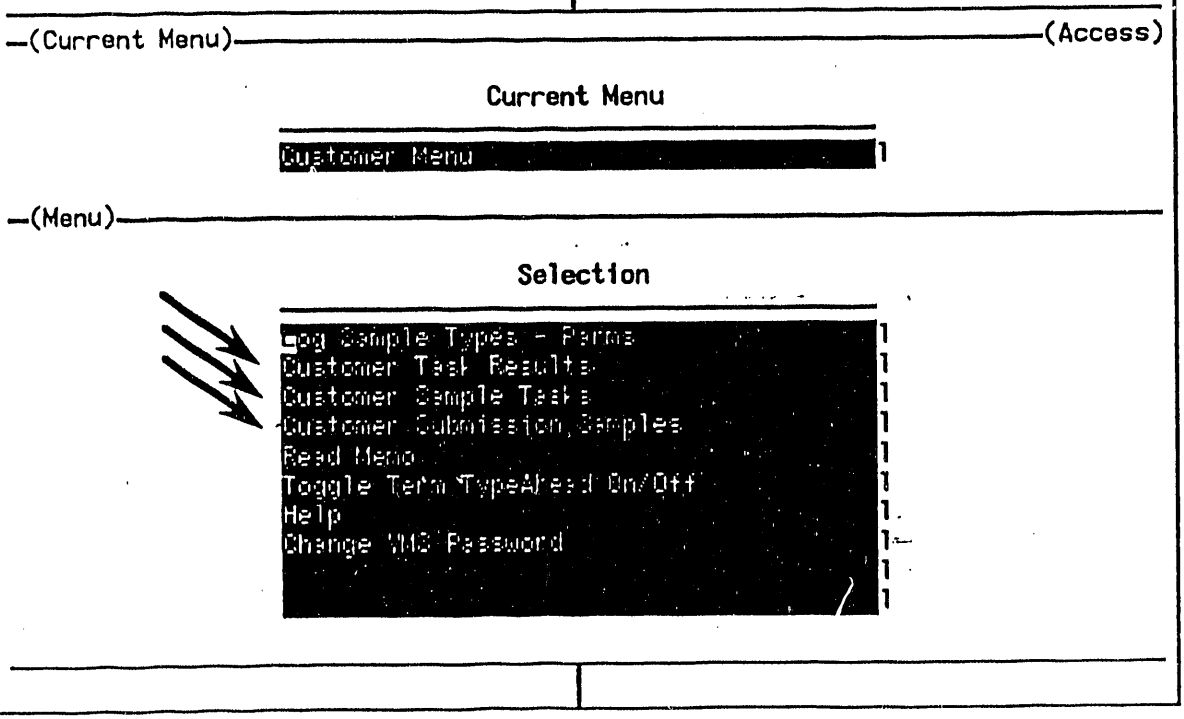
CUSTOMER SAMPLE TASKS:

- Customer Sample Tasks summarizes the work done on all tasks within the sample. Customer Sample Tasks form is a three block form having customized option line features available for additional detail on sample status and condition and task results.

CUSTOMER SUBMISSION SAMPLES:

- Customer Submission Samples provides an overview of all of the samples submitted during a particular submission (terminal) session.

Description and operation of each type of form follows:



CUSTOMER SAMPLE TASKS

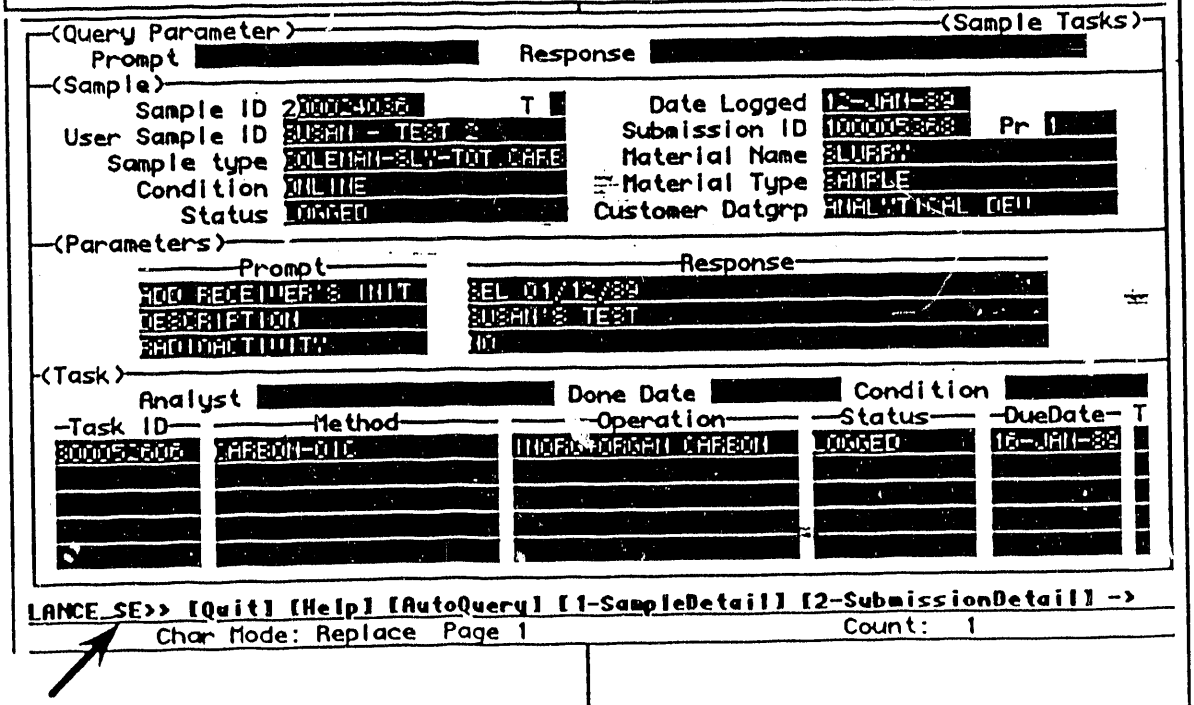
IBM PC	Macintosh

CUSTOMER SAMPLE TASKS:

- Customer Sample Tasks summarizes the work done on all tasks within the sample. Customer Sample Tasks form is a three block form having customized option line features available for additional detail on sample status and condition and task results.

TO TRACK INDIVIDUAL SAMPLES:

- Sample tasks are used to track individual samples through logging, progress of work and reporting of results. The samples remain online for about a year.
- Customer samples tasks uses system Sample ID as the preferred query. Other queries are allowed but are slower.
- Queries also allow wildcards but are slower.
- Sample ID's may be queried as unique numbers as shown below.
- Sample sets are shown on the next page.
- More detailed information may be obtained from the Option Line shown below. Customer Sample Task Options are listed on Page 21 of this manual.



CUSTOMER SAMPLE TASKS - SETS

IBM PC	Macintosh

TO TRACK SAMPLE SETS:

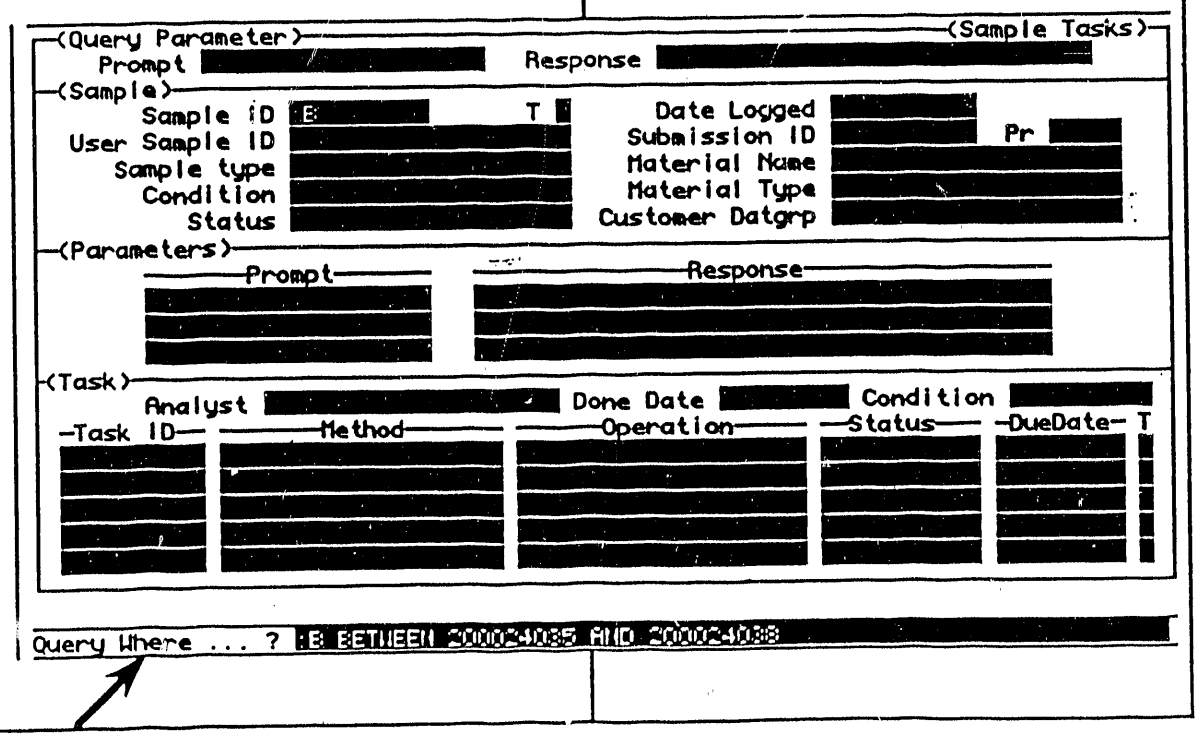
- Range selection uses a ":" marker, and identifier and Execute Query.

  For example:
  Query Where? *:B BETWEEN 200024035 AND 200024038*
  as shown below.

- Additional markers and identifiers may be used for example, in the Date Logged field, Condition and Status fields, etc. Other logical operators may also be used to query the Oracle database.

- The query line is limited to 40 characters

- Queries also allow wildcards but are slower.



CUSTOMER SAMPLE TASKS - OPTIONS

IBM PC	Macintosh

CUSTOMER SAMPLE TASKS:

- Customer Sample Tasks form is a three block form having customized option line features available for each block.

SAMPLE TASKS BLOCK OPTIONS:

[Quit] [Help] [AutoQuery][1-Sample Detail][2-SubmissionDetail] ⟶
⟵[3-ViewParameters][5-Locate Sample][Browse Text]

These option are used to track the progress of samples and give additional detail of samples and submissions.

PARAMETER BLOCK OPTIONS:

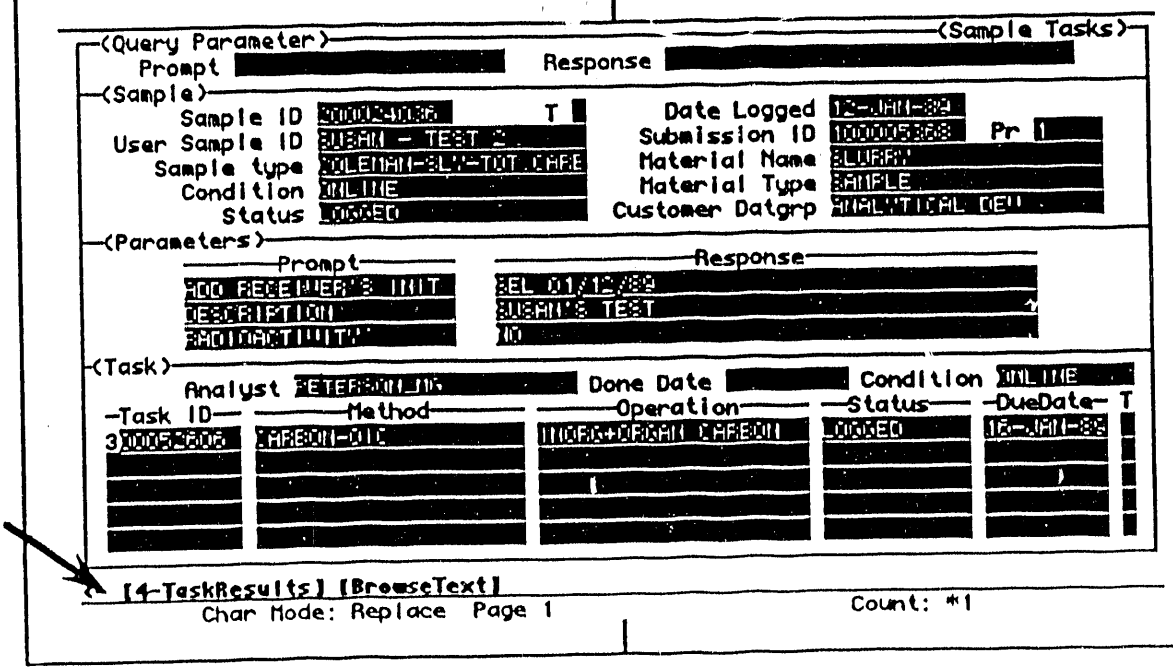
[Quit][Help][AutoQuery]

These options are limited and valueless options.

TASK BLOCK OPTIONS:

[Quit] [Help] [AutoQuery][1-TaskDetail][3-ViewParameters] ⟶
⟵[4-Task Results][Browse Text]

These options are used to retrieve actual results and details of test results.



CUSTOMER TASKS RESULTS

IBM PC	Macintosh

CUSTOMER SAMPLE TASKS:

Customer Tasks Result summarized the work done within the sample in more detail than the Customer Sample Task selection. Customer Tasks Results is a three block form having customized option line features option line features available for additional detail on sample status and condition and task results.

PARAMETER BLOCK:

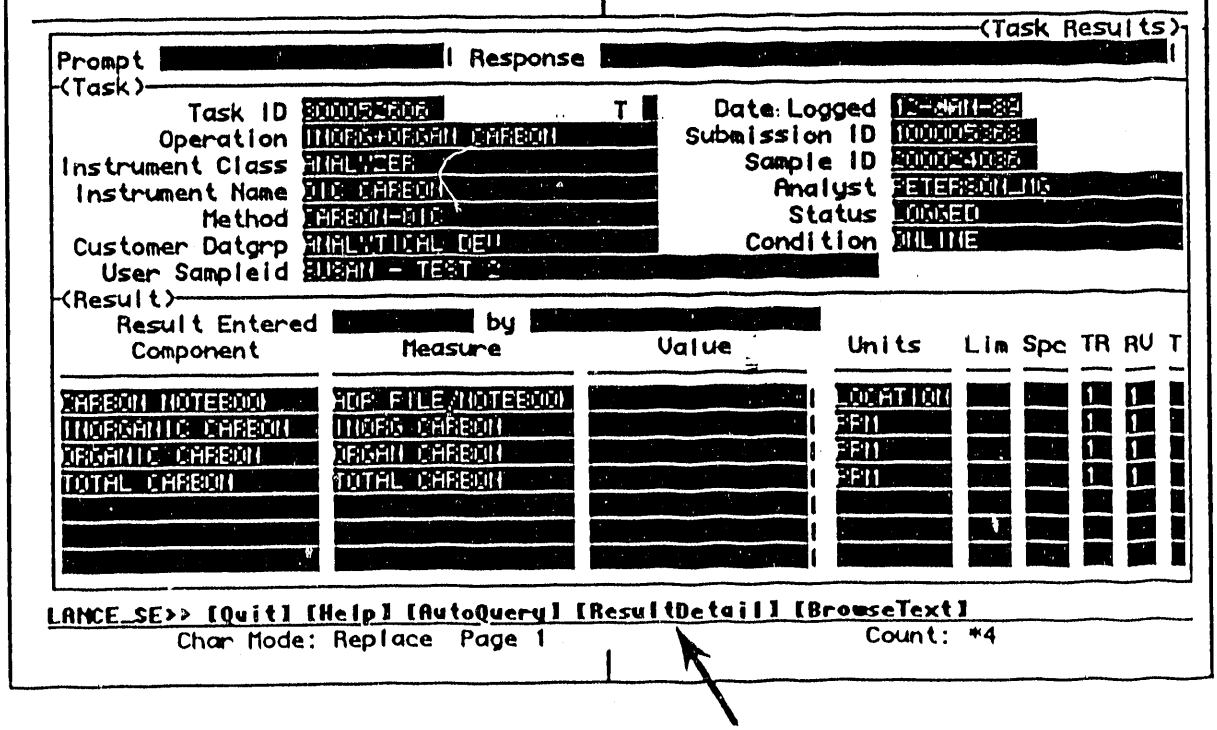
Queries by sample parameters may be made from this block using either prompt or response fields. Useful for clustering all samples of a given type. For example, FISSIONABLE.

TASK BLOCK:

All fields except User Sample ID are queriable. Retrieval by Sample, Submission or Task field is most efficient. Queries for sets use the same procedure as Customer Sample Task Sets in this manual.

RESULT BLOCK:

The Result Block is not queriable. This block displays the detail of results for queries in Task and Parameter Blocks. Result detail is obtained from the Option line.



CUSTOMER TASK RESULTS - OPTIONS

IBM PC	Macintosh

CUSTOMER TASK RESULTS:

PARAMETER BLOCK OPTIONS:

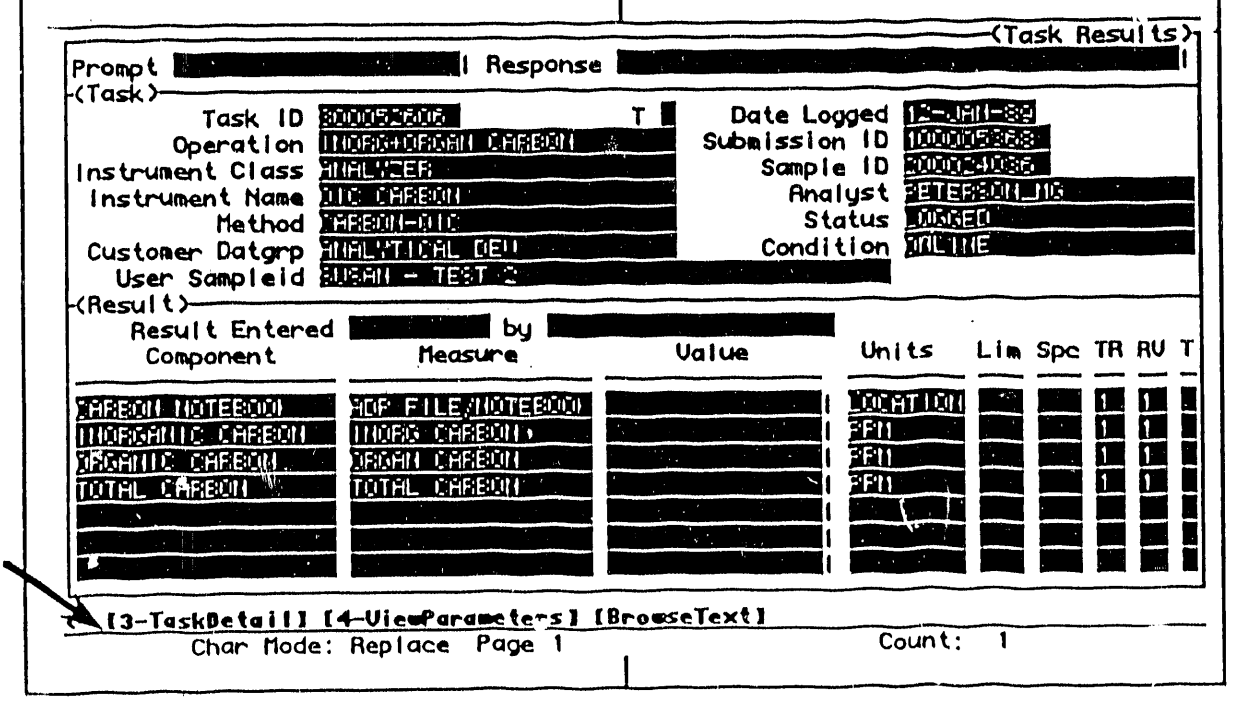
[Quit][Help][AutoQuery]

TASK BLOCK OPTIONS:

[Quit] [Help] [AutoQuery][1-SubmissionDetail][2-SampleDetail] →
←[3-TaskDetail][4-ViewParameters][Browse Text]

RESULT BLOCK OPTIONS:

[Quit] [Help] [AutoQuery][ResultDetail][Browse Text]



CUSTOMER SUBMISSION SAMPLES

IBM PC	Macintosh

CUSTOMER SUBMISSION SAMPLES:

Customer Submission Samples is an overview of Sample Sets.

PARAMETER BLOCK:

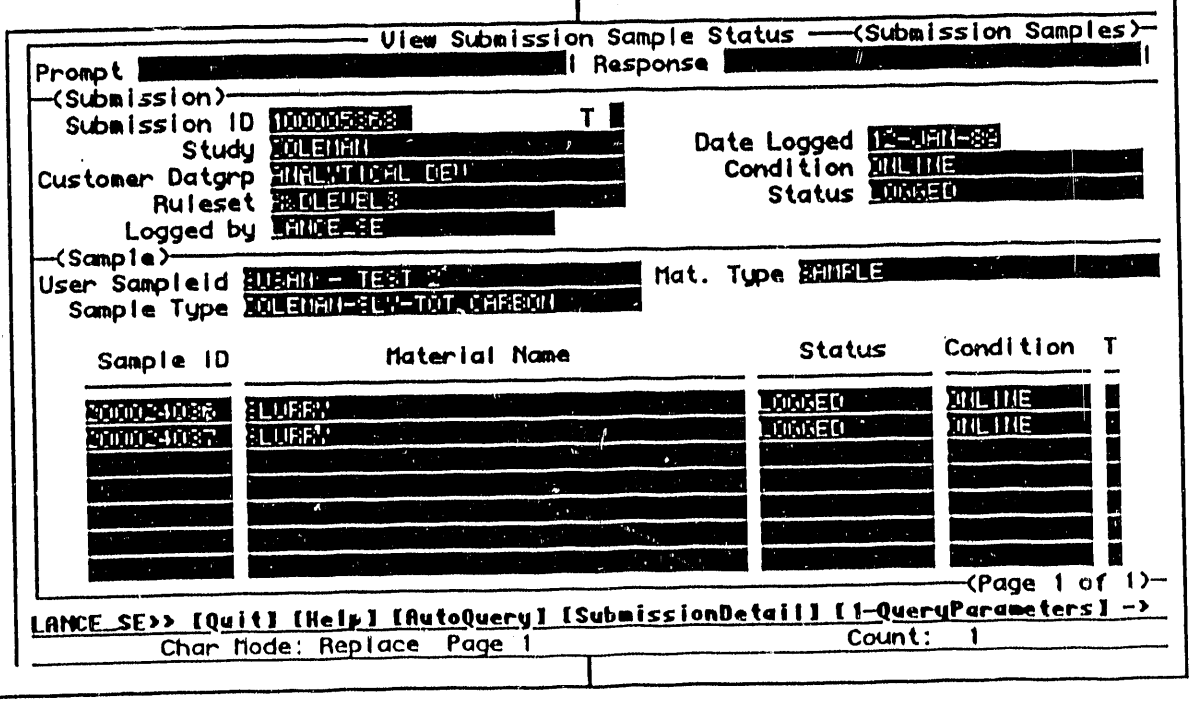
Generally not used for useful queries.

SUBMISSION BLOCK:

Queries used to to retrieve samples logged in sets clustered by Submission ID. The sample sets may be retrieved by Submission ID or Study and may be bracketed by Date logged, Condition or Status.

SAMPLE BLOCK:

The Sample Block is not queriable. The Sample Block displays detail status and condition for each sample. More detail can be obtained from the Option Line.



CUSTOMER SUBMISSION SAMPLES - OPTIONS

IBM PC	Macintosh

CUSTOMER SUBMISSION SAMPLES:

PARAMETER BLOCK OPTIONS:

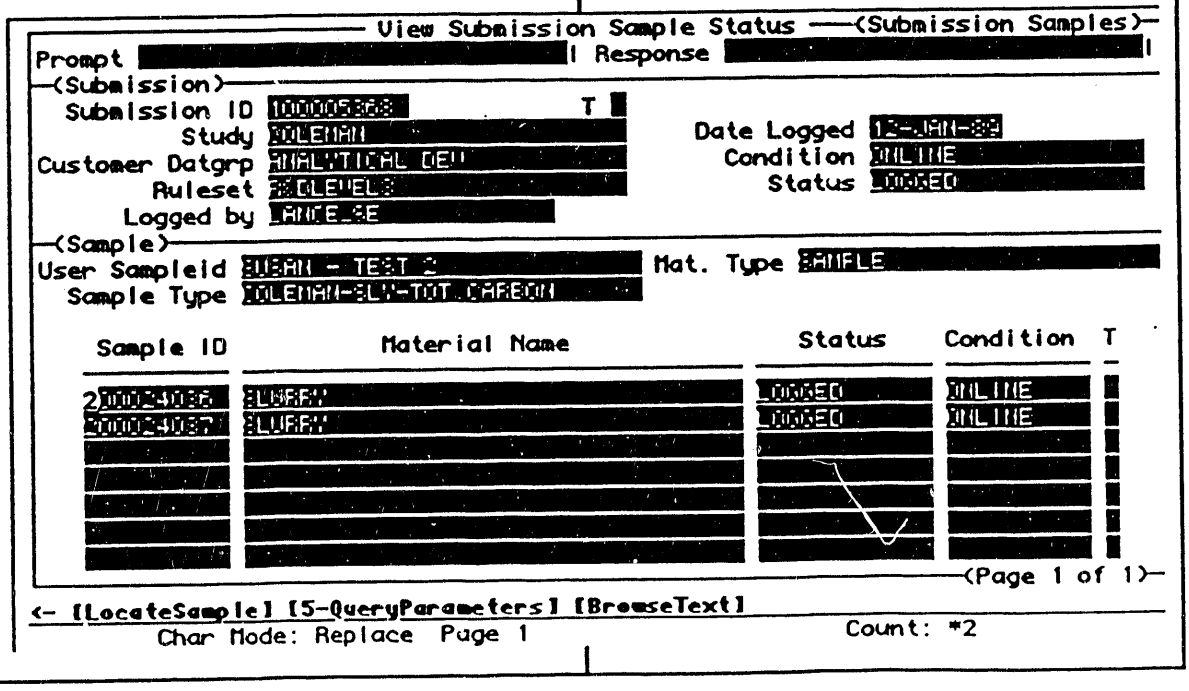
[Quit][Help][AutoQuery]

SUBMISSION BLOCK OPTIONS:

[Quit] [Help] [AutoQuery][SubmissionDetail][QueryParameters]→
←[Browse Text]

SAMPLE BLOCK OPTIONS:

[Quit] [Help] [AutoQuery][1-SampleDetail][3-SampleTask]→
←[LocateSample][5-QueryParameters][Browse Text]



CUSTOMER REPORTS

IBM PC	Macintosh

STANDARD HARDCOPY REPORTS:

- Standard hardcopy report will be sent to your user address when your work is complete.
- The user address is taken from the User Info selection in the Customer Menu. Use this selection to change your address, if necessary.

SPECIAL QUERIES:

- On request, the ADSLIMS system manager, Bill Kerrigan 5-3306, can provide electronic or hardcopy special queries clustering information about the samples your have submitted. Clustering by system identification or your user sample id (see the Logging Samples section of this manual - LOG ST PARMS) is the usual query strategy.

LOG OUT OF ADSLIMS

IBM PC	Macintosh

TO LOG OUT OF ADSLIMS:

- To log out of ADSLIMS from any screen

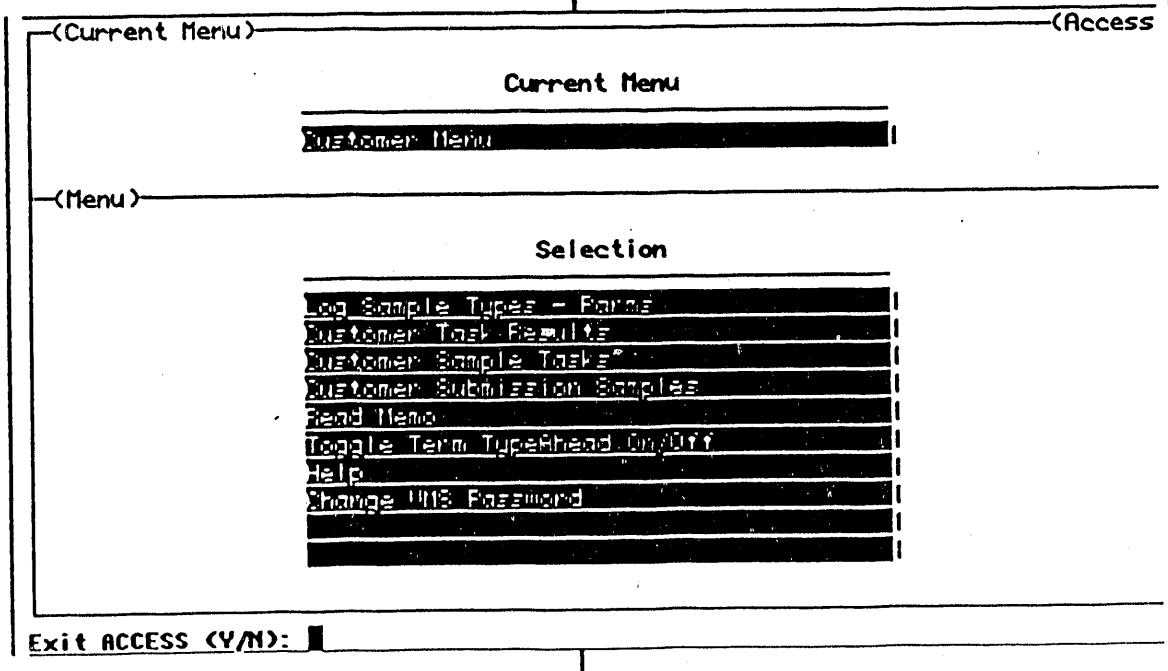
Press <Ctrl Z>	Click on exit in softkeys

until you reach the Customer Menu screen

Press <Ctrl Z>	Click on exit in softkeys

- Answer *Y* for *YES* to to the prompt *Exit ACCESS (Y/N):* to complete your logout.
- You will be returned to the Local > prompt.*

*Softkeys for other applications must be reloaded (ie. All-in-1).



APPENDIX

APPENDIX

LOADING SPECIAL FUNCTION KEYS FOR IBM

TO OBTAIN THE ADSLIMS FUNCTION KEY SET:

- You should receive a diskette with the Function Key Set files for use with pcLINK when you receive your ADSLIMS account. If you did not, contact W. J. Kerrigan, 5-3306.
- Specify what size diskette you need. This will depend on the type of personal computer you have.

PS/2 (IBM Personal System 2)	-	3.5" high density diskette
AT (IBM AT)	-	5.25" high or low density diskette
XT (IBM XT)	-	5.25" low density (dd, ds) diskette

TO LOAD THE ADSLIMS FUNCTION KEY SET:

- You will need to load the Function Key Set from your diskette only once. However, you will need to load the file from your hard disk each time you use ADSLIMS.

Type the responses that appear in ***bold italic.***

1. Turn on your computer. After the computer boots, insert the diskette in your floppy disk drive A: (A: is assumed to be your floppy drive).
2. Change directory to the PCLINK directory

   C:\> *CD \PCLINK*
   Press **<Enter>**

3. Enter the pcLINK program

   C:\PCLINK> *PCLINK*
   Press **<Enter>**

4. Select T. Terminal Mode from the pcLINK Master Menu

   Highlight **T. Terminal Mode** by using the up and down directional arrows
   Press **<Enter>**

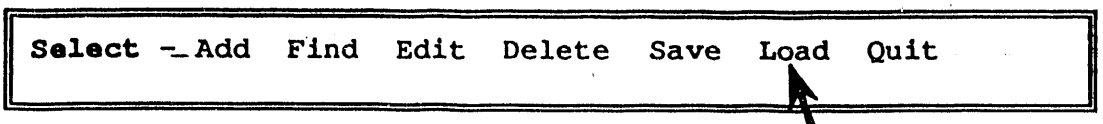
5. At the pcLINK Terminal Mode screen, to reach the Terminal Control Menu

   Press **<Alt M>**

6. Select the Function Key Menu from the Terminal Control Menu

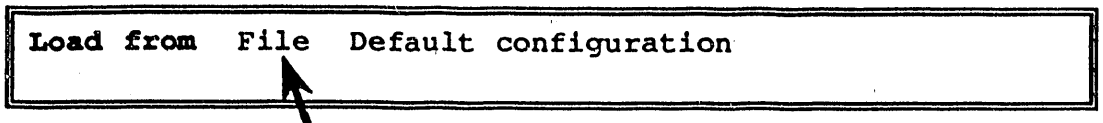
   Highlight **Function Key Menu** by using the up and down directional arrows
   Press **<Enter>**

LOADING SPECIAL FUNCTION KEYS - Continued



7. Select Load from from the Key Mappings - VT220 screen

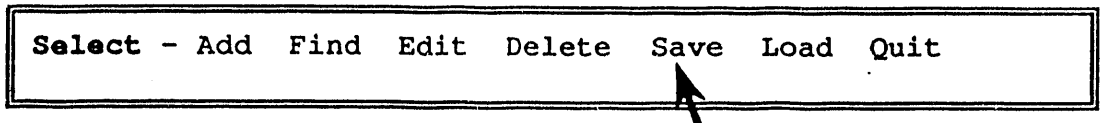
Highlight **Load** by using the left and right directional arrows
Press **<Enter>**



8. Select File

Highlight **File** by using the left and right directional arrows
Press **<Enter>**

9. Type in the drive identifier A: (A: is assumed to be your floppy drive) and filename ***LIMS.FKY***. There are no spaces between the drive and filename.
Press **<Enter>**

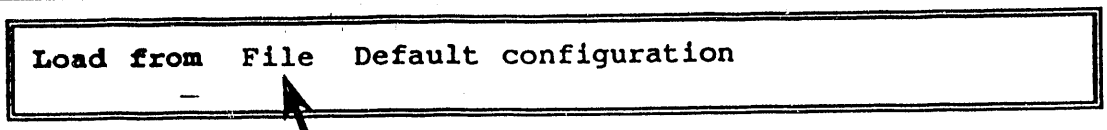


10. Select Save

Highlight **Save** by using the left and right directional arrows
Press **<Enter>**

Instructions are continued on the next page.

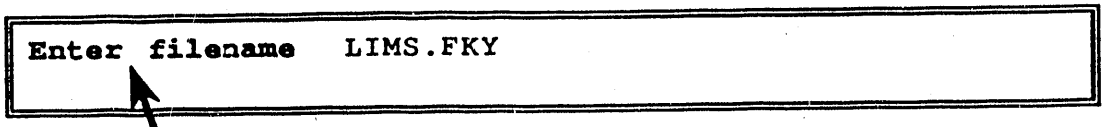
LOADING SPECIAL FUNCTION KEYS - Continued



11. Select File

    Highlight **File** by using the left and right directional arrows

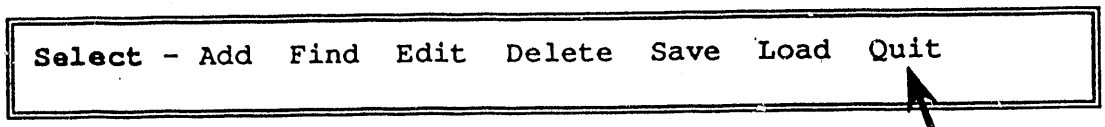
    Press **<Enter>**



12. Type in the filename

    LIMS.FKY

    Press **<Enter>**



13. Select Quit

    Highlight **Quit** by using the left and right directional arrows

- These steps load your function key set file to the pcLINK directory on your hard disk. When you exit pcLINK, the LIMS.FKY file remains on your hard disk but your LIMS special function keys are no longer loaded in pcLINK

TO LOAD LIMS.FKY FOR YOUR ADSLIMS SESSION:

- Follow Steps 2 - 8 of the preceding directions.
- In Step 9, type in the filename ***LIMS.FKY*** instead of A:LIMS.FKY.
- In Step 10, select Quit.
- At the Terminal Control Menu, continue logon as usual.

ADSLIMS KEY MAPPINGS

Key Mappings - VT220

LIMS Functions Keys	IBM Key	Mapping
BACKSPACE	BACKSPACE	o177
pcLINK terminal control menu	ALT-M	{Master menu}
SHOW KEYS	F1	o330P
SELECT VALUE	CTRL-F1	o33[23~
EXECUTE QUERY	ALT-F1	o33[1~
OPTIONS	F2	o330Q
CREATE RECORD	ALT-F2	o33[2~
DISPLAY ERROR	F3	o33OR
LIST OF VALUES	CTRL-F3	o33[25~
EXIT	F4	o33OS
	CTRL-Z	
ENTER QUERY MODE	ALT-F4	o33[4~
PREVIOUS BLOCK	F5	o33[5~
NEXT BLOCK	F6	o33[6~
COMMIT	CTRL-F6	o33[29~
DUPLICATE RECORD	F7	o33[18~
NEXT SET	F10	o33[21~
PREVIOUS FIELD	F12	o33[24~
REPAGE	CTRL-R	

LOADING SOFTKEYS FOR THE MACINTOSH

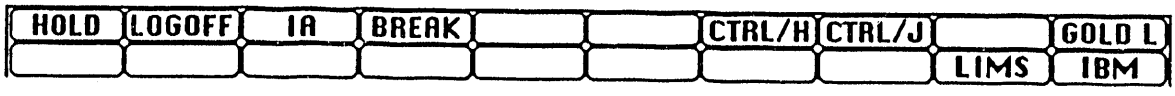
TO OBTAIN THE ADSLIMS SOFTKEY SET:

- You should receive a diskette with the LIMS Softkey Set files for use with pcLINK when you receive your ADSLIMS account. If you did not, contact W. J. Kerrigan, 5-3306.

TO LOAD THE ADSLIMS SOFTKEY SET:

- You will need to load the SoftKey Set only once.
- If you have mapped your softkeys for other uses, loading this set may change your current softkeys.
- Follow these directions:

1. Insert the LIMS Softkey diskette in the floppy drive.
2. Double click on the diskette icon to open.
3. Select and copy the files from the diskette into the pcLINK folder on your hard disk. (Hold down the <Shift> key to select more than one file. Release the <Shift> key. Drag the selected files to the pcLINK Folder.)
4. Close the folders and eject the diskette
5. Double click on the pcLINK icon.
6. Look at the top of the screen. A LIMS button is now on your softkeys.



7. Click on the LIMS button to get the LIMS softkeys.

8. Click on A1 to toggle back to the All-in-1 keys.

Quality Assurance Action Plan

Table Of Contents

WSRC-RP-89-390

QUALITY ASSURANCE ACTION PLAN

FOR THE ANALYSES OF SAMPLES BY THE

ANALYTICAL DEVELOPMENT SECTION
ANALYTICAL SERVICES GROUP (u)

APPROVAL:

W. A. Spencer 6/15/89
W. A. Spencer, ASG Manager
Derivative Classifier

E. F. Sturcken 6/15/89
E. F. Sturcken, ADS QA Coordinator

C. E. Coffey 6/22/89
C. E. Coffey, ADS Manager

REVIEWERS:

J. R. Knight
J. R. Knight, DWPTS Manager
Defense Waste Processing and Fuel Technology

per EW Holtzscheiter
E. W. Holtzscheiter, ATS Manager
Chemical Processes and Environmental Technology

C. W. Smith
C. W. Smith, LSS Manager
Laboratory Operations and Engineering

R. R. Fleming 6/15/89
R. R. Fleming, SRL Quality Section Manager
Quality Assurance

G. T. Wright 6/21/89
G. T. Wright, IWTS Manager
Interim Waste Technology

M. L. Hyder 6/15/89
M. L. Hyder, Associate Chemist
Reactor Safety Research

1.0 ORGANIZATION

1.0 PURPOSE

This section describes the organization of the Analytical Services Group (ASG) in the Analytical Development Section (ADS).

This Quality Assurance Action Plan (QAAP) describes the administrative controls and responsibilities for analyses of samples by the ASG.

2.0 SCOPE

This section applies to the ASG and to those organizations and personnel which interface with the ASG.

3.0 RESPONSIBILITIES

ASG job descriptions are kept on file in the ADS QA office.

4.0 PROCEDURE

4.1 Primary Organization

The ASG is organized as shown in Figure 1.

The SRL Personnel Section maintains and publishes an organization chart for SRL. The ADS Secretary maintains and publishes a list of line management assignments within ADS.

The ASG interfaces with SRL Sections and SRS Departments in providing analytical services. The group interfaces with all support service groups of SRL and with Site Support groups as necessary.

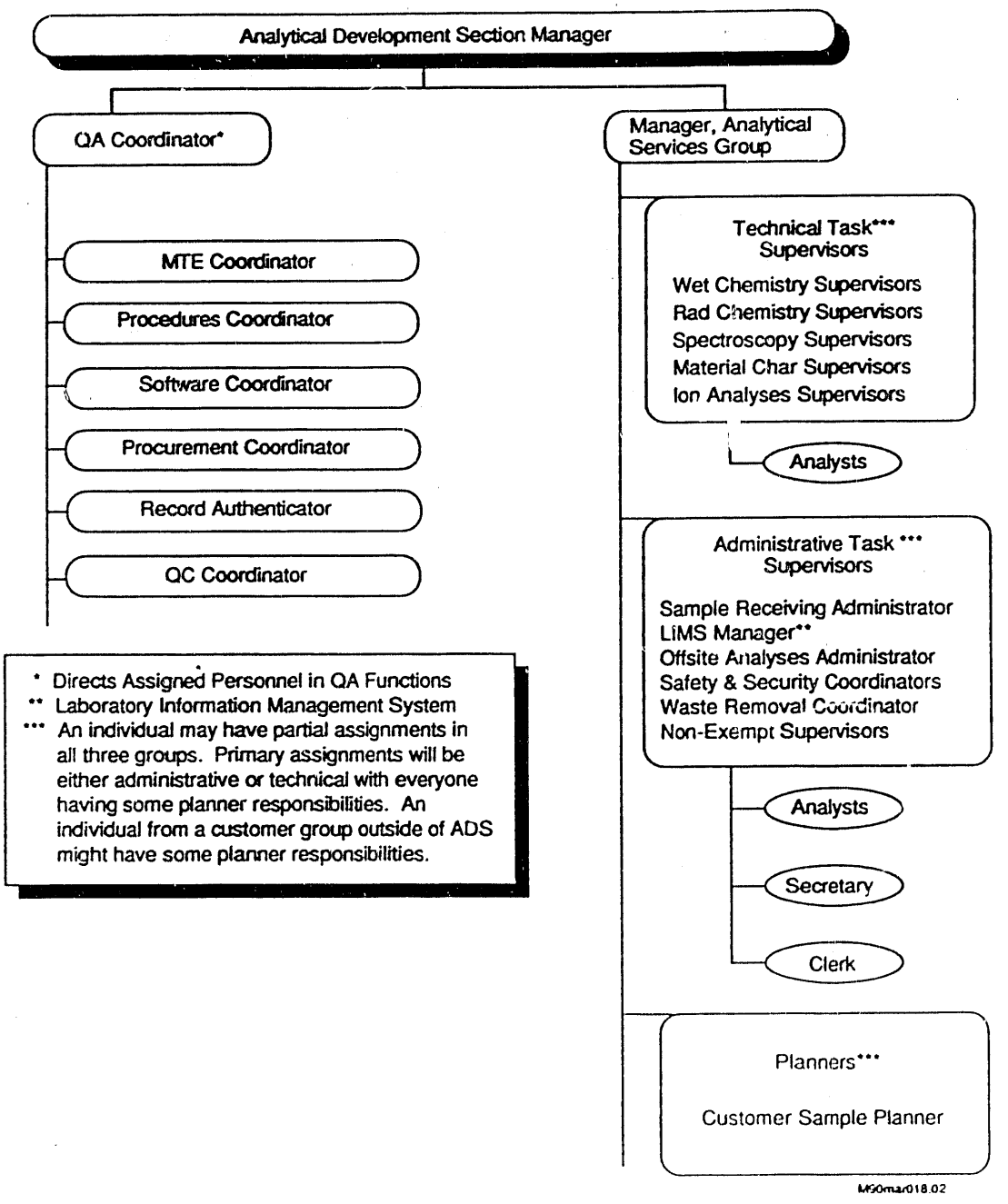
4.2 Assignment to Analytical Services Tasks

The ASG Manager assigns a Planner for customer support and designates cognizant professionals or R&D supervisors as Task Supervisors for analyses of customer samples.

The Task Supervisor is responsible for the analysis, recording, reporting and documentation of the results. He qualifies analysts and selects the method or instrument to be used for analyses according to the qualification and training requirements described in Section 2 and procedure requirements described in Section 5.

The Planner translates customer requirements into an analytical sample plan which is tracked and documented using the ADS Laboratory Information Management System (LIMS). Details on the operation and function of the LIMS are obtained from the LIMS Manager.

Figure 1
ADS Analytical Services Organization



1.1 STOP WORK

1.0 PURPOSE

The purpose of the stop work action is to prevent irreversible or continuing nonconforming conditions or actions.

2.0 SCOPE

This section applies to the Analytical Services Group (ASG) and to all individuals who interact with the ASG.

3.0 RESPONSIBILITIES

ASG personnel must perform inspections according to Section 10 and stop work as appropriate.

Customers have a responsibility to notify the ASG Task Supervisor of problems in samples and analyses as they become aware of them.

4.0 PROCEDURE

General

The ASG follows SRL QA manual items for stop work and accepts verbal and written STOP ORDERS as specified in Section 1.2 of the SRL QA Manual. In addition, any member of the ASG has the responsibility to terminate an analysis in the event of an equipment failure or other condition judged to be adverse to quality and report the condition to the Task Supervisor.

Closure of a STOP WORK Order

The Task Supervisor in charge of an analysis shall determine necessary corrective actions and conditions for restarting the process.

Corrections based on QC chart feedback shall be noted on the QC chart.

Feedback to customers on corrective action is provided by the LIMS sample record.

5.0 RECORDS

STOP WORK orders are lifetime records and details on handling these records are handled as described in Section 17.

2.1 QUALITY ASSURANCE PROGRAM

1.0 PURPOSE

This procedure describes a QA Program for analyses of samples by the Analytical Services Group (ASG).

QA levels for samples are described. The system for training analytical personnel is described. The requirements for procedures, quality control, management reports, and audits are given. Requirements for sample planning and documentation are provided.

2.0 SCOPE

This section applies to the ASG and to users of analytical services.

3.0 RESPONSIBILITIES

Customers - are responsible for defining the QA level for their samples defined in 4.1 below and for implementing QA for samples as defined below and stated in the SRLD Procedures Manual.

Analysts - are responsible for performing the QA duties assigned to them. Analysts are required to train and qualify for use of instruments and methods.

Task Supervisors - are required to establish procedures and Quality Control (QC) for instruments or methods under their direction and are responsible for the training and qualification of analysts for the method or instruments.

The QA Coordinator - is responsible for QA training of all ASG personnel and for communications with the Quality Assurance Division.

The QC Coordinator - is responsible for issuing the monthly QC report and for coordinating the QC program for instruments and methods. The QC Coordinator is also responsible for coordinating records, documents, and other QA assignments as described in this document or directed by the QA Coordinator.

Planners - are responsible for serving as interfaces between ADS and its customers by participating in developing sample analysis plans and assisting in selection of sampling strategies.

4.0 PROCEDURE

4.1 Quality Assurance Levels for Samples

The ASG provides three levels of Quality Assurance: Exploratory, Routine, and Customer-Assisted.

4.1.1 Exploratory

The Exploratory level is for R&D samples of an exploratory or qualitative nature. The Exploratory level requires no controls above those required for all SRLD research. Samples analyzed at this QA level rely on the judgement of the ADS personnel providing the analyses. The Exploratory Level sample is not logged on the LIMS and does not receive a formal QA program. The Exploratory Level is obtained by verbal agreement between the customer and the ASG Task Supervisor.

4.1.2 Routine

The Routine Level is for general R&D support and provides a solid base for customers to develop a more rigorous QA program. The Routine Level provides controls for ADS methods and instruments, but does not control sampling, shelf life, or the effect of a change in sample matrix from that expected or allowed for by the ASG method. Routine Level controls provide permanent records and tracking for samples. It provides a documented QC program for the samples which will verify and record that ADS instruments and methods are performing as provided and meet expected calibration, accuracy, and precision for known materials.

The Quality Control (QC) program provides QC charting of results obtained on standards and allows the customer to obtain accuracy and precision information for instruments and methods. The records include the analyses performed, the actual results or a pointer to them, the persons who analyzed and supervised the samples, and information about the sample provided by the submitter. The actual QC requirements for an analytical method are defined in the written procedures required to use the method.

The Routine Level uses procedures which are controlled by the ADS Records Office and tracked through the LIMS. The Routine Level is automatically provided for all samples logged into the LIMS system. It is not designed for samples resulting from exploratory work but can be used at the customer's discretion. It may not be appropriate for data that will be reported to a regulatory agency.

4.1.3 Customer Assisted

The Customer-Assisted Level is used for samples requiring specialized QA. The level will receive all the controls provided for Routine samples and additional controls specified by the Customer.

Customer controls are the responsibility of the Customer and should be traceable through the customer's QA Task Plans. ADS will assist the Customer with developing special controls upon request of the Customer.

The Customer should prepare a sampling plan and review controls as appropriate with QAD, ASG, and other affected organizations before beginning the sampling and analyses effort. The Customer can establish, and ASG may require, a rigorous chain of custody for the sample by placing a "follower card" on the sample which requires signing at each transfer, including those between the Customer and ASG, within ASG, and upon return to the Customer. The customer's "follower card" must contain the history of the sample.

The Customer is expected to analyze the data from his samples and from submitted standards to determine sampling errors, handling and storage errors, and overall accuracy and precision for his samples, and to identify matrix problems. He must provide feedback to ADS Task Supervisor if a method or analysis appears to be faulty.

Use of procedures specified by the Customer (e.g., EPA protocols) requires ADS approval.

4.2 Control of Samples

ASG Planners assign methods and/or instruments for the analysis of samples. The following information is recorded for samples: customer's name, the study name, cost code, sample description, hazards, radioactivity status, fissionable status, sample size, disposal requirements, and special analysis details.

4.3 Control of Procedures

ADS procedures for analytical methods, instruments, and facilities are prepared and controlled as described in Section 5. Procedures define for each ASG method or instrument, the training and qualification requirements, quality control, and MTE controls in addition to describing safety operations for methods and instruments.

4.4 Quality Control

Quality controls are placed on methods and instruments through the use of standards. The specifics are in written procedures in the ASG Record office. Figure 2 provides a general schematic of the ASG QC processes and indicates the minimum requirements for routine or customer assisted samples.

For analytical methods, a standard is analyzed as required in the method procedure.

For instrument control and calibration, standards are analyzed on a periodic basis as defined in the instrument procedure.

Analysts are required to provide the QC documentation, e.g. give control charts to the QC Coordinator after review and approval by the Task Supervisor.

4.5 QC Coordinator Reports

The quality control coordinator collects method and instrument quality control charts and data. The QC coordinator circulates a QC report of the analysis of these data to management as directed by the Research Supervisor or ADS QA coordinator. The QC coordinator sends a copy of those data that are designated records to permanent storage, as described in Section 17.

4.6 Training and Qualifications of Personnel

4.6.1 Quality Assurance

Training of ASG personnel on the QA Action Plan will be provided by ADS and documented by the ADS QA Coordinator.

4.6.2 Analyst

ADS Analytical Service Group analysts are Technician A's hired competitively based on job resumes and interviews. Analysts have passed a qualification exam for laboratory analysts and successfully complete a six week laboratory training program.

Analysts are qualified for assignment to an analytical method or instrument by the task supervisor according to the requirements defined in the procedure written for the instrument or method.

Analyst in training or substitute analysts may be used provided direct supervision is provided by the Task Supervisor or personnel qualified on the instrument. Analyst qualifications are recorded by the R&D supervisor and documented in the training folder for the individual. This record is maintained in the ADS Records Office. The Training Folder Format is shown in Figure 2 and may be revised by the Records Coordinator as needed as described in Section 17.

4.6.3 LIMS Users

Training for LIMS users is the responsibility of the LIMS manager.

4.6.4 QC Coordinator and QC Training

Training for quality control programs, such as those for balances, pipets, and QC charting, is the responsibility of the QC Coordinator. Training records are kept in the ADS Records office. A summary of QC training is provided with the monthly QC report.

5.0 RECORDS

Analyst training records method and instrument procedures, and QC charts are kept in the ADS Record Office according to Section 17 instructions. LIMS records are kept according to Section 17.

Figure 3

ADS QUALITY ASSURANCE TRAINING

EMPLOYEE NAME:_____ Revision 1
EMPLOYEE SOCIAL SECURITY #:_____ Effective Date: 5/9/89
QA COORDINATOR:_____

		DATE COMPLETED
I.	**General orientation SRL QA (1 time):**	_____
II.	**QA Training (1 time):**	
	Organization	_____
	QA program	_____
	Design Control	_____
	Procurement Doc. Control	_____
	Instructions Procedures & Drawings	_____
	Document Control	_____
	Purchased Items & Services	_____
	Identification & Control of Items	_____
	Inspection	_____
	Test Control	_____
	Control of Measuring and Test Equipment	_____
	Handling, Storage, and Shipping	_____
	Inspection, Test, and operating Status	_____
	Control of Nonconforming Items	_____
	Corrective Action	_____
	Record Management	_____
	Quality Improvement	_____
	Software Quality	_____

Figure 3

ADS QUALITY ASSURANCE TRAINING (Contd)

	DATE COMPLETED
III. **ADS-CPET implementation orientation** (Annual)	_____
IV. **ADS Analytical service group QAAP orientation** (Annual) **DPST-89-209**	_____

GENERAL REQUIRED TRAINING AND EXPERIENCES

1 TIME TRAINING:

8 week F-Area Training School	_____	_____	_____
6 Week SRL Training School	_____	_____	_____
SRL Safety Orientation	OSR A24-206		_____
Division Training	_____	_____	_____

OTHER EXPERIENCES:	YEAR PERIOD FROM	TO	TECHNICIAN'S SIGNATURE
_____	____	____	_____
_____	____	____	_____
_____	____	____	_____
_____	____	____	_____

ANNUAL TRAINING:	DOCUMENT NUMBER	TRAINER/TASK SUPERVISOR SIGNATURE
OHP Training	OSR388	_____
Nuclear Safety Review & Exam	_____	_____
SNM Accountability	_____	_____
WIPP Waste Handling	DPST-88-207	_____
Hazadous Waste Handling	_____	_____
Organics & Aqueous Liquid Disposal	DPSTP 2.10	_____

ANALYTICAL SERVICES
METHOD & INSTRUMENT QUALIFICATION RECORDS

WORK GROUP	PROCEDURE TITLE	PROCEDURE NUMBER	DATE QUALIFIED	TASK SUPERVISOR SIGNATURE
Facility/ Administrative	ADS WIPP Waste	ADS-0102	_____	_____
	Sample Receiving Facility (ADS)	ADS-0108	_____	_____
	Automated Pipet Calibration Analyst Pipet Training Certification	ADS-1000	_____	_____
	Balance Calibration Checks	ADS-1001	_____	_____
	Sample Load, Sample Received, Sample Backlog and Sample Completed Plots	ADS-2000	_____	_____
	_____	_____	_____	_____
	_____	_____	_____	_____

Special Courses	Date Completed
_____	
_____	
_____	

High Level Caves	Sodium Peroxide Fusion of Waste Glass, Glass Frit, And Sludges in Preparation for Anion Analysis by Ion Chromatography and Ion Selective Electrodes	ADS-2300(HLC)	_____	_____
	Sodium Peroxide Fusion Dissolution of Sludge, Glass & Melter Feed for Elemental Analysis	ADS-2502(HLC)	_____	_____

ANALYTICAL SERVICES
METHOD & INSTRUMENT QUALIFICATION RECORDS

WORK GROUP	PROCEDURE TITLE	PROCEDURE NUMBER	DATE QUALIFIED	TASK SUPERVISOR SIGNATURE
High Level Caves	Aqua Regia Dissolution of Sludge For Elemental Analysis	ADS-2226(HLC)	_____	_____
	Microwave-Acid Dissolution of Glass for Elemental Analysis	ADS-2227(HLC)	_____	_____
	Determination of Fe(II)/ Fe(III) Ratio in Glass	ADS-2207(HLC)	_____	_____
	Procedure for Measuring Total Solids, Insoluble Solids & Soluble Solids by Microwave Drying and by Calcining	ADS-2215(HLC)	_____	_____
	Determination of Tetraphenylborate in Precipitate Reactor Feed	ADS-2228(HLC)	_____	_____
	Determination of Total Hydroxide by Auto Titrator	ADS-2308(HLC)	_____	_____
	Acid/Base Determination By Auto Titration	ADS-2301(HLC)	_____	_____
	Yield Stress and Viscosity by Haake Viscometer Measurements	ADS-2229(HLC)	_____	_____
	PH Measurements of DWPF Samples	ADS-2309(HLC)	_____	_____
	_____	_____	_____	_____
	_____	_____	_____	_____

Special Courses	Date Completed
_____	
_____	
_____	

ANALYTICAL SERVICES
METHOD & INSTRUMENT QUALIFICATION RECORDS

WORK GROUP	PROCEDURE TITLE	PROCEDURE NUMBER	DATE QUALIFIED	TASK SUPERVISOR SIGNATURE
Ions	Sodium Peroxide Fusion of Waste Glass, Glass Frit, And Sludges in Preparation for Anion Analysis by Ion Chromatography and Ion Selective Electrodes	ADS-2300	_____	_____
	Acid/Base Determination by Auto Titration	ADS-2301	_____	_____
	Volumetric Determination of Hydroxide, Aluminate & Carbonate in Alkaline Solutions of Nuclear Waste (Adapted for Mettler Auto Titrator)	ADS-2302	_____	_____
	Operating Procedure for Ion 85, Ion Analyzer	ADS-2303	_____	_____
	Free Acid Potassium Fluoride Method	ADS-2304	_____	_____
	Determination of Hydrazine	ADS-2305	_____	_____
	Analysis of Solutions by Ion Chromatography	ADS-2306	_____	_____
	Sodium Bicarbonate Determination	ADS-2307	_____	_____
	Determination of Total Hydroxide by Auto Titrator	ADS-2308	_____	_____
	PH Measurements of DWPF Samples (DWPF DPST-88-281)	ADS-2309	_____	_____
	Permanganate Volumetric Method for Determining Reducing Normality	ADS-2310	_____	_____

ANALYTICAL SERVICE
METHOD & INSTRUMENT QUALIFICATION RECORDS

WORK GROUP	PROCEDURE TITLE	PROCEDURE NUMBER	DATE QUALIFIED	TASK SUPERVISOR SIGNATURE
Ions	Volumetric Determination of Hydroxide, Aluminate & Carbonate In Alkaline Solutions of Nuclear Waste (Adapted for Videotitrator)	ADS-2312	_____	_____
	_____	_____	_____	_____
	_____	_____	_____	_____

Special Courses	Date Completed
_____	
_____	
_____	

Mat Char	Contained Scanning Electron Microscope	ADS-1100	_____	_____
	Contained X-Ray Diffraction Analyzer	ADS-1101	_____	_____
	Microprobe	ADS-1102	_____	_____
	Operating Procedures for Auto Pore II 9220	ADS-1104	_____	_____
	Operating Procedures for the Digisorb 2600	ADS-1105	_____	_____
	Procedure for Transmission Electron Microscopy	ADS-1106	_____	_____
	Procedure for Scanning Electron Microscopy	ADS-1107	_____	_____
	Non-Radioactive X-Ray Diffraction Analyzers	ADS-1108	_____	_____

ANALYTICAL SERVICES
METHOD & INSTRUMENT QUALIFICATION RECORDS

WORK GROUP	PROCEDURE TITLE	PROCEDURE NUMBER	DATE QUALIFIED	TASK SUPERVISOR SIGNATURE
Mat Char	Particle Size Distribution by Sieve Analysis	ADS-2100	_____	_____
	Safe Operating Information Sheet Gatan Dual Ion Mill (C-112)	ADS-0100	_____	_____
	Safe Operating Information Sheet Technics Sputter Coater (C-112)	ADS-0103	_____	_____
	Safe Operating Information Sheet E.F. Fullam Carbon Coater (C-112)	ADS-0104	_____	_____
	Safe Operating Information Sheet Sonic Mill (C-112)	ADS-0105	_____	_____
	Safe Operating Information Sheet Philips Electron Microscope (C-104)	ADS-0106	_____	_____
	Safe Operating Information Sheet ISI Scanning Electron Microscope (C-108)	ADS-0107	_____	_____
	_____	_____	_____	_____
	_____	_____	_____	_____

Special Courses	Date Completed
_____	
_____	
_____	

Offsite	Preparation for Offsite Analyses	ADS-0101	_____	_____
	Analyses SRP	ADS-2701	_____	_____

ANALYTICAL SERVICES
METHOD & INSTRUMENT QUALIFICATION RECORDS

WORK GROUP	PROCEDURE TITLE	PROCEDURE NUMBER	DATE QUALIFIED	TASK SUPERVISOR SIGNATURE
Offsite	Analyses TNX	ADS-2702	_____	_____
	_____	_____	_____	_____
	_____	_____	_____	_____
	Special Courses		**Date Completed**	
	_____			
	_____			
	_____			
Organics	Procedure for Gas Chromatography	ADS-1600	_____	_____
	Qualitated Infra-Red Analysis	ADS-1601	_____	_____
	_____	_____	_____	_____
	_____	_____	_____	_____
	Special Courses		**Date Completed**	
	_____			
	_____			
	_____			
Rad Chem	Operation of the HCL ADS "Rabbit" Sample Transfer System	ADS-1400	_____	_____
	Master Slave Manipulator Operation and Replacement	ADS-1401	_____	_____

ANALYTICAL SERVICES
METHOD & INSTRUMENT QUALIFICATION RECORDS

WORK GROUP	PROCEDURE TITLE	PROCEDURE NUMBER	DATE QUALIFIED	TASK SUPERVISOR SIGNATURE
Rad Chem	Alpha and Beta Liquid Scintillation	ADS-2401	_____	_____
	Alpha Pulse Height Analyses	ADS-2402	_____	_____
	Strontium 90 Determination by Cerenkov Radiation Counting	ADS-2403	_____	_____
	235U Determination By TOPO Extraction and Gamma Scan	ADS-2404	_____	_____
	Alpha & Beta Plate Making Direct Mount and Count	ADS-2405	_____	_____
	Californium Neutron Activation Analysis	ADS-2407	_____	_____
	Tritium Extraction from Radioactive Solutions for Liquid Scintillation Analysis	ADS-2408	_____	_____
	Neptunium TTA Extraction and Alpha Analysis	ADS-2409	_____	_____
	Determination of 79 SE by Distillation and Beta Counting	ADS-2411	_____	_____
	Determination of 99-Technitium by Extraction and Beta Counting	ADS-2413	_____	_____
	Preparation of Selenium Carrier Solution	ADS-2415	_____	_____
	Electrodeposition of Actinides from Aqueous Solution	ADS-2416	_____	_____
	The Separation of Promethium	ADS-2418	_____	_____

ANALYTICAL SERVICES
METHOD & INSTRUMENT QUALIFICATION RECORDS

WORK GROUP	PROCEDURE TITLE	PROCEDURE NUMBER	DATE QUALIFIED	TASK SUPERVISOR SIGNATURE
Rad Chem	Strontium Precipitation Preparation for Liquid Scintillation Counting	ADS-2419		
	Gamma Sample Preparation and Analysis (Gamma PHA)	ADS-2420		
	Plutonium Analysis by TTA Extraction & Alpha PHA	ADS-2421		

Special Courses	Date Completed

Spectroscopy	X-Ray Fluorescence Scan	ADS-1109		
	ICPES-ARL	ADS-1500		
	ICPES-Jarrell Ash	ADS-1501		
	Atomic Absorption Spectrometry Varian AA475	ADS-1502		
	ICPMS	ADS-1503		
	Atomic Absorption Spectrometry SHIMADZU 670	ADS-1504		
	Dissolution of Glass in Hydrofluoric and Hydrochloric Acid in Teflon Lined Bomb	ADS-2500		
	Uranium Extraction	ADS-2501		

ANALYTICAL SERVICES
METHOD & INSTRUMENT QUALIFICATION RECORDS

WORK GROUP	PROCEDURE TITLE	PROCEDURE NUMBER	DATE QUALIFIED	TASK SUPERVISOR SIGNATURE
Spectroscopy	Sodium Peroxide Fusion Dissolution of Sludge, Glass, and Melter Feed for Elemental Analysis	ADS-2502	_____	_____
	Cold Vapor Atomic Absorption for Hg	ADS-2503	_____	_____
	Procedure for Frit Acceptance Elemental Analysis	ADS-2504	_____	_____
	Direct Elemental Analysis by ICP-AFS and AAS	ADS-2506	_____	_____
	_____	_____	_____	_____
	_____	_____	_____	_____

Special Courses	Date Completed
_____	
_____	
_____	

Wet Chem	Leco CS-044 Analyzer	ADS-2200	_____	_____
	OIC-700 Total Carbon Analyzer	ADS-2201	_____	_____
	Determination of Chloride Indirectly by Silver Analysis	ADS-2202	_____	_____
	Chemical Oxygen Demand Determination for Glass Batch Materials Micro Digestion Method	ADS-2203	_____	_____
	Conductivity of Solutions	ADS-2205	_____	_____

ANALYTICAL SERVICES
METHOD & INSTRUMENT QUALIFICATION RECORDS

WORK GROUP	PROCEDURE TITLE	PROCEDURE NUMBER	DATE QUALIFIED	TASK SUPERVISOR SIGNATURE
Wet Chem	Determination of Uranium by Davies-Gray Titration Automatic Titration Method	ADS-2206	_____	_____
	Determination of Fe(II)/Fe(III) Ratio In Glass	ADS-2207	_____	_____
	Flash Point Determination of Liquids or Liquids with Suspended Solids by Pensky-Martens Closed Cup Methods	ADS-2208	_____	_____
	Gravimetric	ADS-2209	_____	_____
	Water Determination With The Karl Fischer Reagent	ADS-2210	_____	_____
	Miscellaneous Dissolution	ADS-2211	_____	_____
	Moisture	ADS-2212	_____	_____
	Measurement of Oil and Grease Content in Aqueous Solution by OCMA-220 Oil Content Analyzer	ADS-2213	_____	_____
	Procedure for the Scintrex UA3 Uranium Analyzer	ADS-2214	_____	_____
	Procedure for Measuring Total Solids, Insoluble Solids and Soluble Solids by Microwave Drying and by Calcining	ADS-2215	_____	_____
	Colorimetric Determination of Ferrous and Total Iron in Glass	ADS-2216	_____	_____
	Colorimetric Method for Thorium	ADS-2217	_____	_____

ANALYTICAL SERVICES
METHOD & INSTRUMENT QUALIFICATION RECORDS

WORK GROUP	PROCEDURE TITLE	PROCEDURE NUMBER	DATE QUALIFIED	TASK SUPERVISOR SIGNATURE
Wet Chem	Determination of Tetraphenylboron by Mercury (II) EDTA Titration	ADS-2218	_____	_____
	Specific Gravity of Liquids	ADS-2219	_____	_____
	Preparation of Pseudonarcotics for Wackenhut	ADS-2220	_____	_____
	Determination of Tributyl Phosphate (TBP) in TBP-Ultrasene Solution Acid Saturation Method	ADS-2221	_____	_____
	Determination of Tetraphenylboron by Potentiometric Titration with Silver Nitrate	ADS-2222	_____	_____
	Organic/Aqueous	ADS-2223	_____	_____
	Determination of Sodium Tetraphenylborate (NATPB)	ADS-2224	_____	_____
	Determination of Tetraphenylborate in Precipitate Reactor Feed	ADS-2228	_____	_____
	_____	_____	_____	_____
	_____	_____	_____	_____

Special Courses	Date Completed
_____	
_____	
_____	

2.3 QUALITY ASSURANCE ACTION PLAN REVISIONS

1.0 PURPOSE, SCOPE AND PROCEDURE

Any revision of this Action Plan proposed by the ADS staff, its customers or QAD shall be performed by the procedures described in Section 5 of this QAAP.

3.0 DESIGN CONTROL

1.0 PURPOSE, SCOPE, RESPONSIBILITIES, PROCEDURE

Design of Analytical Service Facilities is performed by the LSD design group according to their procedures (DPSTOM-48).

4.0 PROCUREMENT DOCUMENTS

1.0 PURPOSE

This section defines the responsibilities and requirements for control of procurements.

2.0 SCOPE

This section applies to the Analytical Services Group (ASG) personnel and to R&D researchers procuring supplies and services for the ASG.

3.0 RESPONSIBILITIES

ASG Researchers and Analysts have primary responsibilities for planning and initiating procurements including the determination of appropriate QA level.

The ASG Manager is responsible for assigning the cost code for procurements, approving the procurement up to his level of authorization, and obtaining higher levels of authorization when needed.

Procurement of offsite analyses is the responsibility of the Task Supervisor in charge of offsite analyses.

4.0 PROCEDURE

ASG procurements will follow the CP&ET and DWP&FT QA Implementing Procedure which addresses Sections 4 & 7 of the SRL QA Manual.

5.0 INSTRUCTIONS, PROCEDURES AND DRAWINGS

1.0 PURPOSE

This section provides information required by the Analytical Services Group (ASG) to implement Section 5 of the Savannah River Quality Assurance Plan.

2.0 SCOPE

This section applies to procedures used by the ASG to analyze samples.

3.0 RESPONSIBILITIES

The Task Supervisor is responsibile for the selection and use of procedures for the analysis of samples.

4.0 PROCEDURE

4.1 Types of Procedures and Category

The ADS Support Group uses three types of QA Category 3 procedures for control of analyses of Routine samples. These are:

- Pre-approved Procedures from outside sources,
- ADS-Approved Method Procedures, and
- ADS-Approved Instrument Procedures.

4.2 Pre-approved Procedures

Some analytical procedures may be used by the Task Supervisor without seeking additional supervisory approvals. The Task Supervisor is responsible for generating additional information required to adapt the procedures to the ASG instrumentation, address ASG safety, and meet QA requirements. The ADS Procedure Coordinator verifies that requirements have been met and notifies the LIMS Manager of any new method or instrument approved for Routine sample use. Most pre-approved procedures will need attachments addressing training and qualification of personnel and a description of the quality control plan in ASG which assures data quality.

The following sources may be used for pre-approved procedures:

Other SR site laboratories, including:
- Health Protection
- Laboratories Department
- Naval Fuel Laboratories
- DWPF Laboratories
- TNX Laboratories

Other DOE Analytical Laboratories including those of:
- Los Alamos National Laboratory (LANL)
- Oak Ridge National Laboratory (ORNL)
- Y-12 and K-25 Laboratories of Oak Ridge
- Westinghouse Idaho Nuclear Chemical Operations (WINCO)
- Pacific Northwest Laboratories (PNL)
- Westinghouse Hanford Operations (WHO)
- Lawrence Livermore National Laboratories (LLNL)
- Argonne National Laboratory (ANL)
- Rocky Flats Plant (RFP)

Other Government Laboratories including those of:
- Environmental Protection Agency (EPA) including RCRA, Ground Water, CLP (CERCLA) procedures
- Food and Drug Administration (FDA)
- Department of Defense (DOD) including Naval Laboratories procedures
- South Carolina Department of Health and Environmental Control (SCDHEC)
- South Carolina Water Resources Board
- National Technical Standards Institute (NTSI) or (NBS)

Procedures approved by professional societies, including:
- American Society for Testing and Materials (ASTM)
- American National Standards Institute (ANSI)
- American Water Works Association (AWWA) including Standard Methods
- American Public Health Association (APHA)
- Water Pollution Control Federation (WPCF)
- Association of Official Analytical Chemists (AOAC)

Analytical Procedures approved for use by a laboratory under contract to the ADS Support Group.

eg. ECS, Galbraith Laboratories, General Engineering, Conoco

Other procedures which are routinely used by recognized laboratories may be treated as pre-approved procedures if requested by the task supervisor and agreed to by the ADS QA Coordinator and the ASG Manager. A written note to this effect shall be provided to the ADS Procedures Coordinator and placed on file in the ADS Records Office.

4.3 Facility, Method and Instrument Procedures

The preparation, review, approval, issuance and control, and revisions of procedures will be performed using the CP&ET and DWP&FT QA Implementing Procedure which addresses Section 5 of the SRL QA Manual using the format described in DPSTM-87-700-6.

The formats in DPSTM-87-700-6 address training, calibration, and procedural controls as appropriate for the different type of procedures. The following gives the general outline for these procedure formats.

- General Administrative Procedures (Follows the CPET QA Procedures Implementing Procedures Format):
  Purpose
  Scope
  Responsibilities
  Procedure
  Records

- Facility Procedures:
  Purpose/Title
  Scope
  Safety Precautions
  Procedure
  References/Records

- Equipment/Instrument Procedures:
  Purpose/Title
  Manufacturer
  Description
  Safety Precautions
  Training Requirements
  Calibration Frequency
  Procedures (Calibration/Operation)
  Preventive Maintenance
  Reference/Records

- Method Procedures:
  Purpose/Title
  General Limitations
  Quality of Data/Data Validation (QC Program)
  Training Requirements
  Description of Method
  Reagents
  Safety Precautions
  Calibration Frequency
  Calibration and Standardization Procedure
  Analysis Procedure
  Calculations
  References/Records

5.0 RECORDS

Analytical procedures are permanent records as defined in Section 6 on Document Control. Copies of analytical procedures are available from the ADS Records office. LIMS links the procedure numbers used to the actual sample which was analyzed. The LIMS data base is kept as a permanent record according to Section 17.

6.0 DOCUMENT CONTROL

1.0 PURPOSE

This section provides a procedure for the preparation, approval, revision, and protection of documents.

2.0 SCOPE

This section applies to all documents used in support of sample analyses.

3.0 RESPONSIBILITIES

The ASG Manager is the authenticator and derivative classifier for analytical support services documents.

The ADS Secretary, Clerk, and Procedures Coordinator have responsibility for Document Control. The ADS Procedures Coordinator is responsible for the ADS Records Office.

Document originators shall use the Document Approval form, OSR 14-357.

Task Supervisors and cognizant professionals are responsible for notebooks and instrument manuals.

4.0 PROCEDURES

Control of Documents created by the ADS Analytical Services Group follows the SRL QA Manual Section 6 as implemented by the Site Services Department, Information Services Division (SSD/ISD). Guidance for use of the document control system is found in the Clerical Manual, DPSPM-GEN-15, Section 3 "Document Handling", and in the SRL Procedure Manual, Section 4 "Handling Information". The Savannah River Plant Security Manual controls handling of classified documents.

Approved ASG analytical procedures are designated Controlled Identification and issued as working copies under the direction of the ADS Procedures Coordinator. The ASG Analytical Procedures Manual is Controlled Distribution.

Sample, instrument, and method logs are Controlled Distribution and are obtained through ISD as registered notebooks. Research notebooks and maintenance logs are Controlled Distribution obtained from ISD.

Paper copies of sample results are designated uncontrolled information, unless classified, in which case the results are handled as instructed by the division security officer, the research supervisor, the LIMS manager, or higher management.

Unless stated, other information documents are designated as uncontrolled information for use onsite. Release of information from the site follows the procedure in the SRL Procedures Manual.

OSR 14-357
(Rev 11-89)

WSRC
SAVANNAH RIVER SITE
DOCUMENT APPROVAL SHEET

Document Number
UC or C Number

1. DESCRIPTION OF DOCUMENT

TITLE _____

AUTHOR(S) _____ BUILDING NO. _____ PHONE NO. _____

TYPE: ☐ INTERNAL DOCUMENT (Within SRS) ☐ EXTERNAL DOCUMENT (To OSTI)
☐ SOFTWARE PACKAGE (To NESC)
☐ Technical Report
☐ Abstract or Paper
☐ Other _____

Additional Information for External Papers/Abstracts

PAPER FOR: Presentation Only _____ Publication Only _____ Both _____

MEETING NAME _____

CITY _____ DATES _____

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APPROVED BY MANAGER _____ DATE _____

3. CLASSIFICATION & PATENT INFORMATION (to be completed by Patent & Classification Reviewer)

CLASSIFICATION (circle one for each)		CLASSIFICATION GUIDE TOPICS	PATENT CONSIDERATIONS
Overall	S C UCNI U	_____	Possible Novel Features _____
Abstract	S C UCNI U	_____	_____
Title	S C UCNI U	_____	Closest Prior Art _____
Cover Letter	S C UCNI U	_____	_____

APPROVED BY DOE PATENT & CLASSIFICATION OFFICER _____ DATE _____

4. PUBLICATIONS PROCESSING

DATE RECEIVED _____ PUBLICATIONS MANAGER _____

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DOE-SR RELEASE DATES: Patent Branch _____ Tech. Info. Office _____

DATE COMPLETED _____ DATE SUBMITTED TO RECORDS/OSTI/NESC _____

7.0 GRADED PROCUREMENT SYSTEM

1.0 PURPOSE

This section describes the Graded Procurement System for procurements supporting analysis of samples by the ADS Analytical Services Group (ASG).

2.0 SCOPE

This section applies to procurements by the ASG.

3.0 RESPONSIBILITIES

The ADS QA Coordinator has responsibility for training and guidance on the SRL Graded Procurement System.

The ASG manager or other supervision authorizing the procurement request have a responsibility to verify that appropriate QA procurement levels have been assigned. (Special training for authorization authority may be required by QAD or Procurement groups.)

QAD has responsibility for verifying and approving procurement level 1 decisions.

4.0 PROCEDURE

ASG personnel will use the graded procurement system as described in the SRL Procedures Manual, Section 3.1 and the CP&ET/DWP&FT QA Implementing Procedure which addresses Sections 4 and 7 of the SRL QA Manual.

Contracts for offsite certified analyses are designated level 1 procurements except as noted below and require use of a laboratory for which a supplier evaluation form has been filed. A copy of the ASG procurement specifications (available from the ADS QA office) for offsite certified analyses is attached to the purchase requisition. The deliverable items or services and the technical requirements (sections 1.1 and 2.0 of the ADS procurement specifications) are defined to reflect the purchase requests. The ADS offsite analyses Task Supervisor maintains a list of previous qualified suppliers and initiates requests for qualifying new suppliers of laboratory analyses.

The SRS Analytical Laboratories may assume responsibility for control of these analyses and the procurement specifications for offsite analyses. In this case procurement level will be the one determined by Analytical Laboratories.

5.0 RECORDS

Offsite certified analyses are permanent records and are maintained according to the SRL document control system.

Purchase orders are records and are maintained according to M&L requirements. Purchase orders are also maintained as computer records according to site requirements.

8.0 IDENTIFICATION AND CONTROL OF ITEMS

1.0 PURPOSE

This section describes control of items used in the analysis of samples by the ADS Analytical Services Group (ASG).

2.0 SCOPE

This section applies to personnel in ASG and users of ASG services.

3.0 RESPONSIBILITIES

The QA Coordinator, the Division Nuclear Material coordinator, and the Division Property coordinator have responsibility for training on control and identification of items.

The receivers of procured items and samples are responsibile to inspect those items and initiate corrective actions if needed.

Customers of ASG have a responsibility to prepare, package, and label samples according to the SRL Procedures Manual on Analytical Services, Section 2.12. They have a responsibility to initiate chain of custody requirements for samples and implement the necessary controls beyond those provided by the ASG. ASG will provide customers a listing which indicates personnel handling a sample in ASG.

HP, SRL Property Management, SRP Property Control, and Receiving groups are responsible for directing the control and identification of materials by the ASG including radioactive materials, nuclear materials, received materials, chemicals, capital equipment, and cost items of a sensitive or controlled nature.

The ADS LIMS manager has responsibility for the identification system for samples in the LIMS. ADS customers and loggers of samples have a responsibility to assure that LIMS identification numbers are labeled on the samples.

4.0 PROCEDURES

ASG shall follow the CPET/DWP&FT QA Implementing Procedures for control of items which states that SRL Property Management Group controls shall be followed and that other existing controls for SNM, Criticality, HP, etc. shall be used as much as possible.

ASG has established special controls for control of samples. These are defined in SRL Procedures Manual Section 2.12. These and a few additional controls are stated below.

Samples submitted for analyses shall be uniquely identified as described in the SRL Procedures Manual section 2.12. Samples shall have either a white (non-hazardous label), yellow (thorium or depleted uranium), or red (hazardous, transuranic, or radioactive) label as defined in the SRL procedures manual. Routine samples shall be tagged with a unique identifying number obtained from the ASG LIMS. Labels and written messages must be compatible with the sample containers.

Samples are delivered to ASG sample receiving for distribution within ASG, or taken directly to the analysis location. Routine samples shall be acknowledged in the LIMS as to who initially received the sample.

Samples which require a chain of custody for movements within ADS shall be tagged with a sample follower card listing those handling the sample and logged in the LIMS as Routine or Customer Assisted samples. Chain of custody initiation is the responsibility of the customer. ASG will provide the customer a listing of personnel involved with a sample in ASG via a LIMS report. OSR 28-20, Distribution Record, may be used as a follower card provided it is protected during circulation. A customer may design a smaller and more rugged card as needed.

Samples which have a limited lifetime are controlled by the customer. The customer shall use the "Customer-Assisted" QA level and shall negotiate controls with ASG personnel. ASG shall not attempt to control lifetime of samples except as negotiated.

Facility custodians are required to inspect their facilities and items including samples as required by the ADS safety program. More frequent inspection is performed under the direction of the facility custodian and the analysts using the work space or handling the controlled items. Method and Instrument procedures may require additional inspection requirements.

Standards used for methods and instruments are to be identified and controlled according to instructions provided by the method and instrument task supervisor or as implemented in the controlling procedures for the instrument or method. Routine samples are analyzed according to written procedures.

5.0 RECORDS

Follower Cards are records to be handled according to customer instructions. If OSR 28-20 is used as follower card it is a lifetime record and a copy is to be sent to Central Files.

9.0 CONTROL OF SPECIAL PROCESSES

A special section on control of special processes is not needed. Analyses performed by the Analytical Services Group (ASG) may be considered as special processes requiring special controls. As indicated in Section 5 on Instructions, Procedures and Drawings, the method and instrument procedure formats define both training and data validation controls for analytical procedures and provide adequate special control. Specialized controls for analytical samples as opposed to analytical procedures were previously defined in Section 2.

10.0 INSPECTIONS

1.0 PURPOSE

This section describes how inspections will be performed and implemented to support analyses of samples by the ADD Analytical Services Group (ASG).

2. 0 SCOPE

This section applies to all analyses and supporting equipment and facilities used by the ASG.

3.0 RESPONSIBLITIES

All personnel in the ASG perform self checking inspections of samples, equipment, standards, chemicals, and facilities under their control.

4.0 PROCEDURES

Self-Checking inspections concerning safety and health are to be performed for all ADS activities as required by groups such as HP and the various safety committees in addition to inspections for samples as described below.

Exploratory Samples

Self-Checking inspections are the only ones required for exploratory samples.

Routine Samples

Inspections for routine samples are de ined in the pertinent method or instrument procedure. The QC aspects of sampl. analyses are considered a form of routine inspection and verification of proper method or instrument performances.

Customer-Assisted Samples

Self Checking and method QC inspections are the only requirements for Customer Assisted Samples except as negotiated with the customer. The customer may set up with ASG approval, additional controls and inspections. For example, Customers may negotiate inspections for samples, storage life, timing, sample matrix, and other factors involving the sample.

Self-Checking - Training

Annual training is given by HP and ADS on many aspects of self checking and monitoring and is documented in the training records. Specific training is given for ionizing and non-ionizing radiation, heat stress, sound exposures, x-ray exposures, hazardous chemical training, nuclear criticality, and radiation monitoring. Training in safety and security are conducted via monthly meetings.

Method and Instrument task supervisors teach analysts, and other personnel associated with their equipment, the self-checking aspects of their analytical equipment.

5.0 RECORDS

The QC charts collected by the QC coordinator are records which are documented by the QC coordinator and stored using the SRL document control system.

11.0 TEST CONTROL

Test controls in addition to those previously defined are not required.

Analytical tests are controlled according to the generalized rules for samples defined in Section 2. Samples which receive the Routine QA level, by being logged into the LIMS, are specifically controlled according to written procedures as defined in Section 5. The procedures define QC, training, and analyses as judged appropriate by the procedure's author, and approved by a peer, the task supervisor, and Analytical Services Group (ASG) management.

Customers are expected to implement additional test controls as required for their programs. Customer requirements are established jointly by an ASG planner and a customer when the LIMS sample plans are prepared.

12.0 CONTROL OF MEASURING AND TEST EQUIPMENT

1.0 PURPOSE

This section defines the requirements and responsibilities for the the control of Measuring and Test Equipment (MTE) used by the Analytical Services Group (ASG).

2.0 SCOPE

This section applies to MTE used to provide data for the analyses of samples by the ASG.

3.0 RESPONSIBILITIES

Task Supervisors have responsibility for implementing the MTE controls for their assigned MTE. This includes the clustering of property IDP numbers into an MTE name. This includes establishing the procedure for calibration, determining maintenance, establishing records (unless transferred to the QC coordinator), determining calibration ranges and acceptable uncertainties, and determining the category as directed in this procedure.

Task Supervisors and the analysts using the MTE have responsibility for segregating, tagging, or otherwise limiting the use of MTE which is out of calibration until repairs can be made.

Task Supervisors have responsibility for selecting and establishing procedures governing the calibration of MTE including the selection and traceability of standards.

The Property Coordinator (MTE coordinator) has responsibility for maintaining a list, by category, of MTE in ADS.

The QC coordinator is responsible for coordinating controls on pipettes, balances, or other MTE which is not directly under the control of a task supervisor.

The facility custodian is responsible for MTE, other than above, in his facilities.

4.0 PROCEDURE

Exploratory Samples

MTE used for Exploratory samples is considered to be Category 2 MTE for these applications and is used as originally procured and set up.

Routine Samples

MTE are calibrated by the analytical method and/or by periodic calibration as allowed for in Item 4.1.8, Section 12, SRL QA manual and directed by the Task Supervisors.

Analytical instruments used for Routine samples are treated as MTE Category 1 unless otherwise specified and documented in the QA MTE file and approved by the QA and research supervisor. The specifics for calibration and control are defined according to the specialized analytical procedure for the instrument or method as defined in Section 5 on procedures. The QC coordinator is responsible for maintaining a list of the instruments and keeping copies of the analytical procedures.

In general, quantitative analytical measurements are calibrated with each batch of samples and confirmed with a check standard that is control-charted as described in their procedure. Specifics for a given instrument or analytical method are given in their written procedures. The control data or charts are collected by the QC coordinator from the Task Supervisors and sent to ASG management on a monthly basis for review. In some cases an instrument will be monitored through multiple control systems. For example, a counting room instrument will name an instrument control chart, could be tested as part of a method control chart, and could be further checked by customer control standards. This is illustrated in Figure 2 in Section 2.

In the event that recalibration of equipment indicates a problem with previous analytical results, the analytical task supervisor has the responsibility to determine the extent of the data which has been made questionable and has the responsibility of notifying customers as appropriate. To assist him, the LIMS date-stamps data entries and samples. The query of the LIMS data base provides a list of samples during the time periods in question. The analytical calibrations and QC checks are date recorded and kept according to the procedure for the instrument or method.

Periodic instrument calibrations are used for those items, such as balances and pipettes, which are necessary for the preparation of method control standards. Periodic calibrations are also used for instrumental analyses. The procedures for each instrument states the frequency of calibration.

The QC Coordinator coordinates balance and pipette calibration and training on the use of those instruments. All balances and pipettes are checked on a periodic basis at least once per year. Additional controls on these devices are specified in the method procedures. Balances receive a random check weighing once per month.

Special analytical instruments such as scanning electron microscopes or mass spectrometers have instrumental procedures describing the periodic controls, maintenance, and training required.

Standards used for calibrating instruments are dated and used as described in the procedures. The QC chart contains the value of the standard, its accuracy range, the precision limits for the method or instrument, the date it was used, the initials of the analyst and reviewer. These charts are permanent records. The information on the charts is the responsibility of the method or instrument task supervisor.

Maintenance logs, repair manuals, and periodic calibration records are kept locally with the instrument or in the office of the instrument task supervisor.

The procurement of standards and calibration services shall be coordinated with the Savannah River Standards Laboratory. Items calibrated offsite are Category 1 procurements as instructed in Item 4.8 Section 12 of the SRL QA Manual. Offsite standards, which are provided routinely as a commercial off-the-shelf item, may be purchased from NIST or standardizing groups as a level 2 procurement.

Storage of standards shall be the responsibility of the task supervisor. MTE equipment storage is the responsibility of the facility custodian in whose facility the equipment resides. Section 13 provides additional information on Storage, Handling, and Shipment of MTE and standards.

Training

Training on instruments and methods are established in the procedures for Routine samples or as described in Section 2.

5.0 MTE RECORDS

QC data is kept as permanent records by the QC coordinator. Record copies are maintained for one year in the ADS QA office and annually transferred to ISS for permanent storage.

13.0 HANDLING, STORAGE, AND SHIPPING

1.0 PURPOSE

This section describes handling, storage, and shipping requirements to prevent damage or loss, and to minimize deterioration of materials and equipment.

2.0 SCOPE

This section applies to materials and equipment used by the ADS Analytical Services Group (ASG).

3.0 RESPONSIBILITIES

ADS personnel are required to be familiar with the proper handling and storage of equipment, samples, and other items under their control as described in the SRL Procedures Manual. LSD, HP and the SRL, SRS Safety Manuals advise additional handling, storage, or shipping if required.

ADS personnel are to handle nuclear materials, RHYTHM items, waste items, security items, chemicals, and other sensitive items as decribed in the controlling manuals for these substances or as described by the oversight committees or organizational groups responsible for coordinating the handling of materials.

4.0 PROCEDURE

All ADS equipment is designated class A material unless designated otherwise by the property custodian. Shipments of samples are coordinated through the offsite analyses task supervisor.

The SRL Procedures Manual is the primary reference document for stating how to handle, store, and ship materials. The handling of samples is described in Section 2.12. The SRL Procedures Manual, Section 3, addresses the handling of items.

Shipment of samples, equipment, and other items or materials are covered by the RHYTHM system and DOT requirements and coordinated with the SRL Property Management Group. Form OSR 1-53 is used to approve all shipments and track the materials.

Nuclear Materials are handled according the SRL accountability manual, DPSTOM 48, and the SRL criticality control manual, DPST-68-108.

5.0 RECORDS

Shipments are recorded and followed by Property Management using form OSR 1-53. Routine QA level samples are reported and tracked for offplant shipment via the LIMS.

14.0 INSPECTION, TEST, AND OPERATING STATUS

1.0 PURPOSE

This section describes status checks and tagging system for the ADS Analytical Services Group (ASG).

2.0 SCOPE

This section applies to equipment, instruments, and facilities used by the ASG.

3.0 RESPONSIBILITIES

Task Supervisors and facility custodians have primary responsibility for control of their facilities and equipment and for establishing appropriate status indicators to assure proper and safe operation, and for the training of analysts and other users of the facility to use the indicators.

Analysts and other ADS support personnel have responsibility to maintain, observe, and take actions based on training in response to status indicators.

4.0 PROCEDURE

ADS Support Services shall use indicators for safety and health alarms as required to meet safety, security, or environmental requirements.

"Caution" and "Do Not Operate" tags are used as described in the safety manuals. Nonconformance tags shall be used as directed by QAD or the ADS QA Coordinator.

Use of indicators of facility status including hood flow alarms, fire alarms, air monitors, or other radiation detectors shall be coordinated with Lab and HP support personnel according to guidance by those groups and tested according to their recommendations.

Instrument maintenance logs and other indicators of equipment status are kept for each instrument and are the responsibility of the task supervisor.

5.0 RECORDS

Maintenance logs are records as described in Section 17.

15.0 CONTROL OF NONCONFORMING ITEMS

The ADS Analytical Services Group controls Nonconforming Items as described in Section 15 of the SRL QA Manual. A copy of OSR 28-12, the Nonconformance Report for Items, is shown in Figure 1.

SAVANNAH RIVER LABORATORY DIVISION
QAAP MANUAL
SECTION 15.0 CONTROL OF NONCONFORMING ITEMS

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Effective 6/9/89
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QA Use Only Trend Code	OSR 26-12 (Rev 1-90) Savannah River Site Nonconformance Report	Date of Report Location of Nonconformance	NCR Number/Rev Page 1 of _____
Issued By (Name/Signature)	Reporting Department	Phone / Date Found	Found By
Title			Number of QA Hold Tags

Specified Requirements

Description of Nonconformance

Reportable to DOE? Yes ☐ No ☐ Custodian _____ (Name/Signature) _____ (Date)

A | Validation Car No. _____ SCAQ? Yes ☐ No ☐ CQF _____ (Name/Signature) _____ (Date)

Disposition Rework ☐ Repair ☐ Use As Is ☐ Reject ☐ Activity NCR ☐

Conditional Release ☐ No ☐ Yes (See Attached)

Disposition Details

Disposition Written By _____ (Name/Signature) _____ (Department) _____ (Date)

Disposition Approval Signatures and Dates	Custodian	CQF	
B Add Other Signatures as Required	CTF (If Applicable)		

Probable Cause and Steps to Prevent Recurrence (As Practical)

C Custodian _____ (Name/Signature) _____ (Date)

Verification of Disposition and Closure Approval

D Implementation Complete _____ (Custodian/Signature) _____ (Date) CQF _____ (Name/Signature) _____ (Date)

16.0 SRL CORRECTIVE ACTION SYSTEM

The ADS Analytical Services Group follows the Corrective Action System as described in the CP&ET and DWP&FT Implementing Procedure for Section 16.

17.0 RECORD MANAGEMENT

1.0 PURPOSE

1.1 An administrative control system for records provides a means of ensuring the availability of evidence that attests to the quality of an item, activity, or the qualifications of an individual.

1.2 This section defines responsibilities and establishes requirements for identification, control, validation, protection, and disposition of documents designated as records.

2.0 SCOPE

This section applies to the ADS Analytical Services Group (ASG).

3.0 RESPONSIBLILITIES

LIMS Records are the responsibility of the ASG LIMS manager.

ADS Procedures are the responsibility of the ADS Procedures coordinator.

QC records are the responsibility of the ASG QC coordinator.

Method and Instrument Task Supervisors are responsible for the raw data, and supporting data files for the methods and instruments under their control.

The ASG Manager, as the records authenticator, is responsible for identifying records, classifying records for security, directing editing of records, and assuring that the Record Index Form (OSR 17-84, 3-88) is completed. The ADS Records Coordinator is responsible for revising the ASG training records format.

4.0 PROCEDURES

Records in ADS will be handled according to DOE Order DOE 1324.2A. Primary records for the ADS Analytical Services Group are the research notebooks, LIMS records, QC, and training records.

Documents and technical reports identified as records by the ASG Manager are prepared by the originator and routed for review and approval using OSR 14-357. Approval of procedure records is described in Section 5.

Training Records

The ASG training records are kept in the ASG Records Office. The general format for these records is shown in Section 2. The training record format may be revised at the discretion of the Records Coordinator to reflect new issues in training such as addition or deletion of supported analytical procedures.

LIMS Records

LIMS records are electronically backed up on tape or disk at a frequency determined by the ASG LIMS manager.

The LIMS manager is responsible for the security plan which covers the backup of the LIMS system and permanent storage and retirement of LIMS data. The LIMS manager is responsible for the documents describing the LIMS records, eg. the LIMS manual.

Because LIMS records are computer records and the technology is changing, a detailed description of current LIMS record storage is left to the LIMS manager. Microfiche, paper, and other optical storage techniques are also acceptable.

LIMS data are considered to be lifetime records and stored in accord with instructions from the Computer and Telecommunications Division (CTM).

ADS Records Office-Interim Storage

ADS training records, QC charts, and analytical procedures are kept in the ADS Records office by the ADS clerk. The ADS procedure coordinator, MTE coordinator, QC coordinator, and QA coordinator provide instructions on the handling of these records.

Method and Instrument Records & Notebooks

Method and Instrument logs, notebooks, and raw analytical data are kept according to instructions by the instrument or Task Supervisor in a form which most readily facilitates their retrieval and use. Many are stored electronically. Research Notebooks are kept by the Researcher at a location convenient to him. Retention of these records is according to the stated retention schedule.

Notebooks of professional personnel and instrument logs and maintenance records are considered permanent records. Technicians notebooks are non-permanent records. Research notebooks are controlled as documents according to Section 6.

Additional Record Controls

Additional control of records may be imposed by QA groups, SSD, or DOE. The ADS Analytical Services Group intends to meet record requirements and shall use the CP&ET and DWP&FT Implementing Procedures which addresses Section 17 of the SRL QA Manual.

5.0 RECORDS

The record retention schedule for ADS is a permanent record kept by SSD/ISD.

18.0 AUDITS

Audits are performed by SRL QAD according to the SRL QA Manual.

The monthly collection of QC reports by the QC coordinator and the distribution of these reports to Analytical Services Group (ASG) management serves as an internal surveillance of ASG activities.

19.0 QUALITY IMPROVEMENT

This section of the QAAP will need to be issued when the SRL QA Manual section 19 becomes available.

20.0 COMPUTER SOFTWARE QUALITY ASSURANCE

This section of the SRL QA Manual has not been issued. In the interim, the following controls will be used.

No control is required for analysis of exploratory samples.

No control is required for analysis of routine samples for which standards are used, as prescribed in the analytical procedure, to verify performance of the method. For analysis of other routine samples, the analytical procedure will describe the controls to be used.

The LIMS manager shall show, for each method, that information entered into the computer by the customer and by ADS personnel is retrievable. The LIMS manager shall document his validation test to the QC Coordinator.

Any testing of the LIMS software following changes in the system shall be at the direction of the ASG Manager if in his sole judgement, testing is required.

END

DATE FILMED

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