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Lawrence Livermore Laboratory

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

Quarterly Progress Report

July through September 1976

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INTRODUCTION

This report summarizes activities and accomplishments (during the July through September 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

- Sample site selection and planning were initiated for a reconnaissance sampling project covering 14,000 square miles in western Utah.
- Reconnaissance sampling contracts were placed for the Humboldt River Basin and six basins in the Lovelock and Winnemucca quadrangles in northern Nevada.
- Planning was completed for two orientation studies in northeastern Washington.

- A technical paper, "Dispersion of Uranium and Selected Trace Elements in Playa Basins," was presented at the 25th International Geological Congress in Sydney, Australia, August 15-25, 1976.
- Installation of the high-throughput instrumental neutron-activation analysis system and a new delayed-neutron counter will be completed in December, 1976.
- An arc source and 22 direct-reading channels were added to the emission spectrometer for sediment analyses.
- The Phase II computer system was purchased with FY 1976T capital equipment funds. This system will provide on-line data reduction for several analytical systems and will perform all data-base-management tasks.
- A computer-readable magnetic tape of the data in the Walker River Basin Pilot Study report was sent to Oak Ridge National Laboratory for entry into the NURE national data base.
- Data reports for five orientation studies were open-filed in this quarter.

GEOLOGY AND SAMPLE ACQUISITION

Reconnaissance Sampling Program

Planning — An updated geological province map of the western United States is shown in Fig. 1. Each province has its own unique set of geological and hydrological variables that determine the optimum sampling and analytical techniques to be used in the reconnaissance sampling program. Our reconnaissance sampling program will conform to these geological boundaries. Each geological province has been assigned a potential for successfully locating new uranium districts based on geology, current industry activity, and the probability of successfully using hydrogeochemical techniques to locate undiscovered district-size anomalies.

The projected reconnaissance sampling schedule (Fig. 2, Table 1) reflects the relative priority assigned to each province as well as projected budgets. High-priority areas, such as the Basin and Range province and the northern Rocky Mountain province, will be sampled early in the program. Low-priority areas, such as the Pacific Coast province, will be sampled in

Fig. 1. Major geologic (physiographic) provinces in the seven western states.

Fig. 2. Projected reconnaissance sampling schedule by fiscal year.

FY 1980 and 1981. Parts of the Colorado Plateau, although a major uranium-producing region, will be sampled later in the program because of intensive exploration activity by industry in this region.

Table 1. Proposed sampling schedule through FY 1981.

	COMPLETED	PROJECTED			
	FY 1975-76	FY 1977	FY 1978	FY 1979	FY 1980-81
Number of sites	3,000	8,000	29,000	24,000	13,000
Square miles covered	42,000	42,000	175,000	161,000	280,000
Percentage of total area	6	6	25	23	40
Areas covered	Nev., Cal.	Nev., Cal. Utah, Ariz.	Nev., Cal., Utah, Ariz., Idaho, Wash.	Cal., Ariz., Utah, Idaho, Oregon, Wash.	Cal., Ariz., Utah, Oregon, Wash.

Sample site selection and planning are underway for the next reconnaissance sampling project, which will cover some 14,000 square miles in west-central Utah. This area, commonly referred to as the "Beryllium Belt," is shown in the two Army Map Service (AMS, scale 1:125,000) series maps shown in Fig. 2. Approximately 3000 sites will be sampled during the second quarter of FY 1977.

The Beryllium Belt lies along the eastern extent of the block-faulted Basin and Range Province. The typical Basin and Range north-south lineations, however, have been subdued and modified by late tertiary volcanics. Most of the mountain ranges in the Beryllium Belt are made up of tertiary granitic intrusive rocks and rhyolitic extrusive rocks. The granites are early tertiary in age and include overthrust strata ranging in age from Precambrian to Silurian. Uranium mineral occurrences in the Beryllium Belt are associated with fluorine and beryllium mineralization and as secondarily enriched deposits.

Current Reconnaissance Projects

Humboldt River Basin - The qualified low bidder on the Humboldt River Basin sampling contract began work July 30, 1976, with four sampling teams. By September 30, 1976, the contractor had collected 1348 dry-sediment, 133 wet-sediment, and 406 water-and-wet sediment samples. The contractor was paid \$11.40 per site for dry sediments and \$19.20 per site for water and wet sediments.

Samples were delivered to a Lawrence Livermore Laboratory representative in the field. After the samples had been received and checked, they were boxed, loaded into a trailer, and shipped to LLL for processing and analysis.

LLL personnel repeated sampling operations at 5% of the sites for the field quality-assurance program.

Six Basins — This area consists of the following six basins, which make up part of the Lovelock and Winnemucca AMS quadrangles:

- San Emido Desert
- Granite Springs Valley
- Buena Vista Valley
- Pleasant Valley
- Jersey Valley
- Buffalo Valley.

Bids closed on August 25, 1976, and the successful low bidder started work on September 11 with three sampling teams. By September 30, 1976, the contractor had collected 407 dry-sediment and 16 water- and wet-sediment samples. The cost was \$18.40 per site for dry sediments and \$38.50 per site for water and wet sediments. The specifications for sample collection in these six basins are presented in Appendix B.

The Desert Research Institute also had two crews in the field sampling springs and wells in both the Humboldt River and six-basin areas. They sampled a total of 133 sites.

Orientation Studies

Northern Rocky Mountain Province — Reconnaissance sampling in the high-priority northern Rocky Mountain Province is scheduled for FY 1978. In keeping with our sampling design philosophy, an orientation study will be conducted in the region to determine optimum sampling and analytical techniques specific to that environment.

Two areas have been selected for study; one contains a known uranium deposit and the other, in similar terrain, has no known uranium deposits (see Fig. 3). The Midnight Mine area contains one of the major U.S. occurrences of uranium in crystalline rocks. Data obtained from various geologic samples

Fig. 3. Two areas are being examined in the Washington orientation study for the northern Rocky Mountain Province. Both areas have similar terrain, but the Midnight Mine area has known uranium deposits while the Newport area does not.

in this area will be compared with data from the Newport area, which does not contain known uranium deposits. This region is part of the northern Rocky Mountain Province and is known locally as the Okanogan Highlands. The eastern portion of this province includes Precambrian Belt Series metasediments and miogeosynclinal lower and middle Paleozoic rocks. These are underlain by numerous bodies of mesozoic intrusives, with minor extensions of Columbia River basalts exposed in the southern section. Discordant uranium deposits occur in both Precambrian metasediments and Tertiary sedimentary rocks, with the probable source rock in both cases being a Cretaceous coarse-grained two-mica quartz monzonite with anomalously high U (average 12 ppm). Ore bodies consist of an oxidized upper zone of autinite and a lower reduced zone of coffinite and pitchblende.

The specific objectives of this study are to:

- Determine the geochemical mobility of uranium in the northern Rocky Mountain Province.
- Determine the potential use and limitations of various geologic media (surface water, ground water, and stream sediments) to identify uranium anomalies.
- Develop sampling and analytical techniques appropriate to the environment.
- Determine any "associated elements" that might be useful in delineating anomalous areas.
- Provide background values for uranium and "associated elements" by comparing a mineralized area with one of similar geology but with no known uranium occurrences.
- Test the applicability of the activity ratio U^{234}/U^{238} as a geochemical tracer.
- Determine optimum sampling-density and site-selection criteria for the regional reconnaissance program.
- Determine the magnitude of temporal variation in uranium content and/or chemical composition of surface waters in this region.
- Compare "mobile" uranium estimates of leaching versus total sample analysis.
- Conduct limited particle-size studies to determine the optimum size fraction for analysis.

Approximately 300 sites will be sampled over an 1100-mi² area, and samples will be taken from water in streams, springs, wells, and seeps as well as from the sediments of active (wet) and inactive (dry) streams.

Basin and Range Province — Analysis of the data from the Walker Basin and Playa Basin orientation studies is providing a fundamental understanding of the dispersion of uranium in the semiarid Basin and Range province. These results have demonstrated the potential as well as the limitation of various sampling mediums, techniques, and interpretation parameters that can be used in the reconnaissance sampling program. Several technical reports on these data are in progress and will be released early in 1977. Meanwhile, the following tentative conclusions have been made based on the data:

- Water from active streams is an effective sampling medium where the stream has a significant ground water input, as is common in or near the ranges. However, once the stream becomes an effluent stream, as is common in the basins, stream water is ineffective in detecting uranium anomalies.
- Significant increases in uranium content observed downstream in certain systems (i.e., the Walker River) can be attributed largely to evaporative processes and agricultural activities.
- The uranium-to-chloride ratio appears to be an effective method for normalizing seasonal and evaporative processes within stream systems. Also, electrical conductivity measurements provide a useful qualitative measure of the maturity of a given water sample.
- Ground water appears to be an extremely useful sampling medium. However, the sparsity of sites and their limited distribution present major limitations.
- Uranium in surface water (i.e., the Walker River) appears to be a very conservative component. The uranium is highly mobile and does not appear to be effectively removed from solution by reaction with stream sediments.
- In stream sediments, dispersion of uranium away from known anomalies seems to be largely mechanical. Therefore, in predominately dry streams, dispersion paths are short.

- Uranium in resistate minerals is, in most cases, the predominant source of uranium in the sediments. Normalization of the uranium with certain elements (i.e., Zr, Hf), therefore, should provide a method to distinguish the "nonresistate" from the "resistate" uranium in a sediment sample.
- The coarser sediment size fraction (i.e., 0.5 to 1.0 mm) appears to be a more useful analytical sample than the very fine size fractions. The finer size fractions are subject to wind transport and are commonly enriched in resistate minerals.
- Near-surface playa-sediment sampling does not look promising for the reconnaissance sampling program. In general, the uranium content of sediments in a playa displays a significant lateral and vertical heterogeneity.

SAMPLE PROCESSING AND ANALYSIS

Analytical Processing

Analyses of representative fractions of all samples collected in four playa basins in Nevada were completed during the transition quarter. The principle analytical methodology employed was instrumental neutron-activation analysis (INAA). Detectable amounts of 22 elements, selected on the basis of geochemical interest and measurement reliability, were reported to the data base. Other elements that may have been detectable were ignored. This selective reporting eliminated the need for extensive checking of gamma spectral interpretation results, thereby significantly reducing the data processing time. Five data reports, based on these measurements, were open filed in early August 1976.

To obtain suitable statistics on the proper size fraction for the dry-sediment samples, a second (finer) seive cut was analyzed as described above. About 70% of the samples were reprocessed at the finer mesh size. These results have been validated and placed in the data base transfer file. Completion of these analyses eliminated the backlog of available samples, so analytical operations were suspended until new samples are submitted.

Table 2 summarizes the types and numbers of samples processed during the quarter. The few delayed-neutron-count (DNC) measurements indicated in

the table were of a developmental nature on selected samples. All reported uranium concentrations were obtained by gamma spectrometry.

Table 2. Samples processed — transition period FY 1976T.

	Number of samples
<u>Instrumental neutron-activation analyses</u>	
Concentrated water (residues)	74
Playa sediments	134
Dry sediments (500 to, 1000 μ)	239
(less than 150 μ)	165
Host rock	32
Control standards	39
Blanks and miscellaneous	5
	<hr/> 688
<u>Delayed-neutron uranium analyses</u>	
Concentrated water (residues)	20
Sediments	24
Blanks	14
Standards	7
	<hr/> 65

Developing Analytical Capability

Installation of the automated high-throughput neutron-activation analysis facility remained on schedule through the transition quarter. All components have been ordered, and most of the equipment had been received at quarters end. In addition, modifications to the reactor building to accept the system were nearly complete (see Fig. 4). A new addition to the loading dock outside our "switch" room was built and readied for the dropout station. Installation of delay stations, detector stations, pneumatic-tubing supports, and switch-equipment mounting brackets is well underway. Switches, valves, and pneumatic tubing will be installed in early October. Hardware is expected to be complete by mid-October. Then, electronics installation and checkout will begin.

Fig. 4. Modifications to the reactor building.

A few conceptual design changes were made to the system as described in the last quarterly report. The "rabbit" transport network being installed (Fig. 5) will provide all of the flexibility described previously. In addition, it will provide a re-irradiation option after either DNC or INAA counting operations. Re-irradiation after the initial measurements will permit the use of larger samples and improve the quality of late-time measurements without sacrificing sensitivity for the initial counts.

The new design reduces the number of "Y" joints from seven to three, providing faster, more reliable operation. In the current design, a 4-way switch replaces the two 2-way switches previously indicated. This should slightly increase reliability and reduce control problems.

Perhaps the most significant changes in previously reported plans were the decisions to build a new delayed-neutron counter and to isolate the NURE radiation system from the presently existing fast-rabbit system. The new DNC facility was made possible by additional funding and should greatly increase the sensitivity and precision of our uranium analyses. As illustrated in Fig. 6, the DNC will have twenty 1-in. $\times$ 12-in. BF_3 counting tubes, shielded from the rabbit "home" position by a 2-in. lead sleeve. A gamma sensor will be installed below the "home" position to provide a go/no-go decision for further measurement in the activation-analysis parts of the system. The entire package will be suspended underground for shielding from reactor and cosmic radiation. Delivery is expected around December 1, 1976.

A new in-core irradiation facility will be provided for the NURE system, an addition clearly needed for some time because of the increased use of the existing in-core terminal by other LLL programs. We could reasonably expect a myriad of problems in sharing the current facility. The new in-core station is expected to be delivered in early December. Checkout of other parts of the system should be nearly complete by the time this final piece of hardware is available. Total system checkout will follow installation of the new in-core terminal.

Purchase of a direct-reading emission spectrometer with an inductively coupled argon plasma source was discussed in the last quarterly report. This instrument is scheduled for delivery in early December. During the transition quarter, a change order was issued to add a dc arc source and a number of new direct-reading channels to the spectrometer for sediment analyses. As were the plasma-source channels, the dc-arc channels were identified from

Fig. 5. Block diagram of the NURE rabbit system.

Fig. 6. Diagram of the delayed-neutron detector.

elements selected on the basis of desirability for uranium geochemical investigations. Elements requiring extraordinary procedures for analyses at "typical" concentration levels were excluded. A few channels were added so that elements useful in characterizing geologic factors and cultural influences could be measured. Table 3 lists the elemental channels and quoted lower detection limits for the two sources in the spectrometer we ordered. The unit should be fully operational in January.

Final design of the sample-processing facility at the reactor building has been approved. Modifications to be made include limited sediment-processing, sample-receiving, and clean-room facilities. Construction began in late September and will be completed by mid-December. The interior of the clean-room facility will be complete by late November, ready for installation of the emission spectrometer when received. Temporary processing facilities are available in the interim. When completed, the sample-processing facility will house sediment-control sample preparation, sample-receipt and logging stations, water analyses, emission spectrometry, and irradiation-capsule preparation.

Approval was also received to order the Phase II computer system, which will provide on-line data reduction for several analytical systems and will perform all NURE data-base-management tasks. System components and software are listed in Table 4. At quarter's end, the computer order was in final budget review, however, capital-equipment money was committed in FY 1976T and the order is expected to be placed early in FY 1977, with delivery in the third quarter of FY 1977. Until then, a borrowed PDP-8I will be used for data acquisition.

Comments On FY 1976T Milestones

In the last quarterly report, we set forth a number of milestones for the FY 1976 transition quarter. The following comments are an assessment of our performance relative to those goals:

- Analysis and final reporting of data from the four playa-basin pilot studies was completed.
- No Humboldt River Basin samples were available in 1976T, but processing of these samples should begin in the first quarter of FY 1977.
- Clean-room design was completed, but installation was delayed to the first quarter of FY 1977.

Table 3. Lower detection limits.— emission spectrograph.

	Plasma (ppb in H ₂ O)	dc arc (ppm in solids) ^a
Al	5	10 000
As	40	—
Ca	1 000	2 000
Cd	1	—
Ce	—	1 000
Co	1.5	10
Cu	2	2
Fe	50	2
Ga	—	Internal standard
K	1 000	100
La	—	20
Li	1 000	—
Mg	10	2 000
Mn	—	1 000
Mo	10	20
Na	1 000	1 000
Ni	10	200
P	30	—
Pb	—	60
Si	30	10 000
Ti	—	1 000
U	60	200
V	1	6
Y	—	10
Zn	5	10
Zr	—	20
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Number of analytical channels	18	22

^aAssumes 50:50 mixture of sample with graphite.

Table 4. NURE Phase II computer system.

Digital Equipment Corp. 11/70-YA computer system consists of:

PDP-11/70 central processing unit
High-speed cache
Real-time clock
128 K words of memory
Bootstrap loader
LA36 DEC writer
Serial interface
RWP04 disc system (80×10^6 bytes)
TWU16 magnetic-tape system
FP11-C floating-point processor
CR11 card reader
LV11 printer/plotter
DH11-AE multiplexer
H960-DH cabinet
TU-16-EE magnetic-tape unit
RX11-BA dual floppy system
QR300-AD IAS operating system
QP100-AD FORTRAN IV plus
QP375-AD DBMS-11
QP010-AD COBOL-11
QP600-AD SORT-11
QJ916-Ad BASIC-PLUS-2

- Installation of the HT-INAA system is on schedule. Most major components of the system have been received. Also the new delayed-neutron counter and in-core irradiation facilities were designed and ordered.
- The Phase II computer system was approved for purchase with FY 1976T funds.

Plans for First Quarter of FY 1977

The following activities are scheduled for next quarter:

- Begin processing Humboldt River Basin, six basin, and east Washington pilot-study samples.

- Complete installation of HT-INAA system.
- Receive and install emission spectrometer.
- Complete clean-room, receiving, and sediment-processing laboratory building modifications.
- Place contract for sample preparation.

Plans for Remainder of FY 1977

- Complete automation of analytical facilities (HT-INAA, DNC, and ICP).
- Tie system components to central data-transfer system.
- Process — 10,000 samples through INAA
— 2,000 samples through emission spectrometry.
- Validate system for the analysis of 25,000 samples per year.
- Install Phase II computer system and bring data-reduction operations on line during fourth quarter of FY 1977.
- Develop automated SO_4^- and Cl^- analyses.

INFORMATION MANAGEMENT AND REPORTING

Site Data Base

Progress on the site data base included expanding the site interface file to include additional field measurements, entering data from sites visited in the four basin pilot studies, and producing the four subsequent open-file reports.

Site locations, currently reported in the Universal Transverse Mercator (UTM) grid system, must be converted to longitude and latitude to produce a national NURE number for each sample. A program to convert the UTM measurements to longitude and latitude was obtained from Argonne National Laboratories and converted to run on LLL's main computers.

A map-digitizer and graphics system were ordered for the NURE program. This system will be used to enter site locations into the minicomputer database-management system.

Sample-Analysis Data Base

Progress on the sample-analysis data base included extending the data base to include conductivity, chlorides, and sulphates for water samples. Tables were also produced for the four basin pilot-study open-file reports,

and a computer-readable magnetic tape containing the data in the Walker River Basin Pilot Study report was sent to Oak Ridge National Laboratory for entry into the National NURE data base.

Programs have been written to statistically and graphically analyze data in the sample-analysis data base. Specific analyses during the past quarter included means, variances, standard deviations, frequency graphs, and Pearson-product correlation coefficients.

Data-Base-Management System

A PDP 11/70 was ordered from Digital Equipment Corporation in FY 1976. This minicomputer system was chosen via open solicitation and will be used to acquire data from the INAA, DNC, and optical-emission-spectrometry systems. The computer will be used to process and store data and to produce reports and graphs. For simple, fast access to the data by multiple users, data-base-management software (DBMS-11) was ordered with the minicomputer system.

Sample Control

During the anticipated lifetime of the NURE program, several hundred thousand separate packages of historical and analytical samples will be generated, analyzed, and stored. Therefore, a suitable sample control system must be implemented so that samples can be located in the system efficiently and reports issued to advise the staff of the status of sample flow. This requires that every sample package in the system have a unique identifier, that changes in the status of the material (movement, splitting, processing) be reported to the data base, and that a reporting system notify appropriate persons of the current inventory or changes in inventory.

An existing sample-location-and-tracking system, developed by the Special Materials Group at Livermore, will be used for this purpose. The system employs a "traveler card" that accompanies the sample package and a computer-based reporting system. The data base is notified of changes in the status of the package or sample by means of "transactions." The traveler card provides a convenient computer-readable sample identifier at the time of the transaction. Information on the traveler card along with the appropriate transaction code will be transmitted to the data base via dataphone links. The use of dataphones, which transmit information by acoustical signals over commercial telephone lines, will provide considerable flexibility

in locating reporting stations both inside and outside the Laboratory. The number of transaction codes (see Table 5) has been minimized to keep the operation simple and efficient. To reduce the number of times the data base is updated, some transactions are combinations of simpler statements. For example, transaction code 5 (new sample and shipment) can be used at the end of a processing cycle to notify the system of a combined code 1 (new sample) and code 4 (shipment).

Table 5. Transaction codes.

Code number	Transaction type
1	New sample
4	Ownership change
5	New sample and shipment
6	Off site shipment
7	Destruction (administrative)
8	Destruction (test)

A traveler card will be attached to a sample package the first time the box of samples is opened after being received from the field. This could be either by LLL or a contract sample processor. As the material is moved through the cycle, from processing to analysis and storage, the complete identifier number is changed and updated to provide a unique number for each package in the system. Field assignments for traveler cards are listed in Table 6. The location and type of field has been constrained to be compatible with the requirements of the existing materials-control system. Most of the definitions are self explanatory. A site-revisit signal (column 6) is required to cover the situation of drawing samples from a site where the previous site number and sample types taken are not known to the field sampler. The rabbit number (columns 75-80) is needed so that the rabbit can be reassociated with its traveler card following neutron-activation analysis. Source (columns 24 and 25) is optional, but can be used to associate samples with the contractor that did the work. Amount (columns 68-73) is a data-processing aid to the neutron-activation-analysis system in that it provides a computer-readable record of the quantity of material being analyzed.

Table 6. Traveler card field assignments.

Column	Name	Code	Type ^a
3-5	NURE Prefix	PFX	A
6	Site Revisit	R	N
8-13	Site Number	SN	N
16-17	Material Type (field)	TP	N
18-19	Treatment	TR	N
21	Field Replicate	FR	N
22	Analytical Replicate	AR	N
24-25	Source ^b	SO	N
68-73	Amount ^b	AMT	AN
75-80	Rabbit Number	RB	N

^a A = alphabetic, N = numeric, AN = alphanumeric.

^b Source and amount are not strictly required to identify a package.

The format and content of the reports to be issued have not yet been specified. However, as a minimum, samples will be reported by location (i.e., processing phase) and type.

Reports

Hydrogeochemical-survey-data reports were completed for five different pilot areas and forwarded to ERDA-GJO this quarter. The report on the Walker River Basin, an approximately 10,000-km² area in western Nevada and adjacent parts of California, was released to the public August 4, 1976. The other four reports presented data from surveys in Nevada playa basins selected to compare results from basins having presence or absence of uranium occurrences. The Winnemucca Dry Lake and Roach Lake basins are bordered by granitic and carbonate rocks containing known uranium occurrences. The Smoke Creek and Cave Valley basins are bordered by similar rocks, but without known uranium occurrences. All of these reports were open filed on August 18, 1976. The reports are entitled:

- *Hydrogeochemical and Stream-Sediment Survey (NURE): Preliminary Report on the Walker River Basin Pilot Study (Nevada), July 1, 1976, GJBX-40(76) [LLL No. UCID-16911-P-1].*

- *Hydrogeochemical and Stream-Sediment Survey (NURE): Preliminary Report on the Winnemucca Dry Lake Basin Pilot Study (Nevada), August 1, 1976, GJBX-41(76) [LLL No. UCID-16911-P-2].*
- *Hydrogeochemical and Stream-Sediment Survey (NURE): Preliminary Report on the Smoke Creek Desert Basin Pilot Study (Nevada), August 1, 1976, GJBX-42(76) [LLL No. UCID-16911-P-3].*
- *Hydrogeochemical and Stream-Sediment Survey (NURE): Preliminary Report on the Cave Valley Basin Pilot Study (Nevada), August 1, 1976, GJBX-43(76) [LLL No. UCID-16911-P-4].*
- *Hydrogeochemical and Stream-Sediment Survey (NURE): Preliminary Report on the Roach Lake Basin Pilot Study (Nevada), August 1, 1976, GJBX-44(76) [LLL No. UCID-16911-P-5].*

In addition, we expect to issue three technical reports during the last quarter of calendar-year 1976. Two of these reports are concerned with the percentage distribution of lithologies in the pilot survey areas mentioned above. The third, entitled *Sampling Natural Waters: Are Filtered Samples True Solutions?*, by J. W. T. Meadows, *et al.*, is a study of the stability of water samples after they have been filtered.

A computer-readable, magnetic tape containing the data in the Walker River Basin Pilot Study preliminary report was sent to Oak Ridge National Laboratory for entry into the NURE national data base. The tape was a nine-track, EBCDIC tape containing 10066 unblocked records with 80 characters per record. Each record was in one of the following two formats:

FORMAT 1

<u>Column</u>	<u>Type</u>	<u>Item</u>	<u>Format</u>
3-7	Integer	Site number	I5, right adjusted
9,10	Integer	Card type, = 10 for Format 1	I2
25-31	Alpha/numeric	UTM north-south measurement	A8
37-43	Alpha/numeric	UTM east-west measurement	A7

FORMAT 2

<u>Column</u>	<u>Type</u>	<u>Item</u>	<u>Format</u>
3-7	Integer	Site number	I5, right adjusted
9,10	Integer	Card type = 20 for Format 2	I2
12,13	Alpha	Sample type:	A2
		RO - rock sample	
		SE - wet-sediment sample	

FORMAT 2 (continued)

<u>Column</u>	<u>Type</u>	<u>Item</u>	<u>Format</u>
		SD - dry-sediment sample	
		WR - river-, creek-water sample	
		WL - lake-water sample	
		WW - well-water sample	
		WS - spring-water sample	
14,15	Alpha	Sample treatment:	A2
		NO - none	
		LA - acidified	
		DS - dried, sieved	
16	Integer	Duplicate number, = 0, 1	I1
19,20	Alpha	Element symbol:	A2, left adjusted
		U Uranium MO Molybdenum	
		SB Antimony ND Neodymium	
		AS Arsenic K Potassium	
		BA Barium RB Rubidium	
		BR Bromine RU Ruthenium	
		CA Calcium SM Samarium	
		CE Cerium SC Scandium	
		CS Cesium NA Sodium	
		CR Chromium SR Strontium	
		CO Cobalt TA Tantalum	
		EU Europium TB Terbium	
		AU Gold TH Thorium	
		HF Hafnium W Tungsten	
		HO Holmium YB Ytterbium	
		FE Iron Zn Zinc	
		LU Lutetium Zr Zirconium	
22-23	Real	Element concentration:	E12.3
		parts per billion (ppb)	
34-45	Real	Absolute error	E12.3

The records on the file were ordered by site number, card type, sample type, treatment, and duplicate number. Thus, for each site number, there was one location record with UTM coordinates followed by: element concentrations for the first sample from a site, element concentrations for the next sample from a site, etc.

Where a concentration was not reported for a given element, no element-concentration card was included. Thus, the number of cards per sample varied.

Plans for First Quarter of FY 1977

- Data on the field-visit forms for the Humboldt River Basin Study will be entered into the site data base and used to prepare status reports on types and quantities of samples collected and to produce "traveler cards" for use in the special-materials location-and-tracking system.
- Additional data from the Desert Research Institute will be added to the NURE sample analysis data base.
- The sample-tracking system will be implemented this quarter.
- The NURE Walker River and four-basin data will be analyzed by pattern-recognition techniques, using the PATTERN* Program.

*Bender, C. F. and Cox, L. A., *PATTERN: A Polyalgorithm for the Analysis of Generalized Data Sets - Principles and Practice*, Lawrence Livermore Laboratory, Rept. UCID-16915 (1975).

APPENDIX A
PRIMARY CONTACTS FOR LLL PORTION
OF NURE HYDROGEOCHEMICAL AND STREAM-SEDIMENT SURVEY

<u>Name</u>	<u>Responsibility</u>	<u>Mail stop</u>	<u>Phone ext.</u>
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-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
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APPENDIX B
UNIVERSITY OF CALIFORNIA
LAWRENCE LIVERMORE LABORATORY
SPECIFICATION UHS-002

1. GENERAL

1.1 Scope: This specification describes the requirements for the collection of water and/or sediment samples from approximately 450 specific locations in the following six drainage basins located in west central Nevada:

Buena Vista Valley
Pleasant Valley
Buffalo Valley
Granite Springs Valley
Kumiva Valley
San Emido Valley

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 University Furnished Supplies: The University will furnish the following materials and/or services:

1.3.1 The University will supply one set of maps and aerial photos showing sample site locations. These maps will consist of
(a) 7-1/2 minute quadrangles, (b) 15 minute quadrangles,
(c) 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)	Digital pH meters	(1 ea)
(2)	Conductivity meters	(1 ea)
(3)	Water Filter Apparatus	(1 ea)
(4)	Squirt Bottles	(3 ea)

(5)	Thermometers	(2 ea)
(6)	Polyethylene bags, 6×12	(500 ea)
(7)	Polyethylene bags, 12×18	(500 ea)
(8)	Stapler (Monel)	(3 ea)
(9)	Alkalinity and CO ₂ Kits	(2 ea)
(10)	Timer, pocket	(1 ea)
(11)	N ₂ 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	(1 ea)

1.3.3 The University will furnish the following expendable supplies:

(1)	Deionized Water	(10 gal)
(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 × 5 cards	
(8)	pH Buffers (4.0, 7.0 and 10.0)	
(9)	Labels	
(10)	Shipping Tags	
(11)	Kimwipes	

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. It is the Seller's responsibility to deliver equipment for repair and/or replacement to the field coordinator at the sample delivery location.

1.4 Seller Requirements: 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 The Seller shall provide a minimum of three sampling teams, which are to be broken down as follows:

(1) A minimum of two sampling teams for dry and wet sediments.

(2) One sampling team for water and wet sediments.

The teams are to work from a common base camp and not more than 50 miles apart and/or in the same valley or individual basin.

Note: Each sampling team shall consist of one or more persons.

1.4.3 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 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 for any and all claims for loss or damage resulting from Seller's sample collection activities.

1.4.4 The Seller shall maintain day to day contact with the University's Field Coordinator, who will restock equipment items and expendable supplies as required. These supplies will be available at the sample delivery location.

1.4.5 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.

1.4.6 The Seller shall take extreme care to leave all sample locations free of trash or debris of any kind.

1.5 Sequence of Operations: 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 60 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.

- (1) Take temperature of water with mercury bulb submerged in the water (Ref.: Para. 2.4).
- (2) pH calibration and determination (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 (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 and 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
 - l) save filter in plastic box provided
- (4) Conductivity (Ref.: Para. 2.7).
 - a) place cell in beaker of water
 - b) switch to appropriate scale and read
- (5) Carbon Dioxide (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 Phenolphthalein indicator in reaction vessel

- d) titrate with standard base to end point
- e) record CO₂ as mg/liter
- (6) Total alkalinity (Ref.: Para. 2.8)
 - 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 Phenolphthalein indicator in reaction vessel
 - d) if solution turns pink, titrate with standard sulfuric acid solution
 - e) calculate Phenolphthalein 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
- (7a) Dry Sediment (Ref.: Para. 2.3.1)
 - 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 (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, take a polaroid photo and attach shipping tag with label to adjacent vegetation

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 or aerial photos. In areas where 7-1/2 and 15 minute topographic maps are not available, locations will be shown on the best available maps or aerial photos. 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 inaccessible 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 maps/aerial photos furnished. 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 and aerial photos, 200 yards for 15 minute maps, and 300 yards for BLM maps that have scales of two inches to the mile. All field maps and aerial photos 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.

The Seller shall fasten one of the University-furnished yellow shipping tags to vegetation adjacent to the site. A label with the site number is to be affixed to the shipping tag.

- 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 Field Containers: 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 Sample Labels: The Seller will be supplied with self adhesive labels marked with sequential site numbers. 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. The Seller shall also mark on each sample the appropriate sample code using an indelible marking pen. The code is:

WR - Water, River

F - Filter

SD - Sediment, Dry or Damp

SW - Sediment, Wet

2.1.5 Site Visit Forms: The Seller will be supplied with site visit forms in triplicate (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 a copy of the form with the sample. Keep the original and one copy of the form filed separately and deliver it to the University's Field Coordinator when required, i.e., weekly. The sample location shall be identified by Township, Range, Section, 1/4 section, and 1/4-1/4 section. The 1/4 section and 1/4-1/4 sections shall be identified by the following code:

NE - A

NW - B

SW - C

SE - D

2.1.6 Field Coordinators: 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 must meet with the LLL Field Coordinator at least once a week and transfer the samples, site visit forms, photographs and completed maps to the Coordinator.

2.1.7 Quality Control: Independent LLL sampling teams will be operating in the area and will be resampling a number of sites for quality control purposes.

2.2 Stream Sediment Sampling: Previous studies suggest that the finer sized fraction (less than medium sand size) offer the best analytical sample to detect anomalous uranium concentrations. The Seller shall collect a representative sediment sample in sufficient quantity to insure at least 25 grams of the <100 mesh fraction will be furnished to LLL.

2.2.1 Dry Sediment

2.2.1.1 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 collapse 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

canyons or the apex of alluvial fans, but the samples must be taken from a recently active area.

2.2.1.2 Equipment:

- (a) Aluminum shovel
- (b) No. 3 sieve (1/4" square openings)

2.2.1.3 Sampling Procedure:

- (1) With clean aluminum shovel, collect a composite 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 weights 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.

2.2.2 Wet Sediments

2.2.1 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.

2.2.2.2 Equipment:

- (a) Aluminum shovel
- (b) Polyethylene Scoop

2.2.2.3 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 on 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.

2.3 Stream Water Sampling: 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 upstream. Recommendations for minimizing contamination are presented elsewhere in this specification. Water samples shall be taken only from flowing streams and from locations where it is possible to obtain a water sample free from "non suspended" sediments. Water samples shall not be taken from stagnant water.

2.3.1 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 in a polyethylene bag. Label the bag with the site number, time, and date.

To prevent a change in the water chemistry, the water sampling analysis must be done immediately adjacent to the water source. The seller is not permitted to transport the samples via motorcycle or similar conveyance to a central location for analysis.

- 2.4 Water Temperature: 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 $\pm 0.1^{\circ}\text{C}$.

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 $+ 0.1^{\circ}\text{C}$.
- (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 sealed buffer bottle in the sample source (caution, prevent contamination). Set the temperature compensation knob to the temperature of the source.
- (3) Calibration: 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 time 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 every 2 days or sooner if accidentally contaminated.

2.6 Filtration: 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 membrane-type 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) Dissassemble the unit by removing the nylon machine screws holding the top and bottom flanges.

- (2) Gently remove the porous support disk from the recess in the funnel. Do not 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, NEVER EXCEED 40 psig. 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 N₂ pressure bottles when pressure drops to 500 psig.

2.7 Electrical Conductivity: 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:

- (1) 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 beaker filled with water from the 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_2 Equivalent:

The concentration of the hydroxide, carbonate, and bicarbonate ions in the sample solution is determined by titrating with standard sulfuric acid solution to two specific ph's. A Phenolphthalein 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 immediately 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 Phenolphthalein indicator to the reaction vessel and swirl to mix thoroughly.

Note: Whenever indicator or reagent is added to water samples (as in the Alkalinity and Carbon Dioxide tests), the dropper bottle must be vertical when the drops are added to the water samples. This is to insure that all drops are of a uniform size.

- (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 blue-gray. 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 (step 10) times 20 to give total alkalinity as mg/liter CaCO_2 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 Carbon Dioxide: 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 8.3, 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 Phenolphthalein 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 instruction provided in each CO₂ kit.
- (2) Keep the standard base reagent sealed when not in use.

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