

2/6-13-78

LA-7175-PR

Progress Report

MASTER

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Issued: May 1978

**Hydrogeochemical and Stream Sediment Reconnaissance
of the National Uranium Resource Evaluation Program**

October—December 1977

**The Rocky Mountain States of New Mexico,
Colorado, Wyoming, and Montana
and the State of Alaska**

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The four most recent reports in this unclassified series are LA-6708-PR, LA-6831-PR, LA-6992-PR, and LA-6993-PR.

This work was supported by the US Department of Energy, Division of Uranium Resources and Enrichment. Program Code B048.

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HYDROGEOCHEMICAL AND STREAM SEDIMENT RECONNAISSANCE
OF THE NATIONAL URANIUM RESOURCE EVALUATION PROGRAM

October-December 1977

by

Wayne A. Morris, Merle E. Bunker, and David W. Steinhaus

ABSTRACT

Early in the quarter the scope of the HSSR for uranium was expanded by the DOE to include the analysis of thorium and lithium in sediment and several other elements selected by the LASL in either sediment or water. Development of the LASL multielement capabilities in the x-ray fluorescence, spectrochemical, and neutron activation techniques was accelerated to achieve the required throughput rate of 200 samples per day by each method early in FY 78. The LASL will report data for 43 elements in sediment samples and 13 elements in water samples. During the quarter, uranium analyses were determined for 7360 sediment samples and 7100 water samples. The priorities and format for HSSR reconnaissance reports were changed to reflect the new DOE guidance received early in the quarter. Reconnaissance report boundaries will coincide with those of NTMS 1:250 000-scale topographic map sheets; existing data bases are being split and combined to conform to these boundaries. Computer output formats and headers for the multielement listings to be used in LASL HSSR reports were developed and are ready to be used on the first multielement report. One reconnaissance report was open filed through the DOE Grand Junction Office. Two NTMS quadrangles were sampled as part of pilot studies; 39 of the 66 quadrangles assigned to the LASL in the lower 48 states have been completely sampled, and bid quotations were requested for sampling all or the remaining parts of the other 27 quadrangles during FY 78. Sampling specifications to be released for bid next quarter were prepared for the 35 quadrangles to be sampled in Alaska in FY 78.

I. INTRODUCTION

This report outlines the activities and progress of the Los Alamos Scientific Laboratory (LASL) in the Hydrogeochemical and Stream Sediment Reconnaissance (HSSR) for uranium, sponsored by the Department of Energy (DOE) Grand Junction Office, during the October-December 1977 quarter. The HSSR program, part of the larger National Uranium Resource Evaluation (NURE), is designed to identify areas favorable for uranium mineralization throughout the conterminous United States and Alaska. The ultimate goals of the NURE program are to provide a comprehensive uranium resource assessment of the entire country and to make the resulting information available on a timely basis to the private sector so that follow-up investigations of the more favorable areas can be conducted. The LASL is now responsible for the HSSR sampling in 66 National Topographic Map Series (NTMS) 1:250 000-scale quadrangles in the Rocky Mountain region of New Mexico, Colorado, Wyoming, Montana, and (since October of 1977) parts of Texas, Oklahoma, Arizona, Utah, and Idaho, and in 153 such NTMS quadrangles in Alaska.

II. REPORT OF PROGRESS

In 1976 and 1977 the LASL sampled 57% of its assigned area, completing 39 quadrangles (12 others are one-half finished) in the Rocky Mountain region (Fig. 1) and 27 quadrangles (4 others are one- to two-thirds finished) in Alaska (Fig. 2). In all, about 115 000 locations have been sampled, providing a total of about 83 000 sediment samples and 77 000 water samples; approximately half of these samples were collected in 1977. During the 1978 field season, all 27 partly or totally unfinished quadrangles of the LASL's Rocky Mountain region (Fig. 1) and about 35 quadrangles in Alaska (Fig. 2) will be sampled, leaving only 90 in Alaska to be sampled.

In October 1977 the DOE expanded the scope of the HSSR for uranium to include the analysis of sediment samples for thorium and lithium, and sediments and/or water samples for several other elements selected by each laboratory. The LASL plans to analyze for and report on 41 elements in addition to the 3 specifically requested by the DOE. Of these additional elements, 40 will be determined in the sediments and 12 (only 1 of which will not be determined in the sediments as well) will be analyzed for in the waters.

The multielement analyses greatly enhance the overall value of the HSSR program; several of the additional elements can be used to aid in the evaluation of the uranium data, while many are important in themselves as essential raw materials. In effect, the HSSR will provide data that can be helpful in assessing the long range mineral resources of the US, both for strategic and energy planning and for general industrial and economic growth.

Survey Work

Some sampling and other field work were done during the quarter. As parts of pilot studies, the University of Colorado collected samples from the Limon 1:250 000-scale quadrangle in northeast Colorado and the University of Montana sampled the White Sulphur Springs 1:250 000-scale quadrangle in central Montana. Two LASL staff members sampled the Grand Teton National Park area in northwest Wyoming to complete the LASL's sampling responsibility in the eastern half of the Driggs quadrangle; that data will now be open filed

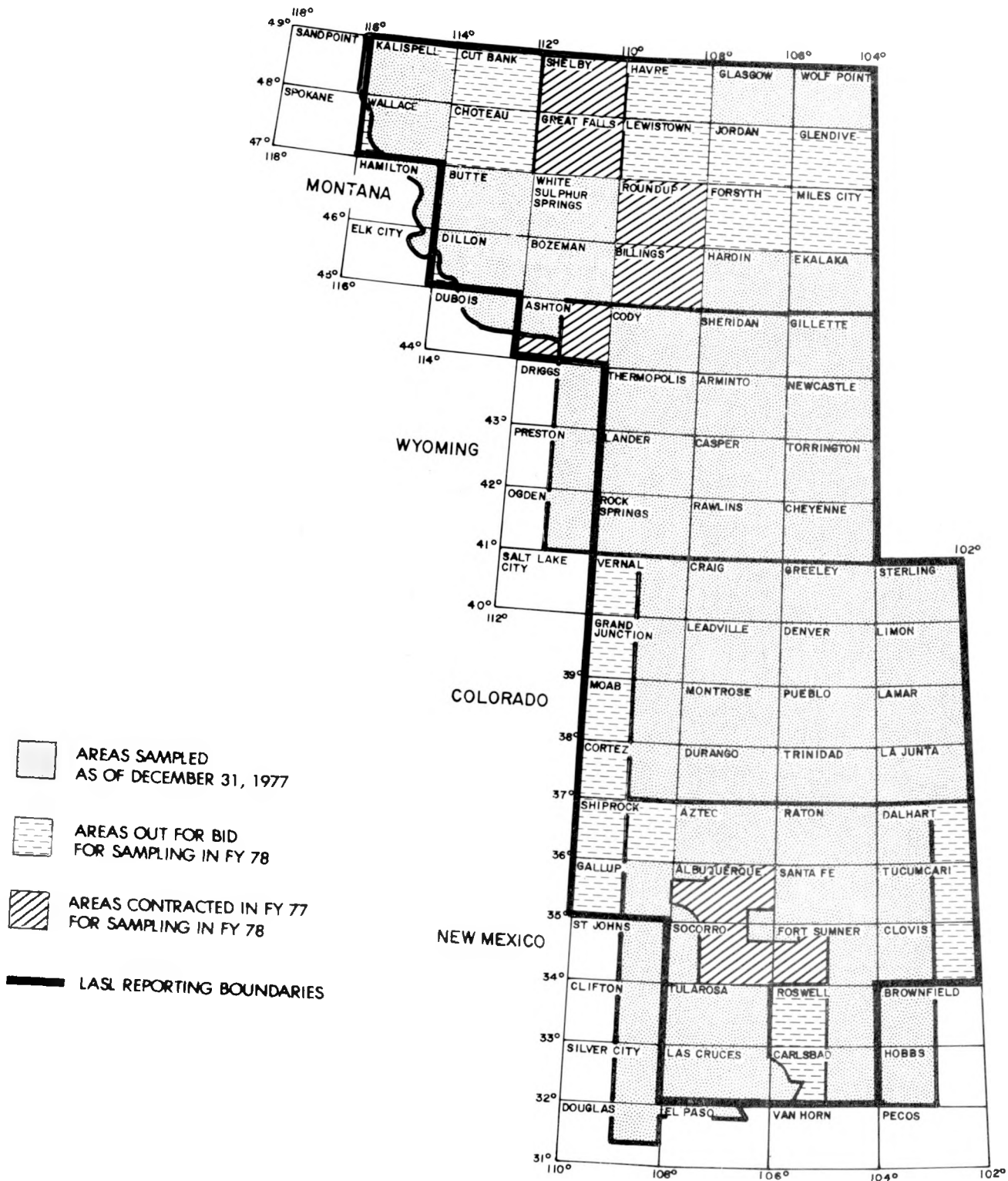





Fig. 1. Sampling status in the LASL's conterminous four states.

-  AREAS SAMPLED AS OF SEPT. 30, 1977
-  FOUR AREAS TO BE LET FOR BID FOR SAMPLING IN FY 78
-  AREA CONTRACTED IN FY 77 FOR SAMPLING IN FY 78

50 0 50 100 150 Miles
50 0 50 100 150 Kilometers

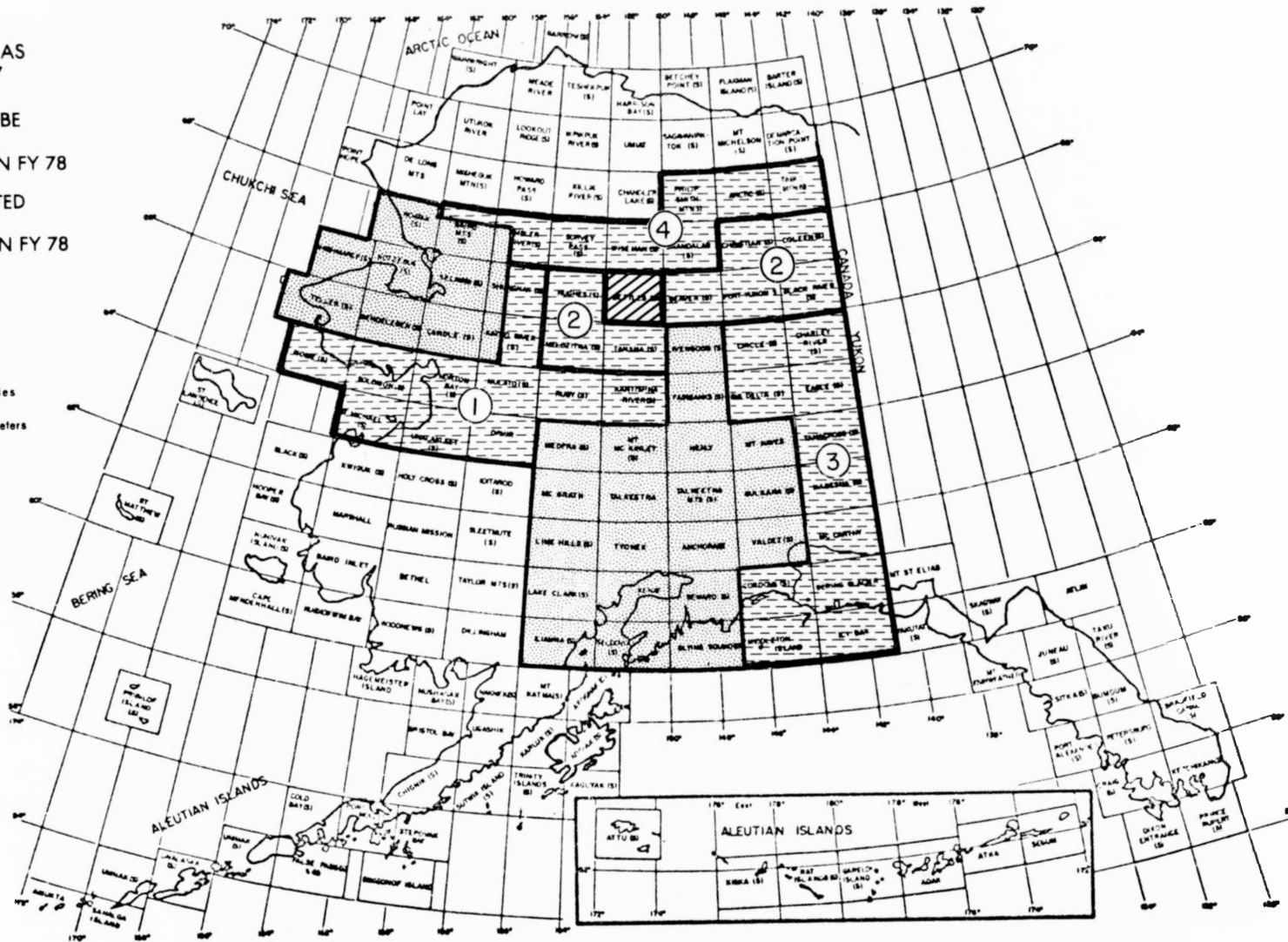


Fig. 2. Sampling status in Alaska.

and made available to the Lawrence Livermore Laboratory, which--under the revised area allocations made by the DOE in October 1977--is now responsible for the HSSR report on that quadrangle. Three LASL staff members made several reconnaissance flights over much of the 777 000 km² in Alaska that will be sampled in FY 78.

Bid Invitations

Bid invitations to sample eight contract areas (25 400 sample locations involving all or parts of 21 NTMS quadrangles) in the Rocky Mountain region during the spring and summer of 1978 were sent out to 23 geochemical and geological consulting firms. At the end of FY 78, when these areas and those of the six pilot studies contracted to state universities for FY 78 are completed, the entire area assigned to the LASL in the lower 48 states will have been sampled (Fig. 1).

Bid specifications were written for sampling four areas (about 30 000 sample locations covering 35 NTMS quadrangles) in Alaska in FY 78 (Fig. 2); the request for bids will be sent out early next quarter. In preparation for sampling these areas, the LASL has sent many letters requesting permission for access to private, native corporation, state, and federal lands in the proposed contract areas.

Publications

In September and October 1977 the DOE gave the LASL specific guidance on the reporting dates for quadrangles and the format of the reconnaissance reports. The most notable change was that the boundaries of report areas must coincide with those of the NTMS topographic map sheets (1:250 000 scale); except for reconnaissance reports that were essentially finished, those not covering full map sheet areas would not be accepted by the DOE. As a result, the work on several reports was stopped and shifted to the new priority quadrangles, and three reports covering most of western New Mexico, each nearing the final stages of preparation, had to be set aside temporarily because they did not cover entire NTMS quadrangles. The southeast Montana report, which covered the Ekalaka and part of the Hardin quadrangles, was rewritten as the Ekalaka report by separating out about 400 sample locations from the Hardin quadrangle.

Even with these DOE changes, the LASL goal for FY 78 is to open file reconnaissance reports for 11 quadrangles in the Rocky Mountain region and 17 in Alaska and to do the same with data releases for parts of 12 others (the equivalent area of 5 quadrangles) that were sampled and analyzed for uranium by the LASL and are to be reported as complete quadrangles by either the Oak Ridge Gaseous Diffusion Plant or the Lawrence Livermore Laboratory.

A LASL reconnaissance report, Uranium Hydrogeochemical and Stream Sediment Reconnaissance of the Northern Part of the Powder River Basin, Wyoming, by Wayne A. Morris, was open filed on November 25, 1977, by the DOE Grand Junction office as GJBX-94(77).

Meetings and Presentations

On November 28, 1977, Robert R. Sharp, Jr., the LASL HSSR Program Manager, gave an invited presentation to the Alaskan Energy Office at Anchorage, Alaska. His talk outlined the national HSSR program and the LASL's approach to it and detailed the LASL HSSR activities in Alaska.

Clean Room Installation

After months of design and contractual delays, the HSSR program clean room was finally installed at the Omega West Reactor (OWR) site in December. Its facilities include two clean benches for sediment sample splitting and rabbit loading, a series of stainless-steel sinks for acid cleaning of reactor rabbits, and a fume hood for chemical operations. The final balancing of the HEPA-filtered air system has not been done because one of the main exhaust blowers was lost in shipment. A replacement blower is expected in late January 1978.

III. SAMPLE ANALYSIS

Uranium Analysis

The analysis of water samples for uranium continued using fluorometry for concentrations in the low parts-per-billion (ppb) range, an evaporative concentration fluorometric method for samples with less than 0.2 ppb, and delayed-neutron counting (DNC) for waters found to have greater than 10 ppb (or interference problems) by fluorometry. During the quarter, uranium concentrations were determined for 6780 water samples by fluorometry and 312 by DNC and for 7360 sediments by DNC.

A laser uranium analyzer (Scintrex UA-3), purchased about six months earlier and capable of detecting uranium directly from water samples in the 50-parts-per-trillion range, was received and its evaluation initiated. Also, a data acquisition system is being fabricated to process output from existing fluorometers or the UA-3 analyzer for automatic transmission of the uranium data to the LASL Central Computing Facility (CCF).

Multielement Analysis

Development of the LASL multielement capability was accelerated with the DOE expansion of the HSSR program to include analyses for thorium and lithium in sediments and other elements in both waters and sediments. The multi-element analyses of waters by a plasma jet-emission spectrographic method (12 elements) and of sediments by an energy dispersive x-ray fluorescence method (9 elements) were started at rates of between 60 and 100 samples per day. A Tracor Northern data analysis system was ordered to increase the x-ray fluorescence throughput to 200 samples per day. An arc-source emission spectrograph is being set up for analyzing sediments (2 elements), and work continued on the modification of the OWR neutron activation analysis (NAA) facility, which is designed to handle 200 sediment samples per day (31 elements). In all, 13 elements for waters and 43 for sediments will be reported.

Spectrochemical Methods. Approximately 1000 HSSR water samples were analyzed for calcium, cobalt, chromium, copper, iron, magnesium, manganese, molybdenum, nickel, lead, titanium, and zinc using the inductively-coupled-plasma source with a 3.4-m direct reading spectrograph. Major improvements were incorporated into the system to find and correct for interfering elements and to decrease the operator time per sample. For each sample, an exposure of 15 s is made under computer control. After the computer determines and reports the results on each element (corrected for the interfering elements), the operator may then save the data on a file, repeat the run, or abort it as

in the case of a control sample. High, off-scale results are flagged and the computer goes into a filter mode in which a 1% transmission filter is inserted between the plasma source and the spectrograph entrance slit. The analysis is then repeated, the high value is recalculated, and the new value is inserted in place of the previously calculated result. The only exception to this procedure is for samples with magnesium concentrations greater than 100 parts per million (ppm); these samples must be diluted with deionized water before the analysis is repeated.

The latest list of lower detection limits and interfering elements is given in Table I. Standard solutions, with the same nitric acid concentration as the HSSR samples, are used to calibrate this system and to determine the correction for the interfering elements. Even with the corrections, the lower detection limits will shift as a function of the composition of the water because of elemental interferences.

In the spectrochemical determinations of lithium and beryllium in sediment samples, the required sensitivity of 1 ppm for each element was achieved. In this procedure a representative portion of the dried sample is ground to a fine powder, 50 mg of the ground sample is mixed with 50 mg of a buffer consisting of one part graphite and one part SiO_2 powder, a 15-mg portion of the sample-buffer mixture is weighed and packed into a graphite electrode that is used as the anode of a 12-A dc arc in air, and an exposure of 80 s is made of the resulting spectra with a 3.4-m Ebert spectrograph; photomultiplier tube signals are used to measure the concentrations of beryllium and lithium. The lower detection limits are somewhat below the expected average value of 6 ppm for beryllium and well below that of 65 ppm for lithium. As with the spectrographic analysis of water samples, these lower detection limits will shift slightly as a function of the composition of the sediment because of elemental interferences.

Great caution and care are being used in starting to analyze the sediment samples; the first few will be run in duplicate with both photographic and photoelectric detection. As experience is gained, the photographic detection and the duplicate photoelectric analyses will be phased out. Standards (synthetic sediment matrixes high in SiO_2 with lesser amounts of Na_2CO_3 , MgO , CaCO_3 , Al_2O_3 , and Fe_2O_3) containing 37 impurity elements in known concentrations are used to calibrate this system.

A large, dual, direct-reading spectrograph is being constructed for versatile spectrochemical analysis; it will have complete optical wavelength coverage and many photoelectric detectors. Because all metallic elements will be observed during each analysis, the detection of and correction for interfering elements will be easier than with the existing spectrographs. The inductively-coupled-plasma source will be the first source to be used with this new instrument. When it is fully operational, there will be photoelectric detectors on several spectral lines for most elements of interest, an increased number of elements determined per run, better detection and correction for interfering elements, and (with an automatic sample changer) an increased number of samples run per day by each operator.

Neutron Activation Analysis. The pneumatic sample-handling facilities at the OWR were modified for the automated multielement analysis of sediment samples using four Ge(Li) γ -spectrum detectors. The new four-clip, 200-sample primary loader was installed; the delay loader, which delays samples between the delayed-neutron and initial γ -ray count, and the sequential unloader, which retrieves samples after several days of decay, have been tested; and the

TABLE I

PRESENT LASL LOWER DETECTION LIMITS FOR THE MULTIELEMENT ANALYSIS
OF WATERS BY SPECTROCHEMICAL METHODS

| <u>Reported Element</u> | <u>Lower Detection Limits (ppb)</u> | <u>Change From Last Quarter (ppb)</u> | <u>Interfering^a Elements</u> |
|-----------------------------|---|---|--|
| Calcium | 20 | None | Cobalt (3.2), Iron (7.1) Nickel (1.8), Titanium (3) |
| Cobalt | 55 | Decreased from 70 | Nickel (2.8) |
| Chromium | 25 | Increased from 13 | |
| Copper | 4 | Decreased from 6 | |
| Iron | 25 | Increased from 20 | Cobalt (20.8) |
| Magnesium | 2 | Decreased from 200 | |
| Manganese | 3 | Decreased from 5 | Cobalt (6.2), Iron (21), Nickel (1.9) |
| Molybdenum | 25 | None | Calcium (12.8), Silicon (5.1), Titanium (6) |
| Nickel | 25 | None | Titanium (6) |
| Lead | 200 | None | Calcium (53) |
| Titanium | 4 | Decreased from 8 | |
| Zinc | 50 | Increased from 40 | Copper (4.3), Nickel (4.2) |

^a The numbers in parentheses are the apparent ppb of the reported element that result from each ppm of the interfering element in the water sample.

new double irradiation port was installed in the OWR. This expanded transfer system should accommodate a throughput of 200 sediment samples per day, each sample being irradiated twice and counted three times (a delayed-neutron count to assay for uranium and two γ -spectrum counts to assay for thorium and 30 other elements).

The NAA control and data acquisition/analysis system has been upgraded. A new PDP-11/60 computer, which will be used primarily to analyze the γ -spectra, will be put on line as soon as a new disk for the existing PDP-11/20 is received. The two computers will transfer data to each other

through a parallel CAMAC-to-CAMAC data link at more than 100 K bits per second. The "uranium-only" analysis programs were modified to run with the expanded pneumatic system and a number of diagnostic sample-handling codes were written to move samples between all components in the reactor room and the equipment trailer. Modification of the multielement data acquisition and analysis program (RAYGUN) is proceeding satisfactorily. All software components of this code were tested and debugged, including an interim magnetic-tape "write" routine which will allow spectra to be analyzed using the LASL CCF rather than the OWR PDP-11/60.

Automated multielement analyses of sediment samples are scheduled to begin in late January 1978. During initial system tests, only one Ge(Li) detector will be employed and the throughput will probably not exceed 50 samples per day. As soon as the "bugs" are eliminated in this mode of operation, all four Ge(Li) detectors will be put on line, raising the throughput to about 200 samples per day. Planned modifications in the software will permit the analysis of water samples by DNC to proceed simultaneously with the multielement analysis of sediment samples by NAA. Also, the pneumatic and data-handling systems could easily be modified to allow the "uranium-only" assay of several hundred sediment samples per day by DNC without interfering with the multielement analyses.

IV. DATA MANAGEMENT

The existing data bases, which were initially set up for the sampling contract areas (the boundaries of which were established by several previous DOE areal priority systems), are being split and combined along the boundaries of the NTMS 1:250 000-scale topographic map sheets to comply with the DOE directive on report area boundaries. All future reconnaissance reports will cover areas conforming to these new boundaries. The first several will include analyses for uranium only; those with multielement data will follow as soon thereafter as possible.

The uranium and multielement data for the water and for the sediment samples will be published as separate appendixes to the LASL HSSR reports; the concentrations of the elements in the waters (in ppb) and in the sediments (in ppm) will be listed alphabetically by the chemical symbol and grouped by the analytical method used. The computer output format and headers for the multielement listings were developed and are ready for use on the first multielement report.

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