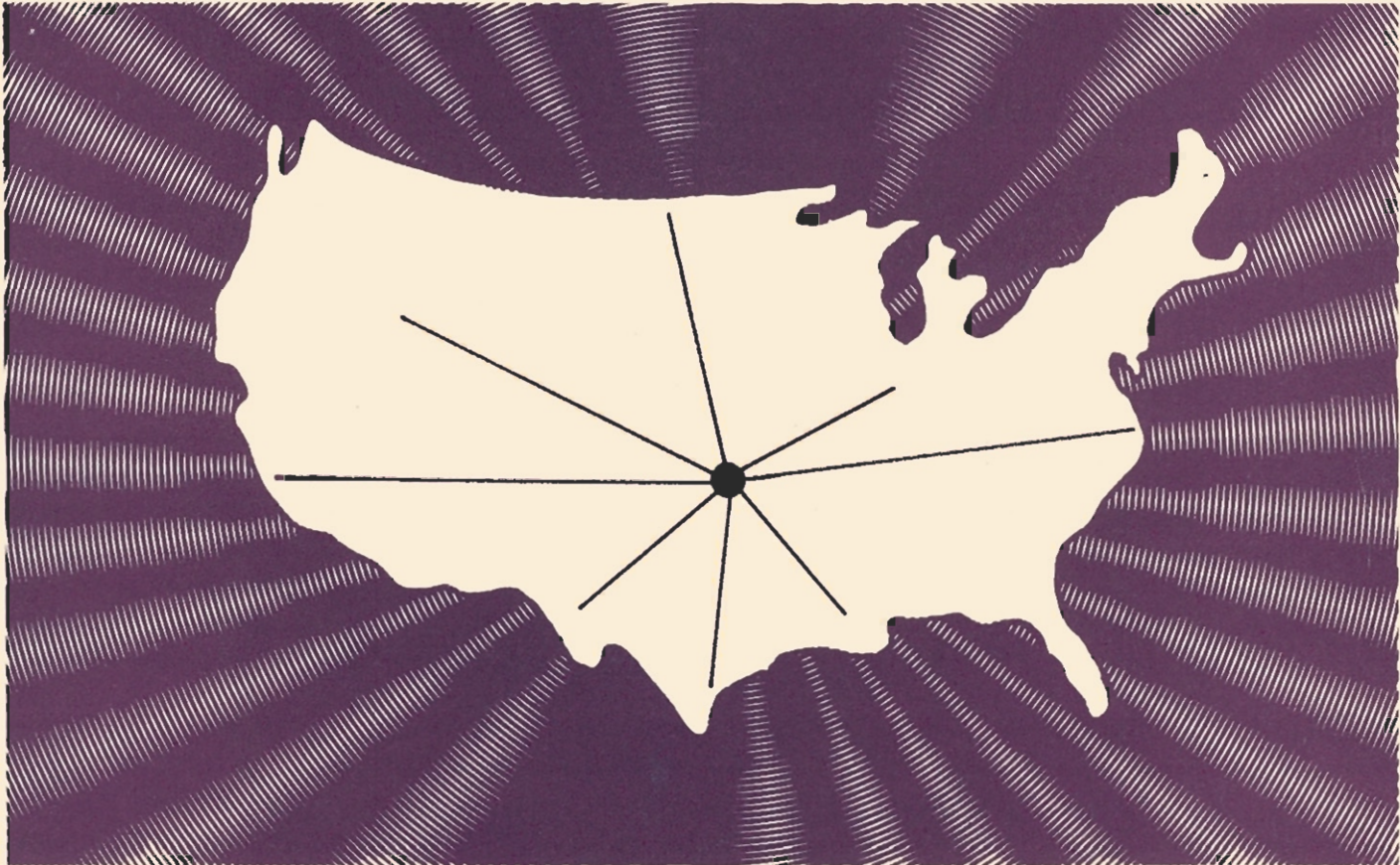




THE UNIVERSITY OF OKLAHOMA

...the completion of this history and
...the
**A TECHNICAL HISTORY
OF THE
NURE HSSR PROGRAM**
...with the
Engineering Corporation in Grand Junction
...for their assistance in locating inf
...a preliminary draft of this report



OFFICE OF INFORMATION SYSTEMS PROGRAMS

ENERGY RESOURCES INSTITUTE

**A TECHNICAL HISTORY
OF THE
NURE HSSR PROGRAM**

**A Technical History
of the
Hydrogeochemical and Stream Sediment Reconnaissance (HSSR) Program
of the
National Uranium Resource Evaluation (NURE)
1973 - 1984**

August 5, 1985

by

**Information Systems Programs
Energy Resources Institute
University of Oklahoma**

for the

**U.S. Department of Energy
Under Contract # DE-AC07-82GJ0176, Modifications # 003-005**

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ACRONYMS AND ABBREVIATIONS

Bendix	Bendix Field Engineering Corporation
BuMines	Bureau of Mines
cm	centimeter
DNC	Delayed Neutron Counting
DOE	Department of Energy
ERDA	Energy Research and Development Administration
g	gram
GJBX	Grand Junction - Bendix
GJO	Grand Junction Project Office, U.S. Department of Energy
HSSR	Hydrogeochemical and Stream Sediment Reconnaissance
ICAP	Inductively Coupled Argon Plasma
INAA or NAA	Instrumental Neutron Activation Analysis
ISP	Information Systems Programs
kg	kilogram
l	liter
LASL	Los Alamos Scientific Laboratory
LLL	Lawrence Livermore Laboratory
LPTR	Livermore Pool Type Reactor
μ	micron or micro
m	meter
mi	mile
ml	milliliter
NAA or INAA	Instrumental Neutron Activation Analysis
nm	nanometer
NTMS	National Topographic Map Series
NURE	National Uranium Resource Evaluation
ORGDP	Oak Ridge Gaseous Diffusion Plant
ORNL	Oak Ridge National Laboratory
ppb	parts per billion
ppm	parts per million
quad	quadrangle
sec	seconds
SRL	Savannah River Laboratory
USGS	U. S. Geological Survey
XRF	X-Ray Fluorescence

CHAPTER 1: INTRODUCTION

1.1 PURPOSE, SCOPE, AND AUDIENCE

The Hydrogeochemical and Stream Sediment Reconnaissance (HSSR) program of the National Uranium Resource Evaluation (NURE) generated a database of interest to scientists and other professional personnel in the academic, business, industrial, and governmental communities. NURE was a program of the Department of Energy Grand Junction Office (GJO) to acquire and compile geologic and other information with which to assess the magnitude and distribution of uranium resources and to determine areas favorable for the occurrence of uranium in the United States.

The HSSR program provided for the collection of water and sediment samples located on the 1° x 2° National Topographic Map Series (NTMS) quadrangle grid across the conterminous United States and Alaska and the analysis of these samples for uranium as well as for a number of additional elements. Although the initial purpose of the program was to provide information regarding uranium resources, the information recorded about other elements and general field or site characteristics has made this database potentially valuable for describing the geochemistry of a location and addressing other issues such as water quality.

The purpose of this Technical History is to summarize in one report those aspects of the HSSR program that are likely to be important in helping users assess the database and make informed judgements about its application to specific research questions. The history begins with an overview of the NURE Program and its components. Following a general description of the goals, objectives, and key features of the HSSR program, the implementation of the program at each of the four federal laboratories is presented in four separate chapters. These typically cover such topics as sample collection, sample analysis, and data management.

The primary audience for this report are those individuals or corporations unfamiliar with the details of the HSSR program, who intend to use the HSSR data and who may wish to read the history or note key references to understand the structure and quality of the data. However, even those who have used the data often may find the information presented here helpful in their work.

1.2 THE ROLE OF INFORMATION SYSTEMS PROGRAMS

The Information Systems Programs (ISP) of the Energy Resources Institute at the University of Oklahoma is working under a U.S. Department of Energy (DOE) contract to enhance the accessibility and usefulness of the HSSR data. ISP first completed the design and creation of a standard format computerized master file to replace the large number (over 800) of original data sets with varying formats. (See GJBX 42(83).)

In addition to this Technical History, ISP produced, under the second phase of the NURE contract, several data files with accompanying documentation to describe and summarize the HSSR data. These data files include an Analysis File containing reconnaissance level data expected to be of primary interest to users and special files of samples collected as part of detailed, pilot, or orientation level studies, and botanical samples or replicate samples. Supporting documentation for these files includes a data summary book

presenting selected reconnaissance-level data for the U.S., the individual states and NTMS $1^{\circ} \times 2^{\circ}$ quadrangles; a users' dictionary; and documentation of selected reports from users regarding their experiences with the HSSR data.

1.3 OVERVIEW OF TECHNICAL HISTORY

The following chapter introduces NURE and provides a broad description of HSSR program goals and implementation. Chapters 3 through 6 present in more detail the implementation of HSSR at each of the four participating laboratories: Lawrence Livermore Laboratory, Los Alamos Scientific Laboratory, Savannah River Laboratory and Oak Ridge Gaseous Diffusion Plant. The references identify reports that provide more detailed descriptions of various aspects of the HSSR program.

CHAPTER 2: THE NURE HSSR PROGRAM

2.1 INTRODUCTION

The National Uranium Resource Evaluation (NURE) program was initiated in the Spring of 1973 by the U.S. Atomic Energy Commission to assess uranium resources and to identify areas favorable for detailed uranium exploration throughout the United States. The NURE program, which spanned a decade, had several planned components including:

- Aerial Radiometric and Magnetic Reconnaissance;
- Subsurface Geologic Investigation Projects;
- Remote Sensing Projects;
- Geophysical Technology Development;
- Intermediate Grade Uranium Study Project;
- World Class Uranium Study Project;
- National Logging Program;
- Topical Geologic Studies; and
- Hydrogeochemical and Stream Sediment Reconnaissance.

This chapter provides an overview of the NURE program and then focuses in more detail on the Hydrogeochemical and Stream Sediment Reconnaissance (HSSR) program which is the source of all the data contained in the NURE HSSR Analysis File produced by Information Systems Programs at the Energy Resources Institute of the University of Oklahoma under contract to the Department of Energy.

Throughout the course of the NURE program, the organizational arrangements, objectives, and available resources changed in ways that affected the type and quality of the data generated from each of these components. The purpose of this chapter is to assist users of the data to place the HSSR program in perspective so that their expectations of the data will be grounded in reality.

The first section describes the goals and objectives of the NURE program. The second section focuses on the structure of the HSSR component of NURE, describing its purpose and scope, the organizational arrangements, the resources, and planned timeline. In the third section, methods of sampling and analysis, data collection and management, and quality control procedures are described. The following four chapters provide more detailed descriptions of how the HSSR program was managed within each of the four analytical laboratories.

2.2 OVERVIEW OF THE NURE PROGRAM

The national and international energy situation in 1973 generally suggested a promising future for nuclear power plants in the face of restricted and expensive fossil fuels for traditional electric power generation plants. Consequently, the U.S. Atomic Energy Commission (AEC) initiated a program to locate new areas for uranium exploration. Following the enactment of the Energy Reorganization Act of 1974, responsibility for this program was assumed by the Energy Research and Development Administration (ERDA) and subsequently by its successor agency, the Department of Energy (DOE).

In June 1976, ERDA produced a preliminary estimate of potential uranium resources that was based primarily on existing ERDA files and some reconnaissance level geologic field work (GJBX 11(77)). The broad goal of the NURE program was to provide the federal government with a more comprehensive set of data with which to make estimates of U.S. uranium resources. Figure 2.1 provides an overview of the conceptual model that guided the NURE Program. Some of the specific goals and target completion dates were (GJBX 26 (77)):

- to achieve a preliminary evaluation, based on existing data of domestic uranium resources and favorable exploration areas, by January 1976;
- to conduct a more comprehensive evaluation of domestic uranium resources and favorable exploration areas by October 1981;
- to develop new and improved procedures, equipment, and technology for uranium search and assessment; and
- to disseminate information on new exploration concepts and technology, and to identify new areas favorable for uranium exploration at the earliest date through publications, open files, documents, and seminars.

In addition to the activities of the HSSR program documented in this report, the NURE program sponsored other important activities of scientific or technological importance. Information available from those programs can be used to supplement the HSSR data or for other geoscience applications. A brief description of each program component follows.

Aerial Radiometric and Magnetic Reconnaissance: Initiated in 1974, this program was intended to acquire surface radiation data and magnetic measurements from airborne instrumental surveys for the uranium resource assessment effort and to identify as rapidly as possible the broad source regions of highest uranium favorability across the contiguous United States and Alaska. Results of the radiometric data from these aerial survey projects were open-filed and typically presented in the form of statistical anomaly maps by geologic unit, histograms, and flight-line stacked profiles of equivalent potassium, thorium, and uranium gamma values and their ratios.

Subsurface Geologic Investigation: These special studies were designed to evaluate subsurface stratigraphic data to confirm uranium resource estimates in known host areas, to obtain additional data for suspected host areas, and to evaluate reserves below currently depleted ore zones. NURE subsurface data were generated by drilling, logging, and analyzing formations; additional subsurface data have been obtained from industry records and by logging holes drilled by industry or other governmental agencies for purposes other than exploration through the National Logging Program (GJBX 11(78)).

Remote Sensing: This program investigated the possibility of using techniques for the interpretation of data from sources such as LANDSAT to meet the objective of delineating areas favorable for uranium exploration.

Geophysical Technology Development: This component was to provide new and improved instrumentation and techniques for uranium exploration and resource

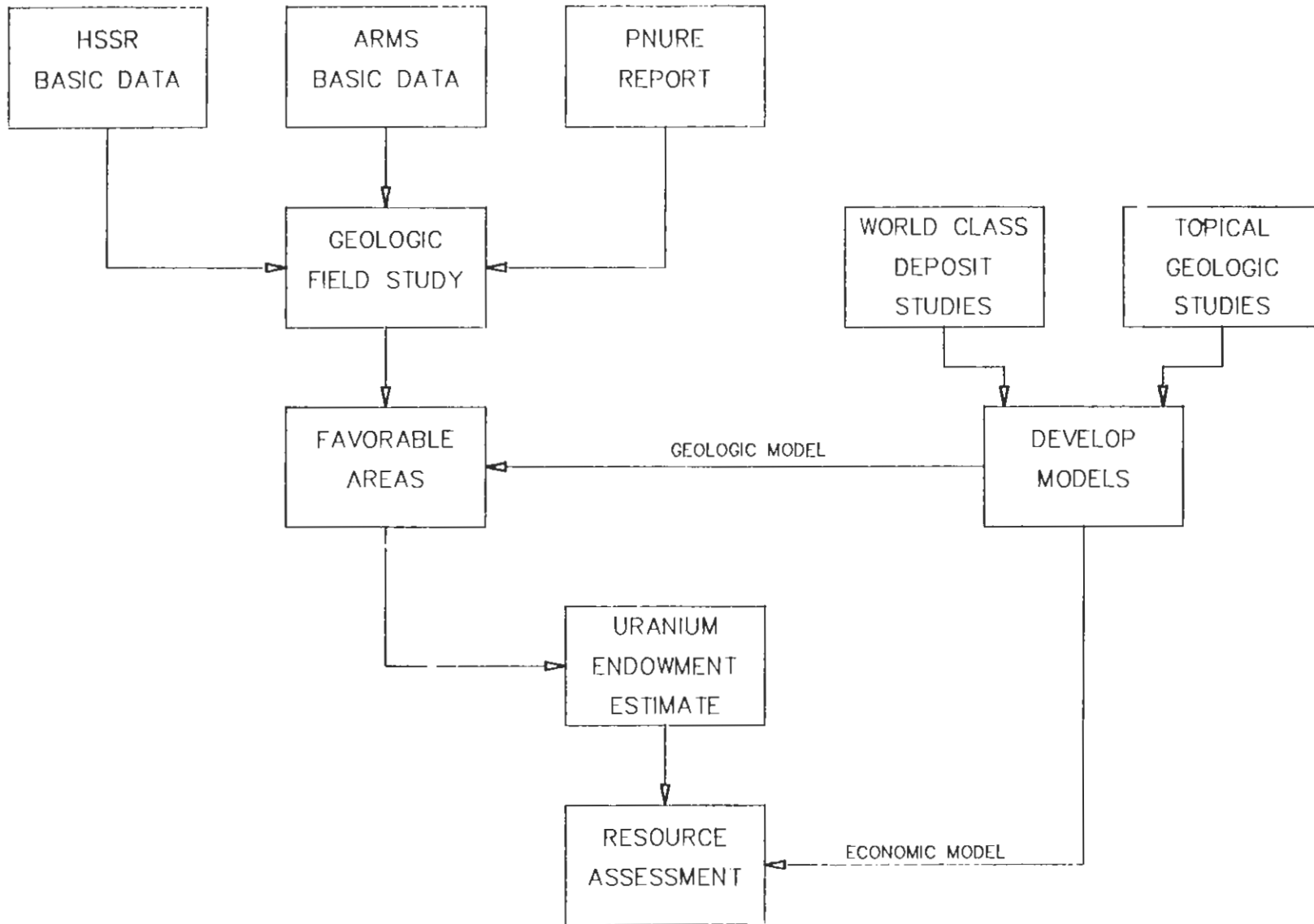


Figure 2.1 Schematic of Conceptual Model for the National Uranium Resource Evaluation (NURE)

Source: D'Andrea, Ralph F., Jr. 1985.

assessment in a range of geoscience areas including conventional and nuclear geophysics, geology, geochemistry, geostatistics, and remote sensing. Examples of activities under this umbrella include development of calibration models, provision of state-of-the-art geochemical analysis, and the improvement of geophysical methodology.

Intermediate Grade Uranium Study: This was a project to evaluate potential uranium resources containing between 0.01 and 0.05 percent U_3O_8 ; study areas included locations in Oregon, Nevada, Wyoming, Colorado and Arizona.

World Class Uranium Study: This project evaluated U.S. environments not yet identified as hosting significant uranium deposits but geologically similar to areas elsewhere in the world known to host economically important uranium deposits. Emphasis was placed on specific U.S. non-sandstone environments.

National Logging Program: This program relogged old boreholes, primarily oil, gas, and water wells, to look for evidence of uranium mineralization.

Topical Geologic Studies: A relatively minor component of the NURE program in terms of resources, these special studies were intended to enhance broad geologic knowledge, especially integrating subjective geologic evaluations into an overall framework of data interpretation based on information from the surveys and subsurface investigations. One major goal of these studies was to develop a framework for assessing uranium resources in locations other than sandstone formations in the United States.

Hydrogeochemical and Stream Sediment Reconnaissance: This major data collection effort was designed to sample systematically ground and surface waters and stream sediments in the U.S. This activity is covered in depth in the following section.

The NURE program also developed information management and dissemination mechanisms that supported the storage of NURE data on tapes and in printed format or microform in open-file reports and maps. This information dissemination function, including users services for sale of reports, maps, and data tapes, is now the responsibility of the U.S. Geological Survey (USGS). Reports and maps may be purchased from the USGS Open File Services Section, Building 41, MS306, P.O. Box 25046, Federal Center Denver, Colorado. Sales of data tapes are handled by the USGS EROS Data Center, User Services, Sioux Falls, South Dakota 57198. Facilities for reading-room examination of NURE reports and maps are available at the USGS Library, Denver West Bldg 3, 1526 Cole Boulevard, Golden, Colorado.

2.3 THE HYDROGEOCHEMICAL AND STREAM SEDIMENT RECONNAISSANCE PROGRAM

2.3.1 Purpose and Scope

As it was initially envisioned in 1974, the Hydrogeochemical and Stream Sediment Reconnaissance (HSSR) program was to sample "the Nation's surface waters, ground waters, and stream sediments, to determine variations and sources of radioactivity as guides for uranium search" (Bowyer, 1974, p. 5, as cited in Averett 1984a). Coverage of the United States was to be systematic rather than selective, with considerable discretion granted the laboratories in carrying out the survey. ERDA's Grand Junction Office was assigned

responsibility for providing project guidance and ensuring that the nation's goals and objectives for the program were met. An HSSR Technical Coordinating Committee met quarterly until 1978 to monitor, evaluate, and direct the program's implementation at a general level.

Initially, the sample analysis effort was limited to uranium. The analysis was expanded, however, to look at a number of additional elements that might have significance as pathfinders for uranium occurrences or that provided additional information about the geology and geochemistry of the sample site.

Within three years, program resource considerations and substantive concerns among geologists and federal policymakers led to a reevaluation of the concept of a broad, systematic survey and a decision to reduce the scope of the project. A Task Force, appointed in 1978 to assess the future direction and activities of the HSSR project, recommended that the remainder of the project field work concentrate more selectively on those "key favorable areas" for uranium resources in the United States that were expected to yield positive results. Thus, HSSR was redirected from a comprehensive and systematic survey to a more selective data collection and assessment.

2.3.2 Project Organization

The organizational meeting for the HSSR program was held in April 1975 and the sampling got underway during 1976. The primary institutions responsible for carrying out the HSSR program were the Lawrence Livermore Laboratory (LLL) in Livermore, California; the Los Alamos Scientific Laboratory (LASL) in Los Alamos, New Mexico; the Oak Ridge Gaseous Diffusion Plant (ORGD) in Oak Ridge, Tennessee; and the Savannah River Laboratory (SRL) in Aiken, South Carolina. The Ames Laboratory at Iowa State University in Ames, Iowa, conducted a limited review program on the uranium analyses run by the other laboratories. (See GJBX 240 (80) for a summary of the lab results.)

Each laboratory was given a specific area of the country for which it had responsibility for sample collection. (See Figure 2.2.) LLL had the Pacific Coast and Basin and Range States. LASL was assigned the Rocky Mountain States and Alaska. ORGD had responsibility for the Great Plains States, while SRL was responsible for the eastern and southeastern states. LLL participation in the program ended in 1979 and SRL was assigned the Western States. (See Figure 2.3.) By 1976, the labs had completed several pilot studies to identify appropriate sampling and analytical techniques. During 1977, 1978, and 1979, field sampling, data analysis and reporting were underway. (See GJBX 5 (84) for a guide to special studies and basic data reports.)

2.4 IMPLEMENTATION OF HSSR

2.4.1 Project Changes

Changes in the scope and organization of the HSSR stemmed from two principal sources. The first was that HSSR was one component of NURE, a very large, national energy program that evolved over a decade of rapidly shifting national energy goals. For example, NURE was initiated during a time of rising national concern over energy independence and under a U.S. energy policy system characterized by cooperation between industry and the government. Over the course of the NURE program three successive

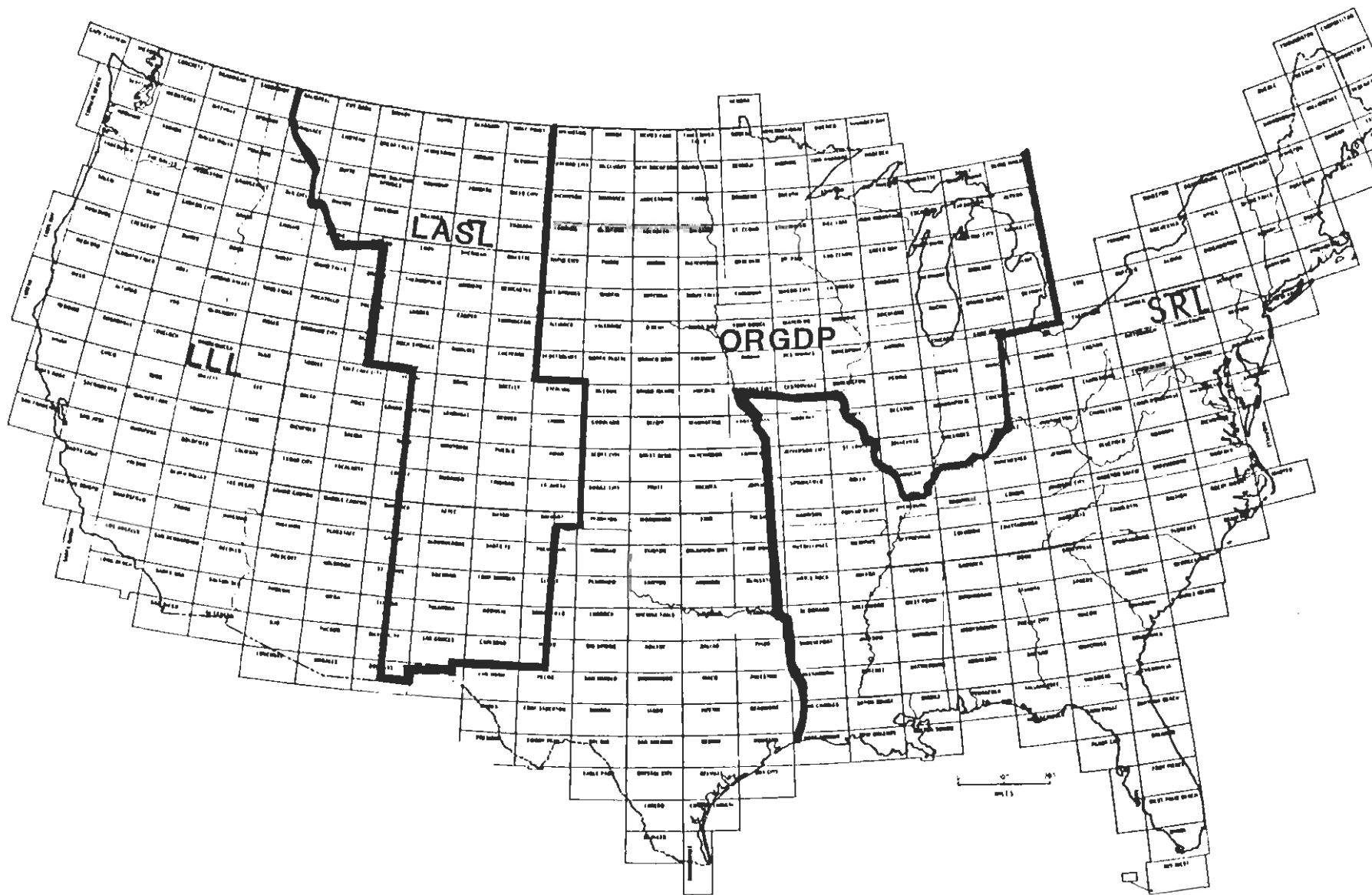


Figure 2.2 Initial HSSR Assignments of Laboratory Areas in the 48 Conterminous States

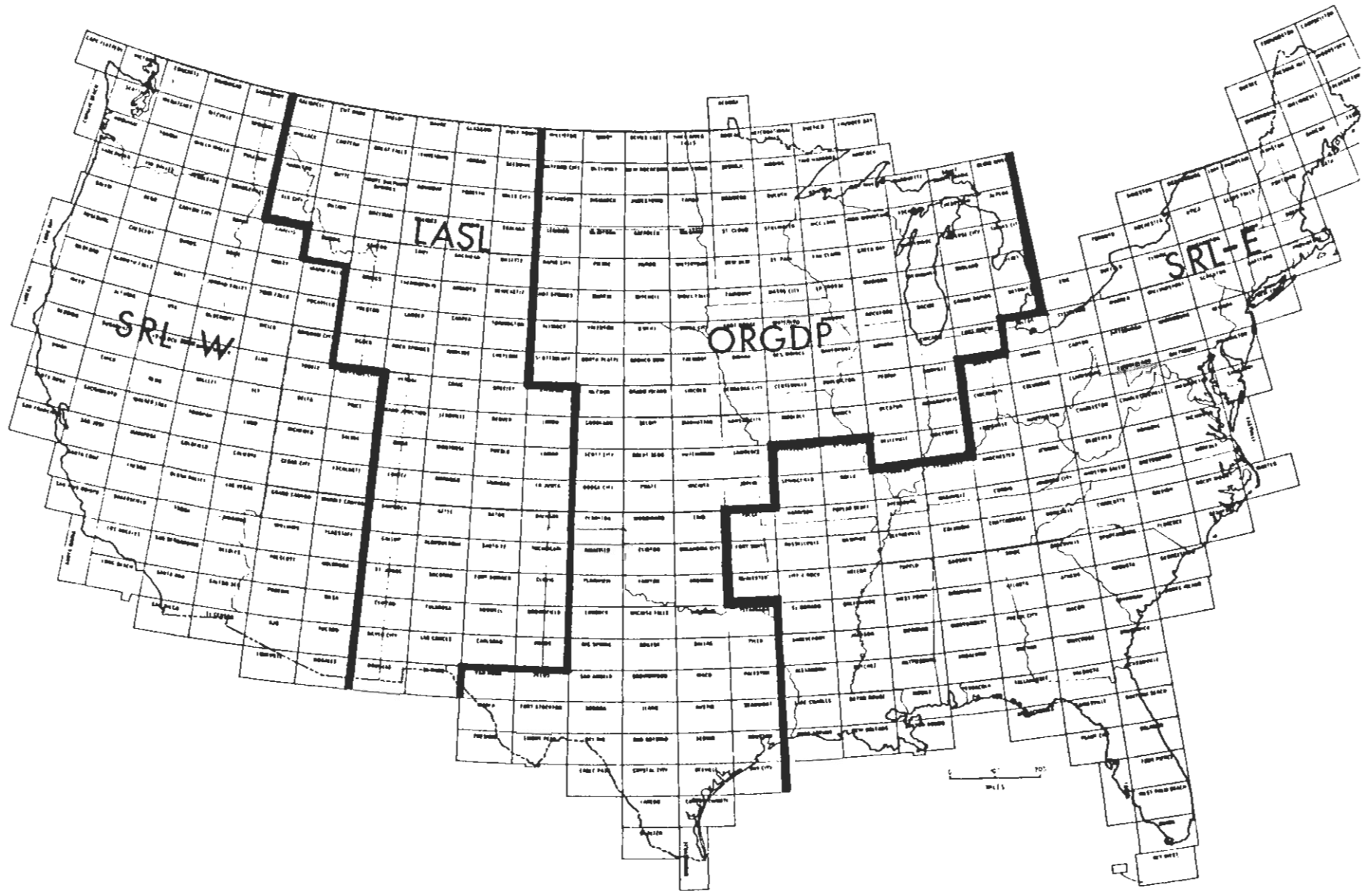


Figure 2.3 Final HSSR Assignments of Laboratory Areas in the 48 Conterminous States
Source: Averett, GJBX 5 (48)

presidential administrations (Presidents Nixon, Ford, and Carter) worked to develop a consensus regarding national energy goals in the face of unstable fuel supplies and rapidly rising prices. During this time, the federal institutional structure for managing energy policy underwent rapid change as the Federal Energy Administration evolved into the cabinet-level Department of Energy (DOE) in 1977. This institutional change was ultimately reflected in programmatic and budgetary revisions sought by the President's Office of Management and Budget and by DOE for individual energy-related projects. Strategic policy changes and resource constraints within NURE ultimately impacted the HSSR effort as well. Thus, the relative emphasis on certain features of the HSSR program changed to reflect the changes in national energy priorities.

A second key factor in HSSR program changes was the variation in the actual implementation of the program among the four laboratories. Although many changes occurred as the laboratories responded to new directives from the DOE Grand Junction Office (GJO), significant differences were also introduced by the laboratories themselves as they sought the most effective way to conduct their programmatic responsibilities. This variation in program development among the laboratories was encouraged by GJO in order to stimulate an imaginative and innovative approach toward meeting the HSSR objective of surveying the nation's surface waters, ground waters, and stream and lake sediments to aid in assessment of resources and identification of areas favorable for uranium exploration (Shannon 1977).

It is important to note in this connection that no consensus existed at that time within the scientific community regarding how best to conduct the sampling and analysis for the HSSR program. Geochemical exploration for uranium at the large scale and magnitude envisioned for HSSR was essentially a new undertaking characterized by the absence of well-defined theory or significant literature in scientific journals. In addition, the unique geographic and physiographic features in the United States indicated the need for some variation in sampling procedures. Thus, the imposition of a standardized approach to the implementation of HSSR during its early stages was considered premature by many of the scientific and professional groups involved in its development.

2.4.2 Sampling and Analysis

As a reconnaissance survey, the HSSR program was designed to search regions for signs favorable for uranium occurrence, using the minimum number and widest spacing of samples necessary to accomplish this task. Originally, the laboratories planned this sampling within their assigned states to correspond to drainage basins or other areas defined by criteria of geologic importance. Several pilot studies were conducted by the laboratories to assess their field and analytical measurement techniques, and sampling was initiated under this plan.

In late 1977, the laboratories were notified by DOE that all sampling and data reporting was to reflect a grid of $1^{\circ} \times 2^{\circ}$ quadrangles ($1^{\circ} \times 3^{\circ}$ north of latitude 59° affecting Alaska) corresponding to the 1:250,000 maps of the NTMS. (See, for example, Figure 2.3.) However, the laboratory boundaries were not immediately changed to reflect this; thus many quadrangles that crossed state lines were sampled only in one portion. Even after a lab was given responsibility for an entire quadrangle, the remaining HSSR funding and DOE priorities did not support a completed sampling of these quadrangles (Averett 1984a). Figures 2.4 and 2.5 indicate the completion status of each quadrangle at the end of the NURE project.

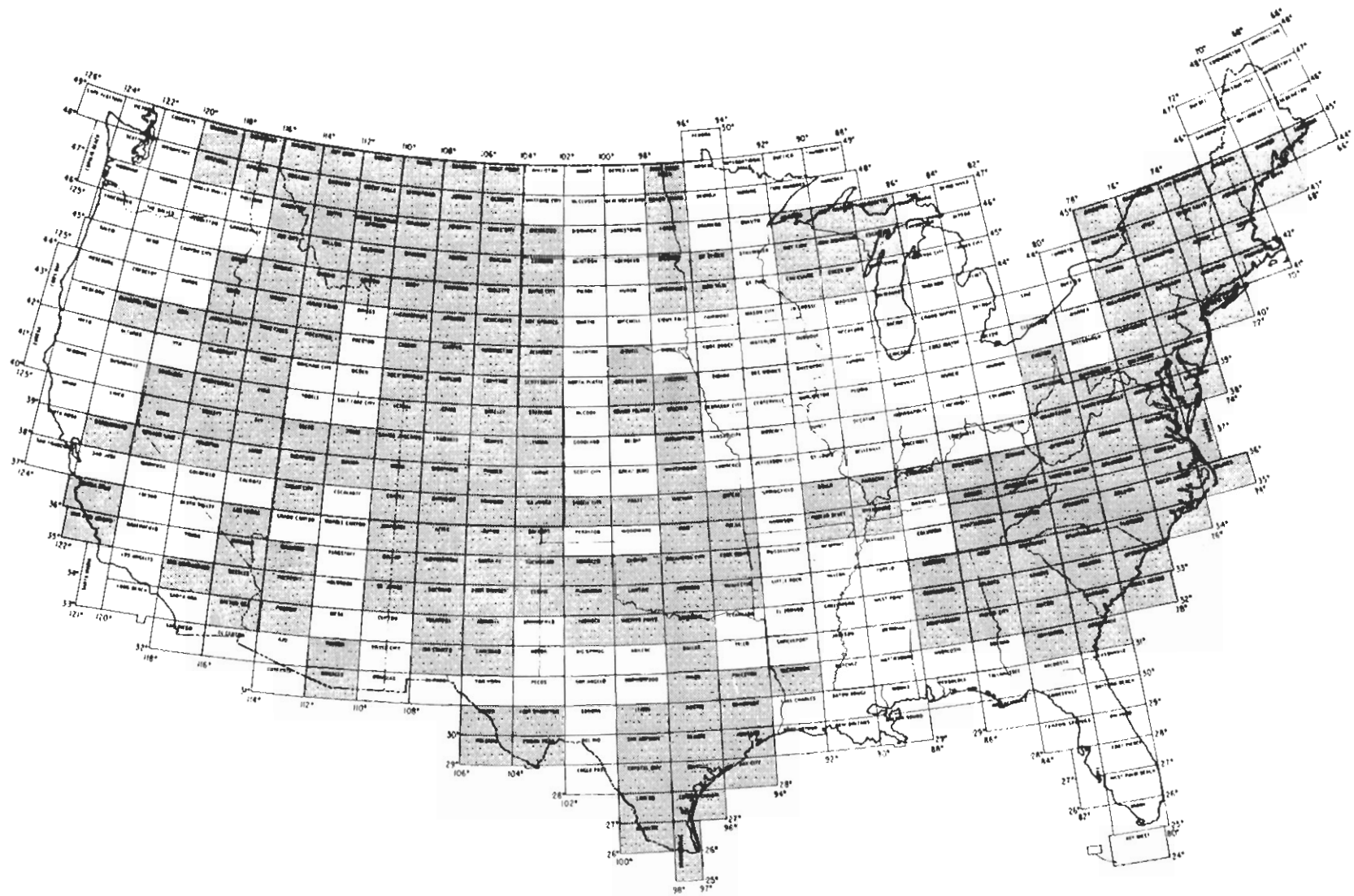


Figure 2.4 Reconnaissance Sampling Completed for the 48 Conterminous States at Termination of HSSR Survey
Source: Averett, GJBX 5 (84)

The reconnaissance sampling was typically performed by contractors and subcontractors, usually personnel from geologic consulting firms and university geoscience departments. Sample sites were marked by the laboratories on field maps that were digitized for site location at the laboratory. Three sample types -- stream, spring and lake sediments, surface water, and ground water -- were authorized for collection. Rock samples and botanical samples were collected at some sites that were designated for special studies. For example, early in the HSSR program, ORGDP collected botanical samples as a part of its orientation sampling program. Heavy sediment aggregates from USGS Wilderness Projects were collected in a few areas.

In general, the HSSR sampling teams were able to obtain permission to enter private, public, and Indian lands. Some individual land owners and Indian tribes denied permission for entry, but these omissions are not identified in the quadrangle reports. The National Park Service initially permitted sampling on its lands but this permission was withdrawn in 1979 (Averett 1984a).

Sample site densities typically ranged from four to fifteen square miles per sample site. A variety of field data was collected to characterize the sample site. Depending on specific laboratory instructions, sampling teams recorded information such as temperatures for air and water; scintillometer or gamma ray spectrometer readings; stream width, depth and flow data; descriptive data on wells; potential sources of contamination; local geologic formation; specific conductance and alkalinity; and date of collection.

Several analytical techniques were applied by the laboratories; these are presented in more detail in each of the chapters on the four laboratories. Table 2.1 provides a brief summary. Initially, samples were to be analyzed for uranium only. The principal method for sediments at SRL, LLL and LASL was delayed neutron counting (DNC) after a period of neutron irradiation. At ORGDP, fluorometric analyses were done on acid soluble solutions of all sediment samples, and DNC analyses were done on all sediment samples. ORGDP typically used fluorometry on all water samples, and isotope dilution mass spectrometry on water with a very low uranium concentration. LASL used DNC for uranium analysis in sediments and in water with high uranium concentrations and used fluorometry for water samples with ordinary or low uranium concentrations.

The analysis of elements other than uranium was authorized as part of the HSSR program in August 1977. Compared to the total HSSR program budget, the cost of this additional analysis was expected to be relatively inexpensive. In addition, scientists involved in the program believed that the results would be useful in interpreting the geochemical significance of uranium concentrations.

A central archive was established for the NURE HSSR samples, maps, and data forms at Oak Ridge Gaseous Diffusion Plant (ORGDP) in 1980; this archive has now been transferred to the USGS. The archive contains approximately 380,000 original sediment samples from all four laboratories, about 250,000 replicates, splits, size fractions or other samples and approximately 500,000 resin samples of waters collected by SRL (Birchfield et al 1981, as cited in Averett 1984b; Grimes 1985).

TABLE 2.1
ANALYTICAL METHODS USED IN THE NURE HSSR PROGRAM FOR INDIVIDUAL ELEMENTS, BY LABORATORY

METHODS OF ANALYSIS	SEDIMENT SAMPLES				WATER SAMPLES			
	LLL	LASL	SRL	ORGDP	LLL	LASL	SRL	ORGDP
Delayed Neutron Counting	U	U	U	U	U	U	U	
Fluorometry			U	U		U		U
Instrumental Neutron Activation Analysis	***	***	***	****			***	
Spectrophotometry								
Inductively Coupled Plasma - Source Emission Spectroscopy	***	***		***	***	***	***	***
Arc-Source Emission Spectroscopy		Be, Li		***				
X-Ray Fluorescence		Ag, As, Bi, Cd, Cu, Mo, Nb, Ni, Pb, Se, W, Zr	Th					
Atomic Absorption			***	As, Se, Sn			As, Se	As, Se
Gamma Ray Spectrometry			eK, eU eTh	eK, eU, eTh				
Mass Spectroscopy							He	He, U
Alpha Counting							Rn, Ra	Rn
Colorimetry			Nb, P, W		Cl, SO ₄	NH ₃ , NO ₃ PO ₄ , SO ₄		Cl, SO ₄
Flame Emission Spectrophotometry			K, Li					

Source: Averett 1984a

*** All remaining elements were analyzed by one of these methods.

**** All remaining elements were analyzed by this method in the first five quadrangles reported.

2.4.3 Data Collection and Management

Each laboratory designed its own data collection and management system, including forms used by the sampling teams, coding formats, database specifications, and software for creating, maintaining and utilizing the HSSR data. This approach was a viable one, allowing each laboratory to be responsible for its own data collection system; but the result has been some confusion for users of the tapes produced by different laboratories.

The basic approach after 1977 was to organize all data reports, whether in print or on tapes, by the 1° x 2° USGS quadrangles. At the end of the program, SRL summarized the eastern data by states; western data were incorporated into one report by quads. These print reports typically included an introductory geologic description of the area, sample location maps, geochemical symbol maps, and the results of the chemical analysis for each sample either in the print copy or on accompanying microfiche tables. Later in the program, limited funding led to reports with little or no text or maps. Data tapes typically contain only field measurements, site and sample identifiers and elemental concentration values. Each quadrangle report typically includes 1,000 to 2,000 site locations; but many include much fewer. (See GJBX 5 (84) for a series of maps indicating the source of the data reports for each quadrangle.)

The quadrangle data files were put on tapes by the laboratories and sent to ORNL or later to the Bendix Field Engineering Corporation (Bendix) in Grand Junction, Colorado. Bendix, the operations contractor at GJO, maintained a database for all four laboratories. Bendix was assigned repository responsibilities to receive, log, and store all tapes sent to it by the laboratories (including the tapes previously sent to ORNL) and make them available to those requesting the NURE HSSR data. More recently, Bendix developed users' aides to the NURE data, including an assessment of problems with the values or formats on existing data files, a compilation of formats used by the different labs and some summaries of the laboratories' activities. (See GJBX 36 (83), GJBX 3 (84), and GJBX 5 (84).)

2.4.4 Quality Control Programs

Each laboratory established quality control procedures for its laboratory analysis. The GJO also established a quality control program by contracting with the Ames Laboratory at Iowa State University in Ames, Iowa. The purpose of the program, initiated in December 1976, was to ensure that measurement variations among the laboratories were detected and controlled to acceptable levels, so that significant variations in uranium content of field samples could be discerned. Ames submitted standard water and sediment samples each month to the laboratories for uranium analysis. Water samples were prepared by adding known amounts of uranium to a natural water sample provided by SRL known to contain about 20 parts per trillion of uranium. Each batch prepared by the Ames Laboratory was sent to the Y-12 Plant in Oak Ridge, Tennessee, for uranium analysis by isotope dilution mass spectrometry to give the "true" concentration of uranium in order to facilitate comparisons with the results from the laboratory analysis.

The sediment samples used for quality control were obtained, homogenized, divided into smaller amounts and packaged to minimize segregation during storage. The "true" concentration was estimated from the accumulation of data from uranium analysis by the laboratories.

Attempts to establish similar quality control procedures for the multielement analysis were unsuccessful. The program ended in September 1980 with the issuance of a final report containing control charts of all determinations reported to Ames Laboratory and histograms of monthly statistics for each lab. (See GJBX 240 (80) for more details.)

Each laboratory also had its own internal quality control program covering a range of concerns from sample collection to data analysis and reporting. LLL and SRL, for example, conducted resampling of selected sites to check site mapping and the representativeness of samples for a specific site. LLL also analyzed splits of field samples to check processing and analysis. ORGDP did data edit checks for data recording errors, and also conducted a resampling program and a multielement quality control program. LASL also developed quality control programs to check field sampling analytical and coding problems; these programs included site visits and resampling (especially where the laboratory was concerned about a specific contractor), use of standard materials or blanks in analysis, and computerized edit checks on the data.

Concerns over the quality of the data collected and reported as part of the HSSR program stem from both the field and laboratory work. The laboratory chapters that follow describe some of the quality control procedures and reference laboratory documentation that describes the laboratory's field and analytical procedures.

CHAPTER 3: LAWRENCE LIVERMORE LABORATORY

3.1 INTRODUCTION

The Lawrence Livermore Laboratory (LLL) was assigned responsibility for hydrogeochemical and stream sediment reconnaissance (HSSR) of 1.8 million square kilometers in ten western states. (See Figure 2.2.) Lawrence Livermore Laboratory's participation in the NURE program began in 1975 and ended in 1979 before the entire assigned region could be sampled. About 38,000 sites in Nevada, Utah, Idaho, Arizona, California and Washington were sampled. About 30,000 of the samples collected were processed and analyzed by LLL, and data from approximately 13,000 sites were formally reported. However, many sample analyses had not been reported when LLL participation ended. (See Section 3.4.) The responsibility for most of the original LLL area was given to the Savannah River Laboratory (SRL); this effort has been referenced as SRL-West. (See Chapter 5.)

3.2 SAMPLE SELECTION AND COLLECTION

3.2.1 Site Selection

Sites were selected so that the sample would be representative of the area upstream from the point of sample acquisition and so that contamination by environmental pollution (farms, bridges, mines, etc.) would be minimized. LLL typically conducted a small pilot or orientation study of a geologic province to develop a sampling density sensitive to varying geological and climatic conditions.

A large part of the LLL region was within the arid to semi-arid Basin and Range province. Stream sediment sampling within this area was concentrated along the range fronts. For relatively small drainage canyons ($< 7 \text{ km}^2$), a sample was collected at the mouth of the canyon. For larger drainage canyons, sites were placed up the canyon and along tributary canyons to obtain representative coverage. Some sample sites were located in well developed stream channels in the flats and valleys to provide adequate coverage of the Pleistocene lake sediments. In areas outside of the Basin and Range province, small-order stream sites were selected to obtain an overall sample density of about one site per 13 km^2 . In the arid to semi-arid regions, ground water was the preferred sample type so that subsurface geochemical information could be obtained in areas of wind blown contamination. Stream sediment samples were taken only along the most active stream channels.

The average site density overall was about one site per 13 km^2 , although the actual site density varied between one per 8 km^2 and one per 18 km^2 because of differences in the scale of geological variability. Sites chosen were in drainage paths so that geochemical measures of surrounding highlands could be obtained. Areas excluded from sampling or areas where the sampling density was lower include the playas and large alluvial valleys. A few rock samples were also taken, primarily in pilot surveys.

LLL observers occasionally accompanied the contract sampling crews. LLL geologists resampled about 5% of the sites. More than 95% of the resampled sites were accurately located on available maps or photos. The remaining sites were plotted within 200 m of the correct locations. Analytical replicates were run on about 10% of all samples. (See GJBX 122 (78) and Higgins (1980) for details).

3.2.2 Sample Collection

A. Ground and Surface Water

Where water was available, water samples were always taken at a pre-designated site. Because of the geochemical importance of ground water, sampling was biased toward ground water samples when they were available. Water samples were collected through a 0.4 μ pore filter. A 1/2 l sample was acidified with nitric acid in a polyethylene bottle and split for uranium and selected cation analysis; a second 1/4 l unacidified sample was bottled for analysis of selected divisions. Surface water samples were hard to find at times. For example, during the 1977-1978 drought, sampling of about 18,134 km² of northern Arizona yielded one surface water sample (GJBX 122 (78)).

B. Sediments

About 2 kg of sediment material were taken at each site, with aluminum or plastic shovels, and placed in a double polyethylene bag.

3.2.3 Field Measurements

A. Ground and Surface Water

Where water was found, conductivity, pH, alkalinity, dissolved CO₂, and temperature were all measured. For well water, a measurement for dissolved oxygen and information about the type, depth and casing of the well were also recorded. Precipitation and stream flow were also recorded where appropriate.

B. Sediments

No special field measurements were taken at sites where only dry sediments were collected. If water was present, the measurements mentioned above were made.

3.3. SAMPLE ANALYSIS

3.3.1 Sample Preparation

Upon receipt at LLL, the sediment samples were oven-dried overnight at 100°C. They were split into two fractions and one was placed in archival storage. The other was sieved for 15 minutes, and, for arid to semi-arid regions, the portion passing a 1000 μ m sieve but retained on a 500 μ m sieve was collected. This choice was made to eliminate fine, windblown material while still keeping enough grains to form a representative sample. In moist regions (Washington and Idaho), a finer size fraction was used. The sieved portion was placed in a polyethylene bottle and blended by tumbling about an axis at 45° to the axis of the bottle. Approximately 1-3 grams were weighed into a 1.85 cm³ (1/2-dram) polyethylene vial, which was heat-sealed by fusing the lid to the polyethylene vials. After heat sealing, this vial was in turn inserted into a polyethylene container ("rabbit") and a lid was screwed in. Each rabbit carried a binary-coded number in the form of holes drilled into a band around its "waist." Prior to use, the rabbits were soaked in reagent-grade methanol and nitric acid, and washed in an ultrasonic cleaner. Handling of clean containers was done with gloves.

For water samples, a 20 ml aliquot was removed from each acidified sample to be used for elemental analyses other than uranium. The site number and volume were recorded for data processing when the aliquot was removed. The remainder was placed in an oven in a polyethylene bottle lined with a polyethylene bag until dry. The bag was then removed, rolled up and placed in a vial which was inserted into a rabbit for uranium analysis. The second field sample taken at water sites was untreated and used for chlorine and sulfate analysis. Rabbits containing dried residue from water samples were retained for historic archiving. Other samples and splits were discarded after validation of analyses.

Rock samples were processed at LLL. The samples were broken into pieces small enough to fit into a jaw crusher ("chipmunk") using a rock hammer or a small sledge and steel plate. The entire sample was passed through the crusher. If a sample was larger than about 2 kg, it was split using a Jones splitter, and one split was archived. The other split (less than 2 kg) was pulverized to -100 mesh (less than 150 μm). The sample was then placed in a clean 250 ml wide-mouth polyethylene bottle and blended in the same way as were the sediments. Cleaning of equipment was performed with compressed air after each sample was processed. After each sample was processed, quartz was passed through the equipment to clean it.

3.3.2 Methods of Analysis

A. Selection of Elements and Methods

LLL analyzed for uranium in both water and sediment samples by delayed neutron counting (DNC). Instrumental neutron activation analysis (INAA) was used to measure elemental concentrations of sediment samples. An automated optical emission spectrometer equipped with an argon plasma source was used to measure elemental concentrations of water samples. A modified spectrophotometric analyzer was used to obtain measurement of chloride and sulfate concentrations of water samples. Table 3.1 summarizes the elements analyzed and their detection limits by sample type and method of analysis.

B. Description of Methods

Instrumental Neutron Activation Analysis: Neutron activation analysis was used for both uranium and multi-element determinations in the sediment samples. The method involved irradiating a sample with neutrons for a fixed period of time in the core of the Livermore Pool Type Reactor (LPTR*), counting delayed neutrons emitted from the sample a short time later to measure uranium concentration, and counting gamma rays at short and long decay times to analyze for other elements. A total of eight Ge(Li) gamma ray detectors were used in this work. However, only four of these were used initially. The timing sequence used prior to January 1, 1979, is shown in Table 3.2.

Thimble #1 had a thermal neutron flux of about 3.1×10^{17} n/m²-sec, and an epithermal flux of about 1.2×10^{16} n/m²-sec. When the reactor was operating, a new sample

* The LPTR was deactivated in 1980; the reactor and neutron activation system described here no longer exist.

TABLE 3.1
RANGE OF DETECTION LIMITS BY SAMPLE TYPE FOR LLL

Element	Sediments ^(a) (ppm)	Water ^(b) (ppb)	Element	Sediments ^(a) (ppm)	Water ^(b) (ppb)
Ag	5 - 500	-	Mo	5 - 30	1
Al	16 - 50	6 - 16	Na	20	4 - 200
As	3	0.2 - 140	Nd	20 - 50	-
Au	0.002 - 0.01	.0005	Ni	2,000	8
B	-	16	Os	5,000	-
Ba	200	8	P	-	0.08 - 170
Br	0.8 - 5	1	Pb	-	15
Ca	5,000 - 20,000	2 - 1,000	Pr	1,000	-
Cd	4 - 50	1	Pt	1,000	-
Ce	15	-	Pu	0.00003 ^(d)	0.003 ^(d)
Cl	50 - 200	2,000 ^(c)	Rb	20 - 150	.5
Co	2	0.01 - 4	Re	10	-
Cr	5 - 50	0.05	Rh	100	-
Cs	1 - 30	0.01	Rn	0.5	-
Cu	5,000	1	Ru	100	0.05
Dy	0.5	-	Sb	0.5	0.01
Er	200	-	Sc	0.03	0.002
Eu	0.1	-	Se	5	-
Fe	1,000	2 - 10	Si	100,000	0.2 - 30
Ga	100	-	Sm	0.3	0.004
Gd	40	-	Sn	200	-
Ge	10,000	-	Sr	40 - 350	0.5 - 20
Hf	2	-	Ta	0.5 - 3	0.004
Hg	300	-	Tb	0.4 - 20	-
Ho	1	-	Th	2	0.005
I	8	-	Ti	10 - 1,000	1
Ir	100	-	U ^(e)	0.8	0.1
K	0.3 - 10,000	0.04 - 300	U ^(f)	0.01	0.036
La	0.3	-	V	0.5 - 10	2
Li	-	1	W	5 - 30	1
Lu	0.07 - 1	-	Yb	0.8 - 3	0.01
Mg	2 - 50	0.01 - 5	Zn	30 - 200	1
Mn	0.2 - 200	1	Zr	80 - 2,000	2

Source: Averett 1984a.

- a. Almost all elemental concentrations in sediments were analyzed by Instrumental Neutron Activation Analysis (INAA). Exceptions to this are indicated by footnotes.
- b. Most elemental concentrations in water were analyzed by a Plasma Source Optical Emission Spectrometer (OES). Exceptions to this are indicated by footnotes.
- c. Chloride analysis was performed with a Technicon Autoanalyzer (TM).
- d. Plutonium was analyzed by delayed neutron counting (DNC), if analyzed.
- e. The detection limits for uranium in sediments are for INAA; for uranium in water the method was DNC. (See Higgins 1980)
- f. The detection limits for uranium in sediments are for DNC; for uranium in water the method was OES. (See Higgins 1980)

TABLE 3.2.
LPTR TIMING SEQUENCE PRIOR TO JANUARY 1, 1979

<u>STEP</u>	<u>OPERATION</u>	<u>ELAPSED TIME (sec)</u>
1.	20-sec irradiation in thimble #1	20
2.	20-sec delay	40
3.	30-sec delayed neutron count	70
4.	18.4-min. delay	1170
5.	540-sec gamma count on one of four detectors, numbered 1 to 4	1710
6.	100-sec irradiation in thimble #1	1810 (30. min)
7.	1-week delay (approximately)	
8.	1800-sec gamma count (nights and week-ends, using detectors #1 to 4)	

Source: Van Konynenberg, McMillan, and Higgins, 1981.

entered the system every 3.48 min., giving a throughput of 17.2 samples per hour. After the last sample of the shift had entered the system, about 30 minutes had to elapse before this sample was ejected into the one-week storage bin, completing the first six steps. When the reactor was not operating, the system counted gammas from one-week-old samples.

At the end of 1978, the system was upgraded to include two thimbles in the reactor core instead of one, and eight Ge(Li) detectors rather than four. The new timing sequence is shown in Table 3.3. Thimble #1 had a thermal neutron flux of about 3.3×10^{17} n/m²-sec, and an epithermal flux of 1.2×10^{16} n/m²-sec. Thimble #2 had fluxes of 2.6×10^{17} and 1.2×10^{16} n/m²-sec, respectively. When the reactor was operating, a new sample entered the system every 2.5 minutes, giving a throughput of 24 samples per hour. After the last sample of the shift had entered the system, about 33 minutes had to elapse before this sample was ejected into the one-week storage bin, completing the first six steps. Most of the second counts were performed by the separate counting system, which operated 24 hours per day.

These timing sequences were arrived at by a compromise involving several factors:

1. High precision was desired for uranium and thorium detection.
2. Elements that would be helpful in elucidating the source and behavior of uranium had priority over other elements.
3. The throughput of samples had to be maximized to meet the program schedule.
4. The sequence chosen had to be amenable to continuous operation, allowing sufficient time for system maintenance.

TABLE 3.3.
LPTR TIMING SEQUENCE AFTER JANUARY 1, 1979

<u>STEP</u>	<u>OPERATION</u>	<u>ELAPSED TIME (sec)</u>
1.	20-sec irradiation in thimble #2	20
2.	20-sec delay	40
3.	30-sec delayed neutron count	70
4.	20.15-min. delay	1279
5.	562-sec gamma count on one of four detectors, numbered 1 to 4	1841
6.	120-sec irradiation in thimble #1	1961 (32.7 min)
7.	1-week delay	
8.	1800-sec gamma count usually using detectors #5 to 8)	

Source: Van Konynenberg, McMillan, and Higgins, 1981.

5. The sequence had to be compatible with the reactor operation schedule.
6. The maximum number of detectable elements was desired, other requirements being satisfied.

The approximately 30,000 sediment samples were irradiated, and gamma ray spectrometry was performed. The dried water samples were analyzed by irradiating them for a short time (30 seconds) and counting the delayed neutron emission to determine uranium. Twenty-four other elements in the water were measured on the RF plasma source optical emission spectrometer (OES).

Except for its high degree of automation, the unique feature of the LLL neutron activation analyses was the method of spectral data reduction. Spectral analysis was accomplished by removing back-ground, splitting complex peaks, and establishing tentative isotopic identities by comparison of the resulting spectrum with a library containing several principal gamma-ray energies and intensities for more than 60 activation product isotopes. A series of computer-controlled comparisons determined the best fit and evaluated the reliability and measurement error for each isotopic assignment. Only the more reliably identified and accurately measured isotopes were retained in further data processing. All elements were not identified in all samples. Detection limits varied from sample to sample, depending on the actual elemental composition. A few "unidentified" minor gamma-rays remained at completion of this analysis. These are due to statistical fluctuations in background, lower abundance (i.e., not part of the library) emissions from detected isotopes, and isotopes present below their "detection limits," as defined by reliability of assignment and measurement error criteria. Output of these analyses was the total number of atoms per sample of each identifiable activation isotope present at the end of irradiation (Leach 1985).

Additional computer programs converted the isotope atoms to elemental fractions of the sample using the sample weights, reactor flux, isotopic cross sections and abundances, and other factors. The programs also accepted the delayed neutron count data, cor-

rected the result for maximum interference, and subtracted fission product production where that was important (e.g., for molybdenum). In case there were two or more isotopes produced from a single element or an element was detected in both gamma spectra, results were weighted and averaged, and a single concentration number and error were estimated. Resultant data were the neutron activation analysis input to one of the data directory files.

Optical Emission Spectrography: Plasma-source optical emission spectrography was used to analyze the dissolved cations in acidified water or acid leaches of sediment samples. Aliquots of each sample were placed in an automatic computer-controlled sample changer which sequentially read an identifying computer card, transferred a premeasured amount of unknown solution to the plasma torch, rinsed the sample tube, and recorded the spectral output for twenty-four elements. This procedure was repeated three times for each sample, and the averaged results were recorded on a floppy disk. A standard solution was run after each sixth sample to measure long-term instrumental drift. The data were entered from the disk into a preprocessing computer where the sample composition was derived, considering such factors as the aliquot ratio and instrumental drift. The results were then transferred to tape and stored in another of the data directory files.

Chloride and Sulfate Analysis: The special chemistry, chloride, and sulfate analyses were performed with an instrument called a Technicon AutoAnalyzer^(TM) on unacidified water samples. This instrument automatically added chemicals to aliquots of samples and determined the anions by photo absorption analysis. These data were computer-processed and recorded on punched cards as concentrations of the ions. The cards are read into still another data directory file by sample number.

3.4 Data Management and Reporting

LLL issued a variety of reports in addition to the basic data reports, including reports on pilot and orientation studies, analysis for gold and silver, and description of equipment and analysis used by the lab. Data from about 13,000 sites were formally reported by LLL. Additional samples collected by LLL were placed in storage and later analyzed and reported by the SRL-West program. SRL only reported analysis on LLL samples for four quadrangles which still leaves at least 32 quadrangles which LLL sampled but for which no reports have been issued (Averett 1984a; Grimes 1985).

LLL geologists resampled about 5 percent of the sites for quality control and reliability and concluded that in 95 percent of the original samples, the location had been accurately mapped; the remaining 5% were within 200 m of the mapped site. The original sediment samples differed no more than 15 percent in trace element composition from the replicate set of sediment samples and had an average variance of 12% for uranium values. Water sample replicates showed a variance of 1% for uranium. During analysis of sediments by the laboratory, every tenth sample was split in half and both splits analyzed for quality control purposes. (See GJBX 89 (78) and GJBX 122 (78) for more detailed descriptions.)

CHAPTER 4: LOS ALAMOS SCIENTIFIC LABORATORY

4.1 INTRODUCTION

The Los Alamos Scientific Laboratory (LASL) had responsibility for 2.7 million square kilometers of the United States, including all of the Rocky Mountain States of Colorado, Montana, New Mexico, Wyoming, Alaska (see Figure 2.3), and parts of Idaho, Utah, Arizona, and Texas. Early in the program, the laboratory's area of responsibility was delineated along state lines, and several quadrangles were shared with either the Lawrence Livermore Laboratory (LLL) or the Oak Ridge Gaseous Diffusion Plant (ORGDP). When some of these quadrangles were assigned as complete units to LASL, other priorities and reduced funding levels in the later years resulted in incomplete sampling for these quads (Averett 1984a).

All sediment samples were initially analyzed by LASL; the water samples and some sediment samples were analyzed at ORGDP. LASL also analyzed some Alaskan samples collected by the U.S. Bureau of Mines (BuMines); the data reports for these samples were written cooperatively by the BuMines and Bendix (GJBX 33 (80) and GJBX 178 (81)).

4.2 SAMPLE SELECTION AND COLLECTION

4.2.1 Site Selection

The LASL based its program on published literature on geochemical surveys conducted in physiographic and climatic regions similar to its areas of responsibility; the actual sampling procedures were tested in a series of pilot studies.

The climate in the Plateau region and southern part of the LASL area is semi-arid; thus, although well water samples were collected when possible, a dry stream bed was often the only sampling medium available. The northern and interior sections of Alaska have extensive lake areas and consequently a high proportion of samples collected were lake water and sediment. In the southeastern parts of Alaska, which are rugged and heavily vegetated, streamwaters and sediments were most frequently collected (Bolivar 1980).

Each collection team was supplied field maps with the desired sample types and locations symbolically premarked at the LASL. The maps were normally USGS Quadrangles (either 7.5' or 15'). Where these were not available, Forest Service, State Highway, or other reasonably detailed maps were provided. As each location was sampled, a unique sample location number, preprinted on transparent adhesive labels provided with the identically numbered field data forms, was pasted over the site on the field map. The latitude and longitude of each location were computed by the sampling contractor.

Every location was later checked (and corrected if necessary) at LASL by overlaying computer-produced location plots on the field maps used. The latitudes and/or longitudes were corrected if the overlay locations were displaced by more than 300 m from the locations marked on the field maps. When a desired location could not be sampled as specified, an alternate sample type or location as near as possible to the original one was picked, and the new sample type and/or location was/were marked on the field map and properly labeled as above. Sample density was nominally 1 per 10 km² (1 per 4 mi²). In low-lying areas of Alaska with abundant lakes, the typical sample density was 1 per 23 km² (1 per 9 mi²).

4.2.2 Sample Collection

A. Ground Water

Ground water samples, collected directly from the source wherever possible, were filtered through a 0.45- μm membrane filter (except in Alaska where this step was omitted) directly into a prewashed and sealed, 41-ml reactor rabbit and 25-ml vial (both polyethylene). Ground water samples in both the rabbit and vial were then acidified to a pH of <1 with 8N reagent-grade HNO_3 . All sample containers were doubly labeled with pre-printed, adhesive labels carrying the same sample location number as that preprinted on the field data form. Springs were sampled as near to their point of emergence as possible. Well waters were taken near the wellhead if the well was pumping or from a holding tank if not.

B. Sediments

Following the collection of a water sample (if any), enough fine-grained, organic-rich, water-transported sediment to yield a composite sample of 25 g after processing (as indicated below) was taken from beneath the water level (where water existed) at three adjacent spots at each spring or stream location. The sediment was put into a new, clean, and originally sealed, rip-top polyethylene bag which was then double-labeled for delivery with the field data form to the contractor's drying facility. After drying at 100°C or less, each sample was sieved through a 100-mesh stainless steel sieve. The -100 -mesh fraction was put into a prewashed, 25-ml polyethylene vial which was then appropriately double-labeled (using labels from the data form) and sealed for shipment to the LASL. In the case of lakes sampled in Alaska, the sediment was taken from as near the center of each lake as possible by dropping a tethered, stainless steel bottom sampler overboard from a pontoon-equipped helicopter. Only a bottom sample from a single location was taken (i.e., it was not a composite). The sampler was rinsed before each use and the raw sample was put into a clean polyethylene bag, labeled, and treated as above.

C. Surface Water

Sample collection procedures for surface waters were essentially the same as for ground water. (See Section A above.) In the case of stream water, samples were taken from the fast-flowing current away from the bank. Ponds, including small lakes and reservoirs, were sampled just below the surface, near their center.

4.2.3 Field Measurements

A. Ground and Surface Water

The air temperature, read in the shade at the time of sampling, was recorded to the nearest whole degree Celsius. The water temperature was measured and recorded to the nearest one-half degree Celsius. The pH of the water was measured with a calibrated, portable pH meter and recorded to the nearest one-tenth of a pH unit. The conductivity (in $\mu\text{mho/cm}$) of the source water was measured with a calibrated, temperature-compensated (25°C), portable meter. The scintillometer readings were measured with a portable scintillometer on a flat, dry spot within a few meters of the sample location at well or spring sites. Two readings were recorded, the first with a radiation shield in place to

block out ground radiation and the second with the shield removed. The readings (in counts per sec) were converted by computer to give the equivalent uranium (eU) value set forth in the data listing.

Recorded field observations included very general descriptions of the local bedrock, sediment, water, vegetation, terrain, weather, possible contaminants, and water well configuration, all based on the best judgement of the field samplers.

B. Stream Sediments

The field measurements for sediments were essentially the same as for ground and surface water, except that measurements related to water at the site were omitted where the sample was taken from a dry stream bed, playa, etc.

4.3 SAMPLE ANALYSIS

4.3.1 Sample Preparation

To prepare water samples for uranium analysis by fluorometry, two NaF (98%)-LiF (2%) flux pellets were prepared and placed on platinum dishes in a controlled laboratory environment. The 25-ml water vial was vigorously shaken, and 0.20-ml aliquots of water were withdrawn and dropped onto the flux pellets. These aliquots were then evaporated under a heat lamp and the sample fluxes heated until fused and then cooled.

Initially waters with uranium concentrations greater than 10 ppb uranium were reanalyzed by the DNC method. This cutoff point was later increased to 40 ppb. The 41-ml rabbits were thoroughly cleaned (exterior) before analysis. Samples received in 25-ml vials (used exclusively in some of the early work) were transferred to clean, labeled, 41-ml rabbits before being analyzed. Each water sample was weighed, and its net weight and location number were recorded. The rabbits were then loaded into a 25-sample transfer clip. The reactor pneumatic transfer system and background radiation levels were checked, and the system was calibrated using four standards.

For DNC and INAA a split of each sediment sample was transferred to a clean 4-ml rabbit, weighed (less the tare), and recorded along with the appropriate sample location number. The readied rabbits were loaded into a 50-sample transfer clip. The reactor pneumatic transfer system and background radiation levels were checked, and the system was calibrated as above. Splits of sediment samples were prepared for additional analysis by grinding 6g of each -100-mesh sample to a -325-mesh powder. A 5-mg portion of the minus 325-mesh sample was analyzed by x-ray fluorescence and then mixed with 10 mg of a buffer consisting of one part graphite and one part SiO₂ for determination of beryllium and lithium by arc-source emission spectrography.

The single uranium concentration of the water samples given in the data reports was the average obtained from the duplicate aliquots. The lower limit of detection for each aliquot by the normal procedure was 0.2 ppb; some water samples from Alaska were evaporated to extend the detection limit to 0.02 ppb. Whenever the uranium concentration in a water sample run by fluorometry was given as some value less than 0.2 ppb or 0.02 ppb as appropriate, one of the two aliquots had a uranium concentration that was too low to detect. If the listed uranium value was 0.1 or 0.01 ppb, both aliquots were

below the detectable limit. Analytical precision at the lower limit of detection is $\sim 30\%$; however, it improved to $\sim 10\%$ at one order of magnitude above the lower limit. The basic fluorometric method as used for uranium at the LASL is described in more detail in GJBX 24 (77).

4.3.2 Methods of Analysis

A. Selection of Elements and Methods

Sediment samples were analyzed at Los Alamos by delayed-neutron counting (DNC), instrumental neutron-activation analysis (INAA), x-ray fluorescence (XRF), and arc-source emission spectrography (Table 4.1). The suite of elements analyzed varied over the course of the analytical effort. In the early stages of the program through the middle of 1978, samples from LASL quadrangles were analyzed only for uranium by DNC. Later, as funds for analytical work were increased, these samples were analyzed for a multi-element suite. All of the quadrangles that were originally analyzed for uranium only were later reanalyzed for a multi-element suite. For water samples, LASL normally reported only uranium concentrations. ORGDP later analyzed LASL water samples for its own multi-element suite and reported the results.

About midway through the analytical effort, improvements to the XRF system allowed the inclusion of arsenic, selenium, and zirconium to the multi-element suite; thus, results for these elements are available for about half of the quadrangles analyzed. The data falling above the detection limits were sparse, primarily because detection limits were very high (the analytical systems had not been optimized for these elements). Of the 46 elements included in the analysis (Table 4.1), 32 had detection limits below the average crustal abundance.

B. Description of Methods

Uranium Analysis: All sediments and waters with high uranium content were analyzed by DNC. For water samples, the typical cycle was a 60-sec irradiation, a 30-sec delay, and a 60-sec count. For sediments 20-sec irradiation, 10-sec delay, and a 30-sec count were used. The uranium concentration was determined by DNC, converted to ppb for water and ppm for sediments, and entered into a data base. Above the 1-ppm level, the standard deviation of uranium values in sediment samples was less than 4%. The specially designed delayed-neutron detectors, built by Los Alamos and used for these analyses, are described by Balestrini et al. (1976).

Instrumental Neutron Activation Analysis:* Concentrations of 31 additional elements were determined by INAA (see Table 4.1) The full DNC/INAA timing sequence used for each sample was: 20-sec irradiation, 10-sec delay, 30-sec DNC analysis, 20-min delay,

*During the course of compilation of "The Geochemical Atlas of Alaska" (GJBX 32 (83)), the authors noted that some elements displayed apparent concentration discontinuities at quadrangle boundaries. Because the NURE HSSR was primarily a uranium detection program, the INAA was not optimized for several of the trace elements. An early calibration error in magnesium caused the first seven quadrangles to have magnesium concentrations approximately 30% greater than actual.

TABLE 4.1
ELEMENTS ANALYZED IN SEDIMENT SAMPLES AT LASL

<u>Analytical Technique</u>	<u>Element(s)</u>	<u>Detection Limit (ppm)^a</u>	<u>Crustal Abundance (ppm)^b</u>	<u>% of Samples >Detection Limit</u>	
Delayed Neutron Counting	Uranium	0.01	1.8	99.98	
Instrumental Neutron Activation Analysis ^c	Aluminum	3200	81300	99.6	
	Barium	150	425	87.6	
	Calcium	1000	36300	89.8	
	Chlorine	50	130	n.d.	
	Dysprosium	0.7	3.0	92.8	
	Short-lived radionuclides counted 21 minutes after irradiation	Magnesium	2700	20900	90.4
		Manganese	55	950	99.9
		Potassium	3400	25900	83.0
		Sodium	1000	28300	99.9
		Strontium	400	375	5.8
		Titanium	750	4400	92.7
		Vanadium	6	135	98.1
		Antimony	1	0.2	5.0
		Cerium	10	60	93.2
Cesium		2	3	51.1	
Long-lived radionuclides counted 14 days after irradiation	Chromium	10	100	92.9	
	Cobalt	1.7	25	94.5	
	Europium	0.4	1.2	90.8	
	Gold	0.05	0.004	1.0	
	Hafnium	1.3	3	87.2	
	Iron	1100	50000	99.0	
	Lanthanum	7	30	79.2	
	Lutetium	0.1	0.5	81.2	
	Rubidium	13	90	n.d.	
	Samarium	0.4	6	n.d.	
	Scandium	0.9	22	99.5	
	Tantalum	1	2	2.4	
	Terbium	1	0.9	4.8	
	Thorium	1	7.2	90.7	
Ytterbium	1	3.4	60.9		
Zinc	100	70	46.4		
Energy-Dispersive X-Ray Fluorescence	Arsenic	5	1.8	73.9	
	Bismuth	5	0.2	9.9	
	Cadmium	5	0.2	n.d.	
	Copper	10	55	90.8	
	Lead	5	13	55.1	
	Nickel	15	75	76.6	
	Niobium	20	20	n.d.	
	Selenium	5	0.05	0.3	
	Silver	5	0.07	0.3	
	Tin	10	2	1.6	
	Tungsten	15	1.5	5.8	
	Zirconium	5	165	85.2	
	Arc-Source Emission Spectrography	Beryllium	1	2.8	70.0
		Lithium	1	20	99.3

^a Parts per million

^b Crustal abundances are average values from Mason (1966), pp. 45-46

^c The lower detection limits for INAA are a complex function of sample composition and weight. Here, the lower detection limits are average values calculated from typical 4-g samples (Minor et al., 1982).

500-sec gamma-ray count for short-lived radionuclides, irradiation for 96 sec, a delay for 14 days, and then a 1000-sec gamma-ray count for long-lived radionuclides. Gamma-ray counting was done by lead-shielded Ge(Li) detectors; 4096 channels of gamma-ray data were recorded and later interpreted for each individual element by computer. Elemental concentrations for each sample were printed out automatically, along with their associated statistical errors. Concentrations for which the statistical counting error exceeded 50% were considered below the detection limit of the system and marked as such. The detection limit reported for each element in INAA was a complex function of the total composition and mass of the individual sample. Therefore, the detection limits in Table 4.1 were average values calculated on the basis of typical 4-g samples (Minor et al., 1982). At concentration values exceeding one order of magnitude above the statistical detection limit for each element in each sample, the relative errors were generally less than 10%. The INAA system used at Los Alamos is described by Garcia et al. (1982) and Minor et al., (1982).

Energy-Dispersive X-Ray Fluorescence: A computer-controlled, energy-dispersive XRF system was used to determine elemental concentrations of cadmium, copper, lead, nickel, niobium, silver, tin, and tungsten, and in the latter portion of the program, arsenic, selenium, and zirconium. The system consists of an automatic 20-position sample changer, a lithium-drifted silicon detector, a pulsed molybdenum-transmission-target x-ray tube, a multichannel analyzer, and a minicomputer. A computer program was used to calculate the position of the 6 g samples in the x-ray beam, to unfold overlapping peaks, to determine peak intensities for each element, and to calculate the ratio of the intensity of each peak to that of the molybdenum $K\alpha$ Compton peak. Concentrations of each element were then calculated using equations obtained by analyzing prepared standards. Detection limits were: 5 ppm for Ag, Bi, Cd, and Pb; 10 ppm for Cu and Sn; 15 ppm for Ni and W; and 20 ppm for Nb. The relative standard deviation was 10% or less at the 100-ppm level and 20% or less at the 20-ppm level. Details of the method and equipment used are described in more detail in GJBX 52 (77).

Arc-Source Emission Spectrography: To determine beryllium (Be) and lithium (Li) by arc-source emission photography, a sample/buffer mixture was placed into a graphite electrode that was used as the anode of a dc arc having a short circuit current of 6 A for 10 sec, then 17 A for 50 sec. Photomultiplier tubes in a direct-reading spectrograph were used to measure the second order 313.0-nm line of Be, the first order 670.7-nm and 601.3-nm lines of Li, the background spectra near these lines, and the 327.6-nm line of V. The 670.7-nm Li line was used for Li concentrations up to 10 ppm, and the 601.3-nm line of Li was used for concentrations above 10 ppm. The vanadium (V) line was used to correct the Be value when V was present. The signals from the photomultiplier tubes were read by a digital voltmeter and were processed by a desk-top calculator. The results were simultaneously printed on paper and written on cassette tape for later transmission to a computer data file. The elemental concentrations of Be and Li were determined from the spectra, based on the results of previously run calibration standards. The lower detection limit for both elements was 1 ppm. Precision at the lower detection limit was $\sim 50\%$ for both elements and improved to $\sim 25\%$ at one order of magnitude above the lower limit. During the ending stages of the program Be and Li analyses were dropped to reduce costs as program budgets became more restrictive.

4.4 DATA MANAGEMENT AND REPORTING

The LASL reports on reconnaissance level data typically include the following sections:

- Abstract
- Text, including geographic, climatic and meteorological descriptions; special site-specific considerations regarding geology, hydrology, water quality, and geochemistry; known uranium occurrences in the area; empirical and statistical evaluations; and conclusions
- Summary of LASL's standard procedures for collecting, treating, and analyzing samples
- Listings of water and sediment samples with selected information
- A coding key
- Maps and overlays
- References

Like ORGDP, LASL issued a number of reports on geochemical and geostatistical interpretations of the data. LASL conducted studies during their work on the HSSR project to assist them in assessing the quality of the data. For example, LASL completed a study of the variation in its analysis and sampling methods by collecting two samples of water from each of 139 sites in the Lordsburg area of New Mexico. These sample pairs were both analyzed by fluorometry and each individual analyzed twice so that data from four fluorometric analyses were obtained. (See GJBX 65 (76) for more details.)

LASL staff also published articles describing their research experiences in evaluating the HSSR data, in integrating the HSSR data with several other large data sets and in display techniques developed to enhance utilization and analysis of the data (see, for example, Freeman, Bolivar and Weaver 1983; Bolivar, Freeman, and Weaver 1983; Byeth and McIntee 1981).

When HSSR Program funding for LASL ended, LASL had a significant amount of analyses completed but not yet issued in report form. Bendix was authorized by DOE to produce reports based on the LASL data tapes. More than 100 of these reports were issued in microfiche. In addition, ORGDP issued 98 reports on LASL samples which they had analyzed.

CHAPTER 5: SAVANNAH RIVER LABORATORY

5.1 INTRODUCTION

The Savannah River Laboratory (SRL) accepted responsibility in 1975 for conducting the HSSR program surveys of twenty-five states in the eastern United States. In 1977, when the program moved to quadrangle reporting, SRL picked up small areas in three adjoining states. When Lawrence Livermore Laboratory's (LLL) participation in the HSSR program ended in 1979, SRL assumed responsibility for portions of the seven western states in which LLL had conducted sampling. This latter area is referred to as "SRL-West".

Overall, SRL was responsible for hydrogeochemical and stream sediment reconnaissance of 3.9 million square km² (1.5 million m²) in 37 eastern and western states. (See Figure 2.3.) In order to carry out this program, approximately four hundred thousand water and sediment samples were collected.

5.2 SAMPLE SELECTION AND COLLECTION

5.2.1 Site Selection

Nominal ground and surface water sampling density in rural areas varied from 13 to 25 square kilometers (5 to 10 square miles) per site, depending upon the geology of the area. In areas of crystalline rock, surface water sampling was generally at greater density than ground water sampling. In areas of sedimentary rock, the ground water sampling was typically at greater density. Areas thought to have relatively high uranium potential were sampled more densely than those thought to have lower potential. Also, in the Eastern states, where both privately held and developed lands are predominant, sampling could not be as uniform as in the Western states.

Coastal Plain areas were sampled for ground water at a higher density than other areas and for sediment at a lower density. Adjacent areas may not be sampled at the same density since later surveys were sometimes made at higher densities. Some areas were not sampled due to urban or industrial contamination, geographic inaccessibility (swamp, lake, etc.), or lack of permission to sample. Additionally, some counties or portions of counties were inadvertently left out of the analytical schedule.

Sampling site locations were marked on compilation maps. The maps were returned to SRL for determination of geographic coordinates. An electronic digitizer was used to measure, verify, and enter latitude and longitude for each site into SRL's HSSR database. These data were recorded to four decimal places but are considered reliable to only three decimal places.

For ground water sampling, field maps were marked with a grid. The well or spring closest to the center of each grid was sampled unless the site proved unsuitable. In such cases, a site further from the center of the grid unit was selected; a site in an adjoining grid unit could be used as long as it was not within one mile of the designated site within that adjoining unit.

For stream sediment sampling in humid areas, streams draining no more than three times the area of a grid unit were designated for sampling. The largest stream beginning in the

grid unit designated for sampling or in an adjoining unit was selected. If a road or stream intersection or any other easy access were apparent, the selected stream was sampled at that point. Otherwise, the stream was sampled as near to the grid center as feasible. Samples were taken upstream from roads, ponds, or other sites with potential for altering the sample. When the criteria for stream sediment site selection would have resulted in two adjacent grid units remaining unsampled, those criteria were not applied.

In arid areas, the grid pattern was followed for sediment sampling except that: (1) where the ground was covered by windblown sands, no sampling was done; and (2) where a well-defined fluvial channel system existed, sites were selected according to criteria for humid areas. If the grid unit contained both mountain and basin, the selected site was as high as was practical up the alluvial fan as needed to avoid windblown material.

SRL authorized supplemental samples where normal grid site selection would miss significant geological features such as volcanic rocks, canyon or cliff exposures, high radioactivity, or hot springs. These sites were flagged on the field forms as supplementary.

Working maps for site selection in the East were typically county road maintenance maps and in the West, either topographic maps, U.S. Forest Service maps or other special maps. As samples were taken the sites were numbered sequentially, and marked on duplicate maps; the maps were then returned to SRL. Coordinates of sampling sites were measured by SRL directly from these maps.

5.2.2 Sample Collection

A. Ground Water

Spring and well-water samples were collected as near the source as possible. Well systems were thoroughly flushed before sampling. All treated waters (e.g., chlorinated) and samples from holding tanks were avoided. After rinsing sample containers, about 2 l of water were collected. Samples were filtered through a 0.8- μm Nuclepore membrane in the field by use of a pressure filter apparatus.

B. Stream Sediments

A sediment sample was collected either by use of a spring-loaded jaw scoop (for silty to rocky stream bottoms) or by a bag sampler consisting of a stainless steel tube with an attached bail and bag. At least five sediment samples spaced along 30 m of the stream bottom in the active part of the stream were composited. A stainless steel sieve was used to field screen the sediments and the -40 mesh fraction was retained. About 0.5 kg of the -40 mesh fraction was then placed into a Kraft paper bag and labeled. Samples were dried at 90 to 100°C before sending them to SRL.

Pilot surveys determined that soil samples from arid areas generally were as effective as stream sediment samples from arid areas, although dry wash areas were avoided when stream beds coexisted in the same grid square. The sample procedure for dry sediments was the same as for wet sediments; however, the sample was usually collected with a shovel or trowel. If significant amounts of windblown sand were present, a composite of at least 15 separate areas was used. The sample was then sieved to -18 to +40 and -40 mesh fractions. Both size fractions were then sent to SRL.

C. Stream Water

In humid areas, sample collection procedures for surface water samples were identical to those for ground water samples. In addition, 60 ml of filtered water were collected in a 2-oz bottle containing 1 ml of 8N ultrapure nitric acid.

5.2.3 Field Measurements

A. Ground Water

Temperature, pH, and conductivity measurements were taken on the unfiltered ground water samples. About 50 ml of filtered ground water were used for alkalinity measurements made with a field titration kit.

Because of both the low uranium concentrations in ground water samples in the eastern United States and because water samples are difficult to preserve, SRL developed a field ion-exchange procedure. About 1 l of water with a conductivity of 500 $\mu\text{mho/cm}$ or less was mixed with 10 g (4 g dry weight) of 100- to 200-mesh high purity cation-anion exchange resin. For samples exhibiting conductivity values greater than 500 $\mu\text{mho/cm}$, a smaller volume of water was used. The ion exchange resin was mixed for about 10 minutes using a battery-operated stirrer. The resin was then allowed to settle into the original 2-oz bottle, which was sent to SRL for analysis of its contents.

Gross radioactivity at each site was measured with a scintillometer. The instrument was held about 30-50 inches above the ground and a reading recorded in counts per second.

Dissolved helium was measured for all ground water samples. The collection technique, modified from Dyck et al (1976), used a clean, 10-oz soft drink bottle. The filled bottle, less 2 ml of water, was capped, inverted, and then sent to SRL for analysis.

B. Stream Sediments

At the collection site for each stream sediment sample, SRL field personnel measured the pH, specific conductance, temperature, and alkalinity of the stream water and recorded general site descriptions. Gross radioactivity at each site was measured with a scintillometer. The instrument was held about 30-50 inches above the ground and the reading recorded in counts per second. One to two liters of water were collected in a plastic beaker from the active part of the stream. Fifty ml of that water were filtered through 37 mm-diameter paper and placed into a 125 ml Erlenmeyer flask for alkalinity titration. After completing the alkalinity titration, a conductivity probe was used to measure the temperature and conductivity of the remaining unfiltered water in the plastic beaker. A pH meter was typically used to measure the pH of the unfiltered water, although Lo-Ion paper was used occasionally if the meter was malfunctioning. The unfiltered water was then discarded.

5.3 SAMPLE ANALYSIS

5.3.1 Sample Preparation

Each ground water sample was treated with a 10-gram portion of ultrapure mixed cation-anion exchange resin that collected all dissolved ions from the water. The volumes of water ranged from 50 to 1000 ml, depending upon sample conductivity. Resin samples were dried at 105°C and packed in ultrapure polyethylene capsules for analysis. The encapsulated samples were loaded into the NAA pneumatic system in batches of 25, including one standard sample and one blank.

Sediment samples were dried at 105°C, sieved to <149 µm (100 mesh), blended, coned, and quartered. One and a half-gram aliquots of the <149 µm material were packed into ultrapure polyethylene capsules for analysis. The encapsulated samples, in batches of 25, including one standard and one blank, were loaded into the NAA pneumatic system. Transport into the irradiation assembly and counting stations and the collection and processing of data were computer-controlled.

5.3.2 Methods of Analysis

Neutron Activation Analysis: The primary method of analysis at SRL was Neutron Activation Analysis (NAA) using the Reactor Activation Facility (RAF). (See Table 5.1 for the planned analysis regime at SRL.)

Each sample received a 2-sec irradiation and 2-sec combined gamma-neutron count to ensure that the sample could safely undergo further irradiation. This test was immediately followed by 10 cycles of 6-second irradiations and 6-second gamma-neutron counts. Uranium concentrations were determined from the neutron counts, and short-lived (< 2 min) activation products were measured by the gamma counts. Following the recycle regime, resin samples were unloaded. Sediments received an additional 400-second irradiation, followed by gamma counting after decay for 600 seconds (to measure intermediate-lived activation products) and 7 days (to measure long-lived activation products). Maximum RAF throughput for mixed sediment and resin runs was about 4000 samples per seven-day week assuming no system failures. In practice, 2500 to 3000 samples per week were normally activated. (See GJBX 56 (80).)

For quality control, every 24th sample was a standard, and every 25th sample was a blank. Analytical results for uranium and other detectable elements were printed in real time for these samples as they were completed. These results were checked to ensure proper sample ordering and correct analyses.

Sediment samples were typically analyzed for uranium and 19 other elements (Al, Br, Ce, Cl, Dy, Eu, F, Fe, Hf, La, Lu, Mn, Na, Sc, Sm, Th, Ti, V, Yb). Uranium was determined by counting delayed neutrons emitted by induced fission products of ²³⁵U in the sample. Other elements were determined by computer reduction of gamma-ray spectra collected at intervals from a few seconds to about 10 days after irradiation. Delayed counts were not made for resin samples. More detailed information about the activation analysis system at SRL can be found in GJBX 77 (77) and GJBX 55 (77).

TABLE 5.1

SRL ANALYSIS REGIMES FOR RECONNAISSANCE SAMPLES^{a, b}

Step	No. of Cycles	T_a	T_d	T_c	T_r
Test	1	2 sec	1.3 sec	2 sec	1.0 sec
Recycle	20(40) ^c	6 sec	1.3 sec	6 sec	1.0 sec
Long Irradiation	1	900 sec (0 sec) ^c	1.3 sec	300 sec (600 sec) ^c	
Delayed Count			10-17 d	900 sec	

- a. 0.5 g of sediment or 5 g of ion exchange resin.
- b. T_a , T_d , T_c , and T_r are durations of activation, decay, counting, and sample return respectively.
- c. Numbers in parentheses apply to resin samples.

Source: GJBX 77 (77) p. 88.

Supplemental Analyses: Some stream sediments were analyzed for additional elements by a subcontractor using several methods including Flame Emission, Atomic Absorption and Colorimetric. (See GJBX 103 (82) for a detailed schematic of this supplemental work.) Extractable uranium was determined after a 30-minute leach using hot (180°C) 4:1 nitric and perchloric acids. Other elements included in this supplemental work were Ag, As, Ba, Be, Ca, Co, Cr, Cu, K, Li, Mg, Mo, Nb, Ni, P, Pb, Sn, Sr, Th, W, Y and Zn. For these analyses, a standard and a blank were included with each group of 40 samples.

SRL did not publish uniform detection limits for elemental concentrations. Thus, Table 5.2 presents a detection limit or minimum reported value for all elements analyzed in water and sediment samples by SRL or subcontractors. In the data reports, elemental values below a detection limit were indicated by a minus (-) sign preceding the limit. For example, for an element reported in ppb, a -3 indicates that the sample contained less than 3 ppb of that element. A period (.) was used to indicate not only that the element was not detected but that the detection limit was unusually high for that sample. The missing data code, designated with an "M", was recorded for those samples which were insufficient for analysis. (See GJBX 128 (80)).

TABLE 5.2

DETECTION LIMITS OR MINIMUM VALUE REPORTED FOR SRL

<u>Element</u>	<u>Sediments (ppm)</u>	<u>Water (ppb)</u>	<u>Element</u>	<u>Sediments (ppm)</u>	<u>Water (ppb)</u>
Ag	0.1	2	Mn	0.01	0.2
Al	0.03	0.8	Mo	-	4
As	1	0.5	Na	0.3	2
Au	0.01	-	Nb	5	4
B	-	8	Ni	1	4
Ba	-	2	P	0.1	40
Be	0.5	1	Pb	1	-
Br	-	0.3	Ra	-	0.1
Ca	0.01	3	Rn	-	50
Cd	-	30	Sc	0.137	0.01
Ce	2.26	-	Se	1	0.2
Cl	-	11	Si	-	3.4
Co	2.5	2	Sm	0.3	-
Cr	25	4	Sn	5	-
Cs	1.4	-	Sr	10	14
Cu	2.6	2	Ta	0.8	-
Dy	0.11	0.001	Tb	0.078	-
F	100	2	Th	0.67	5
Fe	0.02	10	Ti	0.12	2
He	-	2.2	U	0.1	0.001 - 0.33
Hf	0.36	-	V	0.2	0.07
K	0.3	0.4	W	2	-
La	3	-	Y	5	1
Li	3	2	Yb	0.5	-
Lu	0.1	-	Zn	5	4
Mg	0.068	0.1	Zr	-	2

Source: Averett 1984a.

Helium Analysis: Helium was determined for selected areas, particularly the western quads, using a specially developed mass spectrometric procedure. (See GJBX 13 (79).) When available, helium values were reported in standard cubic centimeters of helium per 1000 liters of air ($\text{cm}^3 \text{ He}/1000 \text{ L}$, i.e., ppm by volume). This method introduces a nearly constant 5.2 ppm helium background from air; thus, all samples analyzed are above detection limit. Some of these samples were also analyzed for methane.

5.4 DATA MANAGEMENT AND REPORTING

Although the actual order and format of the information presented in SRL data reports varied, the following sections were typically included in each basic data report for quadrangles, states, or regions:

1. **Geologic Summary and Uranium and Mineral Occurrences:** Describes geologic provinces and history and reviews existing literature on important minerals in the area.
2. **Hydrology:** Discusses local climatic, geographic, drainage and hydrologic characteristics.
3. **Factors Affecting the Data:** Identifies issues such as season of sampling or uneven distribution of sampling that might affect the interpretation of data.
4. **Quality Assurance:** Discusses findings from resampling and analytical standards used to verify the data collection and analysis work.
5. **Results and Discussion of the Data:** Presents data summaries and highlights most important results of the area survey.
6. **Acknowledgement and References.**
7. **User's Guide:** Provides descriptions of formats, abbreviations, and codes used in the data tables.
8. **Appendices:** Either on paper or fiche, these appendices present the actual data for each sample; site location and geologic maps; histograms; cumulative frequency distributions; and a user's guide to the data.

At the end of SRL's participation in the HSSR program, SRL sent unanalyzed water and sediment samples left at the lab after its funding for the HSSR program ended to Oak Ridge Gaseous Diffusion Plant (ORGDP) for multielement analysis; reports for those samples were issued by ORGDP. Subcontractors performed a substantial amount of work for SRL, including the preparation of some reports. At the end of SRL's program funding, more than 30 rough drafts of subcontractor reports were in the SRL files; SRL submitted these through DOE to Bendix for editing and issuance as open-filed data reports. Also, as the program neared completion, SRL began to issue reports with the data presented on microfiche.

CHAPTER 6: OAK RIDGE GASEOUS DIFFUSION PLANT

6.1 INTRODUCTION

The Uranium Resource Evaluation Project was established in 1975 at the Oak Ridge Gaseous Diffusion Plant (ORGDP), Oak Ridge, Tennessee to complete the HSSR program by conducting a systematic determination of the distribution of uranium and associated elements in ground water, stream sediment, and stream water samples in the Central United States. Details of this work are given in GJBX 62 (78), and GJBX 84 (78), and GJBX 32 (80). Oak Ridge was responsible for 154 NTMS $1^{\circ} \times 2^{\circ}$ quadrangles (See Figure 2-3.) Initial reconnaissance was conducted only in geologically favorable areas assigned the highest priority by DOE. Other quadrangles were sampled after completion of the most favorable quadrangles.

6.2 SAMPLE SELECTION AND COLLECTION

6.2.1 Site Selection

The objective of the Oak Ridge Program was to collect samples that were representative of the surface and subsurface geology at an average density of one site per 26 km^2 (10 mi^2), and to accurately evaluate and describe the environment from which these samples were taken. Types of samples collected included well and spring waters which provided information on fluids in contact with units at depth and on stream sediment and water for interpretation of the geochemistry of near-surface units.

Ground water (well and spring) samples and stream sediment samples appear to best represent the subsurface and surface geology, respectively. Stream waters were collected in some of the quadrangles sampled, but these samples were discontinued because of uncertainty regarding their usefulness in the interpretation of surface geology over a 10-state area. The plant samples and other sample types collected during the pilot surveys may have local importance in the interpretation of uranium potential, but their usefulness over the full 10-state area is considered questionable by some reviewers of the project.

Planning for reconnaissance sample collection was done on 15- and 7 1/2-min USGS topographic maps, with county maps and aerial photographs being substituted where topographic coverage was unavailable. The well grid for the quadrangle was drawn onto these maps with a line spacing of 5.1 km (3.2 mi) to identify the ideal ground water site location to the field sampler. For stream sediment sampling, tentative stream sites were also drawn on these maps to identify drainage basins 5.2 to 52 km^2 (2 to 20 mi^2) in an area with an average sample density of one sample per 26 km^2 (10 mi^2).

Once a sample was collected, the exact site location and sample number were plotted on another copy of the topographic map which was sent to Oak Ridge for determination of the latitude and longitude. The site was also plotted on a composite map which covers between 1/8 and 1/4 of the quadrangle at a scale of approximately 1:62,500 with the actual drainage basin sampled drawn for stream samples.

6.2.2 Sample Collection

A. Ground Water

Ground water (wells and springs) reconnaissance samples were collected from the nearest available site to the node of the 5.1 km (3.2 mi) grid. Maximum deviation from the grid node was 2.5 km (1.6 mi). Uniform geographic distribution of data points (one per 26 km² or 10 mi²) was obtained in this way.

Well water samples were taken as near to the well head as possible without disassembling the well-pipe system. Wells in which a sample could not be taken before the water entered a filtering or softening system were not sampled. Water was flushed from the pipes and/or pressure tank for as long as was necessary to obtain a sample that was fresh from the aquifer.

Two polyethylene bottles and their caps were thoroughly rinsed before the sample was taken. Once the sample was taken, all air bubbles were dislodged from the bottles, and the cap was sealed tightly and taped with vinyl tape. The sample was sent back to the laboratory for analysis as soon as possible.

B. Stream Sediments

Sampling sites were located at least 25 m upstream from any man-made contamination (roads, bridges, etc.), with sampling proceeding upstream. Under unusual circumstances, a sample was collected downstream from a road or bridge, usually at a distance of at least 1 km, with sampling proceeding downstream. Sediment samples were collected from the active portion of the stream. Before collecting the sample, the polyethylene sediment scoop was cleaned by sticking the scoop in the sediment several times. At least six scoops of sediment material, taken 2 to 3 m apart, were collected. Sufficient material was collected to obtain 25 g of -100-mesh sediment after sieving. Water was drained from the sample bag, and the top of the bag was folded over and sealed with vinyl tape.

C. Stream Water

Site selection for stream water reconnaissance sampling was the same as for stream sediment sampling. When stream sediment and stream water samples were collected, the same site was used for both samples with the water sample being collected first to avoid collecting any fine-grained material that may have been stirred up in the collection of the sediment.

Two polyethylene bottles and their caps were thoroughly rinsed before the sample was taken. Samples were collected as near the fast moving part of the stream as possible, to avoid collecting organic material that might have been present in the stream. After the sample was taken, all air bubbles were dislodged from the bottles, the cap was sealed tightly and taped with vinyl tape, and the bottle was sent to the laboratory for analysis of the sample.

D. Other Sample Types

Other types of samples, such as botanical species, soils, and rocks, that were available for collection tend to be (1) only of local importance in the interpretation of an area, or (2) predominantly used to delineate specific deposits. Few of these types of samples were collected by this project.

6.2.3 Field Measurements

A. Ground Water

Measurements made at the sample site were specific conductance, dissolved oxygen, temperature, pH (using an Horiba U-7 Water Analyzer or Lo-Ion paper), and alkalinity (using a LaMotte Alkalinity Test Kit). The odor, color, and appearance of the water were also noted. The total depth, and the identity and depth of the producing horizon and discharge were taken from the owner/user or from a publication whenever possible; this information could also have been inferred from other wells in the area. The type of well, power classification, casing, pipe composition, well use and sample location (in relationship to the well head and pressure tank) were also noted.

Contaminants, such as corroded well casing or pipes and precipitate around pipes and faucets, and an estimate of the age of the well were noted in the "Remarks" section of the field form.

B. Stream Sediments and Water

Information recorded in the field included the average stream velocity, width and depth (unless dry), water level, size of the dominant bed material, sample odor, number of grabs taken, and the estimated percentage of organic material in the sample. Information recorded at stream sites also included specific conductance, dissolved oxygen, temperature, pH, and alkalinity, if water is present, and odor and color. Contamination (fertilizer, road material, soil or slump material from channel banks, mining and industrial activities, etc.) that was found within at least 100 m upstream from the site or appeared on the topographic map within the basin were noted in the "Remarks" section of the field form.

6.3 SAMPLE ANALYSIS

6.3.1 Sample Preparation

Water sample preparation and all chemical analyses were performed in a clean room to avoid contamination. The unused portion of the -100-mesh fraction of all stream sediments and a duplicate 250-ml bottle of all water samples were retained in permanent storage for possible future use. Water samples were received in 250-ml polyethylene bottles, vacuum filtered through 0.45- μ m cellulose acetate paper, and then submitted for analysis.

Stream sediment samples were dried overnight at 85°C, placed in a plastic envelope, and disaggregated by impact with a rubber mallet. The fraction passing through a 100-mesh (150- μ m) nylon sieve was collected and blended. Approximately 2 g were loaded into a

polyethylene rabbit for NAA. Another 0.25 g aliquant was placed in a 100 ml Teflon beaker to which was added 5 ml of concentrated nitric acid, then 5 ml of concentrated hydrofluoric acid. The sample was placed on a hotplate at 250°C and evaporated just to dryness. Then, 20 ml of 30% nitric acid were added and the sample warmed. The contents of the beaker were transferred to a 50-ml centrifuge tube, diluted to volume with distilled water and submitted for analysis.

6.3.2 Methods of Analysis

A. Selection of Elements and Methods

The selection of elements to be determined in water and sediment samples was based on a number of different factors. Past experience had shown that important associations for sandstone deposits existed between uranium and arsenic, molybdenum, selenium, and vanadium. A list of 19 other elements considered to be important for the NURE hydro-geochemical survey for uranium was obtained from the USGS. Thirteen additional elements associated with uranium in the minerals of nonsandstone deposits were added to this list. Concentration ranges for each element normally occurring in natural waters and sediments were reviewed to determine the applicability of the available analytical techniques. Priorities were then assigned to each of the elements in the combined list. Elements were eliminated because of low priority as uranium indicators and lack of adequate sensitivity by the analytical techniques used. The result was the list of elements listed in Table 6.1.

A series of ground water and stream sediment samples were collected for use in comparing the sensitivity, cost-effectiveness, and high sample throughput capabilities of several analytical techniques. Each sample was analyzed for uranium by fluorescence spectroscopy, thermal emission mass spectrometry, spark source mass spectrometry (SSMS), and delayed neutron counting (DNC). Other element determinations were made by emission spectrography, sequential atomic absorption analysis, SSMS, and neutron activation analysis (NAA). A comparison was made of costs, equipment, and sensitivities. Because the analysis for uranium was the major effort of this survey, its measurement was optimized. Other procedures were chosen to determine the elements selected for analysis in the most cost-effective way possible. The principal procedures selected for each element and the detection limits for each sample type are listed in Table 6.1.

B. Description of Methods

Uranium Analysis: Uranium was determined in water and stream sediment samples by fluorescence spectroscopy. A 40-ml aliquot of the filtered water sample was transferred to a 60-ml disposable plastic cone and acidified with 1 ml of concentrated nitric acid. The uranium was extracted by adding 1 ml of a 2% solution of trioctylphosphine oxide (TOPO) in Varsol and then shaking the cone. A 50- μ l pipette was used to transfer an aliquot of the organic phase onto a sodium fluoride pellet which was then sintered for 20 min at 900°C. The fluorescence intensity of the sample and standard pellets was measured and read directly by an automated fluorometer, and the sample concentrations computed. Results were reported as ppb in the original water sample. For dissolved sediment samples, 20 ml of each sample were transferred to a 50-ml vial and extracted into 2 ml of 2% TOPO in Varsol. A 50 μ l aliquot was transferred onto a sodium fluoride pellet and processed in the same manner as water samples. The uranium concentration was calculated and reported as ppm of the original sediment sample.

TABLE 6.1
DETECTION LIMITS BY SAMPLE TYPE FOR ORGDP

Element	Method of Analysis	Detection Limits		Detection Limits for
		Sediment (ppm)	Water (ppb)	Sediments by DC Arc Source* (ppm)
U-FL	Fluorometry	0.25	0.2	--
U-MS	Mass-Spectrometry-Isotope Dilution	--	0.02	--
U-NT	Neutron Activation Delayed Neutron Count	0.02	--	--
As	Atomic Absorption	0.1	0.5	--
Se	Atomic Absorption	0.1	0.2	--
Ag	Plasma Source Emission Spectrometry	2	2	1
Al	Plasma Source Emission Spectrometry	0.05(b ^a)	10	--
B	Plasma Source Emission Spectrometry	10	8	20
Ba	Plasma Source Emission Spectrometry	2	2	40
Be	Plasma Source Emission Spectrometry	1	1	--
Ca	Plasma Source Emission Spectrometry	0.05(b ^a)	0.1(b)	--
Ce	Plasma Source Emission Spectrometry	10	30	--
Co	Plasma Source Emission Spectrometry	4	2	4
Cr	Plasma Source Emission Spectrometry	1	4	10
Cu	Plasma Source Emission Spectrometry	2	2	2
Fe	Plasma Source Emission Spectrometry	0.05(a)	10	--
Hf	Plasma Source Emission Spectrometry	15	--	--
K	Plasma Source Emission Spectrometry	0.05(a)	0.1(b)	--
La	Plasma Source Emission Spectrometry	2	--	--
Li	Plasma Source Emission Spectrometry	1	4	4
Mg	Plasma Source Emission Spectrometry	0.05(a)	0.1(b)	--
Mn	Plasma Source Emission Spectrometry	4	2	40
Mo	Plasma Source Emission Spectrometry	4	4	2
Na	Plasma Source Emission Spectrometry	0.05(a)	0.1(b)	--
Nb	Plasma Source Emission Spectrometry	4	--	20
Ni	Plasma Source Emission Spectrometry	2	4	4
P	Plasma Source Emission Spectrometry	5	40	1,000
Pb	Plasma Source Emission Spectrometry	--	--	4
Pt	Plasma Source Emission Spectrometry	--	--	10
Sc	Plasma Source Emission Spectrometry	1	1	2
Si	Plasma Source Emission Spectrometry	--	0.1(b)	--
Sn	Flameless Atomic Absorption	5	--	--
Sr	Plasma Source Emission Spectrometry	1	2	--
Th	Plasma Source Emission Spectrometry	2	--	400
Ti	Plasma Source Emission Spectrometry	10	2	200
V	Plasma Source Emission Spectrometry	2	4	10
Y	Plasma Source Emission Spectrometry	1	1	10
Zn	Plasma Source Emission Spectrometry	2	4	200
Zr	Plasma Source Emission Spectrometry	2	2	40
SO ₄	Spectrophotometry	--	5(b)	--
Cl	Spectrophotometry	--	10(b)	--

(a) Detection limits expressed in percent.

(b) Detection limits expressed in ppm.

* Spectrochemical analysis of stream sediment samples for some of the early quadrangle reports was done using a dc arc source instead of the direct-reading spectrograph with an Inductively Coupled Argon Plasma (ICAP) source used for the remainder of the HSSR Program.

Source: GJBX 32 (80).

For regions where the uranium concentration was expected to be below the detection limit of fluorometric analysis, water samples were analyzed for uranium by isotope dilution thermal emission mass spectrometry. The instrument used for this analysis was a 6-in., 60° magnetic sector instrument with photon counting designed and built in Oak Ridge. A 10-ml portion of a filtered water sample was spiked with 5- μ l of a 2 ppm solution of uranium-233 as an internal standard. The spiked sample was acidified with 100 μ l of concentrated nitric acid and extracted into 1 ml of 2% TOPO in carbon tetrachloride. The aqueous layer was removed and the organic phase air dried, leaving the dry TOPO and uranium. A 250- μ l portion of a 10% ammonium carbonate solution was added to dissolve the residue, and 2 μ l of this solution was placed on a rhenium filament for analysis. The uranium-233, -235, and -238 isotopes were measured. The uranium-233/uranium-238 value was used to calculate the concentration in the sample. Results were reported as ppb of the original sample.

A total uranium value was also determined for stream sediments by neutron activation using delayed neutron counting. Approximately 2 g of -100-mesh sediment material was loaded into a polyethylene rabbit and transferred through a pneumatic transfer system into a reactor where it was exposed to a neutron flux of 3×10^{13} n/cm²-sec for 60 sec and then transferred to a neutron counter. Results were reported as ppm of the dry sediment.

Arsenic and Selenium Analysis: Arsenic and selenium concentrations in water and aliquots of dissolved sediment were determined using hydride generation flameless atomic absorption spectroscopy. An automated, microprocessor-controlled, atomic absorption spectrometer was used for the analysis. Eighty filtered water samples or 60 dissolved sediment samples (diluted from 5 to 35 ml) were loaded in the sample changer. Twenty ml of each sample were pumped into the reactor followed by 15 ml of 15% sulfuric acid, and 10 ml of 0.20% sodium borohydride in 0.5% KOH. The arsenic and selenium hydrides and excess hydrogen gas were collected in a rubber balloon and after 45 seconds were sparged through a quartz atomizer tube heated to 600°C. The absorption of the 193.7-nm arsenic line or the 196.0-nm selenium line was measured and the resulting peak height recorded.

A series of solutions with standard concentrations was analyzed as described and a least-squares fit to a second-order equation applied to the observed peak heights. The calculated coefficients were then used to calculate sample concentrations. During normal operation, every tenth sample was a known standard that must have agreed within $\pm 15\%$ of the expected value.

Sulfate and Chloride Analysis: Sulfate and chloride concentrations in water samples were determined colorimetrically using a Technicon Autoanalyzer (TM). Samples were loaded into an automatic sample changer for simultaneous analysis. The sulfate analysis used the methyl thymol blue-barium chloride reaction. A known quantity of barium was added to the water, and excess barium reacted with methyl thymol blue to form a colored complex. The chloride analysis used the mercuric thiocyanate-ferric nitrate reaction. The chloride reacted with the mercury freeing the thiocyanate ion which formed a colored complex with iron. The intensity of the resulting color was measured for each procedure and compared with the appropriate calibration curve. The concentration was determined and factored to the original sample basis.

Emission Spectrochemical Analysis: Water samples and sediment extracts were analyzed spectrochemically using a direct-reading spectrograph with an Inductively Coupled Argon Plasma (ICAP) source. The sample was aspirated directly into the source with no additional preparation required. This technique offered the advantage of trace-level detection limits with an applicable range of measurement of greater than five orders of magnitude for most elements.

The detection limits for the ICAP source are given in Table 6.1. These limits were determined by analyzing blanks and standards, along with routine samples, during normal operation.

The spectrochemical analysis of stream sediment samples for some of the early quadrangle reports was done using a dc arc source instead of the ICAP. Detection limits for this method of analysis are also reported in Table 6.1. In this procedure, approximately 0.5 g of the -100-mesh fraction of the dried sediment sample were ground in an Al_2O_3 mortar, and 15 mg was weighed and mixed on the balance pan with a level scoop of graphite (approximately 17 mg). This mixture was tamped into a thin-walled, undercut electrode with a 2 mm x 4 mm crater. The electrodes were maintained at 200°C until arced. They were arced until the sample was totally consumed (70 sec), using a current of 15 A dc. The concentration of each element was read from the photographed spectra by visual comparison to standards prepared by grinding oxides of the desired elements into a matrix of 60% SiO_2 , 15% Al_2O_3 , 10% Fe_2O_3 , 10% CaO , 2% Na_2CO_3 , 2% K_2CO_3 , and 1% MgO .

Conductivity: The conductivity of water samples was measured in the laboratory only if it was not measured in the field; a Yellow Springs Instrument Company Model 31 conductivity meter was used with results reported in umhos/cm.

6.4 DATA MANAGEMENT AND REPORTING

Field samples were received in Oak Ridge at a central receiving station, where they were entered into the records management system and distributed for analysis. Quality control samples were added and one of the 250 ml water sample bottles was put into a separate bottle for storage. Computer cards containing information about each sample were provided for use in the laboratory. As each sample was processed through the various procedures in the laboratory, it was identified by the six-digit number appearing on the sample container and the computer card. After processing, the unused portions of sediment samples were returned to the receiving station for storage. These stored sediment samples now reside in the USGS sample archive. (See Chapter 2.)

A quality control program was set up to monitor the precision and accuracy of the procedures used to analyze water and sediment samples for uranium and other trace elements. Control samples at two different concentration levels for each sample type were submitted for analysis on a routine basis. A quality control group, which was administratively separate from the analytical laboratory, prepared the control samples, analyzed the results, and notified the laboratory of any deviation of results from specified control levels. This allowed the laboratory personnel to identify and correct instrumental or procedural problems and to correct questionable results prior to publication. A resampling program was also conducted by ORGDP to evaluate sampling and analytical variations.

In the Oak Ridge HSSR program, approximately 79,000 samples were collected and approximately 231,000 samples analyzed, each having about 60 associated pieces of information. Computerized data verification procedures were initiated to ensure high-quality data. Field forms were completed at the time a sample was collected by the procedures described in GJBX 62(78) and GJBX 84(78). Most of the field information was recorded on a checkoff system to minimize misrecording of field information. Water-resistant labels with preprinted sample numbers were attached to the field form and to the sample container.

An initial review of the field form information was made by geologists when the field forms were filled out. The information was then verified by computerized edit checks to ensure that acceptable data were recorded. Map codes, collector's initials, surface geologic codes, and producing horizon codes were compared with master lists. Numeric data were flagged if any information, such as steam flow or average depth, was not within standard ranges. Reasons for the unusual results were then investigated. Consistency checks which compared two fields were also performed. For example, samples were flagged if the total and M alkalinity differed by more than 10%, or the pH was less than 8 and the P alkalinity greater than zero. The final check on field conductivity, dissolved oxygen, water temperature, pH, and total alkalinity took place in the principal component analyses when the laboratory data were verified. A complete list of continuous data checks is given in Table 6.2.

When samples were collected, the sampling location with the computer preprinted sample numbers were placed on 15- and 7 1/2-min topographic maps. Upon completion, maps were reviewed by geologists and then forwarded to the digitizing technician at Oak Ridge.

The interactive Tektronix digitizing system at Oak Ridge consisted of a minicomputer with supporting peripheral equipment which included an electronic digital tablet sensitive to an electrical pointer. The field map was placed on the tablet (38 in. x 31 in.) and the sampling locations touched by the pointer. Sample numbers were entered for each sampling location. The average rate was about 1 point every 30 sec. On a 7 1/2-min topographic map, a sampled location was accurate to within approximately 50 feet.

After a group of maps was digitized, about 50% of the maps were redigitized and compared with the original results. If errors were encountered, the entire group of maps was redigitized. Additionally, verification of the sampling locations was performed by obtaining computerized plots at the same scale as the topographic maps and overlaying the original digitizer map.

Detection of suspected errors was enhanced by use of automated, statistical procedures such as principal component analysis. (See GJBX 71 (77).) The automated principal component procedure identified errors in samples having unusually high or low values. However, the main advantage was that samples having two or more measurements of an unusual relationship were also identified.

Variables were selected for principal component error analysis that had a minimum of about 70% of the data above the laboratory detection limit. Values below the detection limit were assumed to be one-half the detection limit. To approximate normality, logarithms of the concentrations were used in the analysis for variables other than pH.

Table 6.2

DATA VERIFICATION* FOR CONTINUOUS VARIABLES AT ORGDP

<u>Variable</u>	<u>Verification</u>	
Field Data	Day \leq 31, 1 \leq Month \leq 12, 76 \leq Year \leq 85	
Air Temperature	-15 \leq Air Temp. \leq 40	
Latitude	20 \leq Lat \leq 50, Within one degree of standard for its quadrangle	
Longitude	Within two degrees of standard for its quadrangle	
Stream Flow	\leq 1	
Stream Width	\leq 10	
Average Depth	\leq 4	
Number of Grabs	5 \leq Grabs \leq 20	
Depth of Visibility	0 \leq Depth Vis. \leq 5	
Water Temperature	-5 \leq Water Temp. \leq 40	
Water Conductivity	10 \leq Water Cond. \leq 19,500	
Discharge	\leq 5,000	
Waters		
Dissolved Oxygen	.5 \leq Diss. Oxygen \leq 14	
Only pH	4 \leq pH \leq 11, If P alkalinity > 0 then pH should be < 0 or > 8	
P Alkalinity	0 \leq P Alkalinity \leq 100	The larger of the two measurements must not exceed the smaller by more than .1 times the smaller value.
M Alkalinity	20 \leq Alkalinity \leq 2,000	
Total Alkalinity	20 \leq Alkalinity \leq 2,000	
Wells		
Only		
Depth of Producing Horizon	\leq 2,000	
Well Depth	\leq 2,000	

*Verified for normal ranges; unusual results were checked for accuracy.

Source: GJBX 32 (80)

The principal component analysis produced an ordered list of extreme samples using eigenvalue related statistics. The outlier nature of the sample may have been due to incorrect laboratory results, computer processing errors, sample contamination, errors in field measurements, or to real geochemical anomalies observed for that sample. Additional unusual samples were identified if single element measurements were outside a three-standard deviation confidence interval.

The laboratory and field data from the unusual samples identified by the preceding procedure were reviewed. Approximately 1% of these samples that appear to be the most unusual were selected for reanalysis. These were then compared and any results that were considered to be in error in the original analysis were corrected. A summary of the unusual samples and results of the reanalysis are typically given in the quadrangle reports issued by Oak Ridge.

The compilation of field and laboratory data of a 1° x 2° NTMS quadrangle was presented in the form of a basic data report, as required by DOE, as soon as possible after completion of the quadrangle sampling, laboratory analysis, data verification, and data summarization.

The following major sections comprise the basic data report for ORGDP quadrangles:

1. Geology - Describes the geology of the area, with emphasis on its relation to known or possible uranium occurrences.
2. Sample Collection - Gives the time the samples were collected and analyzed and describes major sources of contamination within the quadrangle.
3. Chemical Analysis - Gives a general description of sample preparation for chemical analysis and provides a reference for the analytical procedures used.
4. Quality Control - Describes the use of calibration control and check samples to check the accuracy and precision of analyses. Also includes a description of the principal component error analysis, which identifies the samples with the most unusual set of chemical data; these samples are then submitted for reanalysis as a check against possible analytical error.
5. Geochemical Results - Summarizes the geochemical results and indicates areas of known or potential uranium occurrences. Uranium and conductivity are discussed for water samples and uranium and thorium for stream sediments. Geographic plots of these elements, a site location map of each sample type and a geologic map are included as plates at 1:250,000 scale.
6. Appendices - Present ground water stream sediment and field and laboratory data.

Data for elements other than those required may have been included in the geochemical results section if one of the following two criteria was met:

1. There was a known or observed relation between the element and the distribution of uranium in the quadrangle or in similar geologic environments elsewhere.

2. If the statistical correlation table indicated a significant correlation coefficient between uranium and the element.

Probability, frequency, and percentile plots along with geochemical distribution maps (at the scale 1:1,000,000) and a partial data listing for selected elements were included in the appendixes for all elements discussed. The reports issued by ORGDP for LASL and SRL samples contain geochemical analysis listings only.

CHAPTER 7: SUMMARY AND RECOMMENDATIONS

7.1 SUMMARY

The National Uranium Resource Evaluation (NURE) was a U.S. Department of Energy (DOE) program initiated in 1973 to provide a set of data from which to assess the nation's domestic uranium resources. DOE's Grand Junction Office (GJO), through a contract with Bendix Field Engineering Corporation, managed the following NURE components:

- Aerial Radiometric and Magnetic Reconnaissance;
- Subsurface Geologic Investigation;
- Remote Sensing;
- Geophysical Technology Development;
- Intermediate Grade Uranium Study;
- World Class Uranium Study;
- National Logging Program;
- Topical Geologic Studies; and
- Hydrogeochemical and Stream Sediment Reconnaissance.

The Hydrogeochemical and Stream Sediment Reconnaissance (HSSR), the focus of this topical report, was to be a systematic survey of the nation's ground and surface waters and stream sediments to assess uranium potential. Initially, samples were analyzed only for uranium; later in the program, however, laboratory analyses were conducted for a range of elements. These additional analyses contributed directly to the current interest in the HSSR database among exploration geologists interested in minerals other than uranium and researchers interested in other issues, such as water quality.

Four laboratories, Lawrence Livermore Laboratory, Los Alamos Scientific Laboratory, Oak Ridge Gaseous Diffusion Plant, and Savannah River Laboratory, were responsible for the sample collection and analysis effort which began in 1976 and ended in 1981. The Lawrence Livermore Laboratory's participation in the HSSR ended in 1979; Los Alamos Scientific Laboratory and Savannah River Laboratory assumed responsibility for that laboratory's remaining assignments.

Each laboratory was assigned a geographic area based on the National Topographic Map Series 1° x 2° quadrangle grid (1° x 3° in Alaska). HSSR data were typically collected and reported in paper copy and on magnetic tapes by the individual quad name. Toward the end of the program, Savannah River Laboratory grouped quad data into a number of multi-state reports. Early in the program, reports were issued on pilot, or orientation studies which were completed to evaluate sampling procedures. Later on, detailed studies were done for specific areas of geologic interest.

At the end of the DOE NURE program, all nonproprietary geoscience data generated during the course of the program, including the HSSR data, were transferred to the United States Geological Survey (USGS); proprietary data relevant to uranium reserves, production, and costs remained with DOE's Energy Information Administration. The HSSR data are available to the public, through the following USGS offices.

Sale of maps and reports:

Open-File Services Section
Building 41, MS 306
P.O. Box 25046
USGS, Federal Center
Denver, Colorado 80225, (303) 236-7476.

Sale of magnetic tapes:

USGS EROS Data Center
User Services
Sioux Falls, South Dakota 57198, (605) 594-6142

Facilities for reading-room examination of HSSR reports and maps:

USGS Library
Denver West Building 3
1526 Cole Boulevard
Golden, Colorado.

Information on the archived samples collected for the HSSR:

B. R. Burger
MS977, P.O. Box 25046
USGS, Federal Center
Denver, Colorado 80225.

Additional repository libraries exist at locations such as universities and state geological surveys throughout the United States.

Information Systems Programs (ISP), a department of the University of Oklahoma's Energy Resources Institute, worked under contract to DOE to complete a reorganization of the HSSR data as reported on magnetic tape. The purposes of this work, completed in June 1985, were to make the data more easily accessible through development of a single, standard format file and to provide documentation and description of this file to assist data users. The final products will be made publicly available.

7.2 CONCLUSIONS AND RECOMMENDATIONS

Reviews of the literature incorporating the NURE HSSR data and discussions with individuals familiar with the database suggest that the HSSR database is generally sound, useful, and valuable. A number of questions remain, however, about the construction and content of the database; resolving these questions is more difficult with this database than might normally be expected for several reasons, including:

- Program History - The data were collected, analyzed, and compiled by four separate laboratories over a six year period. In particular, the differences between the laboratories at all stages of data collection and handling have important implications for the interpretation of the database.
- Construction of the Database - The standardized master file was built from over 49 different formats. Supporting documentation for these formats was often incomplete.
- Size - The database contains almost one million sample records, thus placing technical and financial constraints on the feasibility of traditional quality control work and data manipulation.
- Access - The lack of a machine readable index specific to the HSSR database allowing users to connect specific records to source data sets, GJBX reports and other quality control documentation impedes research efforts.

ISP's experiences with the HSSR data have reaffirmed a need for systematic editing. Even though the ability to determine appropriate corrections does not currently exist, this program would allow the identification of clearly erroneous or questionable data. Results of this work could be made available to the public through a report describing the edit checks and summarizing the number of data elements failing the edits. A copy of the errors detected on each record could be made available on magnetic tape or hard copy.

The documentation associated with the NURE HSSR program activities is extensive, varied, and detailed. While this documentation, which was typically issued as GJBX reports, is generally well done and an important resource to users of the data, the sheer quantity of the material is overwhelming for those individuals unfamiliar with the HSSR program. Currently, the only existing lists of materials are available in paper or microform. The production of a machine-readable catalog of existing GJBX reports by subject would be invaluable to users of the NURE data. Copies of the catalog could be made publicly available on magnetic tape, or in hard copy (sorted according to the topical approach specified by a user).

One of the work products from this contract is a topical report on data quality and characteristics. This report could be extended to a complete data book for each state, summarizing the data contained in the HSSR file. These reports would furnish the basic descriptors to the user community, allowing them to assess the utility of the data for further research without significant investments in data tapes or computer analysis.

The systematic documentation of user's experiences with the database might be helpful. The HSSR database, as a component of NURE, seems to have a mixed reputation. That is, the quality of the data has been both challenged and supported by researchers and geologists. Establishing a procedure for the collection of user's applications, assessments, reanalyses, or evaluations of the database would facilitate conclusions based on comparison and contrast.

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