

K/UR-100

# ORGDP

OAK RIDGE GASEOUS DIFFUSION PLANT



OPERATED BY UNION CARBIDE CORPORATION FOR THE UNITED STATES DEPARTMENT OF ENERGY NATIONAL URANIUM RESOURCE EVALUATION PROGRAM

## HYDROGEOCHEMICAL AND STREAM SEDIMENT RECONNAISSANCE PROCEDURES OF THE URANIUM RESOURCE EVALUATION PROJECT

J. W. Arendt, T. R. Butz, G. W. Cagle, V. E. Kane, and C. E. Nichols Uranium Resource Evaluation Project

December 31, 1979

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## GRAND JUNCTION OFFICE, DOE ISSUES HYDROGEOCHEMICAL PROCEDURES REPORT

The Grand Junction, Colorado, Office, U.S. Department of Energy (DOE), has issued a report, GJBX-32(80), entitled "Hydrogeochemical and Stream-Sediment Reconnaissance Procedures of the Uranium Resource Evaluation Project," as part of the National Uranium Resource Evaluation (NURE).

This report, by J. W. Arendt, T. R. Butz, G. W. Cagle, V. E. Kane, and C. E. Nichols, of the Oak Ridge Gaseous Diffusion Plant (ORGDP), Oak Ridge, Tennessee, is provided as a supplement to HSSR data reports. The supplement provides a description of specific field and laboratory procedures, and of quality control. Different types of data presentations used in the basic data reports are defined and explained in detail.

NURE is a program of DOE's Grand Junction Office 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. ORGDP is operated by the Nuclear Division, Union Carbide Corporation, for the Oak Ridge Operations Office, DOE.

The 56-page report, GJBX-32(80) [ORGDP No. K/UR-100], dated December 31, 1979, has been placed on open file at the following locations:

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This report will be available on microfiche from the Grand Junction Office, DOE, for \$3.00. Prepaid orders should be sent to Bendix Field Engineering Corporation, Technical Library, P.O. Box 1569, Grand Junction, CO 81502, Checks or money orders should be made out to Bendix Field Engineering Corporation, the operating contractor for DOE's Grand Junction Office. For mail orders outside the United States and Canada add \$2.00 per report to cover air mail postage. All foreign orders must be in U.S. dollars or international money orders.

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NATIONAL URANIUM RESOURCE EVALUATION PROGRAM

## HYDROGEOCHEMICAL AND STREAM SEDIMENT RECONNAISSANCE PROCEDURES OF THE URANIUM RESOURCE EVALUATION PROJECT

J. W. Arendt, T. R. Butz, G. W. Cagle, V. E. Kane, and C. E. Nichols Uranium Resource Evaluation Project

> Union Carbide Corporation, Nuclear Division Oak Ridge Gaseous Diffusion Plant Oak Ridge, Tennessee

Prepared for the U. S. Department of Energy Assistant Secretary for Resource Applications Grand Junction Office, Colorado under U. S. Government Contract W-7405 eng 26

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#### PREFACE

The "Hydrogeochemical and Stream Sediment Reconnaissance Procedures of the Uranium Resource Evaluation Project" is provided as a supplement to the basic data reports of the Hydrogeochemical and Stream Sediment Reconnaissance Program issued by the Oak Ridge Gaseous Diffusion Plant (ORGDP). The purpose of this addendum is to provide the reader of the basic data report and the detailed geochemical survey report with a readily obtainable reference explaining the overall ORGDP reporting program in detail. The document describes specific procedures used concerning field and laboratory methodology and quality control. Different types of data presentations used in the basic data reports are defined and explained in detail. This addendum will be updated and modified in accordance with continuing improvements made in the ORGDP program which affect all phases of data collection, verification, and report presentation.

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#### HYDROGEOCHEMICAL AND STREAM SEDIMENT RECONNAISSANCE PROCEDURES OF THE URANIUM RESOURCE EVALUATION PROJECT

#### INTRODUCTION

The National Uranium Resource Evaluation (NURE) Program was established by the U. S. Atomic Energy Commission, now the U. S. Department of Energy (DOE), in the spring of 1973 to assess uranium resources and to identify favorable areas for detailed uranium exploration throughout the United States. The principal objectives of the NURE Program are: (1)to provide a comprehensive in-depth assessment of the nation's uranium resources for national energy planning, and (2) to identify areas favorable for uranium resources. A NURÉ Program report covering uranium resource assessment in 116 National Topographic Map Series (NTMS) 1° x 2° quadrangles, which contain 100 percent of the currently estimated uranium reserves and probable potential resources, is targeted for 1980. The complete resource assessment of the 272 highest-priority guadrangles is scheduled for completion in 1985, and the first comprehensive assessment report of the entire United States is scheduled for completion in This program, which is being administered by DOE, is expected to 1988. increase the activity of commercial exploration for uranium in the United States.

The NURE Program consists of five parts:

- Hydrogeochemical and Stream Sediment Reconnaissance (HSSR) Program,
- 2. Aerial Radiometric and Magnetic Survey,
- 3. Surface Geologic Investigations,
- 4. Drilling for Geologic Information, and
- 5. Geophysical Technology Development.

The objective of the HSSR Program is to provide information to be used in accomplishing the overall NURE Program objectives. This is accomplished by a reconnaissance of surface water, groundwater, stream sediment, and lake sediment. The survey is being conducted by three Government-owned laboratories. Union Carbide Corporation, Nuclear Division (UCC-ND), under contract with DOE, is conducting its survey in 154 NTMS 1° x 2° quadrangles which cover approximately 2,500,000 km<sup>2</sup> (1,000,000 mi<sup>2</sup>) of the Central United States (see Figure 1). This area includes most of the states of Texas, Oklahoma, Kansas, Nebraska, South Dakota, North Dakota, Minnesota, Wisconsin, Michigan, Indiana, Illinois, and Iowa, as well as parts of Arkansas, Missouri, New Mexico, and Ohio. Regional hydrogeochemical studies are being conducted in selected areas throughout the United States. These studies are designed to charac-terize the hydrogeochemistry; stream sediment geochemistry; and/or radiometric patterns of known, or potential, uranium occurrences.



OWG. NO. G-77-530 [U]

Figure 1

INDEX MAP SHOWING THE ORGDP AREA OF RESPONSIBILITY FOR THE HSSR SURVEY

#### URANIUM RESOURCE EVALUATION PROJECT

The Uranium Resource Evaluation (URE) Project was established in 1975 at the Oak Ridge Gaseous Diffusion Plant (ORGDP), Oak Ridge, Tennessee. The purpose of the Project is to complete the part of the HSSR Program assigned to UCC-ND by conducting a systematic determination of the distribution of uranium and associated elements in groundwater, stream sediment, and stream water samples in the Central United States. The URE Project's area of responsibility is 154 NTMS 1° x 2° quadrangles (see Figure 1). Initial reconnaissance will be conducted only in geologically favorable areas assigned the highest priority by DOE. The remainder of the area will be sampled after completion of the favorable Reports for individual  $1^{\circ} \times 2^{\circ}$  quadrangles will be open guadrangles. filed as soon as possible after sampling, analysis, and data verification have been accomplished. In these reports, the significance of the distribution of uranium and associated elements in natural waters and stream sediments will be assessed as indicators of areas favorable for the occurrence of uranium mineralization. Detailed surveys are conducted in favorable geologic terrains to provide detailed geochemical information on specific geologic targets.

#### Program Concept

The URE Project reconnaissance is based on the concept that geochemical techniques can identify promising uranium-bearing areas at virtually any Concentrations of uranium increase as a mineralized area is scale. Figure 2 shows an hypothesized increase in the uranium approached. concentration versus areal extent. As will be noted, the largest area, background, is where uranium concentrations are relatively low. The province may be of the order of 260 km<sup>2</sup> (100 mi<sup>2</sup>) to 2,600 km<sup>2</sup> (1,000 mi<sup>2</sup>), and the uranium concentration an order of magnitude greater in the province. Similarly, for a district, the area is  $26 \text{ km}^2$  (10 mi<sup>2</sup>) to 260km<sup>2</sup> (100 mi<sup>2</sup>) with a corresponding increase of uranium content. At the deposit scale, uranium content increases to ore grade. The objective of the URE Project is to define the areal extent of uranium provinces and districts using geochemical exploration techniques.

The URE Project consists of pilot surveys followed by geochemical reconnaissance with samples of groundwater, stream sediment, and stream water collected and analyzed for uranium and other trace elements which more completely describe geochemical patterns. This allows collection of fewer samples than if analyses were for uranium only.

Pilot surveys are intended to provide information on the following:

- 1. Trace elements indicative of uranium mineralization,
- 2. Relationship between sample types and relative importance of each,
- Range of geochemical concentrations from mineralized to background areas,





GEOCHEMICAL CONCENTRATION VERSUS PROXIMITY TO URANIUM DEPOSIT

- 4. Adequacy of laboratory sensitivity,
- 5. Types of treatments to be given samples,
- 6. Area to which pilot survey applies, and
- 7. Adequacy of sample spacing.

Pilot survey reports have been issued for the Llano area of Texas (Nichols, et al, June 1976), the Texas Panhandle (Nichols, et al, September 1976), and Oklahoma (Bard, et al, February 1979).

#### Planning and Organization

Detailed project planning is an essential element in carrying out an innovative and cost-effective hydrogeochemical and stream sediment survey. The URE Project Office is located at ORGDP. The expertise and capabilities of the Y-12 Plant, Oak Ridge National Laboratory (ORNL), and Paducah Gaseous Diffusion Plant (PGDP) are also available to provide essential services to the project. Functional support services provided within the Nuclear Division are shown in Figure 3, and URE support organizations in Figure 4.

Many of the time-proven production procedures being used by UCC-ND were directly applicable to the URE Project. An example of this activity is the Y-12 Plant production control system, which was used in the design of the URE sample storage and retrieval system. Samples collected during the program are efficiently scheduled, controlled, and placed in retrievable storage for additional tests, if required.

Project plans include the maximum use of automated equipment to minimize costs and potential errors. Automated equipment is included in the analytical laboratories and data management activity. Automatic digitizers are also used to determine accurate latitudes and longitudes of sample sites on field maps.

Sampling is scheduled on a year-to-year basis to provide geochemical data in a timely manner to meet the requirements of other DOE/NURE Program activities. Some considerations in planning include the following: (1) weather, (2) uranium favorability, and (3) availability of funds. Basin sampling is scheduled during time of low runoff, but not during normal periods of freezing.

#### FIELD GEOLOGY PROGRAM

The objective of the URE Reconnaissance Field Geology Program is to collect samples that are representative of the surface and subsurface geology at an average density of one site per  $26 \text{ km}^2$  ( $10 \text{ mi}^2$ ), and to accurately evaluate and describe the environment from which these samples are taken. Types of samples that are collected include well and spring waters which provide information on fluids in contact with units at depth and stream sediment and water for interpretation of the geochemistry of near surface units.





FUNCTIONAL REQUIREMENTS OF THE URANIUM RESOURCE EVALUATION PROJECT



## URANIUM RESOURCE EVALUATION PROJECT SUPPORT ORGANIZATIONS

Figure 4

Groundwater (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 their usefulness in the interpretation of surface geology over a 10-state area is questionable. Plant samples (collected during the pilot surveys) and other sample types may have local importance in the interpretation of uranium potential, but their usefulness over the full 10-state area is doubtful.

#### PLANNING

Planning for reconnaissance sample collection is done on 15- and  $7\frac{1}{2}$ -min U.S. Geological Survey (USGS) topographic maps, with county maps and aerial photographs being substituted where topographic coverage is unavailable.

The well grid for the quadrangle is drawn onto these maps with a line spacing of 5.1 km (3.2 mi) to identify the ideal site location to the field sampler. Tentative stream sites are also drawn on these maps to identify drainage basins 5.2 to 52 km<sup>2</sup> (2 to 20 mi<sup>2</sup>) in an area with an average sample density of one sample per 26 km<sup>2</sup> (10 mi<sup>2</sup>).

Once the sample is collected, the exact site location and sample number is plotted on another copy of the topographic map which is sent to Oak Ridge for determination of the latitude and longitude. The site is 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.

#### Field Form

The majority of the field information is recorded on a form (Figure 5) which utilizes a quick checkoff system that minimizes the misrecording of field information and allows for quick keypunching of the information onto two or more computer punch cards. Procedures for completing the field form, along with a complete breakdown of each item shown on the form, are given in Appendix C of K/UR-12 (Uranium Resource Evaluation Project, March 1978) and K/UR-13 (Uranium Resource Evaluation Project, May 1978).

For basic data reports, the most important information used in verification of data includes the following:

- 1. Map code;
- Sample type;
- 3. Phase (P, 1, 2, or G);
- Location (latitude/longitude) as determined by the Tektroniz digitizer;
- 5. Surface geologic unit code for wells, springs, and sediment;

## OAK RIDGE GEOCHEMICAL SAMPLING FORM







Figure 5

OAK RIDGE GEOCHEMCIAL SAMPLING FORM

Identification of Producing Horizon Use of Well 74 75 76 77 STREAM OR LAKE SEDIMENT (Geologic Unit Code) 26 Sample Condition м Municipal 31 Confidence of Producing Harizon Identification н Household Drv S Stock W wet High Degree ī Irrigation н Probable All of above A Sample Treatment H and S Possible Х S 32 H and I Source of Producing Horizon Identification Ν Y None Ζ S and 1 S Sieved 79 Ρ Publication N None Ø Other Ø Other w Owner 33 34 U User Frequency of Pumping G Geologic Inference Number of Grabs 27 Ø Other Constant (hourly) С F Frequent (daily) 35 36 Infrequent (weekly) % Organic Material (Field Estimate) 1 8 Rare (no recent use) З Card Number Depth to top of Producing Horizon **GENERAL WATER SAMPLES** 28 29 30 31 Water Sample Treatment WELL WATER (Meters) 37 Type of Well N None 81 Confidence of Producing Depth F Filtered Only Drilled D 3 2 Acidified Only С Drive Point н High Ρ Acidified and Filtered Α R Probable Dug G Ø Other S U Unknown Possible Other 0 Depth of Visibility (m) Source of Producing Depth Information Power Classification 38 39 40 33 1.9 C = ClearP Publication A Artesian Flow W Cwner E Electric 41 42 43 44 45 Conductivity U User G Gasoline (µmhos/cm) Geologic Inference G Wind W. Ø Other H Hand 46 47 48 Ø Other Diesolved O<sub>2</sub> (ppm) Total Well Depth Casing 34 36 36 37 2.0 49 50 51 None (Below Water Table) N Temperature (°C) (Meters) S Steel Galvanized G 52 53 54 Confidence of Total Depth P Plastic pН ♦ 38 U Unknown H High Ø Other 55 R Probable **Pipe Composition** Ρ pH by Lo-ton Paper S Possible 2 F Stoel 56 57 58 59 Source of Total Depth Information Z Galvanized Total Alkalinity (ppm) 39 CI Copper Þ Publications P Plastic 60 81 62 63 W Owner U Unknown P Alkalinity (ppm) U User Ø Other Geologic Inference G 84 65 88 67 Sample Location Other Ø M Alkalinity (ppm) 22 23 24 LAKE WATER Meters from Well Head Appearance of Water H = Holding Tank (Use Remarks) Type of Lake 68 55 С Clear Where Sample Taken With Respect To Pressure Tank N Natural м Murky A4 Manmade Algal Α 25 Ø Other Before B Lake Area After A 56 57 58 59 69 70 71 72 73 N No Pressure Tank Discharge (liters/min) From Pressure Tank (Use Remarks) (sq km) REMARKS (Card 4)

Figure 5 (Cont'd)

OAK RIDGE GEOCHEMICAL SAMPLING FORM

20

- Producing horizon code for wells;
- 7. Total well depth; and
- 8. Depth to top of producing horizons.

For interpretive purposes, information of value is as listed:

- 1. Contamination (wells, springs, and sediment);
- Dominant bed material (sediment);
- 3. Sample color (sediment)
- Odor of sample (wells and springs);
- 5. Field measurements for wells and springs [conductivity and temperature, pH, (specific conductance), T-alkalinity, and M-Alkalinity];
- Use of well;
- 7. Confidence of producing depth; and
- 8. Remarks.

#### GROUNDWATER SAMPLING

Groundwater (wells and springs) reconnaissance samples are collected from the nearest available site to the node of a 5.1 km (3.2 mi) grid. Maximum deviation from the grid node is 2.5 km (1.6 mi). Uniform geographic distribution of data points [one per 26 km<sup>2</sup> (10 mi<sup>2</sup>)] is obtained in this way.

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

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

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

Contaminants, such as corroded well casing or pipes and precipitate around pipes and faucets, are noted in the "Remarks" section of the field form, along with the fact of whether the well is very young or old. For more information on URE Project groundwater sampling, the reader is referred to "Procedures Manual for Groundwater Reconnaissance Sampling" (Uranium Resource Evaluation Project, March 1978).

#### STREAM SEDIMENT SAMPLING

The objective of the stream sediment reconnaissance is to collect samples from drainage basins that average  $26 \text{ km}^2$  ( $10 \text{ mi}^2$ ) in area. Because it is impossible to collect samples from basins that are only  $26 \text{ km}^2$  ( $10 \text{ mi}^2$ ), basins between 5.2 and 52 km<sup>2</sup> ( $2 \text{ and } 20 \text{ mi}^2$ ) are sampled (Phase 2). In areas where it is still impossible to obtain the desired coverage of Phase 2 basins, samples may be collected from larger or smaller basins and are designated as Phase G.

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

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 are recorded on the field form. Contamination (fertilizer, road material, soil or slump material from channel banks, mining and industrial activities, etc.) that is found within at least 100 m upstream from the site or appears on the topographic map within the basin are noted in the "Remarks" section of the field form.

For more information on stream sediment sampling, the reader is referred to the "Procedures Manual for Stream Sediment Reconnaissance Sampling (Uranium Resource Evaluation Project, May 1978).

#### STREAM WATER SAMPLING

Site selection for stream water reconnaissance sampling is the same as for stream sediment sampling. When stream sediment and stream water samples are being collected, the same site is used for both samples with the water sample being collected first to avoid collecting any finegrained material that may be stirred up in the collection of the sediment. Two polyethylene bottles and their caps are thoroughly rinsed before the sample is taken. Samples are collected as near the fast moving part of the stream as possible, avoiding collecting any organic material that may be present in the stream. After the sample is taken, all air bubbles are dislodged from the bottles, and the cap is sealed tightly, taped with vinyl tape, and sent to the laboratory for analysis.

#### OTHER SAMPLE TYPES

Other sample types that are 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. These types of samples have not been collected by this project to date.

#### ANALYTICAL PROGRAM

UCC-ND personnel have been routinely analyzing uranium-containing materials for three decades in the analytical laboratories at Oak Ridge, Tennessee. The uranium content of these materials have ranged from trace levels in soil and water samples to high-purity uranium compounds and metal for nuclear reactors and weapons. These capabilities were modified and developed to provide the analytical capacity necessary to support the URE Project. Procedures were selected for use based on the elements to be determined and the relative accuracy and cost.

SELECTION OF ELEMENTS FOR ANALYSIS

The selection of elements to be determined in water and sediment samples was based on a number of different factors. Past experience has shown that important associations for sandstone deposits exist between uranium and arsenic, molybdenum, selenium, and vanadium. A list of 19 other elements considered to be important for the NURE hydrogeochemical survey for uranium was obtained from the U.S. Geological Survey (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 given in Table 1.

#### SELECTION OF ANALYTICAL PROCEDURES

A series of groundwater 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. Other element determinations were made by emission spectrography, sequential atomic absorption analysis, SSMS, and neutron activation analysis. A comparison was made of costs, equipment, and

## Table 1

## DETECTION LIMITS OF VARIABLES DETERMINED IN WATER AND SEDIMENT SAMPLES

	······································	Curr	ent n limits	Previous Detection Limits by dc Arc
		Sediment	Water	Sediment
Variable	Current Method	(ppm)	(ppb)	<u>(ppm)</u>
U-FL	Fluorometry	0.25	9.2	
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
A1	Plasma Source Emission Spectrometry	0.05(a)	10	
В	Plasma Source Emission Spectrometry	10	8	20
Ba	Plasma Source Emission Spectrometry	Z	2	40
Be	Plasma Source Emission Spectrometry	ז	٢	
Ca	Plasma Source Emission Spectrometry	0.05(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	Z	2
Fe	Plasma Source Emission Spectrometry	0.05(a)	10	
Нf	Plasma Source Emission Spectrometry	15		
К	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
Мо	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
Р	Plasma Source Emission Spectrometry	5	40	1,000
РЬ	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	I	2	
Th	Plasma Source Emission Spectrometry	2		400
Ti	Plasma Source Emission Spectrometry	10	2	200
٧	Plasma Source Emission Spectrometry	2	4	10
Y	Plasma Source Emission Spectrometry	ĩ	1	10
Zn	Plasma Source Emission Spectrometry	2	4	200
Zr	Plasma Source Emission Spectrometry	2	2	40
S04	Spectrophotometry		5(b)	
C1	Spectrophotometry		10(b)	
(a)Detection	limits expressed in percent.			
(Detection	n limits expressed in ppm.			

 sensitivities. Since the analysis for uranium is 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 and the sensitivities for each measurement are listed in Table 2. These procedures will be discussed individually.

#### SAMPLE FLOW

Field samples are received in Oak Ridge at a central receiving station where they are entered into the records management system and distributed for analysis. Quality control samples are added, and a duplicate of each water sample is put in storage. Computer cards containing information about each sample are provided for use in the laboratory. After processing, the unused portions of sediment samples are returned to the receiving station for storage. These stored samples will be available for reanalysis or additional analysis as required.

#### DATA FLOW

As each sample is processed through the various procedures in the laboratory, it is identified by the six-digit number appearing on the sample container and the computer card. Results of each determination are recorded either in machine readable form or hard copy output. The laboratory data system will eventually provide for automatic identification of samples from the punched cards and recording of the output in machine readable form to eliminate all nonautomated data transcription processes.

#### QUALITY CONTROL

A quality control program has been 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 are anonymously submitted for analysis on a routine basis. A quality control group, which is administratively separate from the analytical laboratory, prepares the control samples, analyzes the results, and notifies the laboratory of any deviation of results from specified control levels. This allows the laboratory personnel to identify and correct instrumental or procedure problems and to correct questionable results prior to publication.

The results for all quality control samples are maintained in the computer data base and can be summarized in several different ways. Normally, control charts are plotted by computer for each element. Plots of all samples, or of samples analyzed over a selected period of time, can be obtained. The coefficients of variation for measurements made during the period of time when field samples from a specific area were being analyzed are reported along with the results from the area.

#### Table 2

#### PRINCIPAL ANALYTICAL METHODS

- Fluorometric Analyses Uranium Water Samples - 0.2 to 50 ppb Sediment Samples - 0.25 to 50 ppm
- Mass Spectrometry Uranium Water Samples - 0.02 to 25 ppb
- Neutron Activation Analysis (Delayed Neutron) Uranium Sediment Samples - 0.02 to 100 ppm
- 4. Atomic Absorption Arsenic and Selenium
- Emission Spectroscopy Multielement Analysis Using Inductively Coupled Argon Plasma Source (See Table 1 for the list of elements)
- Spectrophotometry Water Samples Sulfate - 5 to 300 ppm Chloride - 10 to 300 ppm

An additional quality control program for uranium measurements is conducted by the Ames Laboratory. Results of uranium analysis of water and sediment control samples obtained from the Ames Laboratory as part of the Multilaboratory Analytical Quality Control for the HSSR Program are reported regularly by D'Silva, et al (1979). Results indicate any bias developed and analyses which were not in control.

#### SAMPLE PREPARATION

Water sample preparation and all chemical analyses are 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 are retained in permanent storage for possible future use. Applicable detection limits for the elements determined and the elements determined for water and sediment samples are shown in Table 1.

Water samples are received in 250-ml polyethylene bottles. They are vacuum filtered through 0.45- $\mu m$  cellulose acetate paper and then submitted for analysis.

Stream sediment samples are dried overnight at  $85^{\circ}$ C, placed in a plastic envelope, and disaggregated by impact with a rubber mallet. The fraction passing through a 100-mesh (150-µm) nylon sieve is collected and blended. Approximately 2 g are loaded into a polyethylene rabbit for neutron activation analysis. Another 0.25 g aliquant is placed in a 100-ml Teflon beaker to which is added 5 ml of concentrated nitric acid, then 5 ml of concentrated hydrofluoric acid. The sample is placed on a hotplate at 250°C and evaporated just to dryness. Then, 20 ml of 30% nitric acid is added and the sample warmed. The contents of the beaker are transferred to a 50-ml centrifuge and diluted to volume with distilled water. This solution is submitted for analysis.

URANIUM ANALYSIS

#### Fluorescence Spectroscopy

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

#### Mass Spectrometry

Water samples from regions where the uranium concentration is expected to be below the lower reporting limit of fluorometric analysis are analyzed by isotope dilution thermal emission (IDTE) mass spectrometry. The instrument used for this analysis is a 6-in., 60° magnetic sector instrument with photon counting which was designed and built in Oak Ridge.

A 10-ml portion of a filtered water sample is spiked with  $5-\mu$ l of a 1 ppm solution of uranium-233 as an internal standard. The spiked sample is acidified with 100µl of concentrated nitric acid and extracted into 1ml of 2% TOPO in carbon tetrachloride. The aqueous layer is removed and the organic phase is air dried, leaving the dry TOPO and uranium. A 250-µl portion of a 10% ammonium carbonate solution is added to dissolve the residue, and 2 µl of this solution is placed on a rhenium filament for analysis. The uranium-233, -235, and -238 isotopes are measured. The uranium-233/uranium-238 value is used to calculate the concentration in the sample. Results are reported as ppb of the original sample.

#### Neutron Activation

In addition to the hot-acid leach process described for fluorescence spectroscopy, a total uranium value is determined for stream sediments by neutron activation using delayed neutron counting. Approximately 2 g of <100-mesh sediment material is loaded into a polyethylene rabbit. It is transferred through a pneumatic transfer system into a reactor where it is exposed to a neutron flux of 3 x  $10^{13}$  n/cm<sup>2</sup>-sec for 60 sec and then transferred to a neutron counter. Results are reported as ppm of the dry sediment.

#### ARSENIC AND SELENIUM ANALYSIS

Arsenic and selenium concentrations in water and aliquots of dissolved sediment are determined using hydride generation flameless atomic absorption spectroscopy. An automated, microprocessor-controlled, atomic absorption spectrometer is used for the analysis. Eighty filtered water samples or 60 dissolved sediment samples (diluted from 5 to 35 ml) are loaded in the sample changer. Twenty ml of each sample is 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 are collected in a rubber balloon and after 45 sec are 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 is measured, and the resulting peak height is recorded. A series of solutions with standard concentrations is analyzed as described and a least-squares fit to a second-order equation applied to the observed peak heights. The calculated coefficients are then used to calculate sample concentrations. During normal operation, every tenth sample is a known standard that must check to within  $\pm 15\%$  of the expected value.

#### EMISSION SPECTROCHEMICAL ANALYSIS

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

Detection limits for the ICAP source are given in Table 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, see Table 1. In this procedure, approximately 0.5 g of the <100-mesh fraction of the dried sediment sample is ground in an  $Al_2O_3$  mortar, and 15 mg is weighed and mixed on the balance pan with a level scoop of graphite (approximately 17 mg). This mixture is tamped into a thin-walled, undercut electrode with a 2 mm x 4 mm crater. The electrodes are maintained at 200°C until arced. They are arced until the sample is totally consumed (70 sec), using a current of 15 A dc, The concentration of each element is read from the photographed spectra by visual comparison to standards prepared by grinding oxides of the desired elements into a matrix of 60% SiO<sub>2</sub>, 15%  $Al_2O_3$ , 10% Fe<sub>2</sub>O<sub>3</sub>, 10% CaO, 2%  $Na_2CO_3$ , 2%  $K_2CO_3$ , and 1% MgO.

#### SULFATE AND CHLORIDE ANALYSIS

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

#### CONDUCTIVITY

The conductivity of water samples is measured in the laboratory using a Yellow Springs Instrument Company Model 31 conductivity meter. The results are reported in  $\mu$ mhos/cm.

#### DATA VERIFICATION

The large quantity of field and laboratory data collected by the NURE HSSR Program necessitates the use of computerized data verification procedures. In a reconnaissance program, a sample represents a relatively large area and, therefore, must properly reflect the environment from which it is taken. Thus, it is important that errors associated with data collection and laboratory analyses are minimized. In the Oak Ridge HSSR Program, as many as 190,000 samples may be collected, each having about 60 associated pieces of information. While it is not practical to have totally error-free data, the outlined procedures do ensure high-quality data. If these procedures were not performed by the organization which collected the data, many errors would go undetected and suspected errors might not be checked. These errors would reflect adversely on the NURE Program.

#### FIELD DATA VERIFICATION

Field forms (Figure 5) are completed at the time a sample is collected by the procedures given in the section on the "Field Geology Program". Since the majority of the field information is recorded on a checkoff system, misrecording of field information is minimized. A waterresistant label is attached to the field form and to the sample container. These preprinted sample numbers are entirely computer controlled.

Verification of the field form information is performed by a computerized system, except for a general review made by geologists when the field forms are filled out initially. The information that is checked in boxes is verified by a computerized system to ensure that acceptable codes have been keypunched. Map codes, collector's initials, surface geologic codes, and producing horizon codes are compared to master lists.

Samples are flagged if any numeric information, such as water width, is not within standard ranges. Reasons for the unusual results are then investigated. Consistency checks which compare two fields are also performed. For example, samples are flagged if the total and M alkalinity differ by more than 10%, or the pH is less than 8 and the P alkalinity is greater than zero. The final check on field conductivity, dissolved oxygen, water temperature, pH, and total alkalinity takes place in the principal component analyses when the laboratory data are verified. A complete list of continuous data checks is given in Table 3.

## Table 3

## DATA VERIFICATION FOR CONTINUOUS VARIABLES

	Variable	Verificat	ion	
Field Data		Day $\leq$ 31, $1 \leq$ Month $\leq$ 12	2, 76 ≦ Year ≦ 85	
	Air Temperature	-15 ≦ Air Temp. ≦ 40		
	Latitude	20 ≦ Lat ≦ 50, Within on standard for its quadr	e degree of angle	
	Longitude	Within two degrees of st quadrangle	andard for its	
	Stream Flow	≦1		
	Stream Width	≦10		
	Average Depth	≦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. $\leq$ 19,00	00	
	Discharge	≦5,000		
Waters	Dissolved Oxygen	.5 ≦ Diss. Oxygen ≦ 14		
Only	Н	4 ≦ pH ≦ 11, If P alkali should be < 0 or > 8	nity > 0 then pH	
	P Alkalinity	$0 \leq P$ Alkalinity $\leq 100$	The larger of the two	
	M Alkalinity	20 $\leq$ Alkalinity $\leq$ 2,000	measurements must not	
(	Total Alkalinity	$20 \leq Alkalinity \leq 2,000$	more than .1 times the	
Wells (	Depth of Producing Horizon	≦2,000	smaller value.	
Unity (	Well Depth	≦2,000		

Generally, field form errors stem from illegibility of recorded information or from keypunching. Since the preceding checks are made both at the time of data entry and prior to the data analysis, the error rate of data given in reports should be minimal.

#### MAP VERIFICATION

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

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

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

#### LABORATORY DATA VERIFICATION

Errors can be expected in processing the large volume of laboratory The source of the errors can stem from sample contamination, data. selective recording of sample numbers, or keypunching laboratory While the percentage of laboratory errors is small, these results. The large magnitude errors create errors can be large in magnitude. false anomalies and can seriously interfere with the evaluation of an Unfortunately, it is not possible to merely examine entire data set. Background values can be important in the the anomalous samples. evaluation of anomalous values. For example, background uranium values in a group of high uranium values can be an indication of a zone of precipitation.

Detection of suspected errors is automated by use of statistical procedures such as principal component analysis (Kane, et al, July 1977). The automated principal component procedure identifes errors in samples having unusually high or low values. However, the main advantage is that samples having two or more measurements that have an unusual relationship are also identified. Variables are selected for principal component error analysis which have a minimum of about 70% of the data above the laboratory detection limit. Values below the detection limit are assumed to be one-half the detection limit. To approximate normality, logarithms of the concentrations are used in the analysis for variables other than pH.

The principal component analysis produces an ordered list of extreme samples using eigenvalue related statistics. The outlier nature of the sample may be 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 are identified if single element measurements are outside a three-standard deviation confidence interval.

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

#### GEOSTATISTICS

DATA ANALYSIS

Statistical methodology is used in the HSSR Program for identifying local and regional geochemical trends which may be related to uranium provinces and districts. However, prior to detailed interpretation appropriate statistical summarization of the data is an important first step for any subsequent data evaluation. Since as many as 190,000 samples are scheduled to be collected by URE Project personnel, it is necessary to computerize most statistical procedures to enable timely release of the data. These automated procedures are developed through the interaction of geologists, statisticians, and computer scientists. This team effort is designed to efficiently produce meaningful geologic results that are statistically sound.

In the analysis of any data set, it may be useful to identify the association between chemical parameters to construct geochemical models or to associate sampling stations to construct regional patterns. Techniques such as correlation analysis, factor analysis, regression analysis, and cluster analysis of variables are helpful in the determination of the relationship between chemical parameters. These analyses identify possible chemical associations, which are used to construct uranium-related geochemical models. Using the uranium-related parameters and other selected variables, it is then desirable to determine the regional patterns in the data. Techniques such as contour plotting, trend surface analysis and weighted sum contours are used in conjunction with summary statistics, histograms, and probability plots. Finally, local differences or patterns can be assessed by a cluster analysis of stations or by a principal component analysis.

A brief description is presented of the statistical methods that have been found useful in the analysis of the URE geochemical data sets. Figure 6 shows how these procedures relate to the entire report preparation process.

#### DATA SUMMARIES

#### Tabular Statistical Summary

Tables of standard statistical measures are given for each chemical parameter determined in each sample type in the appendixes of the Basic Data Reports. The "Measurable Values" column in each table refers to the number of values above the laboratory limit of detection. The "Below Detection Limit" column refers to the number of samples with concentrations below the laboratory limit. If the chemical parameter was not measured in several samples, the measurable values and the values below the laboratory detection limit do not total the number of samples. The explanation and use of the summary statistics given within the table can be obtained from Snedecor and Cochran (1967). With the exception of the minimum, maximum, median, and mode, all computations in the data summaries use only determinations above the detection limit.

The mode of the population was estimated by the midpoint of the interval having the greatest number of observations. The interval size was t = [x(0.75) - x(0.25)]/15 where x(p) is the sample p-th percentile. If any concentration was below the laboratory detection limit, c, the interval size was the maximum of c and t.

Statistics computed from the logarithms of the concentrations are often The final four columns give means and useful for geochemical data. standard deviations computed from the natural logarithms of the elemental concentrations. The "Mean" and "Standard Deviation" use only data above the detection limit. The deletion of less than values in the computations results in a mean that is too high and a standard deviation that is too low relative to the overall population mean and standard deviation. Also, it is possible that large erroneous values would have the opposite affect. The final two columns give robust estimates of the mean and standard deviation of the chemical parameters. "Robust" implies that the influence of less-than values and unusually large values is minimized. If there are no less-than values, the robust mean computes the mean of the middle 90% of the data. If p>5% of the values are below the detection limit, the mean of the middle (100 - 2p)% of the data are used. Analogous estimates of the standard deviation are given, but the data are assumed to be lognormally distributed. The estimation procedure is not used if p > 40% of the data are below the detection limit since unreliable estimates might be obtained.





BLOCK FLOWCHART FOR PROCESSING OF URANIUM RESOURCE EVALUATION PROJECT DATA

#### Probability and Histogram Plots

The use of normal and lognormal probability plots is a standard technique for determining the distribution of chemical concentrations in nature (e.g., Sinclair, 1976 or Tennant and White, 1959). For parameters other than pH, the lognormal probability plot is presented. For plotting convenience, when the sample size was greater than 70, only 40 to 50 points were plotted from the 20th to 80th percentiles.

The histograms identify the concentration range and frequency with which ranges of values occur. Combined with the probability plots, it is possible to relate individual values to the sampled population. For both of the preceding plotting procedures, values below the lower laboratory detection limit were assumed to be one-half the detection limit.

#### Percentile Plots

It is possible to assess differences in levels of elemental concentrations in geologic units by plotting the minimum, 25th percentile, median, 75th percentile, 85th percentile and the maximum for each major geologic unit. The resulting percentile plots enable the evaluation of anomalous concentration levels in the different geologic units. The percentile plot is analogous to a probability plot (although normality or lognormality is not assumed), but is more useful in displaying a visual image of population differences.

Two types of plots are used depending on the range of the data. The usual plot displays in entire range of the data from the minimum to the maximum with the upper percentiles labeled by an angled line to the right of the centerline. The numerical value of the 85th percentile is given with a dash appearing on both sides of the centerline. A few very large values may make the range of variation too large for effective display. In these cases, the data are plotted from the minimum to the 85th percentile. The maximum value for the displayed geologic units are given at the top of the display, the remainder of the labelling is the same as the first type of percentile plot. Percentile plots are not given for a geologic code if the number of samples with that code is less than five; however, these samples are included in the plot using all codes.

#### Contoured Canadian Symbol Maps

Geochemical maps which use symbols to represent ranges of concentrations are presented primarily as a basic summary of the data and are useful in indicating areas of interest in uranium exploration (Jenks, 1973). The Canadian Geological Survey symbol system (e.g., Hornbrook and Garett, 1976) is used where the symbols are divided into two groups of seven and one additional symbol (a shaded star, as shown in Table 4).

## Table 4

## STATISTICAL DISTRIBUTION SYMBOLS AND PERCENTILE RANGES FOR GEOCHEMICAL PLOTS

Symbol	No.	Percentile Range
+	1	c < D.L. <sup>(a)</sup>
×	2	D:.L. < <c 2.0<="" <="" td=""></c>
•	3	2.0 < c <u>&lt;</u> 5.0
•	4	5.0 < c <u>&lt;</u> 9.0
0	5	9.0 < c <u>&lt;</u> 16.0
0	6	16.0 < c <u>&lt;</u> 28.0
0	7	28.0 < c <u>&lt;</u> 42.0
$\odot$	8	42.0 < c <u>&lt;</u> 58.0
$\odot$	9	58.0 < c <u>&lt;</u> 72.0
$\odot$	10	72.0 < c < 84.0
۲	11	84.0 < c <u>&lt;</u> 91.0
۲	12	91.0 < c <u>&lt;</u> 95.0
•	13	95.0 < c <u>&lt;</u> 98.0
	14	98.0 < c <u>&lt;</u> 99.0
*	15	99.5 < c

 $(a)_{\text{Detection Limit}}$ 

The first group of seven increase in size from a small cross to a large open circle. The cross denotes samples with concentrations below the laboratory detection limit. In the second group of seven symbols, the dot in the circle becomes progressively larger with the increase of the concentration range. The final shaded star symbol denotes samples with concentrations appreciably above the typical concentration range. Thus, the symbols increase in size and/or shading as the concentration range increases.

It is desirable that intervals associated with symbols be the same over large areas, regardless of the particular data set being reported. Thus, all symbols may not appear on a given map, but over a wide geographic area the entire symbol range would be used.

Wherever possible, the same symbol intervals are used for adjacent quadrangles to aid comparison of the two areas. A common symbol range is used if the 50th percentile value for an element in the new quadrangle falls within the 7th, 8th, or 9th symbol interval for that element in a previously reported adjacent quadrangle. If the new data set is not compatible with the old symbol intervals, new intervals are determined for that element. The new symbol intervals are taken from the histogram plots. The mid points of the intervals represent the 0.5, 1.05, 3.5, 7.0, 12.5, 22, 35, 50, 65, 78, 87.5, 93, 96.5, 98.75, and 99.5 percentile values for that data set.

To anotate the high and low areas on the Canadian symbol plots, the 85th and the 15th percentile (or the detection limit if more than 15% of the data are below the detection limit) for each parameter is contoured.

The algorithm used in generating the contours involves interpolating the random sample points from a rectangular grid having intervals of size equal to plotting scale  $\div$  250,000 min. The value of each grid point is established by weighting all the sample points by the weighting function

 $w = \begin{cases} 0 \text{ if } d > c \text{ mi} \\ d^{-p} \text{ if } d \leq c \text{ mi} \end{cases}$ 

where d is the distance between the grid point and a sample point. Values of c = 10 mi (16 km) and p = 3 were determined to produce the most appropriate contours. Contour lines are not given if a grid point is not within 10 mi (16 km) of a sample point. For variables other than pH, the logarithm of the concentration is used in the interpolation computations.

#### ASSOCIATION BETWEEN CHEMICAL PARAMETERS

#### Correlation

Correlations can be used to determine the degree of association between two variables. Pearson and Spearman correlations are computed for all pairs of variables for each individual sample type (Kane, et al, 1977). A sample is omitted in calculating a correlation between two variables if either variable has a concentration below the laboratory detection limit.

It is of interest to test the hypothesis that there is no correlation between two variables. If this hypothesis is rejected, some degree of association may be assumed for two variables in the region of sampling. For the Pearson correlation, a t-statistic is computed and the significance range indicated (Snedecor and Cochran, 1967, Page 184). Significance levels for the Spearman correlation are computed by a procedure described in Zar (1972).

A standard Pearson correlation (Snedecor and Cochran, 1967, Page 172) is meaningful only for normally distributed data. Thus, logarithms of all element concentrations are taken where the probability plots indicate that the lognormal distribution is appropriate. It is important to note that the Pearson correlation measures only the linear relationship between the variables and that possible nonlinear relationships are not assessed.

The Spearman nonparametric correlation is also computed (Snedecor and Cochran, 1967, Page 193) because the normal and lognormal approximation may not always be appropriate. The Spearman correlation is based on a ranking of concentrations and is not as sensitive to outliers or erroneous data as is the Pearson correlation.

#### Cluster Analysis of Variables

The cluster analysis of the correlation matrix (Parks, 1966) provides an efficient method to determine groups of correlated variables. Dendrograms, such as Figure 7 are examined in conjunction with the correlation matrix to determine groups of variables that are related. In general, variables in different groups are not as highly correlated as variables within the same group. The correlation matrix should be examined directly for significant relationships between pairs, as well as whether the correlation is positive or negative.

The Pearson correlations are computed as described in the Correlation section and the variable groups are formed using the metric one minus the absolute value of the correlation. With uranium first, the variables are ordered in the correlation matrix according to the indicated grouping. The correlation matrix is divided by lines to indicate the groups. The clustering in no way alters the correlations and can be thought of as a convenient organization of the correlations.

#### Factor Analysis

R-mode factor analysis is a means of summarizing the relationships among a group of variables with a reduced number of new variables called



1. -ABS (PEARSON CORRELATION)

Figure 7

COMPLETE LINKAGE CLUSTER ANALYSIS OF VARIABLES

\*Total Alkalinity \*\*Specific Conductance factors (Jöreskog, et al, 1976; McCammon, 1975, Page 21; Morrison, 1967, Page 259). If the statistical models are appropriate, the set of factors will provide a concise description of the intervariable structure of the original variables. Each factor represents some trait common to those original variables that correlate highly with the factor. A factor score is a realization of this common trait and is just a linear combination of the original variables.

An iterated principal axis factor analysis is performed for a sample type (Barr, et al, 1976). The resulting matrix of loadings, correlations of original variables with factors, called the factor pattern matrix is rotated using the VARIMAX method (Morrison, 1967, Page 284) and is presented in table form. To test model adequacy, a residual matrix (Morrison, 1967, Page 271) is computed by subtracting the reproduced correlation matrix from the observed Pearson correlation matrix. This aids in the final model selection process. The factor scores are then geographically plotted and contoured.

#### ASSOCIATION BETWEEN SAMPLING STATIONS

#### Weighted Sum Maps

General regional patterns can be obscured by automatic contouring algorithms because of natural variations in single-element concentrations. Combining several uranium-related elements on a contour map may smooth the data sufficiently and enable the regional patterns to be identified. This technique differs from most smoothing methods in that the multielement characteristics of a sample, rather than mathematical methods, are responsible for the smoothing.

In forming a weighted sum of elements, it is first necessary to select the elements that are related to uranium. Additionally, a relative importance for each element must be assigned. To sum the uraniumrelated variables, which may have large differences in the range of variation, it is also necessary to standardize all variables. The difference in scale can be adjusted by dividing each observed concentration for a variable by the interquartile range of the variable. The interquartile range (75th percentile - 25th percentile) was used instead of the standard deviation because of the large amount of less than (censored) data typically present in geochemical studies. If the 25th percentile was a censored value one-half the detection limit is used in the calculations. Robust estimates of the mean and standard deviation are used when less than 40% of the data for each element is below the laboratory detection limit.

After the standardizing constants are determined, the weighted sum for each sample is computed by multiplying the concentration of an element by the weight used for the element and then dividing the interquarile range for the element. This sum is multiplied by a constant (e.g., 10 or 100) to enable plotting numbers of the desired magnitude. Finally, the weighted sum numbers are plotted geographically and contoured.

#### Cluster Analysis of Sampling Stations

The similarity in compositon of samples can be used to formulate a geochemical pattern for a survey area. Cluster analysis can be used in geology (Parks, 1966) to associate stations which are most similar, with respect to an array of chemical parameters. Dendrograms, as in Figure 8, can be used to construct a sample station association hierarchy. Samples representing stations which are connected by lines on the dendrogram can be considered associated at the level indicated on the The lower the line connecting samples the more closely vertical scale. associated the samples. Dividing the dendrogram into different parts produces a natural grouping of the sampling stations. Each group is assigned a symbol and the stations plotted geographically. The resulting cluster analysis plot enables geographic or geologic significance to be attached to station groupings. Additionally, clusters of stations can be compared with other clusters by comparing the summary statistics for the station groups.

Variables in the cluster analyses are weighted so that levels of importance can be assigned to different variables. For example, if it is desired to cluster samples that have similar uranium concentration, uranium would have a high weight. However, it is still possible that samples having similar uranium concentrations could cluster in different groups because of differences in other elements.

For all variables, a log transformation was considered in an attempt to make the standard deviation constant for different concentration levels. The transformation selected and the subjective weights appear in the margin of the cluster plots. All cluster analyses use either the rank score metric (Bayne, et al, 1979) or the Euclidean distance with the minimum standard deviation within the clusters as the clustering criterion (Kane, et al, November 1977).

#### REPORT PRESENTATION AND DATA RELEASE FORMAT

The compilation of field and laboratory data of a  $1^{\circ} \times 2^{\circ}$  NTMS quadrangle is presented in the form of a basic data report, as required by DOE as soon as possible after the completion of the quadrangle sampling, laboratory analysis, data verification, and data summarization.

#### REPORT REQUIREMENTS

Within the Basic Data Report, the following major sections are required:

- 1. Geology A section describing the geology of the area, with emphasis on its relation to known or possible uranium occurrences.
- 2. Sample Collection A section which gives the time the samples were collected and analyzed. This section also contains a description of major sources of contamination within the quadrangle.





Figure 8

- 3. Chemical Analysis A section which gives a general description of sample preparation for chemical analysis. This section provides a reference for the analytical procedures used.
- 4. Quality Control A section describing the use of calibration control, and check samples to check the accuracy and precision of analyses. Also included is a description of the principal component error analysis, which identifies the samples with the most unusual set of chemical data which are submitted for reanalysis as a check against possible analytical error.
- 5. Geochemical Results A section summarizing the geochemical results, with areas of known or potential uranium occurrences pointed out. Uranium and conductivity are required to be discussed for water samples and uranium and thorium are required for stream sediments. Geographic plots of these variables plus a site location map of each sample type and a geologic map is included as plates at 1:250,000 scale.
- 6. Appendixes Groundwater and stream sediment data are presented in Appendixes A and B, respectively. A microfiche of all field and laboratory data is included in Appendix C.

ADDITIONAL INFORMATION

Data for variables, other than those required, may be included in the geochemical results section if one of the following two criteria are met:

- 1. There is a known or observed relation between the variable and the distribution of uranium in the quadrangle or in similar geologic environments elsewhere.
- 2. If the statistical correlation table indicates a significant correlation coefficient between uranium and the variables.

Probability, frequency, and percentile plots along with geochemical distribution maps (at the scale 1:1,000,000) and a partial data listing for selected variables are included in the appendixes for all variables discussed.

COMPUTER TAPE FORMAT FOR DATA RELEASE

The ORGDP URE Project record is normally processed as a variable length record with a mixture of character, binary, and floating point data. However, for ease of processing by other installations, an alternate format is used for the data tapes which are released by the URE Project. The data on the tape is arranged in a sequential file consisting of 2,668-byte fixed-length records ordered by a six-digit sample number. The first record on the file is an identification record which contains

approximately 70 characters of bibliographic information describing the quadrangle being reported. This identification record is the same length as all other in the file and is provided for identification. Each additional record corresponds to a geochemical sample and contains field and laboratory data arranged in a fixed-length header section followed by fixed-length segments. The header portion (526 bytes) of the record contains general field site information for each sample type followed by selected laboratory analytical results. Indicators in Positions 495 to 526 of the header segment indicate the presence or absence of data within the fixed length segments. These additional segments of the record contain general water field data, well water field data, botanical field data, multielement laboratory analysis, field analysis data, or comments, depending on the sample type described in the header section.

A brief description of each data element within a sample record is presented in Table 5. Included are the specific data elements and standard variable codes, the field format, and the relative location of data within the record. The field form shown in Figure 5 identifies the field data codes appearing in a record. For field measurements, missing values will be indicated by a -1. Specific procedures, which may be used to determine geochemical measurements reported for a sample, are listed in Table 6. For these measurements, missing values are stored as -99999. The IBM-created tape specifications are: 9 Track, LABEL= (1,SL), DEN=800 BPI, DCB=(RECFM=FB,LRECL=2668,BLKSIZE=5336), EBCDIC characters on tape - (no binary or floating point data).

### Table 5

#### LOCATION OF DATA ELEMENTS WITHIN A SAMPLE RECORD FOR URANIUM RESOURCE EVALUATION PROJECT GEOCHEMICAL SAMPLES

	Data Description	Variable Code	Field Format	Relative Position
FIX	ED HEADER INFORMATION - LENGTH 526 BYTE	ES		
1.	Sample Number	S-N	X(6) <sup>(a)</sup>	1-6
2.	Record Length Counter		χ(4)	7-10
3.	Map Code: 1:250,000	MAP	X(6)	11-16
4.	Site: Unique within Map Code	SITE	X(4)	17-20
5.	Fill Area		X(3)	21-23
6.	Replicate Number	REP	X(1)	24
7.	Initials of Sampler	COLL	X(3)	25-27
8.	Sample Phase	PHAS	X(1)	28
9.	Type of Quality Control Sample	CONT	X(1)	29
10.	Sample Type	TYPE	X(1)	30
11.	Geologic Unit Code at Site	S-GC	X(4)	31-34
12.	Contaminants at Site	CAMT	X(1)	35
13.	Dominant Bed Material at Site	BED	X(1)	36
14.	Sample Color	SC	X(5)	37-41
15.	Type Vegetation at Site	TVEG	X(1)	42
16.	Density of Vegetation at Site	DVEG	X(1)	43
17.	Local Relief at Site	RELF	X(1)	44
18.	Weather Conditions	WIND/SKY	X(2)	45-46
19.	Water Level at Site	SLVL	X(1)	47
20.	Flag to Indicate if Analytical Results are Requested by Landowner		X(1)	48
21.	Sample Odor	ODOR	X(1)	49
22.	Filler Area		X(1)	50
23.	Hour Sample Taken	HOUR	X(2)	51-52
24.	Date Sample Taken (Stored as YYMMDD)	YEAR/ MON/DAY	X(6)	53-58

 $(a)_{X(n)}$  denotes an alphanumeric string of length n.

	Data Description	Variable Code	Field <u>Format</u>	Relative Position
25.	Air Temperature at Site (Centi- grade)	ATEM	<u>+</u> XXXXX.XXX	<b>59-</b> 68
26.	Latitude of Site (Stored as fractional degrees)	LAT	<u>+</u> XXXXX.XXX	<b>69-</b> 78
27.	Longitude of Site (Stored as fractional degrees)	LONG	<u>+</u> XXXXX.XXX	79- 88
28.	Stream Flow at Site (-l=stagnant)	FLOW	<u>+</u> XXXXX.XXX	89- 98
29.	Stream Width at Site	WIDE	<u>+</u> XXXXX.XXX	99-108
30.	Stream Depth at Site	S-DP	<u>+</u> XXXXX.XXX	109-118
31.	Arsenic & Selenium Lab Batch Number:	BTAS	X(5)	119-123
32.	Sulfate & Chloride Lab Batch Number:	BTSC	X(5)	124-128
33.	Lab Procedure Number for Selected Measurements		X(2)	129-130
	Current Lab Procedures:			
	<ul> <li>O1 Uranium by Mass Spectro- metry</li> </ul>	U-MS		
	<ul> <li>02 Uranium by Fluorescence</li> </ul>	U-FL		
	<ul> <li>05 Uranium by Neutron Acti- vation</li> </ul>	U-NT		
	<ul> <li>10 Orthophosphate</li> </ul>	OPO		
	• 11 Nitrate	NO3		
	• 12 Tin	SN-A		
	• 13 Mercury	HG		
	<ul> <li>14 Helium/Neon Ratio</li> </ul>	H/N		
	<ul> <li>15 Radon</li> </ul>	RN		
34.	Lab Batch Number for Selected Measurements		X(5)	131-135
35.	Selected Element Concentration		<u>+</u> XXXXX.XXX	136-145

	Data Description	Variable Code	Field Format	Relative Position
<del></del>			Tormac	103101011
36-74.	The preceding three data elements repeat 13 more times (next 221 bytes)			146-366
75.	Fill Area		X(10)	367-376
76.	Alternate Sample ID	ASID	X(12)	377-388
77.	Stream Order Number	STOR	X(6)	389-394
78.	Basin Identification	BNID	X(4)	395-398
79.	Chloride Concentration	CL.	<u>+</u> XXXXX.XXX	399-408
80.	Sulfate Concentration	S04	<u>+</u> XXXXX.XXX	409-418
81.	Laboratory Conductivity	CT-L	<u>+</u> XXXXX.XXX	419-428
82.	Arsenic Concentration	AS	<u>+</u> XXXXX.XXX	429-438
83.	Selenium Concentration	SE	<u>+</u> XXXXX.XXX	439-448
84.	Lake Area	AREA	<u>+</u> XXXXX.XXX	449-458
85.	Type of Lake	TLAK	X(1)	459
86.	Sediment Condition	COND	X(1)	460
87.	Sediment Treatment	STRT	X(1)	461
88.	Fill Area		X(1)	462
89.	Number of Grabs to Collect Sediment Sample	GRAB	X(2)	463-464
90.	Filler		X(6)	465-470
91.	% Organic Material	ORGA	X(2)	471~472
92.	Fill Area		X(2)	473-474
93.	Basin Centroid Latitude (Stored as fractional degrees)	CLAT	<u>+</u> XXXXX.XXX	475-484
94.	Basin Centroid Longitude (Stored as fractional degrees)	CLON	<u>+</u> XXXXX.XXX	485-494

	Data Description	Variable <u>Code</u>	Field Format	Relative Position
The f data conta	ollowing 7 count fields indicate the for the segment. If the count is 0, ins the data will contain blanks.	presence (> the segment	0) or absence which normal	e (=0) of  ly
95.	General Water Count:		X(4)	495-498
96.	Well Water Count:		X(4)	499-502
97.	Botanical Count:		X(4)	503-506
98.	Extra Multielement Analysis Count:		X(4)	507-510
99.	Multielement Count:		X(4)	511-514
100.	Field Analysis Count:		X(4)	515-518
101.	Filler Area		X(4)	519-522
102.	Comment Count:		X(4)	523-526
GENER	AL WATER SEGMENT - LENGTH 112 BYTES			
103.	Treatment of Water Samples	WTRT	X(1)	527
104.	Appearance of Water	APPR	X(1)	528
105.	Filler		X(2)	<b>529-53</b> 0
106.	Depth of Visibility (-l=Clear)	VISB	<u>+</u> XXXXX.XXX	531-540
107.	Water Temperature	WTEM	<u>+</u> XXXXX.XXX	541-550
108.	Ph Measured by a Meter	РН	<u>+</u> xxxxx.xxx	551-560
109.	Ph Measured by Lo-Ion Paper	LIP	<u>+</u> XXXXX.XXX	561-570
110.	Field Conductivity	CT-F	<u>+</u> XXXXX.XXX	571-580
111.	Discharge Rate of Water	DISC	<u>+</u> xxxxx.xxx	581-590
112.	Total Alkalinity	T-AK	<u>+</u> xxxxx.xxx	591-600
113.	P Alkalinity	P-AK	<u>+</u> xxxxx.xxx	601-610
114.	M Alkalinity	M-AK	<u>+</u> XXXXX.XXX	611-620
115.	Geologic Unit Code of Producing Horizon	P-GC	X(4)	621-624

	Data Description	Variable <u>Code</u>	Field Format	Relative <u>Position</u>
116.	Confidence of Unit Code	CPGC	X(1)	625
117.	Source of Unit Code	SPGC	X(1)	626
118.	Fill Area		X(2)	627-628
119.	Dissolved Oxygen (-l=no reading)	DO	<u>+</u> XXXXX.XXX	629~638
WELL	WATER SEGMENT - LENGTH 42 BYTES			
120.	Type of Well	TWEL	X(1)	639
121.	Well Power Classification	PCLS	X(1)	640
122.	Well Casing	CASE	X(1)	641
123.	Well Pipe Composition	PIPE	X(1)	642
124.	Well Sample Location with Respect to Pressure Tank	PRT	X(1)	643
125.	Use of Well	WUSE	X(1)	644
126.	Frequence of Use	PUMP	X(1)	645
127.	Fill Area		X(1)	646
128.	Meters from Well Head Where Sample Taken	WLOC	<u>+</u> XXXXX.XXX	647~656
129.	Depth of Well Producing Horizon	D-P	<u>+</u> XXXXX.XXX	657-666
130.	Confidence of Depth	CD-P	X(1)	667
131.	Source of Depth	SD-P	X(1)	668
132.	Confidence Well Depth	CWDP	X(1)	669
133.	Source Well Depth	SWDP	X(1)	670
134.	Well Depth	W-DP	<u>+</u> XXXXX.XXX	671-680
BOTAI	NICAL SEGMENT - LENGTH 36 BYTES			
135.	Number of Plants Sampled	NP	X(2)	681-682
136.	Type Deciduous Tree	DECD	X(1)	683
137.	Type Conifer	CONF	X(1)	684

	Data Description	Variable Code	Field Format	Relative Position
138.	Type Bush	BUSH	X(1)	685
139.	Type Moss	MOSS	X(1)	686
140.	Type Algae	ALGE	X(1)	<b>6</b> 8 <b>7</b>
141.	Fill Area		X(9)	688- 696
142.	Trunk Diameter	TDIA	<u>+</u> XXXXX.XXX	697- 706
143.	Plant Height	HITE	<u>+</u> XXXXX.XXX	707- 716
EXTRA	MULTIELEMENT SEGMENT - LENGTH 297 BY	TES		
144.	Date of Analysis (YYMMDD)		X(6)	717- 722
145.	Procedure Used (Procedure 03 only)		X(2)	723- 724
146.	Batch # Lab Used		X(5)	725- 729
147.	Multielement Concentrations: (in the order: AG, AL, B, BA, BE, CA, CO, CR, CU, FE, LI, MG, MN, MO, NA, NB, NI, P, PB, PT, SC, TH, TI, U, V, Y, ZN, ZR)		28( <u>+</u> XXXXX.XXX)	730-1009
148.	Fill Area		X(4)	1010-1013
MULTI	ELEMENT SEGMENT - LENGTH 517 BYTES			
149.	Number of Elements Measured		X(2)	1014-1015
150.	Fill Area		X(2)	1016-1017
151.	Date Multielement Spectrographic Work Received from Lab (Stored as YYMMDD)	DAYL	X(6)	1018-1023
152.	Lab Procedure Used to Obtain Multielement Analysis (Procedure O8)	)	X(2)	1024-1025
153.	Lab Batch Number for Multi- element Measurements	BATC	X(5)	1026-1030

		D	ata Description	Variable <u>Code</u>	Field Format	Relative Position
154.	Mui Or CR NI ZN A (or the of	ltie der: , CU , P, , ZR nega ther e co det	<pre>lement Concentrations in AG, AL, B, BA, BE, CA, CO, FE, LI, MG, MN, MO, NA, NB, PB, PT, SC, TH, TI, U, V, Y, K, SR, SI, SN, CE, HF, LA. tive value for an element than -99999) indicates that ncentration was below level ection.</pre>		50( <u>+</u> XXXX.XXX)	1031-1530
FIELD	IELD ANALYSIS SEGMENT					
155.	Number of Fields for this segment			X(4)	1531-1534	
156.	Measurement Procedure Number			X(2)	1535-1536	
	Presently, the field analysis lab					
	pr	oced	ures are:			
	•	31	Total Gamma-Scintillometer	TGAM		
	•	32	Gamma Spectrometer (Total Counts, e Potassium, Potassium, e Uranium, Uranium, e Thorium, Thorium)			
			Total Counts	QTOT		
			e Potassium	ΕK		
			Potassium	СРК		
			e Uranium	EU		
			Uranium	CPU		
			e Thorium	ETH		
			Thorium	CPTH		
	•	34	Uranium	FU		
	•	35	Fluoride	FFL		
	•	36	Nitrate	FN03		
	•	37	Sulphate	FS04		

	Data Description	Variable Code	Field Format	Relative Position
	<ul> <li>38 Phosphate</li> </ul>	FPO		
	<ul> <li>39 Ferrous Iron</li> </ul>	FFE2		
	• 40 Total Iron	FFET		
	• 41 Turbidity	TURB		
157.	Count of Elements Analyzed by this Procedure		X(2)	15 <b>37-</b> 1538
158.	Field Elements		8( <u>+</u> XXXXX.XXX)	1539-1618
159-173.	The preceding three data elements may repeat up to five more times (next 420 bytes) depending on the count of the total number of fields present.			1619-2038
COMMENT SEGMENT				
174.	Comment (Comments are left justified and padded on the right with blanks)	COMM	630 <b>(X)</b>	2039-2668

## Table 6

## QUANTITATIVE MEASUREMENTS ASSOCIATED WITH ORGDP SAMPLE

Procedure Number	Data Description	Location in Computer Record
01	Uranium by Mass Spectrometry	129- 366
02	Uranium by Fluorescence	129- 366
03	Multielement Measurements by DC Arc Source Spectrography (Ag, Al, B, Ba, Be, Ca, Co, Cr, Cu, Fe, Li, Mg, Mn, Mo, Na, Nb, Ni, P, Pb, Pt, Sc, Th, Ti, U, V, Y, Zn, Zr)	730-1009
04	Arsenic/Selenium	429- 448
05	Uranium by Neutron Activation	129- 366
06	Sulfate/Chloride	399- 418
07	Laboratory Conductivity	419- 428
08	Multielement Measurements by Inductively Coupled Plasma Source Spectrography (Ag, Al, B, Ba, Be, Ca, Co, Cr, Cu, Fe, Li, Mg, Mn, Mo, Na, Nb, Ni, P, Pb, Pt, Sc, Th, Ti, U, V, Y, Zn, Zr, K, Sr, Si, Sn, Ce, Hf, La)	1031-1530
10	Orthophosphate	129- 366
11	Nitrate	129- 366
12	Tin (SN-A) by Atomic Absorption	129- 366
13	Mercury (HG) by Atomic Absorption	129- 366
14	Helium/Neon Ratio	129- 366
15	Radon	129- 366
31	Total Gamma Measured in the Field	1539-2038
32	Field Gamma Spectrometer Measurements (Total Counts, e Potassium, Potassium, e Uranium, Uranium, e Thorium, Thorium)	1539-2038
34	Uranium Measured in the Field	1539-2038
35	Fluoride Measured in the Field	1539-2038
36	Nitrate Measured in the Field	1539-2038
37	Sulfate Measured in the Field	1539-2038
38	Phosphate Measured in the Field	1539-2038
39	Ferrous Iron Measured in the Field	1539-2038
40	Total Iron Measured in the Field	1539-2038
41	Turbidity Measured in the Field	1539-2038

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