# GJBX-59 '76

UCID- 16911-76-2

# Lawrence Livermore Laboratory

HYDROGEOCHEMICAL AND STREAM-SEDIMENT SURVEY OF THE NATIONAL URANIUM RESOURCE EVALUATION (NURE) PROGRAM -- WESTERN UNITED STATES

Quarterly Progress Report April through June 1976





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NEWS

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#### ERDA ISSUES APRIL-JUNE-1976 WESTERN UNITED STATES

#### HYDROGEOCHEMICAL AND STREAM SEDIMENT SURVEY QUARTERLY REPORT

The Grand Junction (Colorado) Office, Energy Research and Development Administration (ERDA), has placed on open file a quarterly report entitled "Hydrogeochemical and Stream Sediment Survey of the National Uranium Resource Evaluation (NURE) Program - Western United States," dated April through June 1976.

The report, prepared by ERDA's Lawrence Livermore Laboratory (IJL), Livermore, California, for the Grand Junction Office, gives the status and summary for the quarter, and highlights pilot and geochemical studies, sampling, site selection, and sample analysis.

The LLL study is part of EEDA's National Uranium Resource Evaluation program to provide data which can be used to dermine systematically the distribution of uranium in surface and underground waters and stream sediments as possible indicators of areas favorable for the discovery of uranium. LLL is operated for EEDA's San Francisco Operations Office by the University of California.

The 71-page report, GJBX-59(76) [LLL No. UCID 16911-76-2], is on open file for public inspection at the following locations:

Grand Junction, Colorado		Technical Library, Grand Junction Office, Energy Research and Develop- ment Administration
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Anchorage, Alaska	May lost	Division of Geological & Geophysi- cal Surveys, 323 E. 4th Avenue
Atlanta, Georgia	80° - 188	U. S. Energy Research and Develop- ment Administration, Suite 438, 1365 Reachtree Street

Austin, Texas	<b></b> .	Bureau of Economic Geology, Geology Building, University of Texas
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Lewrence, Kansas	denne i v se	Kensel Coological Survey, 1930 Avenue "A", Campus West, The University of Kenses
Monlo Pack, California		U. S. Geological Survey, Library, 345 Middlefield Road
Cakland, California		U. S. Energy Research and Develop- ment Administration, Library, Wells Fargo Bldg., 1333 Broadway
Phoenix, Arizona		State of Arizona, Department of Mineral Resources, Mineral Bldg., Fairgrounds
Pittsburgh, Ponnsylvania		U. S. Energy Research : 21 Develop- ment Administration, Feleral Bldg., Ro. 1524, 1000 Liberty Avenue
Rono, Nevada		Nevada Eureau of Mines and Goology, Mackay School of Mines, University of Nevada
Reston, Virginia		U. S. Geological Survey Library, Gifts & Exchange Unit, National Center
Salt Lake City, Utah		Documents Division, Marriott Library, University of Utah

Socorro, New Mexico		New Marico Bureau of Mines, Campus Station
Spokane, Washington	·	U. S. Geological Survey Library, U. S. Court House, Rm. 678
Tucson, Arizona	<b>L.</b>	Geological Survey Branch, Arizona Europu of Mines, 845 Park Avecue
Washington, D. C.		U. S. Energy Research and Dovelop- ment Administration, Library, 20 Massachusetts Avenue, NW

This report is available on microfiche from the Grand Junction Office, ENDA, for \$3.00. Prepaid orders should be sent to: Eendix Field Engineering Corporation, Technical Library, P. O. Eox 1569, Grand Junction, Colorado 81501. Checks or money orders should be made out to <u>Bendix Field Engineering Corporation</u>, the operating contractor for <u>FRDA's Grand Junction Office</u>.

Printed copies of this report may be made at the requestor's expense from a number of commercial copy centers in Grand Junction, a list of which is available from the Bondix Technical Library, telephone 303/242-8621, Ext. 278.

After 60 days, the report will be available from the National Technical Information Service (NTIS), Springfield, Virginia 22161.

NOTE: Microfiche reports issued by MEDA's Grand Junction Office will cost \$3.00 each as of October 1, 1976, based on similar pricing by the National Technical Information Service; each black-and-white map or oversize chart on 35 km film will be an additional 50 cents; each color transparency of a color map or color chart will be an additional \$1.00.

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# HYDROCHEMICAL AND STREAM-SEDIMENT SURVEY OF THE NATIONAL URANIUM RESOURCE EVALUATION (NURE) PROGRAM — WESTERN UNITED STATES

Quarterly Progress Report April through June 1976

#### INTRODUCTION

This report summarizes activities and accomplishments (during the April through June 1976 quarter) of the Hydrogeochemical and Stream Sediment Survey being conducted by the Lawrence Livermore Laboratory (LLL) in support of the National Uranium Resource Evaluation (NURE) Program. The NURE Program is administered by the Grand Junction Office (GJO) of the U.S. Energy Research and Development Administration (ERDA). The LLL study is part of a nationwide systematic investigation by ERDA to determine uranium distribution in surface and underground waters and in stream sediments as a possible indication of areas favorable for the discovery of uranium. Included in the LLL portion of this study are the seven western states of Arizona, California, Idaho, Nevada, Oregon, Utah, and Washington. Similar reconnaissance surveys are being conducted in the rest of the continental United States, including Alaska, by Los Alamos Scientific Laboratory (LASL), Oak Ridge Gaseous Diffusion Plant (ORGDP), and Savannah River Laboratory (SRL). The primary contacts for the LLL portion of the NURE program are listed in Appendix A.

#### HIGHLIGHTS

- The Walker River Basin pilot study preliminary data report was completed on June 30, 1976.
- Smoke Creek Desert, Cave Valley, and the Roach Lake Basin were revisited to obtain water, sediment, and rock samples. These samples, along with samples from the Winnemucca Dry Lake Basin, are being analyzed for preliminary reporting on August 9, 1976.

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Reference to a company or product name in this report does not imply approval or recommendation of the product by the University of California or the U.S. Energy Research & Development Administration to the exclusion of others that may be suitable.

- Geochemical studies were initiated to: a) determine the best sieved size fraction to detect uranium anomalies, b) normalize seasonal variations in the uranium content of streams, and c) develop the methodology for using multielement analysis to define the geological and/or geochemical setting in which the sample was derived.
- Sampling sites were selected for the Humboldt River Basin and request for quotations (RFQs) for sampling were sent to potential contractors.
- Sampling site selection has been initiated for the region covered by the Lovelock Army Map Service (AMS) quadrangle.
- The clean-room facility to house rabbit preparation, water-sample analysis, and the emission spectrometer has been designed; construction should be completed by December 15, 1976.
- Installation of the high-throughput instrumental neutron-activation analysis (INAA) system should be completed in November 1976. This system will enable the multielement analysis of some 20,000 sample sites per year.
- A technical paper, "Geochemical Reconnaissance for Uranium in the Arid Regions of the Western United States," was presented at the Rocky Mountain Section Meeting of the Geological Society of America held in Albuquerque, New Mexico, May 20-21, 1976.

#### GEOLOGY AND SAMPLE ACQUISITION

#### Orientation Studies

During this quarter Smoke Creek Desert, Cave Valley, and the Roach Lake Basin were revisited to collect additional water, sediment, and rock samples. This work terminated the field phase of our orientation studies.

A number of geochemical studies have been started on the NURE data from the orientation studies. These studies, designed to optimize the sampling, analytical, and interpretive methods used in the hydrochemical program, include:

- <u>Transport of Uranium in Stream Sediments</u> A number of dry and wet sediments have been sieved into several size fractions to determine the best analytical sample to detect uranium anamolies. For each size fraction, the following information was obtained:
  - Uranium content
  - Uranium content of an acetic acid-hydrogen peroxide leach
  - Mineralogy of the heavy-mineral (p>3.2) fraction
  - Uranium content of the heavy-mineral fraction

Trace-element concentrations were also determined for selected samples.

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- Temporal Variation of Uranium in the Walker River System The purpose of this study is to identify geochemical techniques for normalizing seasonal variations in the uranium content of a stream. Preliminary results suggest that such seasonal variations may be normalized by the U/Cl ratio.
- <u>Geochemical Data from INAA of Sediments</u> This study is designed to demonstrate how elemental abundance data from activation analyses can be used to define the geological and/or geochemical setting in which a sample was derived. This knowledge is expected to provide a better indication of the uranium potential of an area than does an analysis of uranium alone.

#### Regional Reconnaissance Program — Humboldt River Basin

Selection of sampling sites in the Humboldt River Basin was completed, and RFQs for sampling were sent to 50 potential contractors. (Sampling specifications are presented in Appendix B.) Bid awards are expected to be completed by mid-July, with sampling to begin on or about August 1, 1976. Approximately 1900 sites will be sampled by contract personnel in the 17,000mi<sup>2</sup> drainage system. The Desert Research Institute has completed a literature survey of potential ground-water sites in the Humboldt River Basin and will sample approximately 300 ground-water sites not sampled by contract personnel.

Selection of sampling sites has begun for the center of the region covered by the Lovelock AMS quadrangle. The area to be completed in FY'76T includes the region between Winnemucca Dry Lake and the Humboldt River Basin. This will complete about 75% of the Lovelock AMS sheet. In addition, the remaining area in the Winnemucca AMS sheet not sampled in the Humboldt project is scheduled to be sampled in September 1976, completing about 95% of the Winnemucca AMS quadrangle. Sample acquisition plans for FY'76T and the first quarter of FY'77 are presented in Fig. 1. Additions to this sampling schedule in FY'77 will depend on availability of funds and contract costs.

#### SAMPLE ANALYSIS

Analysis of the Walker River sample set was completed in April, and the results were transferred to the data base. These measurements were made by instrumental neutron-activation analyses according to previously reported



Fig. 1. Map shows sample-acquisition plans for FY'76T and the first quarter of FY'77.

procedures.\* Data processing was facilitated by a new computer control program that can operate on a list of computational requests by linking three existing, but separate, processing codes. This program can perform systems calls, retrieve data and library information from memory, store intermediate and final results, generate hardcopy reports, and provide real-time status reports on its progress — all automatically. With this system, it was possible to complete data processing of all samples as a self-consistent data set and report the results in both man- and computer-readable form, according to the scheduled deadline. Much of this information was included in the preliminary Walker River Pilot Survey Data Report (issued July 1, 1976).

Processing samples from four dry-lake (playa) regions of Nevada began late in the quarter. These samples will be analyzed according to the procedure described above. No data are yet available.

Preparatory to the playa-sample analyses, it was necessary to define the particle size and sample depth below the playa surface that would be most useful for the pilot survey. A series of parameter studies were conducted, using uranium concentrations determined by delayed-neutron counting. Considerable effort went into preparing the counter and rabbit system to make these measurements; some of the problems and solutions are described in the following section. Results, however, indicated that subsurface, intermediate size fractions were most representative of the surrounding region and that severe problems could arise if airborne sediments were included. Accordingly, we have begun analyses of the 1000-to-500- $\mu$  size fraction of samples collected 5 and 10 ft below the surface.

Water samples from wells, springs, and a few streams will be concentrated and processed by INAA. In addition, we have determined  $Cl^-$ ,  $S0_4^-$ , and conductivity in these waters. Chloride was measured by titration with mercuric nitrate in ethanol to a diphenyl carbazone end-point determination. Sulphate was measured potentiometrically after cation cleanup by ion exchange using a  $Pb(Cl0_4)_2$  titration in methanol. Conductivity was determined from the measured resistance between standard 1-cm<sup>2</sup> electrodes at 25°C.

<sup>\*300-</sup>mg sediment samples packaged in a polyethelene "pillow" or 100-ml water sample residues collected on cellulose and pelletized with one 10,000-sec gamma ray analysis 10 days after irradiation.

Table 1 summarizes the types and numbers of samples processed during the quarter.

#### Developing Analytical Capability

Much progress has been made on the planned high-throughput neutronactivation analysis facility. Hardware design is complete, and several units have been ordered. Other hardware was in the bidding stage at quarter's end. Also by the end of the quarter, most of the Phase I<sup>\*</sup> electronics and the four Ge(Li) detector systems had been purchased and the Phase II computer system was nearly ready to release for bidding. Phase I is on schedule for operation this calendar year. Phase II is on schedule for operation in late FY'77.

Details of the proposed pneumatic-transfer system are shown schematically in Fig. 2. The network is built around an existing four-way switch, an incore facility, and a delayed-neutron counter to provide both sequence flexibility and nominal 20,000-sample-per-year throughput.

The simplest mode of complete high-throughput operation involves:

- Selecting a rabbit capsule from the dispenser
- Positively identifying the capsule by sensing its waist-ring identification code using reflected laser light
- Inserting the capsule into core for 3-min irradiation
- Returning it to the delayed-neutron counter for specific uranium measurement and sample-strength determination by gamma-ray monitor
- Transferring the capsule to one of four delay stations for a nominal 10-min delay prior to the first gamma ray count
- Transferring the capsule to a fixed-geometry counting position for a nominal 10-min gamma spectral determination
- Discharging the capsule to a storage hopper dropout for seven-day decay
- Re-inserting it into the pneumatic system via dispenser, identify, and send stations for transfer to detectors (close geometry) and 1-hr gamma spectral determination (night and weekend operation)
- Discharging the capsule to storage.

In addition, other system operating modes include:

<sup>\*</sup> Phase I is complete automation, except for online data reduction. Phase II adds minicomputer data-reduction and data-base-management capabilities.

Table 1. Samples processed during fourth quarter FY'76.

Number of samples

Instrumental neutron-activation analysis	
Walker Basin	
Dry sediment	60
Wet sediment	49
Concentrated river water	6
Concentrated snow melt	10
Host rock	3
Playa_basins	
Host rock	13
Standards	4
TOTAL	145
Delayed-neutron uranium analyses	
Walker Basin (host rock)	14
Playa Basin (host rock)	4
Playa sample fractions	125
Leached-dry sediment-solution residue	56
Standards & blanks	74
TOTAL	273
Delayed-neutron development analyses	
Special chemistry for C1, S0, and conductivity	60



Fig. 2. Block diagram of NURE rabbit system.

- Bypass of the delayed-neutron counter
- Delayed-neutron count only
- Multiple irradiation sequenced with any of the detection modes.

The control and data collection system is shown schematically in Fig. 3. Microprocessors are used both for system control and as multichannel analyzers. Data transfer will be initially through a borrowed minicomputer to cartridge disk for transport to the LLL computer center. When the Phase II computer comes on line, it will receive data directly from the microprocessors and will provide on-line data reduction and data-base management functions. A detailed description of the Phase II system is given in Appendix C.

Developing this system necessitated designing a "universal" rabbit capsule (Fig. 4). The internal volume of the rabbit is sufficient to contain commercially available 2-dram polyethelene vials, which have proven useful as secondary sample containers. The waist ring of each capsule is encoded with unique designators that can be read by the automated identification station. Other features include "stars" on each end of the capsule for rotation in the identifier and a "v" notch to facilitate precise alignment. The outside diameter of the capsule will fit into our existing 1.25-in-ID rabbit system. The rabbits will be injection molded, using a specially purchased highpurity linear polyethelene.

Readying the delayed-neutron facility proved more difficult than expected. The transport system had not been used for three years, and the counter had been disassembled. Most of the transport-reliability problems were solved by reworking the pneumatic system and designing the new rabbit. The cause(s) of these problems, however, are still not understood. Therefore, a new in-core facility is a contingency plan for the high-throughput system. In addition, the delayed-neutron counter itself is not well suited to its present use. We are considering replacing the twenty 2-in. by 3-ft  $\mathrm{BF}_3$  tubes with 1-in. by 1-ft tubes to reduce background and increase efficiency. This would result in one to two orders of magnitude reduction in detectable levels of uranium. Current operation involves a 1-min irradiation (with a 4-sec transit and delay time for consistent count sequencing with small transit-time variations) followed by a 6-sec count and a 1-min count. The 1-min count provides an internal consistency check on the measurements. Calibration using both known samples as well as uranium standards was necessary because of gamma-ray-induced events. We had to install a 2-in. lead "nose" piece around the rabbit home position

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Fig. 3. Block diagram of data collection and control system.



Fig. 4. New universal rabbit design can be used with existing pneumatictransport system. to eliminate a nonlinearity problem with heavier samples. Small samples give poor measurement precision because of the high background signal from the larger detector available to us. Currently, 80 mg is the minimum acceptable sample size.

Significant improvements have been made to the counter and the rabbit system. A photo sensor has been installed to indicate when the rabbit is in "home" position. Also, by installing remote switches, valves, and datarecording equipment, we have reduced the number of operating personnel required from three to one.

An optical emission spectrometer, with direct reading capability and inductively coupled argon plasma source, has been ordered. This instrument will complement the INAA system for water-sample analyses. The spectrometer will be equipped with an arc source to measure selected elements in sediments and to support the activation analyses and quality-control program. We expect to receive the instrument in November 1976 and to have it operating by the end of the year. (The purchase specification is presented in Appendix D.)

A clean-room facility has been designed to house rabbit preparation, the emission spectrometer, and water-sample processing. An existing activation-analysis preparation laboratory is to be used for this facility. The layout of the clean room is shown in Fig. 5. Modifications include a positivepressure filtered-air supply, an entry airlock, cleaning and sealing of all interior surfaces, exhaust ducts for instruments and mechancial equipment, temperature and humidity control, and a logging-receiving area. We anticipate class 10,000 (less than 10,000 particles/m<sup>3</sup>) clean-room performance.

# FY'76T Milestones

During the next quarter, we plan to:

- Analyze and prepare final reports on approximately 600 samples representing the four playa pilot study areas in Nevada
- Process Humboldt Basin samples (as received)
- Complete clean-room installation
- Begin installing HT-INAA system (as components become available in late August 1976)

<sup>&</sup>quot;Uranium analyses in water will be by delayed-neutron counting of residue from evaporation in polyethelene sample bags.



Fig. 5. Layout of clean-room facility that will house rabbit preparations, water-sample processing, and emission spectrometer.

• Order Phase II computer system.

#### Plans for FY'77

Present plans for FY'77 include:

- Continue processing Humboldt samples by standard methods until Phase I HT-INAA and emission spectrometer are operational
- Complete installation of Phase I HT-INAA system in first quarter of FY'77
- Receive and install emission spectrometer in first quarter of FY'77
- Tie system components to central data-transfer mechanism thereby completing analytical processing automation in second quarter of FY'77
- Let contract for sample preparation and special water chemistries during first quarter
- Process  $\sim$  10,000 sample sites through INAA;  $\sim$  2,000 sample sites through emission spectrometry
- Validate analysis system for 20,000-sample-site load expected in FY'78
- Install Phase II computer, and bring data reduction operations on line during fourth quarter FY'77 (provided funding becomes available).

## INFORMATION MANAGEMENT AND REPORTING

#### Site Data Base

Progress was made on the site data base. Data-collection forms were developed and programs were written for validating the data, entering it into the data base, and linking site data to the sample-analysis data base. The site data base uses the Master Control Data Base in the LLL Octopus computer system.

Several versions of the site-visit form have now been tested in the field. We have found that a free field-data format (as opposed to columnar format) is easier to use and adds no appreciable delay in keypunching and verifying the data. Also, the free field format permits changes to the form without rewriting the data base entry and validating programs. (The form presently used is shown in Appendix E.)

Location data for sites visited were initially recorded in townshiprange-section codes. A program has been written to add universal transverse mercator measurements to the site data base from a map digitizer or a card

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file. The site data base was used to prepare a cross index of site identifiers, LLL sample codes, dates samples were collected, and LLL reactor numbers.

Data from samples collected through April 1976 have been keypunched and are now being entered into the data base. This includes data from the Desert Research Institute.

#### Sample-Analysis Data Base

Progress on the sample-analysis data base included refining the sampleanalysis interface file, programming the preprocessor to validate the data, using the link from the site data base to enter the data, and producing tables for the Walker River Basin report. The Octopus Master Control is also used for this data base.

Refinements to the sample-analysis interface file included adding several new fields and storing all numbers in floating-point format in terms of parts per billion. Upon retrieval and reporting, these numbers are automatically converted to the appropriate units.

Several special reports for internal analysis were produced, including error analysis graphs for different sample types for each trace element detected by INAA.

#### Data-Base-Management System

Careful attention has been given to the flow of information from the time samples are collected in the field to the release of open-file reports. Based on experience with the samples collected and analyzed in FY'76, the informationflow process shown in Fig. 6 has been adopted.

In FY'76T, a stand-alone minicomputer system will be purchased for the LLL Uranium Hydrogeochemical Program. This system will support real-time applications, interactive users, and batch programs simultaneously. A typical configuration is shown in Fig. 7. Appendix C gives a detailed description and analysis of the hardware and software being developed for the new data-base system.

#### Reports

A preliminary data report was prepared on the Walker River Basin and sent to the printer on June 30, 1976. The report consisted of the six tables outlined in Table 2.

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Fig. 6. Block diagram of proposed NURE data-base-management system.



Fig. 7. Typical configuration of minicomputer system to be purchased for NURE Program.

Table 2. Tables presented in Walker River Basin preliminary data report.

Sample/site type	Locations	Chemical analysis
Water/rivers and streams	Table l.l (95 sites)	Table 1.2 (120 samples)
Water/springs and wells	Table 2.1 (63 sites)	Table 2.2 (63 samples)
Water/lakes and reservoirs	Table 3.1 (ll sites)	Table 3.2 (11 samples)
Wet sediments	Table 4.1 (102 sites)	Table 4.2 (122 samples)
Dry sediments	Table 5.1 (124 sites)	Table 5.2 (126 samples)
Rock sites	Table 6.1 (19 sites)	Table 6.2 (19 samples)

Multielement analysis based on INAA included up to 25 element concentrations and errors for water samples and up to 32 element concentrations and errors for sediment and rock samples. Uranium concentration was included on each page for easy comparison with other measurements. In total, the 104page Walker River Basin preliminary report involved sixteen different page formats.

To expedite open-file reporting of future pilot studies, we have divised a standardized reporting format. This format is shown in Appendix F.

## Plans for FY'76T

- The site data base will be used to produce location tables and, for water samples, field-measurement tables. In addition, data for the Humboldt River Basin Study will be entered for samples collected in FY'76T.
- The sample-analysis data base will be used to produce analytical tables for the four orientation study reports (Winnemucca Dry Lake, Smoke Creek, Roach Lake, and Cave Valley) due this quarter. New data fields for specific laboratory anion measurements will be added to the data base description.
- The archival data base will be implemented for the Humboldt study.
- A system will be developed to record and report sample location and status as samples are received and moved to various locations at LLL. An existing special-materials location and tracking system is being investigated for this purpose.
- In conjunction with the ERDA-Grand Junction Office, specifications will be developed for producing machine-readable reports for data entry into a national NURE data base.

# APPENDIX A

# PRIMARY CONTACTS FOR LLL PORTION

# OF NURE HYDROGEOCHEMICAL AND STREAM-SEDIMENT SURVEY

Name	Responsibility	Mail stop	Phone ext.
K. Street, Associate Director, Energy & Resource Planning	Principle laboratory contact for NURE Program	L-209	8301
J. Tinney, Radiochemistry staff	Program leader	L-540	7321
D. Leach, Earth Sciences staff	Geology and geochemistry	L-10	8510
R. Lake, Mechanical Engineering staff	Operations, contracts, and field support	L <b>-</b> 7	7156
C. Smith, Radiochemistry staff	Sample analysis	L-233	8754
B. Holder, General Chemistry staff	Information management and reporting	L-404	3730
D. Ries Computations staff	Data-base management	L-380	4061

Name of Primary Contact (L- ) Lawrence Livermore Laboratory P.O. Box 808 Livermore, Ca. 94550

Telephone: (415) 447-1100 + extension

#### APPENDIX B

# SPECIFICATION UHS-001

# for the

# COLLECTION OF SEDIMENT AND STREAM WATER SAMPLES in the

# BASIN AND RANGE PROVINCE

# University of California Lawrence Livermore Laboratory Earth Sciences Division

June 1976

# U.C.L.L.L. SPECIFICATION UHS-001

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June 1976

# UNIVERSITY OF CALIFORNIA LAWRENCE LIVERMORE LABORATORY

## SPECIFICATION UHS-001

#### 1. GENERAL

- 1.1 Scope: This specification describes the requirements for the collection of water and/or sediment samples from approximately 1800 specific locations within the Humboldt River Basin of the State of Nevada.
- 1.2 Intent: It is the intent of this specification that the Seller collect and deliver the required samples to the University's Field Coordinator in strict accordance with the requirements set forth below and at the unit prices specified in the purchase order of which this specification is a part.
- 1.3 <u>University Furnished Supplies</u>: The University will furnish the following materials and/or services:
  - 1.3.1 The University will supply one set of maps showing sample site locations. These maps will consist of (a) 7-1/2 minute quadrangles, (b) 15 minute quadrangles, (c) BLM maps to a scale of 2" to the mile, (d) AMS series topographical maps.
  - 1.3.2 The University will furnish the following Government owned equipment for use by each sampling team. Title to equipment, material and supplies shall remain in Government and all said items shall be returned to University on completion of work.

(1) (2) (3) (4) (5) (6) (7) (8) (9) (10) (11)	Digital pH meters Conductivity meters Water Filter Apparatus Squirt Bottles Thermometers Polyethylene bags, 6 x 12 Polyethylene bags, 12 x 18 Stapler (Monel) Alkalinity and CO <sub>2</sub> Kits Timer, pocket N <sub>2</sub> Pressure Bottles	(1 ea) (1 ea) (1 ea) (3 ea) (2 ea) (500 ea) (500 ea) (3 ea) (2 ea) (1 ea) (2 ea)
(11)	N <sub>2</sub> Pressure Bottles	(2 ea)
(12)	Filters	(100 ea)
(13)	Filter Boxes	(100 ea)
(14)	Sieves	(2 sets)
(15)	Aluminum Shovel	(2 ea)
(16)	Polyethylene Sample Bottles	
	250 ml	(100 ea)

- (17) Polyethylene Sample Bottles 500 ml (100 ea) (18) Spare Parts Kit (l ea) The University will furnish the following ex-1.3.3 pendable supplies: (1)Deionized Water (10 gals) (2)10% Nitric Wash (1 liter + 500 ml squirt) (3) Cardboard shipping boxes (4) Site Visit Forms (5) Pencils and marking pens (6) Tape (7) 3 x 5 cards pH Buffers (4.0, 7.0 and 10.0) (8)1.3.4 The University, through the Field Coordinator, will
- maintain, repair and/or replace, as required, the equipment listed in Paragraph 1.3.2 except for daily calibration and cleaning, which will be the Seller's responsibility. However, the Seller will be responsible for any damage to University furnished equipment, materials or supplies resulting from the fault or negligence of the Seller's employees or agents.
- 1.4 <u>Seller Requirements</u>: The Seller shall provide all required sampling and supervisory personnel, transportation to and between all sampling locations, and all equipment, materials and supplies other than herein specified to be furnished by the University.
  - 1.4.1 The Seller must have a full time geologist in charge of sampling teams, with the responsibility for certification of all samples collected by Seller's personnel.
  - 1.4.2 As provided in Article XXVI, Permits, Seller shall have full responsibility for gaining legal access to sample locations, except sample locations within geographical areas under control of U.S. Forest Service and the U.S. Bureau of Land Management. University will acquire permits for access by Seller's personnel to these Government owned lands. Seller shall hold University and Government harmless from any and all claims for loss or damage resulting from Seller's sample collection activities.
  - 1.4.3 The Seller shall maintain day to day contact with the University's Field Coordinator, who will restock equipment items and expendable supplies as required.

- 1.4.4 The Seller shall deliver all samples (including completed site visit forms and photographs) weekly to a designated location in Winnemucca or alternate city selected by University. University will be responsible for further transport and disposition of samples.
- 1.4.5 The Seller shall take extreme care to leave all sample locations free of trash or debris of any kind.
- 1.5 <u>Sequence of Operations</u>: The Seller may use any sequence he chooses to conduct sampling operations. A typical sequence, which LLL has found to be efficient, is presented below for information only and is not to be construed as a requirement of this specification. For this sequence, an experienced field person will spend approximately 40 minutes at a stream site and approximately ten minutes at a dry stream site. Some of the operations, e.g., the pH calibration, temperature and conductivity determinations, and filtration can be performed almost simultaneously and save time. Steps listed are necessarily abbreviated; for complete procedures refer to the paragraphs of this specification referenced in parentheses.
  - Take temperature of water with mercury bulb submerged in the water (estimated time - one minute), (Ref.: Para. 2.4).
  - (2) pH calibration and determination (estimated time five minutes). (Ref.: Para. 2.5)
    - a) check battery
    - b) compensate for temperature of water
    - c) calibrate with appropriate buffer
    - d) record pH after five minutes of equilibration
  - (3) Filtration (estimated time 10 minutes) (Ref.: Para. 2.6)
    - a) disassemble the unit
    - b) place filter onto support disk
    - c) replace lower flange
    - d) rinse funnel with water sample
    - e) fill funnel with water sample
    - f) apply pressure, not to exceed 40 psig
    - g) discard first 100 ml of filtrate
    - h) rinse the 250 ml container with approximately 100 ml of filtrate
    - i) collect the two water samples (250 ml and 500 ml)
    - j) turn regulator off, disconnect pressure hose
    - k) disassemble and clean filter unit
    - 1) save filter in plastic box provided

- (4) Conductivity (estimated time two minutes) (Ref.: Para. 2.7)
  - a) place cell in water
  - b) switch to appropriate scale and read
- (5) Carbon Dioxide (estimated time one minute) (Ref.: Para. 2.9)
  - a) collect a fresh sample
  - b) rinse the dipper and reaction vessel with the water sample
  - c) place five ml of sample solution and two drops Phenophthalein indicator in reaction vessel
  - d) titrate with standard base to end point
  - e) record CO<sub>2</sub> as mg/liter
- (6) Total alkalinity (estimated time three minutes) (Ref.: Para. 2.8)
  - a) collect a fresh sample
  - b) rinse the dipper and reaction vessel in solution
  - c) place five ml of sample solution and two drops
    - Phenophthalein indicator in reaction vessel
  - d) if solution turns pink, titrate with standard sulfuric acid solution
  - e) calculate Phenophthalein alkalinity and record as mg/liter
  - f) add two drops of bromocresol green-methyl red indicator
  - g) titrate with standard sulfuric acid solution
  - h) calculate total alkalinity and record as mg/liter total alkalinity
- - a) collect a composite sample from five locations, five yards apart
  - b) sieve using the No. 3 sieve (1/4" square openings)
  - c) bag and label
- (7b) Wet Sediment (estimated time five minutes) (Ref.: Para. 2.3.2)
  - a) collect a composite sample from five locations along the active stream channel
  - b) bag and label
  - (8) Complete site visit form

# 2. TECHNICAL SCOPE OF WORK

The Seller shall perform the following work in the locations listed and in strict accordance with the procedures and methodology described herein.

- 2.1 General Provisions
  - 2.1.1 Selection and Location of Sites: The sample site locations have been selected by LLL geologists and are marked on topographic maps. In areas where 7-1/2 and 15 minute topographic maps are not available, locations will be shown on the best available maps. The sample site locations are based on the best geological information available. Therefore, the Seller will be required to visit the sites shown, unless a site is inaccessable or sources of local contamination are present, in which case the Seller will record on the map the reason/s why the site was not sampled. The field personnel must then select an alternate site as close as possible to the original site. Alternate sites should be in the same drainage as the original site; if this is impossible then the next stream drainage with similar geology should be sampled. In any case, alternate sites selected shall not exceed 10% of the total number of designated sample sites.

All sampled sites and their respective numbers must be recorded accurately on the field maps. The accuracy of the location will vary depending on the topography. However, plotting accuracies shall be within 100 yards for 7-1/2 minute maps, 200 yards for 15 minute maps, and 300 yards for BLM maps that have scales of two inches to the mile. <u>All field</u> maps must be returned to LLL.

The Seller shall take a black and white Polaroid photograph of the exact sample collection site, record the site number on the back of the photograph, and staple it to the site visit form.

2.1.2 Sources of Potential Contamination: Sampling obviously contaminated sites must be avoided at all times. This requires some judgment and "common sense" on the part of the field personnel. A simple criterion is that each sample site must be as close to natural conditions as possible. We will be measuring uranium in the parts per billion range, therefore, seemingly insignificant sources of contamination could mask the natural background. Water samples must be collected upstream from cattle guards, roads, bridges, rubbish, drainage pipes, and any potential sources of contamination. Heavily fertilized lands shall be avoided, since phosphate fertilizer can contribute significant quantities of uranium to the system. Potential sources of contamination or departure from "natural" conditions shall be recorded on the field data sheet.

- 2.1.3 <u>Field Containers</u>: Polyethylene bags will be provided the Seller for sediment samples. For water sampling sites, the Seller will be supplied with sealed polyethylene bags containing 500 ml and 250 ml polyethylene bottles, a plastic box for storage of the filter, and an index card for recording the site number.
- 2.1.4 <u>Sample Labels</u>: The Seller will be supplied with self adhesive labels marked with sequential site numbers and sample type codes. These labels shall be placed on each sample. In addition, the Seller will mark on each container the time and date the sample was collected, using an indelible marking pen.
- 2.1.5 <u>Site Visit Forms</u>: The Seller will be supplied with site visit forms in duplicate (refer to paragraph 3.0 for copy of site visit form). Before leaving each sampling location, the Seller shall fill in the appropriate field data and enclose the carbon copy of the form with the sample. Keep the original of the form filed separately and deliver it to the University's Field Coordinator when required, i.e., weekly.
- 2.1.6 <u>Field Coordinators</u>: LLL personnel will operate in the same general area as the Seller's personnel and will exercise inspection rights as provided under Article XIII, Inspection, of the purchase order terms and conditions. The LLL Field Coordinators will have the responsibility to insure that Seller's operations are performed in compliance with this specification, with particular attention to water sampling.

Should problems occur in the field, such as instrument malfunction, the Seller should contact the LLL Field Coordinator. The Seller's field personnel <u>must meet with the LLL Field Coordinator at least once</u> a week and transfer the samples, site visit forms, photographs and completed maps to the Coordinator. In addition, LLL sampling teams will resample a number of the sites for quality control purposes.

2.2 <u>Stream Water Sampling</u>: The Seller has the responsibility to insure that each water sample is an uncontaminated and representative sample. Where sampling sites have been selected on streams with tributaries, it is important to sample sufficiently far down stream to insure that adequate mixing has occurred. For large slow moving (laminar flow) streams, incomplete mixing may persist for many miles downstream. Normally LLL has selected sites sufficiently far downstream for proper mixing. If the Seller's personnel find it necessary to select alternate sample sites on slow moving streams, sample at least one mile downstream from tributaries. For small, rapidly flowing streams with extreme turbulence, the sample sites shall be placed at least 50 yards down stream from tributaries. Always select the more rapidly flowing, most turbulent part of the stream for collecting the water sample. Always avoid areas where bank collapse material may be entering the stream. Recommendations for minimizing contamination are presented elsewhere in this specification.

- 2.2.1 Procedure
  - (1) Select a site that (a) minimizes contamination, and (b) one that is representative (i.e., proper mixing has occurred).
  - (2) Collect the sample with a clean polyethylene bottle that has been rinsed three times with the water which is to be sampled.
  - (3) Pour approximately one liter of the solution into the filtration funnel, swirl, and discard the solution.
  - (4) Fill filtration funnel and pressure filter sample (see instructions for filtration).
  - (5) Collect a 500 ml sample.
  - (6) Collect a 250 ml sample.
  - (7) Package the filter and the two water sample containers into a polyethylene bag. Label the bag with the site number, time, and date.
- 2.3 <u>Stream Sediment Sampling</u>: Previous studies suggest that the finer sized fraction (less than medium sand size) offers the best analytical sample to detect anomalous uranium concentrations. The seller shall collect a <u>representative</u> sediment sample in sufficient quantity to insure at least 25 grams of the <100 mesh fraction will be furnished to LLL.
  - 2.3.1 Dry Sediment
    - 2.3.1.1 Sample Collection Sites: Dry sediment shall be collected at locations identified on the maps provided by LLL. The field personnel must insure that the sediment is representative of the sediment being actively transported down a particular drainage path. Avoid sampling sites where there is extensive wind deposited material. Avoid sampling where there is considerable bank collapsed material. In general, the ideal sample sites are located at the mouth of drainage canyons near the apex of alluvial fans.

For larger dry streams which show evidence of being active for extended periods of time, sampling sites can be chosen that are not necessarily at the mouth of canyons or the apex of alluvial fans, but the samples must be taken from a recently active area. 2.3.1.2 Equipment:

(a) Aluminum shovel

(b) No. 3 sieve (1/4" square openings)

- 2.3.1.3 Sampling Procedure:
  - (1) With a clean aluminum shovel, collect a <u>composite</u> sample from five locations, five yards apart.
  - (2) Sieve this material with the No. 3 stainless steel sieve (first remove the large fragments by hand).
  - (3) Be sure that the 1/4" fraction weighs at least five pounds.
  - (4) Place the sieved sample in a polyethylene bag, sealed by twirling a wire-reinforced strip around the end. Place the appropriate site labels on the bag. Write the date and time the sample was collected on the bag. Then place the sealed bag in another polyethylene bag and staple the end closed.
- 2.3.2 Wet Sediments
  - 2.3.2.1 Sample Collection Sites: At active stream sites, a composite sample shall be collected from the stream bottom, close to the main current. Avoid sampling bank collapse material. Selectively sample the finer size material from the stream bottom. Sand and mud bars located in midstream, and fine grained deposits located on the inside of meander curves provide good sampling sites.
  - 2.3.2.2 Equipment:
    - (a) Aluminum shovel
    - (b) Polyethylene Scoop
  - 2.3.2.3 Sampling Procedure:
    - (1) A composite sample shall be collected from five sites spaced about five yards apart. For sandy or muddy bottom streams, use a clean polyethylene scoop to gather the sample. A clean aluminum shovel shall be used to sample a stream having a coarse grained bottom sediment.
    - (2) For slow moving streams containing algae, sample those areas with minimum algae.
    - (3) After sampling the sediment, remove any large rocks by hand, place the remaining sample directly into a polyethylene bag, drain off most of the excess water, avoiding loss of the finer sediment fraction.
- (4) Place the appropriate site label in this bag. Seal by twisting a wire-reinforced strip around the top of the bag. Record the site number, time, and other data on an index card and place the sealed bag and index card in a second polyethylene bag and tightly staple the end closed.
- 2.4 <u>Water Temperature</u>: The temperature of the water must be measured in situ and from a representative portion of the stream. The most important consideration is to insure that the thermometer is near equilibrium with the water at the time the measurement is made. The procedure listed below will permit temperature determination to be made to within <u>+</u> 0.1°C.
  - 2.4.1 Procedure:
    - (1) If the water is flowing, submerge the thermometer in an active part of the stream; wait five minutes. Keeping the mercury bulb in the water, read the thermometer to  $\pm$  0.1°C.
    - (2) If the water is stagnant, submerge the thermometer for five minutes, then gently stir the thermometer several seconds before measurement. Always keep the mercury bulb submerged in the water while reading the thermometer.
- 2.5 Determination of the pH for Streams and Lakes: The pH of a solution is a measure of the effective hydrogen-ion concentration or activity. Because the hydrogen-ion activity is determined by reactions with the atmosphere, sediment, hydrolysis, and oxidation of certain ionic species, it is necessary to take the pH measurement at the sample collection site. Most surface waters will have a pH between 5.5 and 8.5. We expect the field determination to have a reproducibility of ± 0.2 pH unit.

## 2.5.1 Equipment:

- (a) Portable pH meter with spare battery
- (b) Polymer body, combination electrode with holder
- (c) pH buffers, 4.0, 7.0 and 10.0
- (d) Timer
- 2.5.2 Procedure:
  - (1) Check battery.
  - (2) Temperature Compensation: The temperature differential between the water source and the buffer shall not be greater than 20°C. Should the temperature differential be greater than 20°C, bring the temperature of the buffer within this range by placing the <u>sealed</u> buffer bottle in the sample source (caution, prevent contamination). Set the temperature compensation knob to the temperature of the source.

- (3) <u>Calibration</u>: Remove the electrode from holder, rinse with deionized water and blot dry with a Kimwipe; place electrode in 7.0 buffer; turn meter on; set timer for five minutes. After five minutes calibrate meter to the 7.0 buffer. If you suspect the water to have a pH between 5.5 and 8.5 (which will be the case in the great majority of stream sites) you may now determine the pH.
- (4) Measurement: The measurement should be taken in situ. In the event that the water must be sampled at great distance from the meter, collect the sample in a tightly closed bottle and make the measurement as soon as possible. The electrode should be rinsed with deionized water and blotted dry with a Kimwipe before placing the electrode in the sample. Set the timer for five minutes and record the pH to the nearest 0.1 pH unit at exactly five minutes. Should the pH of the water fall outside the 5.5 to 8.5 range, you must recalibrate it with the nearest buffer.
- 2.5.3 Precautions:
  - (1) If possible, keep meter and electrode in the shade during calibration and measurement.
  - (2) Always have the meter in the OFF position when the electrode is out of the solution.
  - (3) Never allow the electrode tip to dry out. Always store the electrode in deionized water provided in the electrode holder. Should the electrode tip dry out, replace it with the spare electrode and return the "old" electrode to the LLL coordinator.
  - (4) Handle the electrode with great care as the tip is very fragile.
  - (5) Maintain "fresh" buffers. The buffers will be provided in sealed containers and color coded. The buffers should be replaced after 25 determinations or sooner if accidentally contaminated.
- 2.6 <u>Filtration</u>: The purpose of the filtration operation is to remove particulate matter greater than the pore diameter of the filter. This is done to insure a representative and consistent set of water samples for analyses.

The pressure filtration unit is a lightweight, rugged unit made of plastic that accepts a four inch membranetype filter.

- 2.6.1 Equipment:
  - (a) 4" filtration unit with spare disks and seals
  - (b) Nitrogen pressure vessel with regulator
  - (c) Filters, 0.40 micrometer pore size

## 2.6.2 Procedure

- (1) Disassemble the unit by removing the nylon machine screws holding the top and bottom flanges.
- (2) <u>Gently</u> remove the porous support disk from the recess in the funnel. <u>Do not</u> pry this out as it can easily be damaged by careless handling.
- (3) Blot the disk dry with a Kimwipe and then return it to the funnel.
- (4) With a minimum of handling, place a membrane filter on top of the support disk so that it covers the recessed area in the funnel.
- (5) Fasten the lower flange by carefully aligning the screw holes and check to see that the membrane filter is in correctly.
- (6) Rinse the funnel with a small amount of the water sample and then fill the reservoir with the sample.
- (7) Secure the top flange and connect the device to the pressure unit.
- (8) Apply pressure gradually, normally 25 psig will suffice to filter most water, <u>NEVER EXCEED 40 psig</u>. Discard the first 100 ml of filtrate. If the filtration slows to a trickle because of filter clogging, it is recommended that the pressure be released and a fresh filter used.
- (9) Rinse the 250 ml bottle with the first filtrate.
- (10) Collect the 250 ml sample, filled to capacity and tightly capped.
- (11) Fill the polyethylene bag inside the 500 ml bottle to the capacity of the bottle. Make sure that air pockets have not been trapped in the bag. Tightly screw the cap over the end of the bag.
- (12) Turn pressure off at regulator, disconnect pressure hose from filter.
- (13) Disassemble filter unit, gently remove membrane filter with teflon tweezers, and place in plastic container provided.
- (14) Wash filtration unit with weak nitric acid solution and rinse three times with deionized water.
- (15) Return to storage case.
- 2.6.3 Precautions:
  - (1) NEVER exceed 40 psig operating pressure. Although each unit has been pressure tested at 85 psig, your personal safety demands that this pressure should not exceed 40 psig.
  - (2) Always turn regulator off before disconnecting the pressure hose to the filtration unit.
  - (3) Improper cleaning, handling, etc., can induce contamination to the sample.
  - (4) If the water sample must be collected at great distance from the filtration unit, the water must be filtered as soon as possible.
  - (5) Replace the  $N_2$  pressure bottles when pressure drops to 500 psig.

- 2.7 <u>Electrical Conductivity</u>: The electrical conductivity of a water sample is a measure of the ability of the water to carry an electrical current. Therefore the measured conductivity is a function of temperature as well as the composition of the solution. It is important that the temperature recorded on the field data form is the same temperature at which the conductivity is measured. The unit of measure shall be micromhos/cm.
  - 2.7.1 Equipment:
    - (a) Conductivity, salinity, and temperature meter and cell with spare battery.
  - 2.7.2 Procedure:
    - With the switch in the OFF position, adjust the meter to 0 micromhos if necessary.
    - (2) Remove the cell from the deionized water and plug into the meter jack.
    - (3) Check the cell with the CELL TEST BUTTON; return to LLL coordinator if cell is defective.
    - (4) Place the cell into water source.
    - (5) Switch to RED LINE, and with the RED LINE control knob adjust the needle to red line on the water scale.
    - (6) Switch to TEMPERATURE, record temperature in °C when needle is steady (usually after several minutes).
    - (7) Switch to appropriate conductivity range (X1, X10, or X100).
    - (8) MULTIPLY scale meter reading by range to obtain conductivity as micromhos/cm at the temperature of measurement.
    - (9) SWITCH TO OFF, wash probe with deionized water, return probe to deionized water.
  - 2.7.3 Precautions:
    - (1) Always keep the cell stored in deionized water provided in the electrode holder, NEVER allow the cell to dry out. If the cell is broken, damaged, or allowed to dry, return it to the LLL coordinator for replacement.
    - (2) Return cell to LLL coordinator for maintenance at least once every two weeks.
- 2.8 Alkalinity as CaCO<sub>3</sub> Equivalent: The concentration of the hydroxide, carbonate, and bicarbonate ions in the sample solution is determined by titrating with a standard sulfuric acid solution to two specified pH's. A phenophthalein indicator is used for measuring the hydroxide and half of the carbonate alkalinity test at pH 8.2 while a bromocresol green-methyl red indicator is used for measuring total alkalinity.
  - 2.8.1 Equipment:
    - (a) Bausch and Lomb Alkalinity Kit

- 2.8.2 Procedure:
  - (1) Collect a fresh water sample and immdeiately start the determination. Should the water be sampled at a great distance from the kit, collect the water in a tightly sealed bottle and make the determination as soon as possible.
  - (2) Rinse the five ml dropper and reaction vessel with the sample solution.
  - (3) Fill the five ml dipper to overflowing with the sample.
  - (4) Transfer the five ml to the reaction vessel.
  - (5) Add two drops of Phenophthalein indicator to the reaction vessel and swirl to mix thoroughly.
  - (6) If pink color appears, add standard sulfuric acid solution a drop at a time, swirl solution, continue until solution is clear.
  - (7) Phenophthalein alkalinity is determined by multiplying drops of standard sulfuric acid solution times 20 to give mg/liter Phenophthalein alkalinity.
  - (8) After the Phenophthalein alkalinity determination or if the pink color did not appear in step 6, add two drops of bromocresol green-methyl red indicator to the reaction vessel.
  - (9) Add standard sulfuric acid solution a drop at a time with swirling until the solution turns bluegray. If the solution turns pink, you have added one drop to many of standard sulfuric acid solution.
  - (10) Add the total number of drops added in the Phenophthalein alkalinity titration to the number of drops added to the bromocresol green-methyl red indicator (subtract one drop if you overshot the end point).
  - (11) Multiply the sum total drops added (spep 10) times 20 to give total alkalinity as mg/liter CaCO<sub>3</sub> equivalent alkalinity.
- 2.8.3 Precautions:
  - (1) Review the instructions provided in each alkalinity kit.
  - (2) Keep the standard solution sealed when not in use.
- 2.9 <u>Carbon Dioxide</u>: The concentration of aqueous carbon dioxide in water is determined by titrating the sample with a standard sodium hydroxide solution to form sodium bicarbonate. A Phenophthalein indicator added to the test solution gives a characteristic pink color at the completion of the reaction. For water samples with a pH greater than approximately <u>8.3</u>, carbon dioxide cannot be determined.
  - 2.9.1 Equipment:
    - (1) Bausch and Lomb Carbon Dioxide Kit

- 2.9.2 Procedure
  - (1) Collect a fresh water sample and immediately start the determination. Should the water be sampled at great distance from the kit, collect the water in a tightly sealed bottle and make the determination as soon as possible.
  - (2) Rinse the five ml dropper and reaction vessel with the solution.
  - (3) Fill the five ml dipper to overflowing with sample.
  - (4) Transfer the five ml to the reaction vessel.
  - (5) Add two drops of Phenophthalein indicator to the reaction vessel and swirl to mix thoroughly.
  - (6) Add standard base reagent a drop at a time with swirling until the solution becomes a very light pink.
  - (7) Multiply the number of drops of standard base reagent by two to obtain carbon dioxide as mg/liter.
- 2.9.3 Precaution:
  - (1) Review the instructions provided in each  $CO_2$  kit.
  - (2) Keep the standard base reagent sealed when not in use.

#### APPENDIX C

#### DESCRIPTION OF THE ANALYSIS

#### AND DATA-BASE-MANAGEMENT COMPUTER SYSTEM FOR

## THE URANIUM HYDROGEOCHAMICAL AND STREAM-SEDIMENT SURVEY PROGRAM

P. Siemens, D. Ries, C. Smith, R. Gunnick, L. Maynard

## Introduction

The National Uranium Resources Evaluation (NURE) program is an ERDA sponsored, multidisciplinary investigation of the uranium minerals resource in the United States. Its goal is to publish an assessment of national uranium reserves in mid 1981.

Lawrence Livermore Laboratory is conducting a geochemical survey of the seven western-most states as part of the NURE program. The national survey is divided among LLL, Los Alamos Scientific Laboratories (LASL), Savannah River Laboratory (SRL), and Oak Ridge Gaseous Diffusion Plant, with about equal areas of the country being assigned to each laboratory. The objective of this survey is to identify regions of promising uranium presence. The operation consists of: (1) systematic sampling of water and sediments, (2) analysis of these samples for uranium and other elements useful in verifying anomalous uranium presence, and (3) interpretation of the results in support of the NURE report and the prospecting industry. Current projections indicate sampling site visitations will number 8,000 in FY'77; 20,000 each in FY'78, '79 and '80; and 12,000 in FY'81. A total of 100,000 samples will be collected, 80,000 of which will be sediment samples. The program must be completed during FY'81 to permit final evaluations and reporting in time for inclusion in the 1981 NURE report. Where possible, two types of samples will be collected at each site - water and sediment. Automation of the analyses and reporting systems is required to meet our programmatic commitments.

The LLL analytical facilities being assembled to meet the required processing capabilities include an automated instrumental neutron-activation analysis facility (INAA), delayed-neutron counting system (DNC), and directreading optical emission spectrometer with inductively coupled argon plasma source (ICP). Each will feed data to a central computer system for reduction to analytical report format. Typically, water samples will be analyzed by

-C1-

ICP, by DNC, and by special chemistries for selected anions. Sediments will be analyzed by DNC and INAA. Each system has a design capacity of about 26,000 samples per year. Supplemental data sources include field measurements, specific element analyses, and quality control analyses by other laboratories.

One of the more formidable problems associated with the NURE program is the management of the large quantities of data. It will be necessary to maintain at least two working data files to accommodate sampling information and field data that are related to the laboratory-generated analytical information. These files will be updated continuously and corrected until finalized. They will then be merged with the master (archival) data base. Throughout this process it will be necessary to provide status reports for both field and laboratory activities as well as a number of formatted data reports useful in data checking and assessment or in geochemical investigation. Final reports will be generated periodically from the master data files for submittal to the ERDA Grand Junction information system data base and for open filing at ERDA repository libraries. Until these final reports are generated, information relating uranium concentration to geographical location must be considered company confidential, and access to this information will be limited.

Here, we describe a computer system that will meet both the analytical and data-base-management requirements of the NURE program. This system appears to the most efficient means of fulfilling our programmatic requirements.

## INAA and DNC Instrumentation

Instrumental neutron-activation analysis is the principal analytical method for assaying samples collected for the NURE program. Two variations of this technique will be used, one involving the detection of delayed neutrons and the other the detection and analysis of gamma rays resulting from the decay of radioactive species induced in the sample by reactor irradiation.

Samples to be neutron activated are placed in polyethylene capsules called "rabbits." The rabbit becomes the transport vehicle for moving the samples in a microprocessor-controlled pneumatic transport system. Rabbits containing samples are loaded into a dispenser attached to the pheumatic system. From this point on, the system is a fully automated, hands-off system. A flow-chart description of the INAA process is shown in Fig. Cl.

By operating values and switches in proper sequence, the microprocessor causes the rabbit, which carries a machine-readable code for sample identification

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Fig. Cl. INAA process flow.

to enter the reactor for neutron irradiation for a predetermined time. It is then sent to a delayed-neutron counter (DNC) for a short period (i.e., one minute) and then to a delay or holding station to allow some time for the decay of the more intense shortlived isotopes.

After the delay period, the rabbit is moved to one of four microprocessorcontrolled lithium-drifted-germanium detectors to acquire a gamma-ray spectrum of the activated elements. The microprocessor acts as a multiple pulseheight-analyzer capable of acquiring four 4096-channel gamma spectra simultaneously.

After the gamma counting, the rabbit is ejected from the pneumatic system into a special container for further decay. Approximately seven days after initial irradiation, the sample is reinserted into the gamma-counting portion of the pneumatic system for analysis of the long-lived activated elements. This counting is done on nights and weekends when the reactor is not operating.

The analytical instrumentation system can process some 26,000 samples per year per shift (one sample every 3 min, 7 hr a day, 4 days a week, and 48 weeks a year). Each sample will produce one delayed-neutron count (which requires a trivial analysis) and two gamma spectra (approximately 25,000 bytes of raw data) for a total of 78,000 analyses per year.

## INAA Gamma-Spectrum Analysis

The two 4096-channel gamma spectra produced by INAA exhibit different spectroscopic features and provide distinctive sets of analytical information. The spectrum from the first count is typically quite simple and does not require a complex computer program to reduce and interpret the spectral contents. However, this is not true for the spectrum from the second count, which typically exhibits over 100 peaks representing 20 to 40 radionuclides. A proper analytical interpretation of such a spectrum requires much of the power and sophistication emobodied in the GAMANAL program, which runs on the CDC-7600 computers. In its present configuration, this program uses some 6000 FORTRAN statements, about 70,000 memory locations, and has typical execution times of 3 to 30 sec per spectrum.

Analyzing a NURE spectrum does not require all of the flexibility and power that is built into the GAMANAL program. Furthermore, memory requirements can be reduced by chaining and overlaying program segments. The two most critical requirements are execution speed and the memory size available for an executing program and its data arrays. In view of the projected sample throughput, it will be necessary to limit total execution time to 5 to 10 min per sample. This means machinecomputation speeds of 2 to 5% of the CDC-7600's speed would be adequate. (See System Hardware section.)

Memory-size requirements are largely determined by two 10,000-floatingpoint variable matrices, which must be resident during successive stages of the analysis. However, the two matrices are not required to be resident simultaneously.

Using the matrix requires that a copy be preserved before arithmetic operations are performed. Two options are available, one in which the copy is preserved on a mass-storage device with an attendant reduction in execution speed and the other in which sufficient usable core is available to contain both arrays. It is estimated that an additional 5000 to 8000 memory locations will be required by the executing programs at these calculational points.

It is essential that the entire GAMANAL-processing and data-reduction operation be accomplished concurrently with sample analyses. This is the only means available to ensure validity of the results and to maintain the sample-processing schedule.

#### NURE Data-Management Requirements

A data-base-management system (DBMS) will enable centralized control of NURE data. This feature will reduce the redundancy of stored data, lower the frequency of inconsistent stored data, and provide for shareable data. More important, perhaps, the DBMS will provide data independence. That is, application programs which access the data base will be immune to changes in the storage structure and access strategy. In contrast, with a data-dependent structure, knowledge of the data organization and access technique is built into the application logic.

Data management for the NURE program can be divided into three interrelated data-base subsystems, each of which can logically be further divided into subareas, depending on the information sources and purposes for which the data were collected.

The first major data-base subsystem is being organized around the site visits and will be used to ensure unique site identifiers, validate and store field measurements, store and protect information on site locations, record samples taken at each site, and monitor their progress through various

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processing and analytical systems at the laboratory. Data are entered via a terminal, punched cards, magnetic tape, and possibly serial-line interfaces to map digitizers, bar-code readers, etc. The data will be queried interactively and used to generate daily, weekly, and monthly operational status reports and industry open-file reports.

The second major data-base subsystem is being organized around the sample analysis data and consists primarily of trace-element compositions and mineral analyses. For each component, a concentration and standard error is stored. This data base will collate the data from the different analytical methods. In addition, it will compare different sample sizes, types, and treatments and will compile statistics on the analytical throughput. Data are mainly from the high-throughput INAA data-reduction programs and emission spectrometer (ICP) determinations. Additional data are entered from specific chemistry measurements via magnetic tape or punched cards. This data base is also interactively accessed for verification, modification, and update of the analysis data. Daily, weekly, and monthly status reports will be produced automatically, with selected items and formats as needed. These data will be used as input to the industry open-file report.

The third major data base will be the data-analysis and archival data base, which will contain final data from both the site and sample analysis data bases. These data will be used by statistical analysis and graphic display routines on a selected basis. Information in this data base will be used to generate a machine and man-readable open-file report for industry.

The three major data bases must be integrated to reduce data redundancy, allow for flexible querying and report generation, and provide predetermined access subschemes for the different users. Due to the sensitive nature of the data, controlled access is required to portions of the data base (especially those linking uranium concentration with geographical location). The high cost of generating and collecting the data indicates the need for back-up and recovery procedures for each data base. These factors, together with the varied and changing access and reporting requirements, necessitate the use of a database-management system.

#### DBMS Requirements

Because the data must be filed for industrial query, CODASYL standard data organizations are required The results obtained during the program will

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dictate future directions of the survey. Therefore, it must be possible to modify data definitions and organization without requiring redesign of the application programs. To meet this flexibility requirement, the following DBMS features are needed:

- Independent data-structure definitions user-access paths to the data must be decided at run time. The multiple sources of data and varied requirements on its use will continually require new definitions and extensions of the data base.
- The data must be accessible through COBOL- and FORTRAN-callable modules.
- Access to the data base must be restricted to protect sensitive geographical location information.
- The data in the multiple data bases must be interrelated. The field measurements and analytical measurements must often be interrelated.
- Multiple users and applications programs must be able to run concurrently without violating the integrity of the data.
- Update transactions must be recorded to allow for rollback and recovery. Procedures must be provided for automatically dumping and restoring the data base.
- Hierarchical data structures with variable-length fields and data are needed to compress and interrelate the data elements.
- Internal floating-point representation of the data is needed to avoid conversion costs (in terms of accuracy and time) for the analytical programs.
- Adherence to CODASYL data base standard is necessary in that the NURE program will generate extensive quantities of data which must be made available to private industry. The costly efforts involved in exchanging and translating data will, in the near future, necessitate adoption of standard data representations. The Data Base Task Group CODASYL standard currently appears to be a logical choice.

The following options are also highly desirable:

- Usage statistics both in terms of space and type of use, would be extremely beneficial in monitoring access to the data base and in deciding which portions of the data to archive.
- With multiple analyses of given samples, duplicate record keys would simplify the handling of replicates and quality control checks.

A description of the data base is given in Table Cl. Using this description,

a projection can be made of the amount of disk storage required. This projection assumes that the minimum on-line storage required will contain one year's collection of data and that a total of 80,000 sites will be sampled, with a maximum of 20,000 per year. It is further assumed that there will be one water sample for every four sediment samples. Estimated disk-storage requirements for the three major data bases are given in Table C2. It is highly desirable to have the entire data-base network on-line.

#### System Software

Of critical importance to the NURE program is the ease with which the necessary software systems can be generated and maintained. It would take an estimated two years to generate all of the system software required. Therefore, to meet programmatic commitments, we plan to purchase as much of the software capability as we can. The operating systems, languages, and related utilities must provide the programmer as much support as possible.

The operating system will be required to support three classes of operations simultaneously: real-time, batch, and time sharing. Real-time facilities are required for data acquisition and experiment control. The realtime monitor portion of the operating system must promptly recognize an external event or stimulus and start the appropriate software task at a specified priority. This task can then modify an external process, collect data, etc. For example, consider the following: Gamma-ray spectra are collected by a microcomputer and sent to the central NURE computer. This transmission from a serial communication line to a disk file is a high-priority task. Periodically, a lower-priority task checks for disk files needing processing (by checking for unique names of files). Finding such a file, the program processes the data through a matrix spectral-analysis and data-reduction program to identify radionuclides and determine the elemental concentration of the sample. The report generated is then spooled onto disk for printing later at a lower priority.

Important characteristics of the real-time monitor include:

- Assignable task priority.
- Fully protected task partitions (i.e., tasks cannot modify memory outside of their own partition).
- File-management facilities.

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Table C1. Data base descriptions.

Site data Base

Name	Frequency	Disk I/O
Site number control	Weekly	6/site
Site log-sheet entry	Daily	12/site
Map-digitizer input	Daily	6/site
Query and update	lO tímes daily (30-min sessions)	30/task
Status reports	Daily	20/site
Site-analysis data base		
INAA data input	Twice daily	18/sample
Other data input	Once daily	12/sample
Query and update	lO times daily (30-min sessions)	50/task
Status reports	Daily	40/sample
Data-analysis or archival data	base	
Data from site and sample data bases	Weekly	90/sample
Query and selective reporting	Twice daily (l-hr sessions)	100/task
Graphical map generation	Every other week	20/sample
Selected graphical plot	Weekly	10/sample
Man-readable open-file report	Monthly	20/sample
Machine-readable open-file report	Monthly	20/sample
Statistical analyses	Weekly	20/sample

Table C2. Estimated disk-storage requirements.

Data base	Record size	Minimum storage (bytes)	Total (bytes)
Site data base	256 bytes/site	5,120,000	20,480,000
Sample analysis	1024 bytes/sample	25,600,000	102,400,000
Data analysis	512 bytes/sample	12,800,000	51,200,000
	Total	43,520,000	174,080,000

The number of disk accesses can also be estimated from the information in Table Cl. This estimate is summarized in Table C3.

Table C3. Estimated disk accesses to data base per day.

Data base		Average	Maximum
Site data base		3,000	9,000
Sample data base		6,300	18,000
Data analysis		20,000	60,000
	TOTAL	29,300	87,800

 Dynamic, on-line task builder (i.e., tasks must be able to be generated, installed, and run at any time; not just at system-generation time).

The operating system must support a single batch stream to aid the programdevelopment cycle and regular production analyses. The priority of the batch stream should be assignable. Normally, the batch stream will run at a low priority (lower than interactive jobs), but the system should be able to guarantee a minumum time to the batch stream.

The operating system should also provide for interactive users in a timesharing mode. Initially, the system will be required to support users at four interactive terminals (in addition to the batch and real-time functions), but it must be possible to expand the system to 16 users. Each interactive user will require access to the utilities, assembler, editor, compilers, filemanagement system, etc. Interactive programs should reside in fully protected memory partitions.

Program development will be an important ongoing part of the NURE program. The operating system should provide a complete keyboard monitor, or commandlanguage interpreter, to support program-development activities. This command language should allow the user (or the batch stream) to invoke all of system utilities (such as delete a file), languages, and user-written programs as well as control (schedule, terminate, etc.) real-time tasks and I/O assignments.

Utility programs should be available to copy files, delete files, list entries in file directories, report system status, dump memory, file backup to magtape, link object programs (build tasks), etc.

In addition to these utilities, a debugging program and a text editor are required. In particular, the text editor must be very interactive, preferably in a character-oriented mode instead of a line-oriented mode.

The operating system should be supplied with FORTRAN IV, BASIC, and a macro assembler language that produces a relocatable object code. As an option, COBOL should be available for addition to the system at a later time.

The cost of the software described above is estimated to be \$12,000. The addition of COBOL may add another \$7,000.

The data-base-management tasks described earlier will require a database-management system, costing an estimated \$15,000. This system should be a subset of the 1971 CODASYL Data Base Task Group specification; conforming to these standards will provide us with an adequate, well-defined system.

#### System Hardware

A very capable computer system is required to meet the NURE Program's data-base-management needs. This section describes such a system, including peripheral equipment and performance. A block diagram of the proposed system is shown in Fig. C2.

Due to the projected size of the stored data bases, at least one 80mega-byte disk will be necessary. This capacity will provide storage space for the data bases, the operating system, and application programs. In addition, this disk should have a high transfer rate and fast access time. Average seek time should be less than 30 msec, average rotational latency should be less than 8.5 msec, and the transfer rate should be greater than 800K bytes/ sec. The system could operate with only one drive, but a second drive would bring added benefits. First most of the data base could be separated from the operating system thereby allowing for seek overlaps during task swapping, which would improve system performance. A second drive would also allow for shortterm archival storage of raw data from the gamma-ray spectrometers (gamma-ray data will be acquired at a rate of about  $5 \times 10^6$  bytes per week). Finally, having two drives would enable long-term archiving of portions of the data base in a very convenient manner (on disk packs).

Industry-standard nine-track magnetic tape will be used for archival storage and data transmission. Raw data from the instrumentation will be archived for long-term storage on magtape. Data-base backup (i.e., dumping the disk to magtape) will be performed periodically. A transaction log of data base activity will be kept on magtape. Finally, magtape provides a data transmission facility to other computers (perhaps OCTOPUS). Two drives will be required: one dedicated to the DBMS and the other to be available for generalpurpose use.

A low-speed card reader (300 cards/min) will be used during program development to enter batch jobs. Data processed before this computer goes into service will be entered into the data base through the card reader.

During program development, a medium-speed line printer (500 lines/min) will be required for program listings, etc. Reports to be generated during sample analysis consist of one or two pages for each gamma spectra plus a minor amount for the delayed-neutron counter. DBMS reports will be much longer (150 to 200 pages), but will occur less frequently.

Since a graphics capability is needed, this printer should be an electo-

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Fig. C2. Block diagram of proposed NURE data-base-management system.

static matrix printer/plotter. Hard-copy plots will be generated of selected gamma spectra and DBMS data (for instance, maps showing element concentrations at the sample sites).

Both DBMS tasks and gamma-spectra analyses will require an interactive CRT graphics display system. Gamma-spectra analyses involve a large amount of sophisticated peak fitting. During program development, an interactive CRT would be an indispensable aid to determining the quality of the peakfitting routines. Moreover, current standard practice is to review manually each analysis performed by GAMANAL on OCTOPUS. Due to the large number of samples to be analyzed in the NURE program, manual review is clearly impractical. This problem can be resolved by two simultaneous approaches. First, the GAMANAL program will be revised to provide a more dependable result and to identify those analyses that need to be reviewed: Second, the interactive CRT can be used to rapidly review the results of an analysis in graphic form.

The intial system should support six asynchronous lines (EIA standard RS232, up to 9600 baud). These lines will be used for connection to: (1) the INAA and DNC data-acquisition instruments, (2) the ICP instrument, and (3) four terminals (the NURE program currently has three TI Silent 700's - the fourth should be an upper/lower case CRT terminal). In a second phase, more serial lines may be required to connect to additonal instruments (such as map digitizers, bar-code readers, etc.) and more terminals.

Many characteristics of the central processing unit (CPU) have already been mentioned, but will be summarized here. Computing speed is obviously important to process gamma spectra. Experience with such processing on the OCTOPUS CDC 7600 shows that we will need 2 to 5% of the CDC 7600's computing speed. The CDC 7600 has a reported benchmark performance figure of 8,000 for the commonly referenced 'WHETSTONE' test. Sixteen-bit minicomputers with fast floating-point processors reportedly have exceeded a 'WHETSTONE' performance figure of 500 (6% of the CDC 7600). An estimated time breakdown of CPU use is given in Table C4.

A large memory configuration (with memory management for task protection) is required to support simultaneous resident tasks (program development, DBMS, gamma-spectra analysis, etc.). It is estimated that a total memory capacity of 256 to 384K bytes will be required.

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Table C4. Estimated time breakdown of CPU use.

Day shift (four 7-hr days, total 28 hr/w	eek
Process 400 simple gamma spectra at 1 min/sample	6.7 hr
Receive 400 gamma spectra 12,000 bytes/spectra at 100 µsec/byte	0.1 hr
Receive spectrometer data	Negligible
Update data base CPU time + ≃20,000 disk accesses	<u>10</u> hr
SUBTO	TAL 16.8 hr
Remaining time available for interactive users doing application programming and generating reports TO	TAL $\frac{11.2}{28}$ hr
Nights and weekend shift (140 hr)	
Receive 400 gamma spectra 12,000 bytes/spectra at 100 µsec/byte	0.1 hr
Process 400 complex gamma spectra at 10 min/sample	66.7 hr
Update data base CPU time + ≃10,000 disk accesses SUBTO	TAL $\frac{5}{71.8}$ hr
Remaining time available for batch-stream processing TO	68.2 hr TAL 140 hr

#### Implementation Method

The instrumentation system should start producing data by late 1976: Obviously, however, the proposed NURE computer system will not be available by that time. Therefore, the following is proposed as an interim solution.

The NURE program intends to borrow a PDP-8/I computer system (including a Diablo cartridge disk system) from LLL's Radiochemistry Division. Data acquired from the INAA system will be recorded on these cartridge disks. Once or twice a day, the data from these cartridge disks will be transferred to industry-standard magnetic tape on another computer system in the Radiochemistry Division. The raw data on the magtape can then be processed on the OCTOPUS computer system.

This is intended only as an <u>interim</u> solution because: The PDP-8/I is on temporary loan only, this method is quite expensive, and trying to use OCTOPUS as an on-line analysis and data-management tool poses severe restrictions on our mode of operation. ESP:76-127 5/20/76

## APPENDIX D

## SPECIFICATIONS FOR AN

# INDUCTIVELY COUPLED PLASMA, DIRECT-READING SPECTROMETER

# 1. GENERAL

1.1 The system shall provide simultaneous determination of the elements shown in Table I under a single set of operational parameters.

## TABLE I

Elements and Minimum Concentration Ranges in ppm (µg/cm<sup>3</sup>) in Acidified Natural Water to be Covered with Maximum RSD (%) at the Lower Concentration Value

Element		Range		RSD	Element		Range		RSD
Al	0.005	_	10	ppm 1%	Mg	0.01		100	ppm 6%
As	0.04	-	10	3	Мо	0.01	-	٦	3
В	0.005	-	200	40	Na	1	- 20	,000	6
Ca	ſ	- 20	,000	6	N 1	0.01	-	1	3
Cd	0.001	-	۱	6	Si	0.03	-	100	15
Со	0.001	-	1	6	Sr	0.01	- 1	,000	40
Cr	0.002	-	1	3	U	0.06	-	1	1
Cu	0.002	-	200	3	V	0.001	-	٦	15
Fe	0.05	- 3	,000	1	Zn	0.005	-	<b>5</b> 00	6
К	1	- 20	,000	2	Ρ	0.03	-	100	10

Note: Vendor shall indicate in the bid the actual analytical range which can be covered by a single output channel for each element. If the lower limit of the range is not the detection limit (two times the standard deviation of the background), the vendor shall indicate the detection limit. For Na and K, indicate the range covered by an ultraviolet line; if a visible region line would be required to meet the lower limit, list it as an option. If the entire range for any other element can not be covered, preference should be given to the lowest portion of the range.

- 1.2 Desired Option: Same as 1.1, but delete B, Cr, and or from Table I.
- 1.3 The electronic portions of this system shall be constructed of solid state components preferably mounted on easily removable circuit boards. The circuit boards and associated hardware shall be flame retardant.
- 1.4 All equipment shall be so constructed and fabricated as to permit accessibility to any and all component parts for ease of maintenance and replacement.
- 1.5 The vendor shall furnish any special tools or materials required for maintenance and adjustment.
- 1.6 All terminals, plugs, connectors, circuit wiring, etc., shall be uniquely labelled on the equipment where practical and shall be accurately referenced in the system drawings.
- 1.7 Care shall be given to minimizing the acoustical noise level of the system. Where reasonable choices exist in the selection of noise-producing elements (fans, motors, etc.) the quieter elements shall be used.
- 1.8 The instruments shall be fully interlocked for operator safety and protection of the instruments against damage. All safety hazards shall be minimized and the presence of any potential hazard shall be indicated by proper warning signs.
- **1.9** All equipment shall be provided with an electrical ground separate from the ac power line neutral.
- 1.10 Air cooling shall be provided as needed, adequate to maintain all components within their respective allowable operating temperatures.
- 1.11 The system shall be completely assembled by the vendor, who shall provide all equipment necessary for the operation of the system.
- 1.12 All equipment shall be in compliance with relevant OSHA regulations.
- 1.13 Delivery shall be within 120 days of receipt of order.
- 1.14 The system shall include necessary protection to insure that momentary interruptions of the commercial power source, power line transients, or line frequency fluctuations will not cause damage to system components.

- 1.15 The system shall operate as specified with rapid powerline voltage fluctuations or transients of up to one volt/microsecond and with amplitudes not exceeding 10 volts.
- 1.16 The system shall operate as specified in an environment of 18° 27°C and 20% - 60% relative humidity.
- 1.17 The vendor shall provide comprehensive training in the operation of the system for up to two LLL personnel. The schedule and content of such training shall be mutually agreed upon by LLL and the vendor prior to award of the order.
- 2. INDUCTIVELY COUPLED PLASMA SOURCE POWER
  - 2.1 The high frequency power supply shall have the following characteristics:
    - (a) Frequency = 27.14 ± 0.02 MHz.
    - (b) Frequency stability = 0.05%.
    - (c) Power output = 750 2000 W (minimum).
    - (d) Envelope ripple = 5% (maximum)
  - 2.2 The high frequency power supply shall have the following:
    - (a) Automatic feedback control or other system to maintain output power to within ±1% of the set point. This system shall have provision for manual override.
    - (b) An automatic tuning network with manual override. The impedance match at the coupling box shall be shown to be such that the reflected power is less than 5 W at a forward power of 1000 W.
    - (c) Circuit protection on forward and reflected power including automatic overload shut-down circuits.
    - (d) Controls, with manual override, for pre-setting the amount of incident power and limiting the amount of reflected power.
    - (e) Meters showing forward and reflected power.
    - (f) Ability to withstand a mismatch as great as 2 to 1 at full power output.
    - (g) Operating controls within 1 meter of the plasma torch location.
    - (h) Spurious RF radiation within FCC specifications and not deleterious to nearby electronic equipment.

## 3. PLASMA TORCH

- 3.1 The plasma facility shall be provided with two torch assemblies and two aerosol generators.
- 3.2 Individual regulators capable of controlling the coolant gas flow in the range of 5 15 liters Ar/min. and of controlling the aerosol carrier gas flow in the range of 0 3 liters Ar/min. shall be provided.
- 3.3 The vertical range adjustment shall allow any position up to 40 mm above the load coil to be observed by the spectrometer.
- 3.4 The horizontal range adjustment shall allow any position up to 10 mm right and left of the torch center line to be observed by the spectrometer.
- 3.5 The torch shall be provided with Tesla coil ignition.
- 3.6 The induction coil shall be water cooled.
- 3.7 The discharge shape shall be annular (toroidal).
- 3.8 Sample solution uptake rate shall be adjustable and controlable over the range of  $1 4 \text{ cm}^3/\text{min}$ . (ml/min.).
- 3.9 The spray chamber shall have provision for being heated.
- 3.10 Memory effects of Na or Mn shall be reduced by at least a factor of 5000:1 with a 30-sec. wash.
- 3.11 The system shall be compatible with a field-installed automated sample presentation system.
- 3.12 Spurious RF radiation shall be within FCC specifications and shall not affect nearby electronic equipment.

## 4. SPECTROMETER

- 4.1 The spectrometer grating shall be blazed for analysis of the elements and concentration ranges shown in Table I and shall provide the maximum dispersion for these conditions.
- 4.2 The spectrometer exit slits shall be aligned by the vendor.
- **4.3** The spectrometer shall contain an appropriate number of background measuring phototube assemblies.
- **4.4** It shall be possible to expand or change the number and/or location of the exit slits and photomultiplier assemblies in the field.
- 4.5 The spectrometer shall be capable of holding at least 48 photomultiplier tubes and related assemblies.
- 4.6 The phototubes shall be exposed to a low level of white light between analytical cycles.
- 4.7 The spectrometer shall remain in optical alignment with all exit slits on their respective wavelengths over the ambient temperature range of 18° - 27°C. It shall be possible to compensate for temperature fluctuations of 1°C/hr.
- **4.8** A single control shall allow the operator to optimize the optical alignment.
- 5. READOUT SYSTEM
  - 5.1 A photomultiplier and appropriate measurement electronics shall be provided for each exit slit contained in the spectrometer.
  - 5.2 Each photomultiplier shall be individually screened from extraneous stray light sources.
  - 5.3 The interior of the spectrometer shall have no unnecessary reflective surfaces which may contribute to extraneous stray light.
  - 5.4 The electronic measuring system shall be linear over at least a five decade range without switches or operator intervention.
  - 5.5 In case of power failure, computer memory shall be protected. Simple re-start procedures shall be available.
  - 5.6 The system shall have a diagnostic program for testing major system components such as the readout electronics (dark current test) and the photometric quality of the spectrometer (white light test).

- 5.7 Any analytical channel shall be useable as an internal standard channel at the operator's option.
- 5.8 The system shall be calibrated for the elements and analytical ranges given in Table I or for alternate elements and ranges agreed to by vendor and LLL.
- 5.9 The data processing system shall consist of a minicomputer, a data printer, and all interfacing and software necessary to control the spectrometer and process data received from the analytical system.
- 5.10 It shall be possible to expand or change the analytical program in the field.
- 5.11 Dynamic background correction shall be provided.
- 5.12 The system shall be capable of storing analytical curves for 35 elements in at least two matrices.
- 5.13 The system shall permit the operator to enter new analytical matrices, change standardization samples, change analytical curves, and edit output format.
- 5.14 Output shall be presented with four significant figures.
- 5.15 The analytical program shall permit spectral interference corrections involving at least 15 interdependent constants (no. of constants = no. of interfering elements times no. of channels interfered).
- 5.16 At the operator's election, output data shall be available as absolute intensity, intensities ratioed to an internal or external standard, background corrected intensity ratios, or concentration in percent, ppm. or ppb.
- 5.17 The analytical curve for each element in Table I shall be linear over its concentration range without adjustment, operator intervention or sample manipulation.
- 5.18 Concentration readout on up to 30 elements shall be obtained in less than 60 seconds.
- 5.19 Precision (expressed as % RSD) obtainable with 10 exposures at a concentration of 1 ppm shall average not more than 1% for all elements determined at that concentration.

- 5.20 Precision obtainable without additional standardization or adjustment over
  a 4-hour period at a concentration of 10 ppm shall average not more than
  3% for all elements determined at that concentration.
- 5.21 Precision obtainable on 10 integrations of the grating lamp shall be 0.1% or better as averaged over all channels.
- 5.22 The system shall allow for at least ten replicate multi-element determinations to be made per sample and the average value and the relative standard deviation for each element to be computer calculated and printed.
- 5.23 The system shall be compatible with automated sample presentation.
- 5.24 Vendor shall state how many programs will be core resident, method of loading programs, and loading time necessary for each program.
- 6. DOCUMENTATION (A Desired Option)
  - 6.1 The vendor shall supply two complete copies of the operating instructions for the system. Separate manuals for sub-systems are acceptable.
  - 6.2 The vendor shall supply two complete copies of maintenance manuals and schematics with sufficient information that a technician with experience with similar equipment could maintain and repair the system and understand the details of its operation. The maintenance manuals shall include all material which would be supplied to the vendor's own maintenance personnel and shall include at least the following:
    - (a) Specifications of the system and sub-assemblies.
    - (b) Theory of operation with block diagrams.
    - (c) Recommended spare parts lists including manufacturer's part numbers for all house-numbered semiconductors or other components, if available.
    - (d) Detailed schematics including logic diagrams, part names, manufacturer's part numbers, wiring tables, timing diagrams; voltage values, waveforms, and timing information at critical circuit points as required for checking, adjustment, or servicing of the system.
  - 6.3 The machine-readable medium furnished shall be readable and operable on the system as delivered.

- 6.4 The vendor shall supply one copy of each of the following:
  - (a) A manual describing all CPU instructions and timing, I/O and interrupt techniques, peripheral instructions, and other programming techniques required for efficient operation of the system.
  - (b) Published manual and other descriptions pertaining to all software delivered.
  - (c) The object code, on machine-readable medium, of all software delivered.
  - (d) The source code, on machine-readable medium, of all software delivered.
  - (e) The assembled source listings, in hard-copy printout or published form, of all software delivered.
- 7. TESTS FOR ACCEPTANCE (A Desired Option)
  - 7.1 At LLL's option, vendor shall perform a system checkout test at the vendor's plant to demonstrate to representatives of LLL that the system meets the requirements of the specifications. LLL shall be notified two weeks in advance of the date of such tests.
  - 7.2 A second system checkout test similar to the first test shall be performed and passed at LLL after the system has been installed. This test shall be performed by the vendor or under his supervision.
  - 7.3 System checkout schedules and procedures shall be proposed by the vendor and will be subject to approval by LLL.
  - 7.4 The final acceptance test at LLL shall require performance, for a 30consecutive-day period, within the operating and performance characteristics as defined in the specifications. Accurate performance records shall be maintained by LLL during this period.
- 8. TECHNICAL PROPOSAL REQUIREMENTS
  - 8.1 To permit a fair and adequate evaluation of proposals received, vendor shall submit information or material, correlated with the paragraphs of the specifications, which respond to those paragraphs in one or more of the following ways:
    - (a) Describes components, software, operational characteristics, vendor action, etc., which satify the requirements.
    - (b) Gives and explains exceptions or alternatives to the specifications.

# **ICP Specifications**

- (c) States the actual performance if the requirements are either exceeded or not met.
- (d) Lists additional features provided at no additional cost.
- (e) Gives vendor's suggestions, such as additional features offered, which would increase the capabilities or performance of the system for the intended application.
- 8.2 Vendor shall include in his proposal the following information:
  - (a) Current and voltage ratings required for the ac power source(s) which LLL must furnish, including both inrush or peak and steadystate values.
  - (b) Dimensions of the total system as installed, including clearances required for operation and maintenance. Drawings are preferred.
  - (c) Any restrictions on cable lengths or placement of components.
  - (d) Description of standard maintenance service offered, both during and after the warranty period, including point of origin for such service, time required for response, and hours available.
  - (e) All required services and facilities such as compressed air, cooling water, exhaust fans, drains, RF grounding, etc.
  - (f) Performance data and calculations to provide assurance that the system will perform reliably and the names of installations where comparable systems have been installed with names of persons who may be contacted.

# 9. DEFINITION OF "DESIRED OPTION"

A feature, component, or performance requirement which LLL may elect to include or exclude from a resulting order. It must, therefore, appear as a separately identifiable item in the proposal. Failure to include the option does <u>not</u> amount to an exception to the specifications. However, preferential consideration may be given to proposals which include desired options.

APPENDIX E

# LLL HYDROGEOCHEMICAL SITE-VISIT FORM

				Site No
	(PLEASE PRINT)	otion		73-80
	Site visit identific	ation		
Col.				
<u>1&amp;2</u>	Brainst		Teem	
02	Tourship-Paper	10	Ormori	40
02	Section 1/4 Section		Address:	40
04	Jection, 174 Jection	10	Address.	40
05	Man Name:			40
05	Date:	10	Time:	40
00	bucc.	10		40
	Samples Collected (E	nter Sample Type	, Treatment)	
07	S1	S5	S9	
08	s2 <sup>10</sup>	s6 <sup>20</sup>	\$1030	
09	s3 <sup>10</sup>	s7 <sup>20</sup>	S1130	
10	s4 <sup>10</sup>	s8 <sup>20</sup>	S12	
	10	20	30	
	Field Data (All Wate	er Sites)		
	<u> </u>	·····		
11	Temperature (°C)	10	Phosphate	(mg/l)
12	Specific Conductivit	у	Nitrate (	mg/l) 40
13	(µmhos/cm)	10	Ammonia (	$mg/l$ ) $\frac{40}{40}$
14	pН	10	Other	40
15	Total Alkalinity (mg	$(10) \frac{10}{10}$		40
16	Plenal. Alkalinity (	(mg.l) 10		40
17	CO <sub>2</sub> (mg/l)	10		40
	Ground Water, Lakes	10		40
18	Eh (mv)		Dissolved	Oxygen (ppm)
		10		40

Stream Water and Sediment

19	Stream Width (nearest ft) Depth (nearest ft)
20	Water Level: DryLowNormalHighFlood
21	10     11     12     13     14       Evidence of Wind Blown Material at Sample Site:     YesNo
	Not Sure12
22	Nature of Bottom: Rock Gravel Sand & Gravel Sand Silt
23	14 Refuse or other contamination visible
24	10 Precipitate or Stain: Fe bydrovides
24	$\frac{10}{10} \qquad 11 \qquad 12$
25	SulphurSulfidesNone 10 11 12
26	Vegetation: SparseModerateHeavyForest 10 11 12 13
27	Odor: Hydrogen SulfideNone
28	Color of Sediment: Black Gray Brown Dark Brown Red
29	Red Brown Yellow Yel. Brown Buff White 14
30	Suspended Matter: Heavy Light
31	Relief: High Medium Low Flat
32	Weather: Clear Cloudy Light Rain Moderate Rain
	Heavy Rain
33	Evidence of Recent Rain
34	Activity in Area: Industrial Farming Mining
35	Residential None
	13 10
	For Vollo Only
	For werrs only
36	Well Type: Municipal Domestic Farm/Livestock Irrigation 13
37	Well Location:
38	Casing: Iron PVC Galvanized Iron Other
39	Depth of Well (ft)
40	Discharge Type: Artesian Non Flowing Wind Gas/Elect. Pump

41	Hand Drawn Other
42	Discharge Rate (gal/min)
43	Odor: Hydrogen Sulfide None Other 10 10 11 20
	For Springs Only
44	Type: Hot Cold Undeveloped Developed
45	Use: Domestic Livestock None Other
46	Odor: Hydrogen Sulfide None Other
47	Precipitate of Stain: Fe hydroxides Mn hydroxides Organic
48	Sulphur Sulfides None
49	Evidence of Degasing: YesNo
50	Evidence of Mineral Precipitation: Yes No
51	Discharge (ft <sup>2</sup> ) Discharge Rate (gal/min) 10 For Lakes/Reservoirs
52	Type: Natural Lake Man-Made Lake Pond Other
53	Sample Taken: Shore Middle In-flow Out-flow Other 10 11 12 20 Sample Taken: Shore Middle In-flow Out-flow Other 20 11 12 20
Commen	ts:
	·

## APPENDIX F

# STANDARDIZED REPORTING FORMAT FOR LLL PILOT STUDIES UCID-16911-P-xx

## PRELIMINARY REPORT ON THE (-----) BASIN PILOT STUDY (State)

## INTRODUCTION

The Lawrence Livermore Laboratory (LLL) is conducting a hydrogeochemical and stream-sediment survey in the seven western states as part of ERDA's National Uranium Resources Evaluation (NURE) Program. The objective of this survey is to develop a geochemical data base for use by the private sector to locate regions of anomalous uranium content. Prior to wide area coverage, several pilot studies are being undertaken to develop and evaluate sampling and analytical techniques. The ---------- studies were conducted in ---- playa basins in (state), selected to represent different regional geology and uranium occurrence. This study in the ------ Basin, located on Figure 1, characterizes ------ geology (with/without) known uranium deposits.

	THIS SPACE CONTAINS A 150 WORD DESCRIPTION
	OF THE REGIONAL, GEOLOGIC, AND TOPIGRAPHIC
(	SETTING OF THE STUDY.

)))))

-Fl-

This report contains only the analytical data and sample locations. Details of the sampling methods and equipment, analytical sensitivity, errors, interferences, and geochemical interpretation will be reported separately.


FIGURE 1: LOCATION OF THE ----- BASIN

## SAMPLE COLLECTION AND TREATMENT

Samples of the playa were collected at depths of five and ten feet from shallow hand-drilled holes. Sediment samples were obtained from dry stream beds and alluvial fans which surround the playa. Relatively unweathered rock judged to be characteristic of the locale was also collected near many of the sediment sampling locations. In addition, all of the flowing streams, springs, and active wells which could be found were sampled. The pH, temperature, dissolved oxygen, eH, alkalinity, phenolphthalein alkalinity, and carbon dixoide were measured in-situ on filtered but otherwise untreated samples. Collected samples were filtered through a 0.45  $\mu$ m pore membrane and one portion acidified to pH  $\sim$ 1.0 with HNO<sub>3</sub> in the field and the other untreated.

In the receiving laboratory the sediments were dried and the portion passing through a 1000  $\mu$ m sieve, but retained on a 500  $\mu$ m sieve, was analyzed. The playa samples were similarly handled except that they were first blended and the "cake" formed on drying was pulverized before sieving. Essentially all of the playa material passed through the sieve. Rocks were ground and passed through a 250  $\mu$ m mesh sieve before analysis. Unacidified water samples were analyzed for chloride and sulfate content. The acidified sample was mixed with cellulose and dried, and the residue was analyzed. Archival samples were retained from all material collected.

Locations of water, rock, and sediment samples are shown in Figure 2. This figure is scaled to the 1:250,000 AMS map, ---- xx xx-x, and shows the Universal Transverse Mercator 10 km grid for Zone xx.

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## CHEMICAL ANALYSIS

All data in this report except for the water measurements discussed above were obtained by neutron activation analysis using the Livermore  $P\infty$ l-type Reactor. Samples were counted after predetermined decay periods following irradiation. The counting data were converted to elemental concentration data by use of a computer code which also derived an estimate of the statistical errors. Other errors are not specified.

# DATA PRESENTATION

Results of the study are presented in the appended six tables. Each table is divided into two parts, the first of which gives the site number, its location by UTM coordinates, and the field measurement data. The second part contains the site number and neutron activation analytical data. The tables are titled and numbered as follows:

Title	Location & Field Data	Analytical Data
Streams	1.1	1.2
Spring and Well Water	2.1	2.2
Playa Samples	3.1	3.2
Sediments, Wet Sites	4.1	4.2
Sediments, Dry Sites	5.1	5.2
Rocks	6.1	6.2

Certain sites were revisited during different parts of the season and replicate samples collected. These appear in the N.2 tables, with a four-letter sample type and treatment code followed by a numeral. Zero indicates the first sample; 1-the second, or duplicate; 2-the third, etc. The sample type and treatments applicable to this study are: SWwet sediment; SD-dry sediment; WW-well water; WS-spring water; RO-rock; NO-no treatment; DS-dried and sieved; and LA-large acid. Thus, WWLA-4 would be the fifth acidified well water sample.

The location and field measurement tables have the site number, UTM coordinates, date collected, and data as described below.

### Field Measurements

- <u>H O TEMP</u>: Measurement of water temperature, in situ, by mercury thermometer to nearest 0.1 degree C.
- <u>PH</u>: Acidity in pH units, reported to nearest 0.1 pH unit at ambient water temperature.
- TOTAL ALK: Measurement by titration to a bromcresol green-methyl red indicator endpoint (pH = 4.8) with acid. Reported as an equivalent amount of CaCo<sub>3</sub> in mg per liter.
- <u>PHENO ALK</u>: Measurement by titration to a phenolphthalein indicator endpoint (pH = 8.3) with acid. Reported as an equivalent amount of  $CaCO_3$  in mg per liter.
- DISS CO<sub>2</sub>: Measurement by titration to a phenolphthalein indicator endpoint (pH = 8.3) with base. Determination is not made if pH > 8.3. Reported as equivalent dissolved CO<sub>2</sub> in mg per liter.
- <u>DISS 02</u>: Measurement with a commercial dissolved oxygen meter. Accuracy is estimated to be <u>+</u> 0.1 ppm.
- EH: Measurement with a portable commercial eH meter. Reported in millivolts, referenced to a standard colomel electrode at 25 degrees C.

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# Laboratory Measurements

- <u>COND/LAB</u>: Measurement with a commercial conductivity meter. Reported as specific conductivity in micromhos per cm (µmho/cm) at 25 degrees C. CHLORIDE: Measurement of the chloride content reported in parts-per-
- million (ppm). Detection limit estimated to be 5 ppm; accuracy  $\pm$  10 ppm.
- SULFATE: Measurement of the sulfate content reported in parts-per-million (ppm). Detection limit estimated to be 10 ppm; accuracy + 20 ppm.

In the neutron activation analytical data tables the site number, date collected, sample type and treatment, and uranium content are always listed as the first four columns of each page, followed by other trace elements. The other elements are listed alphabetically and are: Sb, As, Ba, Br, Ca, Ce, Cs, Cr, Co, Au, Hf, Fe, Mo, K, Na, Sr, Ta, Th, W, Zn, and Zr. These are divided in groups of four to a page so each table contains several repetitions of the site information and uranium columns. The data tabulated are parts by weight of the element, with the symbols PPK, PPM, PPB, and PPT standing for parts per thousand, million, billion, and trillion, respectively. Those cases where no value is listed indicate that the measurement was below the limit of detection for that specific sample. Because of variation in measurement conditions, the limit of detectability fluctuates, and thus a missing value is not necessarily smaller than the smallest value listed for some other sample.

It must be emphasized that this data is preliminary and may contain undetected errors. No warranty of accuracy can be implied or is intended. The user must be aware that any conclusion he may draw from it is entirely and totally his own, and that neither the LLL nor the University of California accepts any liability for costs incurred by a user because of such errors.

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