GJBX-64 '76

K/UR-3
Part 1

NATIONAL URANIUM RESOURCE EVALUATION PROGRAM

HYDROGEOCHEMICAL AND STREAM SEDIMENT RECONNAISSANCE PROGRAM IN CENTRAL UNITED STATES

FY 1976A
July 1, 1976 Through September 30, 1976

J. W. Arendt, Project Manager
Uranium Resource Evaluation

November 24, 1976

UNION CARBIDE
OAK RIDGE GASEOUS DIFFUSION PLANT
OAK RIDGE, TENNESSEE

prepared for the **U.S. ENERGY RESEARCH AND DEVELOPMENT ADMINISTRATION**
under **U.S. GOVERNMENT** *Contract W-7405 eng 26*

HIGHLIGHTS

NORTHWEST TEXAS PILOT SURVEY (Page 10). The pilot survey report for Northwest Texas was prepared for open filing by the U.S. Energy Research and Development Administration.

FIELD FORM (Page 12). The final major revision for field forms was completed.

STATUS OF PRODUCTION SAMPLING PROGRAM (Page 13). Maps are presented which depict the areas sampled in the Phase I and Phase II Production Program.

STATUS OF PILOT SURVEY (Page 13). Pilot surveys were conducted in Minnesota, Wisconsin, North Dakota, and South Dakota. A pilot survey was begun in the Upper Peninsula of Michigan.

CLEAN ROOM LABORATORY (Page 21). Modifications to the Uranium Resource Evaluation (URE) Project Clean Room Laboratory were completed.

QUALITY CONTROL (Page 35). Results of the URE Project Quality Control Program are presented.

CONTENTS

NATIONAL URANIUM RESOURCE EVALUATION PROGRAM
QUARTERLY PROGRESS REPORT

INTRODUCTION

The National Uranium Resource Evaluation (NURE) Program is being administered by the Grand Junction Office (GJO) of the U. S. Energy Research and Development Administration (ERDA). One part of this program consists of a Hydrogeochemical and Stream Sediment Reconnaissance Program (HSSR) of the United States, including Alaska. The Nuclear Division of Union Carbide Corporation (UCC-ND) is conducting this survey over a 2,500,000-km^2 (1,000,000 mi^2) area of the Central United States. The survey includes the states of Texas, Oklahoma, Kansas, Nebraska, South Dakota, North Dakota, Minnesota, Wisconsin, Michigan, Indiana, Illinois, and Iowa.

SUMMARY

The production sampling program for the Uranium Resource Evaluation (URE) Project continued in the Texas Gulf Coast and was started in Northwest Texas. Pilot surveys were completed in North Dakota, South Dakota, Minnesota, and Wisconsin; and a pilot survey was started in the Upper Peninsula of Michigan.

The Northwest Texas Pilot Survey was prepared for open filing by ERDA. Modification of the URE Clean Room Laboratory was completed. The laboratory layout is presented, and the analytical instrumentation is described.

A project status and summary of UCC-ND activities during the quarter are presented in the following sections of this report. Also included is a listing of plans for the First Quarter FY 1977.

PROJECT STATUS

1. The UCC-ND URE Program is on schedule, and technical progress is satisfactory.

2. FY-1976A expenditures were $557,933 operating funds and $25,000 capital funds.

3. Sample teams are working in the field, samples are being analyzed in the URE Clean Room Laboratory, and results are being recorded in the URE data base.

4. The quality of field data remains satisfactory.

5. Quality control and data management programs are satisfactory.

6. URE archival samples are being placed in retrievable storage.

7. Samples for determination of the natural variation of uranium concentration with time are being received on a regular schedule from Texas.

FY-1976A PLANS VERSUS ACCOMPLISHMENTS

The fourth quarter plans versus accomplishments of the URE Project are as follows:

1. All pilot surveys were completed as-planned, with the exception of Michigan. The Michigan Upper Peninsula survey was started in September 1976 and will be completed in October 1976. An additional 30 well samples will be collected in Wisconsin in early October 1976.

2. Tentative agreements for the collection of base station samples by the United States Geological Survey (USGS) personnel at gaging stations were reached with USGS personnel in Wisconsin, Minnesota, North Dakota, Michigan, Indiana, and Illinois. Final agreements are pending URE Project evaluation of satellite photographic data for final site selection.

3. Planning for the Indiana Pilot Survey was delayed until FY 1977, based on information obtained from personnel of the Indiana and Illinois Geological Surveys.

4. Television, radio, and newspaper releases were not sent to Indiana, due to the rescheduling of the pilot survey.

5. The URE Clean Room Laboratory modification and equipment installation were completed as-planned. All equipment is installed and operational, with the exception of the Jarrell-Ash* Emission Spectrograph which is in the final stages of calibration and automation.

6. A few contacts were made with private industry to obtain comments on the URE Project. It has been decided that a more meaningful sample could be obtained by formally requesting comments after the Northwest Texas Pilot Survey has been open filed. Results of this survey are expected in the second quarter of FY 1977.

7. The remainder of the FY-1976A plans, identified in the Fourth Quarter FY-1976 progress report,** were accomplished as-planned.

FIRST QUARTER FY-1977 PLANS

1. The Michigan Pilot Survey will be completed.

2. A production report on South Texas, which includes 1-1/2 quadrangles and verifies the UCC-ND Project concept, will be completed.

3. The URE Project work plan will be updated.

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*Reference to a company or product name does not imply approval or recommendation of the product by Union Carbide Corporation or the U. S. Energy Research and Development Administration to the exclusion of others that may meet specifications.

**Arendt, J. W., *National Uranium Resource Evaluation Project, Hydrochemical and Stream Sediment Reconnaissance Program in Central United States, Fourth Quarter FY 1976, April 1, 1976 through June 30, 1976,* Union Carbide Corporation, Nuclear Division, Oak Ridge Gaseous Diffusion Plant, Oak Ridge, Tennessee, August 6, 1976 (K-TL-524, Part 5). UNCLASSIFIED.

4. Production sampling will continue in Northwest Texas and the Texas Gulf Coast.

5. Final selection of base stations will be concluded for all 12 states. Negotiations will begin with the USGS for collection of samples.

6. Calibration and automation will be completed on the Jarrell-Ash Emission Spectrograph.

7. An information meeting to present an in-depth review of the URE Project will be held.

ADMINISTRATIVE

PROJECT PERSONNEL

URE Project personnel, as of September 3, 1976, includes the following:

URE Staff(a)	6
Geologists	4
Temporary Field Geologists	2
Field Geology Co-ops	4

(a)Includes Project Geologist

SAMPLING VEHICLES

Four-wheel drive sampling vehicles were ordered for the collection of samples in sandy areas, hilly forests, and boggy areas. Nine vehicles were in the field this quarter.

PUBLICITY

Representatives of the URE Project were interviewed by personnel from TV Station WZZM, Channel 13, Grand Rapids, Michigan. A briefing on the URE Project was given, and a film sequence was taken which consisted of an interview and sample collection activities.

STATE CONTACTS

The ERDA-Oak Ridge Operations sent letters to the Governors of the 12 states in the UCC-ND HSSR area of responsibility requesting the names of individuals and agencies which should be kept advised of survey activities. As of September 31, 1976, 9 of the states have provided lists of contacts and agencies to be kept advised of the program activities.

INFORMATION MEETING

A URE Project information meeting is planned for December 1976. The UCC-ND URE Project will be reviewed in depth and will include areas of interest, such as Geochemical Reconnaissance Approach, Field Sampling Plan, Geochemical Plan (includes advantages of multielement analysis over uranium analysis only), Chemical Analysis Plan, Geostatistical Plan, Data Management Plan, Base Station Monitoring Plan, and Project Status and Plans. The Northwest Texas Pilot Survey will also be reviewed in detail. ERDA will be given the meeting agenda and date in early November 1976.

GEOLOGY AND GEOCHEMISTRY

SUMMARY OF SAMPLES RECEIVED AND ANALYZED

A summary of FY-1976 and FY-1976A water, stream sediment, and botanical samples received and analyzed is given in Table 1. The increase of samples received at Oak Ridge during recent months represents production sampling in Texas and pilot surveys in the Dakotas, Minnesota, and Wisconsin. The recent increase in the difference between samples collected and analyses completed results from new equipment being installed in the URE Project Clean Room Laboratory.

NORTHWEST TEXAS PILOT SURVEY REPORT RELEASED

The pilot survey report for northwest Texas was prepared and sent to ERDA in Grand Junction, Colorado for open filing. After December 1976, it will be available through the National Technical Information Service, U. S. Department of Commerce, 5285 Port Royal Road, Springfield, Virginia, 22161.

Stream sediment, stream water, well water, and ash of tree limbs were analyzed for approximately 25 chemical parameters. The samples were collected in Lynn, Garza, Crosby, Kent, Dickens, Stonewall, and King Counties, Texas over an outcrop area from the Blaine Formation (Permian) to the Ogallala Formation (Tertiary).

A geochemical model of uranium and associated trace elements in groundwater associated with roll-type mineralization is proposed for the interpretation of the reconnaissance-scale sample spacing of approximately 5 km (3 mi) which is used in this survey. Based on the geochemical model, areas of potential uranium mineralization are identified. The size of the interpreted trends suggests that mineralization in this area may not be restricted to small deposits as previously surmised.

Table 1

SUMMARY OF SAMPLES RECEIVED[(a)] AND ANALYZED

SAMPLES COLLECTED

Month	Stream Water	Stream Sediment	Botanical	Well	Other	Total	Cumulative Total
Feb	78	197	214	104	6	599	599
Mar	90	185	212	107	0	594	1,193
Apr	82	171	152	162	3	570	1,763
May	158	358	262	269	5	1,052	2,815
June	307	431	293	230	13	1,274	4,089
July	252	298	139	276	0	965	5,054
Aug	195	285	178	345	2	1,005	6,059
Sept[(b)]	145	191	213	154	1	704	6,763
TOTAL	1,307	2,116	1,663	1,647	30	6,763	

ANALYSES COMPLETED[(c)]

Month	Water	Sediment	Botanical	Total	Cumulative Total
Feb	-	-	18	18	18
Mar	-	196	99	295	313
Apr	156	176	168	500	813
May	292	125	105	522	1,335
June	271	109	103	483	1,818
July	276	170	120	556	2,374
Aug	287	160	152	599	2,973
Sept	338	260	183	781	3,754
TOTAL	1,620	1,196	948	3,754	

(a) Does not include duplicate samples in archival storage.
(b) Will be adjusted when all September field forms are processed.
(c) Includes 1 quality control sample for each 14 samples collected.

STATUS OF BASE STATION PROGRAM

A program is under way to quantify the seasonal variation in the concentration of uranium and associated trace elements in stream waters and stream sediments over a period of 3 to 5 years. Base stations are being established at USGS gaging stations. The sites are selected to measure the variation of trace elements where natural levels of uranium are detected and where uranium is introduced through agricultural activity associated with the use of phosphate fertilizers.

Mr. Quentin S. Paulson (Assistant District Chief of the Water Resources Division, USGS, Bismarck, North Dakota) indicated that his office may be able to collect samples on a regular basis from 2 to 4 sites in North Dakota.

A URE Project geologist visited the 4 base station samplers in Texas that are taking samples in cooperation with the Texas Water Quality Board. He found their procedures to be satisfactory, but did recommend some changes to make their sample collection easier and to allow for less chance of contamination.

About 8 tentative base station sites, representing basins of between 50 and 500 mi^2 in area, are being selected in each of the following states: Iowa, Nebraska, Oklahoma, Kansas, Minnesota, Indiana, Illinois, and Michigan. These sites will be discussed with members of the USGS Water Resources Division in each state and about 4 stations will be selected for sampling. Agreements for collection of the samples will be negotiated with the USGS offices.

Dr. M. E. Ostrom (Wisconsin State Geologist) suggested that the State Geological Survey could cooperate with the URE Project and enter into a cooperative agreement with the USGS to collect base station samples.

BASE STATION SAMPLING PROCEDURES OUTLINED

To assure that high-quality samples are obtained in the base station sampling program, a procedure for sampling has been developed. These instructions will be given to all base station samplers contracted in the future. A geologist field checked each of the four base station samplers in Texas and instructed them in proper procedures.

GEOLOGIC UNIT IDENTIFIER CODE IMPROVED

Guidelines for developing a 4-letter code to identify geologic units have been added to the URE Project Operations Manual. The application of these guidelines has resulted in a systematic geologic unit code which simplifies the identification of formations within the 12-state area.

FIELD FORMS REVISED

The last major revision planned for the field forms has been completed (Appendix A). Most of the changes involve the addition of new items and a rearrangement of the form for greater convenience.

OPERATIONS MANUAL PROGRESS

The first part of the section on Field Forms of the Operations Manual has been drafted and distributed for review. In addition, the section entitled *Preparation for Field Sampling* has been drafted.

FIELD OPERATIONS

STATUS OF THE PRODUCTION SAMPLING PROGRAM

Phase I sampling of 100-mi^2 drainage basins continued in the Texas Gulf Coast with completion of the Crystal City, Lubbock, San Antonio, and the Western Coast half of the Beeville Quadrangles. Figure 1 indicates the area which has been sampled during Phase I.

Phase II sampling of 10-mi^2 basins was completed in the Sequin, Crystal City, and the western half of the Beeville Quadrangles. Figure 2 indicates the area which has been sampled during Phase II.

Both Phase I and Phase II follow-up sampling has been conducted in the Crystal City and western half of the Beeville Quadrangles. This completes all sampling for the report on the reconnaissance geochemical survey in South Texas.

STATUS OF PILOT SURVEYS

Prepilot survey activities were carried out in Minnesota, Wisconsin, North Dakota, South Dakota, Indiana, Illinois, and Michigan. These activities included information-gathering visits to State Geological Survey and USGS personnel, selection of a pilot survey area, orientation of geologists in the field and scheduling of the sampling program. During this quarter, pilot surveys were conducted in Minnesota, Wisconsin, and the Dakotas. An additional pilot survey was begun in the Upper Peninsula of Michigan.

MINNESOTA PILOT SURVEY

Summary of Discussions with Contacts

Dr. Matt Walton (Director of the Minnesota Geological Survey at St. Paul, Minnesota) has suggested development of a cooperative program with the URE Project on a proposal to ERDA by the Minnesota Geological Survey. The proposal involves an extensive groundwater sampling program to establish baseline geochemical parameters for major bedrock aquifers in the state. The program would include sampling up to 30,000 wells to bedrock in Minnesota. Dr. Walton believes that in the Minnesota survey, well water samples could be collected to URE specifications without cost to the URE Project. In return, ORGDP would supply sample bottles and perform multielement analyses of the waters.
Dr. Walton proposed a meeting this winter to discuss selection of the well sampling sites and training of personnel in sampling procedures. Dr. Walton feels that there is considerable potential for uranium deposits in Minnesota, although much of the geology of the state is known only from widely-spaced drill holes and geophysical inference.

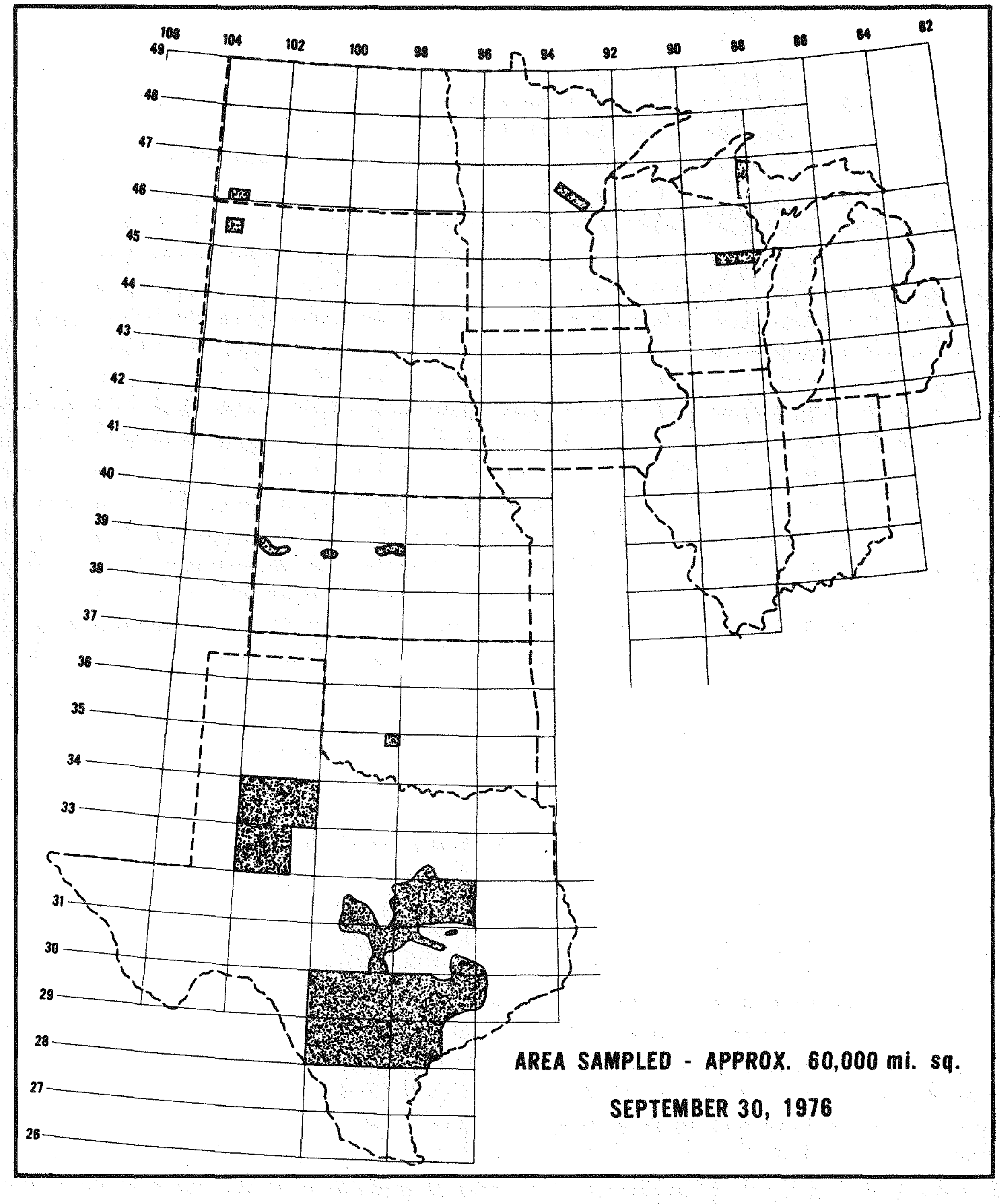
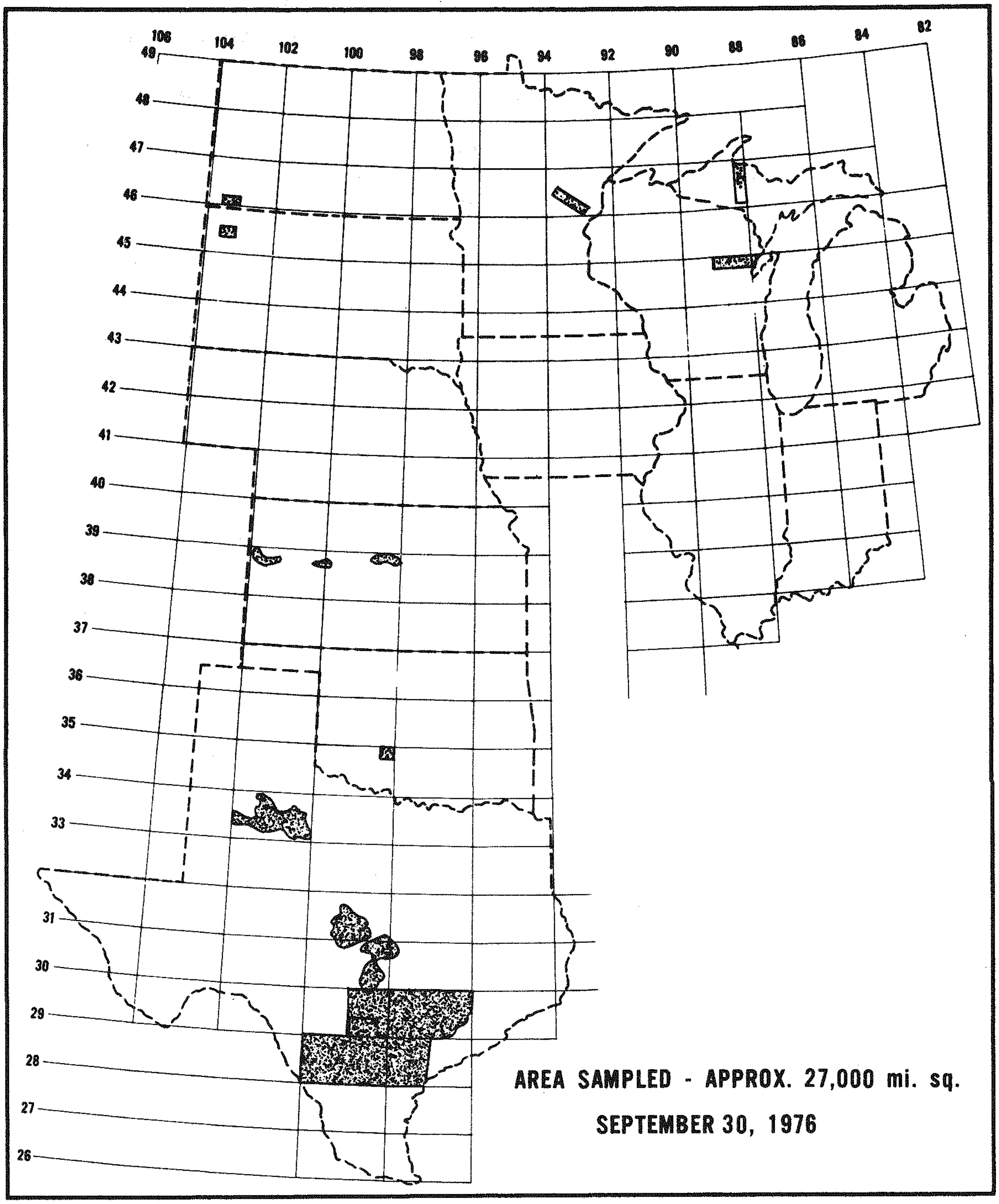


Figure 1

PHASE I AREA SAMPLED



AREA SAMPLED - APPROX. 27,000 mi. sq.
SEPTEMBER 30, 1976

Figure 2
PHASE II AREA SAMPLED

Drs. Joe Shappiro and Hubert Wright (Limnology Institute in the Department of Geology at the University of Minnesota, St. Paul) mentioned possible contamination of the stream water, stream sediment, and lake water with arsenic, copper, and sulfate. They explained that prior to 1967, insecticides used in central Minnesota contained large amounts of arsenic. As a result, the arsenic concentrations in many lake sediments are artificially high. In addition, copper sulfate has been used extensively as an algicide.

Mr. Ralph W. Lamson (Chief of Operations, Water Resources Division, USGS, St. Paul, Minnesota) provided a list of currently active USGS gaging stations on drainage basins between 50 and 500 mi^2 in area. Base stations in Minnesota will be selected from this list. Stream flow data at these sites generally will be available within 1 to 2 months from the date of measurement.

A description of the URE Project and the proposed Minnesota Pilot Survey was presented to a group of 10 people at the University of Minnesota, St. Paul. Two of the participants were from the Minnesota Geological Survey and 8 from the Department of Geology.

As a part of the orientation for bog sampling in the pilot survey area, a URE geologist spent 2 days observing bog sampling techniques and sample site selection strategy with Dr. Cornelia Cameron of the USGS in Reston, Virginia. During this time, Mr. Morris T. Eng [Minnesota Department of Natural Resources (Hibbing Office)] gave an orientation on the characteristics of bogs.

Field Sampling

Sampling for the Minnesota Pilot Survey began in July and was completed in September. An area of approximately 1,100 mi^2 in Aitkin, Carlton, and Pine Counties was surveyed with samples taken of stream water, stream sediment, well water, and tree limbs. The pilot survey covers Precambrian sediments and metasediments, as well as some Middle Precambrian intrusives. During the week of September 13, a geology co-op student was sent to the Minnesota pilot area to assist in the sampling of peat bogs.

WISCONSIN PILOT SURVEY

Summary of Discussions with Contacts

Dr. Meredith E. Ostrom (Director and State Geologist, Geological and Natural History Survey, Madison, Wisconsin) suggested numerous valuable contacts related to the URE work in Wisconsin.

Dr. Michael G. Mudrey, Jr. (Geologist of the Geological and Natural History Survey, Madison, Wisconsin) briefly described known uranium anomalies in Wisconsin. His orientation on the geology of areas having uranium potential in the state provides reason for optimism that uranium districts will be found.

Mr. Albert Harr (District Water Quality Hydrologist, Wisconsin District Office of the Water Resources Division of the USGS) recommended 6 possible sites for the URE base station network and has agreed to provide information on possible agricultural contaminants in each of the basins.

Field Sampling

A 20- x 50-mi area was selected to be sampled in Waupaca, Shawano, Outagamie, and Oconto Counties in east central Wisconsin. The pilot survey covers an area from the Precambrian granites around Big Falls eastward to the Ordovician at Green Bay. Because prepilot survey orientation work identified problems in urban and agricultural contamination in the pilot survey area, special attention was given to sampling in such a manner as to minimize the effects of contamination. An additional problem addressed by this pilot survey (as well as the Minnesota Pilot Survey) was how to interpret the effects of high-organic content in stream sediments. The basic technique involves gathering two sediment samples at each site. One consists of a fine-grained inorganic sediment, while the other is an organic sediment from the bank. At the time of collection, field personnel estimate the organic content of each sample. These 2 sediment types are being analyzed to determine the most effective sample type to be used in production sampling.

Sampling began in July with 1 geologist assigned to the area. By the end of the quarter, the Wisconsin Pilot Survey was near completion with all but approximately 30 well samples collected.

DAKOTAS PILOT SURVEY

Summary of Discussions with Contacts

Dr. Ned Noble (State Geologist of the North Dakota Geological Survey, Grand Forks, North Dakota) provided an extensive list of contacts related to URE work in North Dakota. These included state survey and university personnel, USGS Water Resources personnel, and experts in agricultural development. Dr. Noble also provided information on the location and distribution of uraniferous lignite deposits in the western part of the state to aid in selecting a pilot survey location.

Mr. Lee Clayton (Geologist of the North Dakota Geological Survey) provided a detailed unpublished geologic map of the pilot survey area in the southwest part of the state, as well as valuable information on the stratigraphy of the area finally selected for the pilot survey.

Mr. Quentin Paulsen (Assistant District Chief, Water Resources Division, USGS, Bismarck, North Dakota) provided information on the observation well network in the state that may be utilized by the URE Project in production sampling. His discussion included details of incomplete county hydrologic and hydrogeochemical reports in the area selected for the pilot survey. Mr. Paulsen also discussed the basic groundwater geochemistry in the state and provided detailed geochemical characteristics of the aquifers to be investigated in the pilot survey.

Dr. Bill Harkness (Chief of the Hydrologic Records and Information Section, Water Resources Division, USGS, Bismarck, North Dakota) discussed data available for the URE Project from the surface water and groundwater monitoring networks in the state.

Mr. Orlo Crosby (Chief of Hydrologic Studies, Water Resources Division, USGS, Bismarck, North Dakota) described current hydrologic investigations being conducted in the state, as well as those recently finished but not yet published. He also provided copies of logs for wells located in the area of the pilot survey.

A geologist visited Mr. Duncan McGregor (State Geologist of the South Dakota Geological Survey, Vermillion, South Dakota) and obtained information on the geology of northwestern South Dakota that was utilized in planning the South Dakota Pilot Survey.

Field Sampling

Sampling for the pilot survey of the Dakotas was conducted by 1 geologist, starting in July, and was completed on schedule in September. Approximately 500 mi^2 of Tongue River and Sentinel Butte Formations was sampled in Hettinger and Grant Counties, North Dakota, and another 500 mi^2 of the Ludlow and Cannonball Formations was sampled in Harding and Perkins Counties, South Dakota. Preliminary analysis of measurements taken of well waters at the time of collection indicates that the major aquifers have a distinctive geochemistry and well samples may be very useful in interpretation. A problem encountered was a lack of trees. Thus, few botanical samples were obtained.

MICHIGAN PILOT SURVEY

Summary of Discussions with Contacts

Dr. J. Kalliokoski (Head of the Department of Geology, Michigan Technological University, Houghton, Michigan) described work related to uranium exploration in Michigan. He gave an encouraging orientation on the uranium potential of Michigan, discussed mechanisms of uranium mineralization, and gave a field tour of some of the more interesting outcrops.

Dr. S. C. Nordeng (Professor in the Department of Geology at Michigan Technological University) discussed many problems associated with interpretation of values obtained from geochemical sampling in Michigan, including potential sources of contamination, such as small iron mines where ash and slag were dumped from charcoal furnaces. Ms. Sue Jacobson (a student of Dr. Nordeng) is writing a thesis on geochemical sampling of peat bogs, which is a subject of particular interest to the URE Project. A cooperative program is being arranged with Ms. Jacobsen to evaluate results of her work for future UCC-ND sampling in the northern states.

A briefing on the NURE Program and the UCC-ND geochemical sampling by the URE Project was presented on August 30, 1976 to representatives of the Michigan Department of Natural Resources (DNR) Geological Survey Division in Lansing, Michigan and the District Chief, USGS Water Resources Division in Okemos, Michigan. Persons attending the briefing included the following:

Mr. Harry O. Sorensen - Geologist, Mining and Economic Geology Unit
Mr. William A. Walden - Geologist, Mining and Economic Geology Unit
Mr. Richard P. Bissel - Geologist-in-Charge, Groundwater Geology
Mr. T. Ray Cummings - District Chief, USGS, Okemos, Michigan

Further meetings were held on August 30, 1976 with DNR personnel relating to details of the geology of Michigan, DNR projects and studies, DNR data, and possible pilot survey locations.

Meetings were held on August 30-31, 1976 with Mr. Steve Buda and Mr. Tom Newell (DNR Comprehensive Studies Section) to discuss (1) the availability of surface and groundwater data, (2) sample collection locations, (3) well sampling programs, and (4) possible collection of base-station samples for temporal variation studies.

Mr. T. Ray Cummings (District Chief) provided details of USGS activities in Michigan. He reviewed USGS sampling procedures and provided some documents on the geology of Michigan.

Field Sampling

The Pilot Survey in the Upper Penninsula of Michigan was begun in the second half of September in a 1,025-mi^2 area in Iron and Baraga Counties with 2 geologists assigned to the survey. Several days of field orientation were given to prepare the geologists for sampling in northern Michigan. Included in the orientation were training in bog sampling techniques and the identification of northern trees. In the Michigan Pilot Survey, a second set of water samples will be collected and acidified with nitric acid in the field to evaluate the need to acidify stream water and well water samples in northern areas. Sampling problems identified in the orientation were the lack of good roads for access to sampling sites, lack of wells in certain areas, and cold weather.

CONTACTS FOR THE INDIANA PILOT SURVEY

URE Project personnel visited Indiana for the purpose of making initial contacts and obtaining information to aid in planning a pilot survey. A briefing on the NURE Program and the UCC-ND URE Project was presented to representatives of the Indiana Geological Survey. Persons attending the briefing were:

Dr. John B. Patton	Director and State Geologist
Dr. Maurice Biggs	Assistant State Geologist, Head, Geophysics Section
Dr. Donald Carr	Head, Industrial Minerals Section
Dr. Richard Leninger	Head, Geochemistry Section
Dr. Robert Shaver	Head, Geology Section
Mr. Nelson Schafer	Geologist

Further discussions were held with each of these people on an individual basis. Topics discussed included naturally occurring uranium anomalies, sources of contamination, regional stratigraphy, previous research, other types of mineralization, persons to contact, and possible locations for a pilot survey.

Mr. Henry Gray (stratigrapher with the Indiana Geological Survey) is completing the revision of a series of 1 x 2° quadrangle maps of the bedrock geology for Indiana. This detailed information will be very useful for planning and interpretation.

Dr. Jerry Lineback (currently with the Illinois Geological Survey) wrote his doctoral thesis on the stratigraphy of the New Albany Shale in Indiana. He noted that it contains anomalous uranium values throughout much of the section.

On August 24, a presentation about the NURE Program and the UCC-ND URE Project was given to Mr. Tom Cook (District Chief, USGS Water Resources Division) and Mr. Bill Shampine (Hydrologist, USGS Water Resources Division). They provided information on current and past hydrogeochemical programs conducted in Inidana, as well as contamination problems. Mr. Shampine gave UCC-ND a list of all currently active USGS gaging stations on drainage basins between 50 and 500 mi^2 in Indiana for the purpose of selecting sites for the base station program.

CONTACTS FOR THE ILLINOIS PILOT SURVEY

Representatives of the Illinois Geological Survey, who were briefed August 25 on the NURE Program and the UCC-ND geochemical sampling project, included:

Mr. Jack A. Simon - Chief, Illinois Geological Survey
Dr. Robert Bergstrom - Principal Geologist, Head, Earth Materials Technology Section
Dr. Rodney Ruch - Head, Analytical Chemistry Section
Dr. Keros Cartwright - Head, Hydrogeology and Geophysics Section
Dr. James Bradbury - Head, Industrial Minerals Section
Ms. Dorothy Brown - Research Assistant, Hydrogeology and Geophysics Section

A general discussion of the URE Project followed the briefing, as well as a discussion of the Illinois Geological Survey contract with ERDA to study the New Albany Shale. Further discussions were held on an individual basis.

Dr. Bergstrom discussed the stratigraphy of Illinois and possible host sandstones for uranium deposits. He noted that the Illinois Geological Survey has over 225,000 well logs filed by township, range, and section. Possible anomalous zones may be found associated with sandstones in the northern part of the state.

Dr. Dave Gross (Associate Geologist, Stratigraphy and Areal Geology Section) described methods used by the Illinois Geological Survey to conduct lake and stream sediment sampling programs. He displayed devices to sample small streams, as well as equipment used to sample bottom sediments from Lake Michigan.

Dr. Ruch discussed sampling procedures and sample treatments used by the Illinois Geological Survey. He was joined by Dr. Gary Dreker who discussed analytical methods and problems of analysis.

Dr. Cartwright provided information on geochemical studies carried out by the State Health Department on water wells reaching to bedrock. He reported that 4 wells had anomalously high gross-alpha counts. Dr. Cartwright also discussed the distribution of bedrock wells around the state and pointed out that there were widespread areas in Illinois that had few bedrock aquifers.

Dr. Bradbury discussed information on known radioactive occurrences in Illinois and provided copies of open-filed reports of Geiger counter surveys conducted in the 1950's on some of the bedrock formations in the state.

A briefing on the URE Project was also given to Mr. Bill Ackerman (Chief, Illinois State Water Survey, Urbana, Illinois) and Mr. Robert Harmson (Assistant Chief, Head of the Data Bank, Illinois State Water Survey). They described hydrogeochemical surveys being carried out by the Illinois State Water Survey, including a program of surface water sampling in which 30 stations are sampled monthly for 5 years. This program, which has been active for 30 years, has completed 6 cycles. Results from the first 5 cycles are published and may be useful to the URE Project in evaluating the temporal variation of trace elements.

On August 27, a briefing on the NURE Program and the UCC-ND URE Project was presented to Mr. Larry Martens (District Chief, USGS Water Resources Division, Champaign, Illinois) and Mr. Larry Toler (Hydrologist, USGS Water Resources Division). They discussed USGS programs being carried out in hydrogeochemistry and provided information on drainage basins that could be used in the base station program.

ANALYTICAL CHEMISTRY

URE CLEAN ROOM LABORATORY

Modifications to the URE Project Clean Room Laboratory were completed in FY 1976A. Analytical instrumentation and equipment necessary to provide the required analytical services has been installed and is now in operation. The laboratory layout is shown in Figure 3. The analytical instrumentation is described briefly in this section.

A laboratory staff of 4 technicians and 1 supervisor has been selected. With the present equipment and staff, a throughput capacity of approximately 400 samples/week is expected. Additional capacity can be taken care of as needed by adding an additional shift of technicians.

WATER SAMPLE FILTRATION

Water samples are currently being received by the URE laboratory in 250-ml polyethylene bottles. These samples must be filtered in the laboratory before analysis to remove suspended particulate matter from the sample. The battery of 5 vacuum filtrations, shown in Figure 4, is used for this purpose. The s sample is filtered through a 0.45-micrometer Millipore filter into an 8-oz plastic cup. It remains in this container through the various stages of analysis.

AUTOMATED SULFATE ANALYSIS

The Technicon AutoAnalyser II for determining the sulfate content of water samples is shown in Figure 5. The instrument consists of 5 modules shown from right to left: sample changer, proportioning pump, mixing coils, colorimeter, and recorder. A sample is first pumped through an ion-exchange column to remove alkaline earth metal interfering ions and then through a mixing column where the pH is adjusted. Methyl-thymol blue is then added, and the absorbance of the resulting blue color is measured by the filter colorimeter and displayed on the recorder. Peak heights from standards and samples are entered into the programmable calculator. Computed sample concentrations and the corresponding sample identification numbers are recorded on a magnetic tape cassette. Once the sample tray is loaded, the instrument can operate unattended in analyzing 30 to 40 samples/hr.

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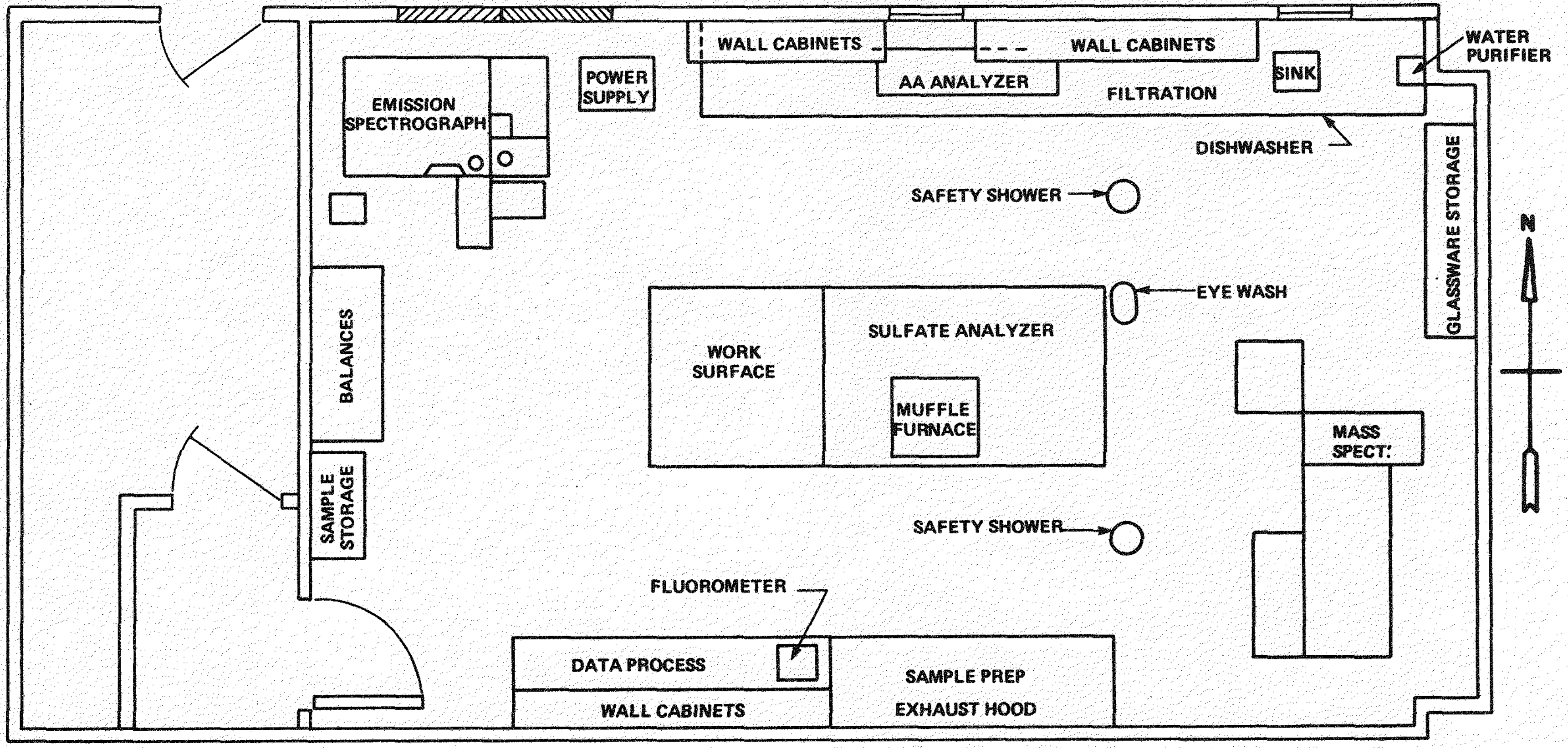

Figure 3

URE PROJECT LABORATORY CLEAN ROOM

Y-12 PHOTO NO. 170131
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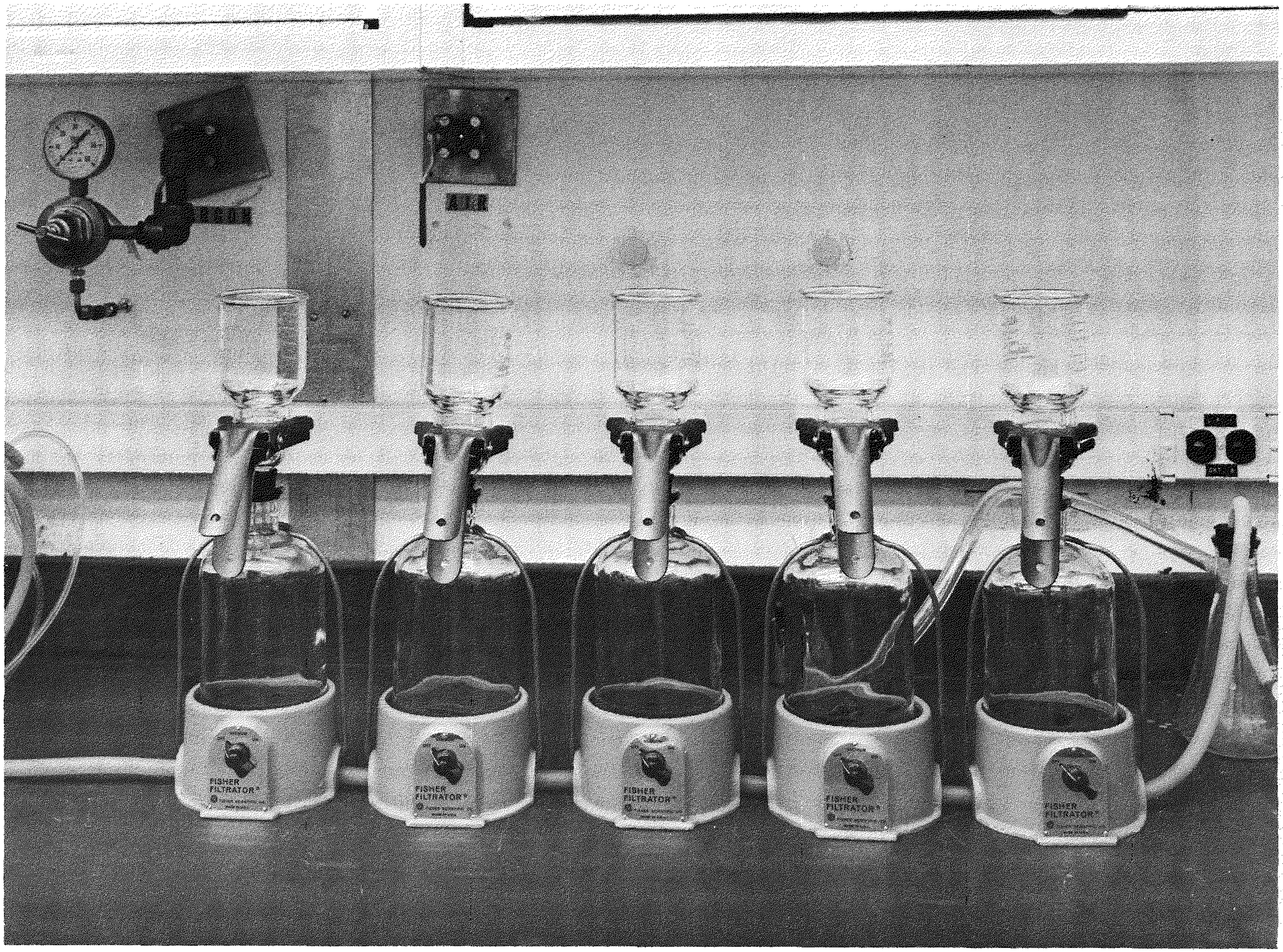

Figure 4
VACUUM FILTRATION APPARATUS FOR WATER FILTERING

Y-12 PHOTO NO. 170132
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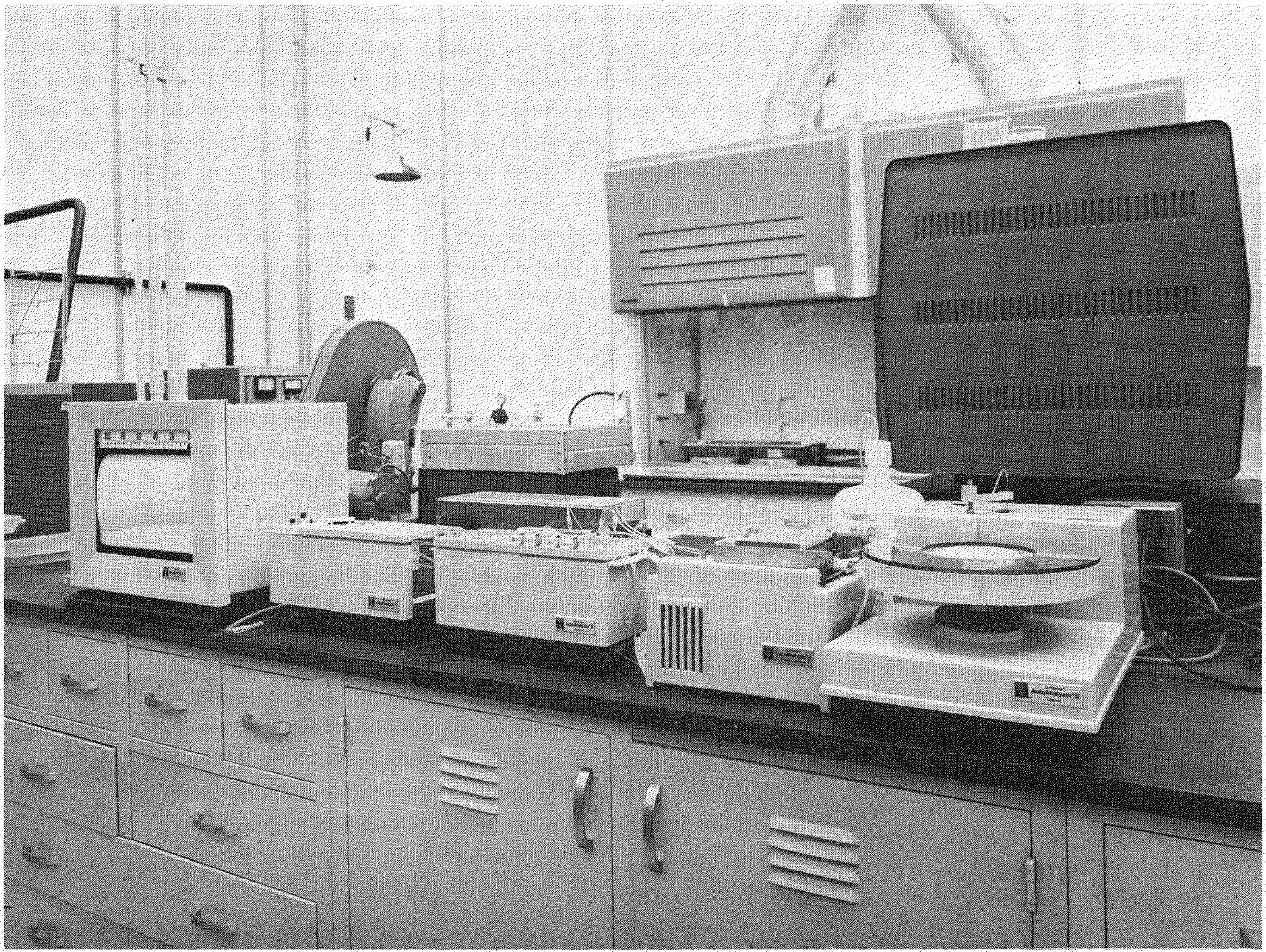

Figure 5
TECHNICON AUTOANALYZER USED FOR DETERMINATION OF SULFATE IN WATER

AUTOMATED ATOMIC ABSORPTION SPECTROMETER

The automated atomic absorption spectrometer, shown in Figure 6, will analyze simultaneously the arsenic and selenium content of natural waters and dissolved sediments. Samples can be analyzed at a rate of 37/hour with a lower reporting limit in water of 0.2 ppb (ug/liter) for both arsenic and selenium. A description of the hydride-generation procedure using sodium borohydride as a reductant has been previously given.

A micropressor is used to control the analysis sequence. A block diagram of the instrument showing the devices under control of the micropressor is presented in Figure 7. All sample and reagent pumps, the sample changer, recorder, and the solenoid valves controlling the argon flow through the reactor are sequentially controlled for automated analysis.

The micropressor controller is shown in the lower left corner of the assembled instrument shown in Figure 6. Immediately adjacent are the 3 tubing pumps, followed by the reaction vessel and gas collection balloon. A portion of the automatic sample charger is shown in the right side of the photograph. To the left center are the optical components of the instrument. The 2 atomic spectral sources are positioned so that as the chopper-mirror rotates, the arsenic beam is transmitted while the selenium beam is reflected and vice versa. In the background is the monochrometer with the electronics readout module on top.

Some precision and accuracy data for arsenic and selenium standard solutions anonymously analyzed over a 3- to 4-week period are shown in Table 2. This demonstrates good precision and accuracy using the automated instrument for arsenic and selenium determinations in the 1- to 10-ppb range.

Table 2

PRECISION AND ACCURACY DATA FOR
ARSENIC AND SELENIUM

Standard, µg/liter	1.0	2.0	4.0	6.0	10.0
Arsenic Mean, µg/liter	0.97	1.99	3.92	6.13	9.5
Arsenic, % RSD	19	11	9	5	6
Selenium Mean, µg/liter	1.04	2.16	4.40	6.30	10.7
Selenium, % RSD	17	5	5	7	4

AUTOMATED FLUOROMETER

The automated fluorometer used to analyze dissolved sediment and water samples for uranium to concentrations as low as 0.2 ppb is shown in Figure 8. Up to 40 prepared sample pellets can be loaded in the metal sample tray and passed

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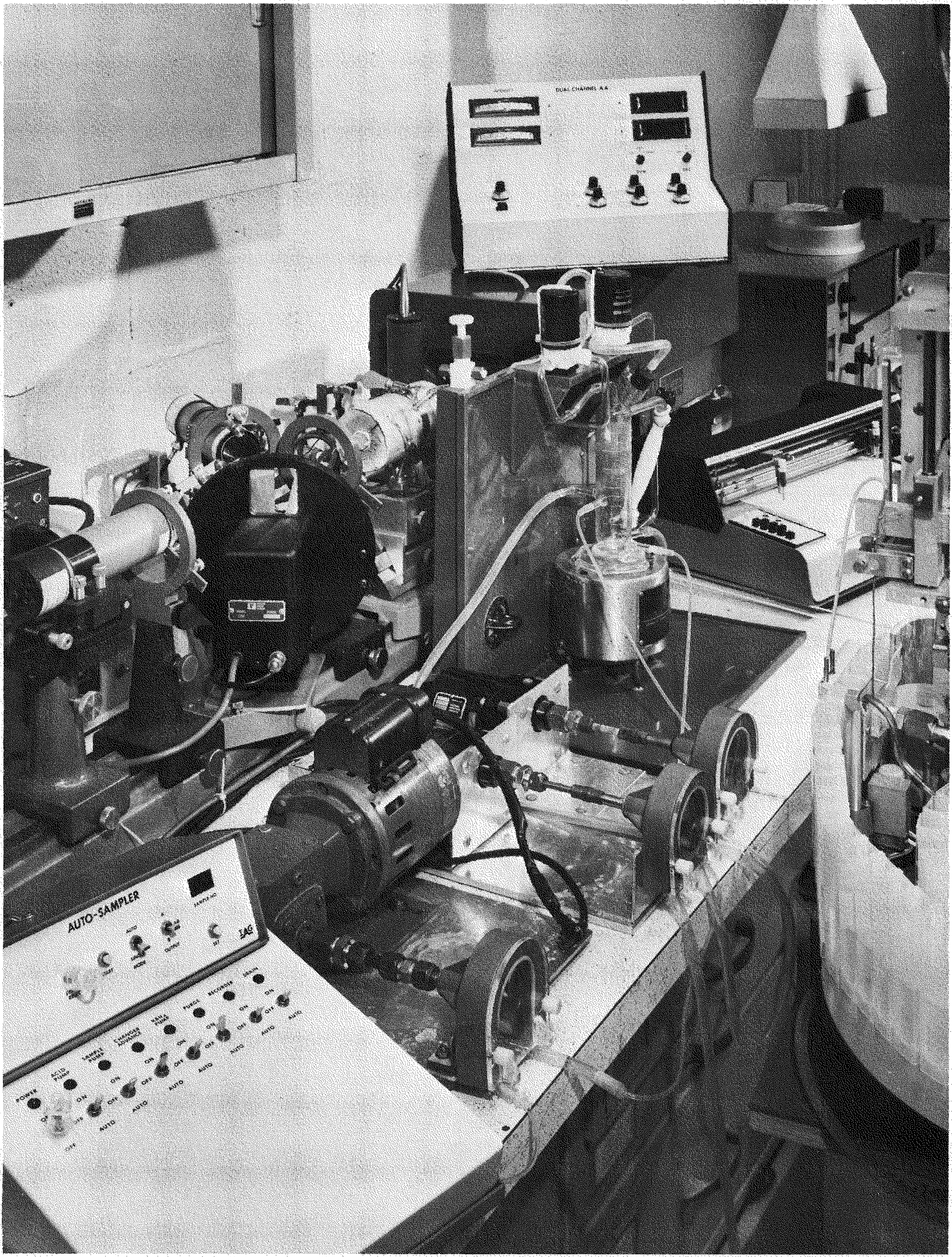

Figure 6
AUTOMATED ATOMIC ABSORPTION SPECTROMETER FOR DETERMINATION OF ARSENIC AND SELENIUM IN WATERS AND DISSOLVED SEDIMENTS

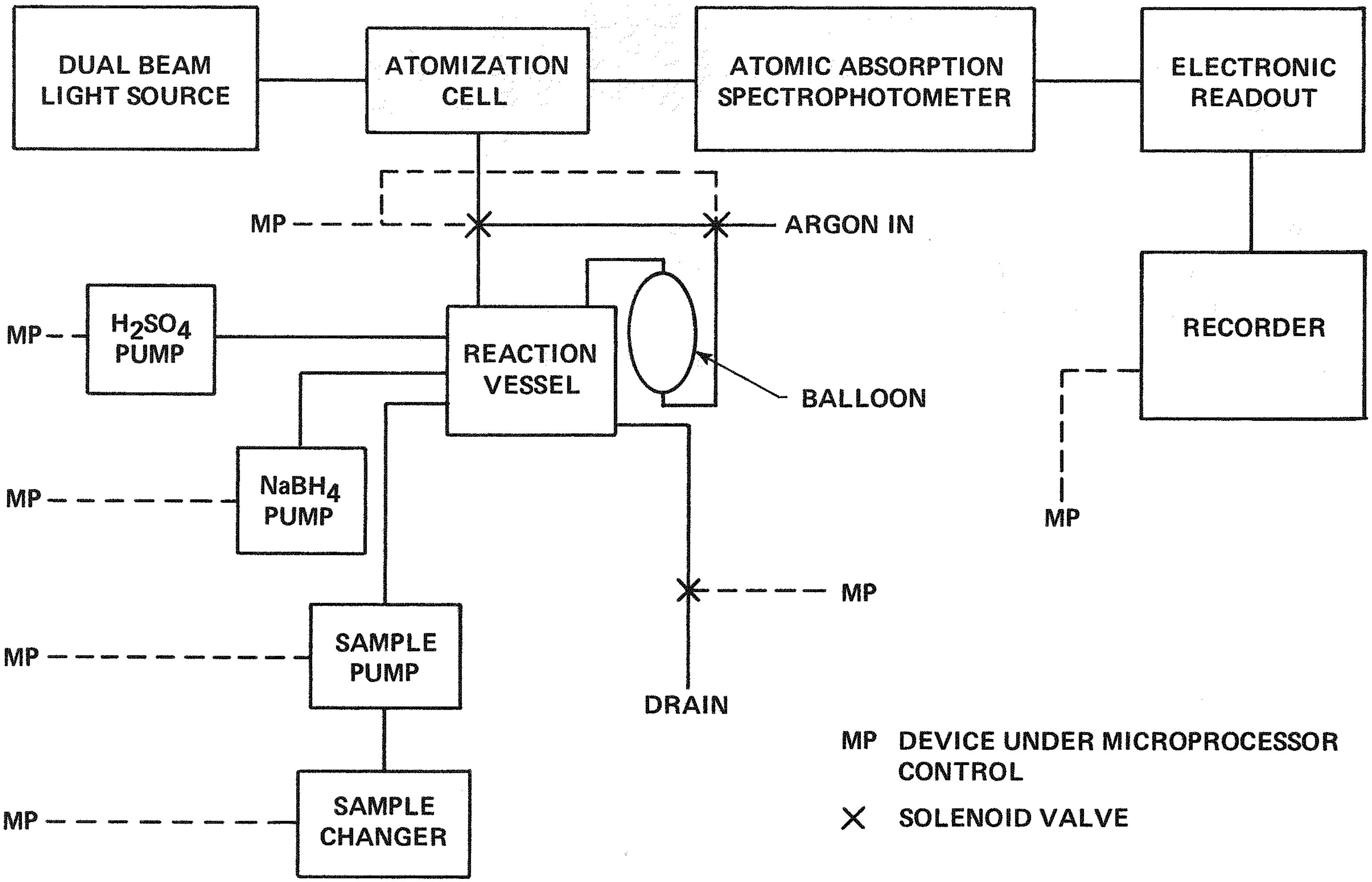

Figure 7

BLOCK DIAGRAM OF AUTOMATED ATOMIC ABSORPTION SPECTROMETER FOR DETERMINING ARSENIC AND SELENIUM

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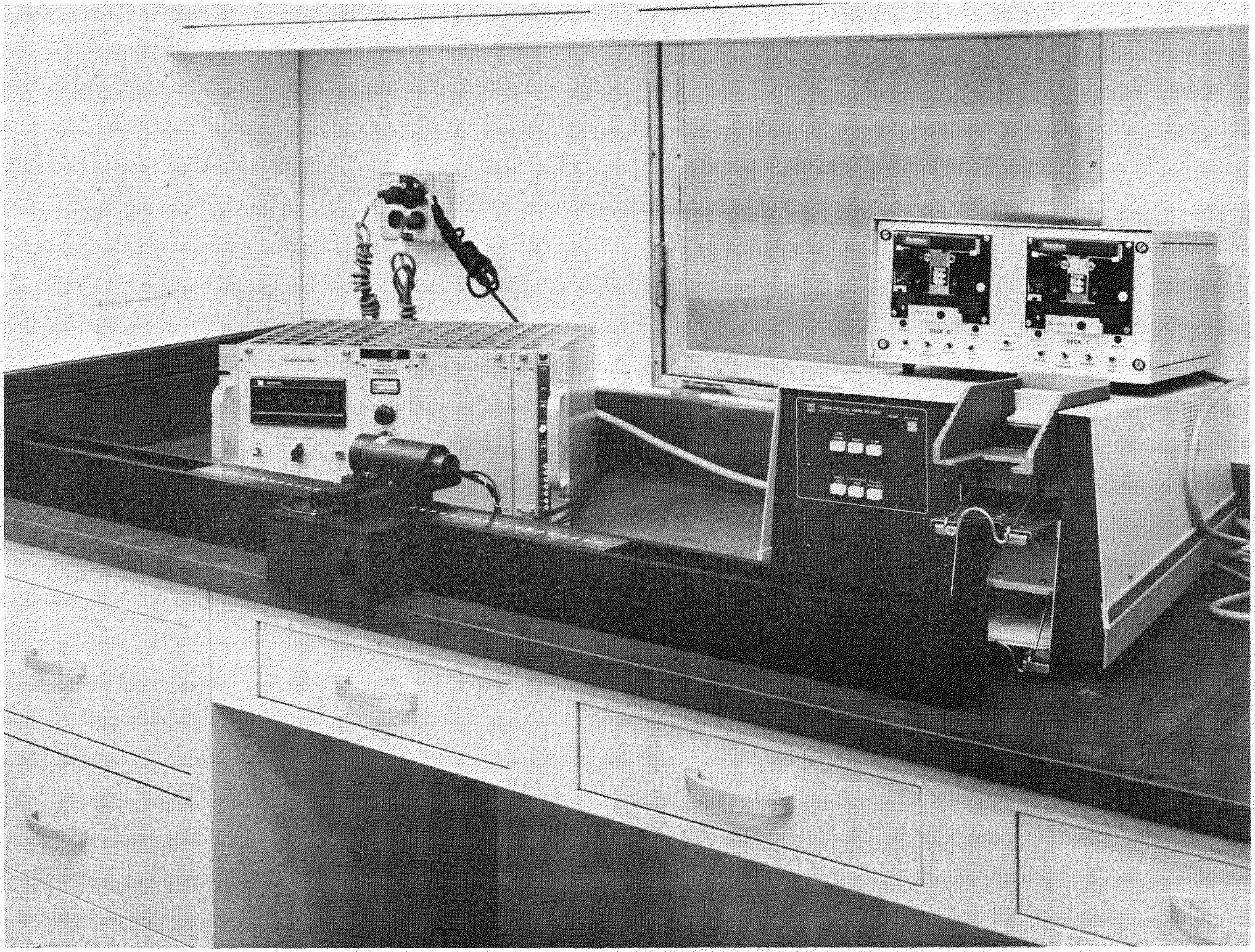

Figure 8
AUTOMATED FLUOROMETER FOR DETERMINATION OF URANIUM

under the ultraviolet light source and photomultiplier detector. Uranium in the pellet will fluoresce with an intensity proportional to its concentration. The electronics module shown directly behing the detector head amplifies this signal and transmits it to a programmable desk calculator (not shown). The calculator computes the uranium content of the water sample, reads the sample identification number from the punched card reader shown to the right, and transmits the data to the cassette tape recorder shown on top of the card reader. This instrument will analyze the 40 pellets in the sample tray in 3 to 4 min. Using the extraction procedure and pellet preparation previously reported and the automated fluorometer, a technician can process and analyze up to 80 water samples/day.

ISOTOPE DILUTION THERMAL EMISSION MASS SPECTROMETER

The isotope dilution-thermal emission (IDTE) mass spectrometer is shown positioned in the URE Clean Room Laboratory in Figure 9. In the module to the right, are the photon counters with channels for uranium-238, uranium-235, and uranium-233, the rate meter, electronics for peak switching, and detector controls. A loading station for pipeting the extract from the water sample onto a filament is contained in the adjacent module. Controls for the instrument vacuum system are also in this module. The mass spectrometer sample entry system, source housing, and analyzer diffusion pump are in the next module. A dual pumping arrangement in the source enables the instrument to reach operating pressure of 5×10^{-6} torr within 2 to 3 min after the sample is introduced. The fourth module in the photograph contains the instrument power supply and additional controls.

A detailed description of the analytical procedure used to analyze water samples for uranium has been given in an earlier report. Uranium in the water sample is extracted with an organic solvent and then back extracted into an aqueous medium. An aliquot of this aqueous phase is then analyzed by comparison of the uranium-238 count to a uranium-233 isotopic spike count. A skilled analyst can prepare and analyze up to 50 water samples in 8 hr.

The precision and accuracy of this method was demonstrated by analyzing standard solutions of uranium varying in concentration from 0.005 to 0.80 ppb (µg/liter). The results of this experiment showing the percent relative standard deviation of the mean are shown in Table 3. Five groups of samples containing nearly identical uranium content were analyzed by IDTE-mass spectrometry, with the results shown in Table 4. Within a given group, the samples received different combinations of acidification and filtration. This variation in treatment and/or the differences in water composition, plus analytical imprecision, must account for the variation of the percent relative standard deviation from 1.4 to 18.6%. Thus, the measurement precision of this method is adequate for uranium measurements in the sub-ppb range.

The lowest uranium concentration reported by IDTE-mass spectrometry is determined by the background level, which is approximately 0.005 ppb. Water samples containing uranium above 0.2 ppb are normally analyzed by fluorescence spectroscopy. To obtain a comparison of the two methods, several samples containing from 0.2 to 10 ppb uranium were analyzed. The results are plotted in Figure 10. The slope indicates good agreement between the two methods.

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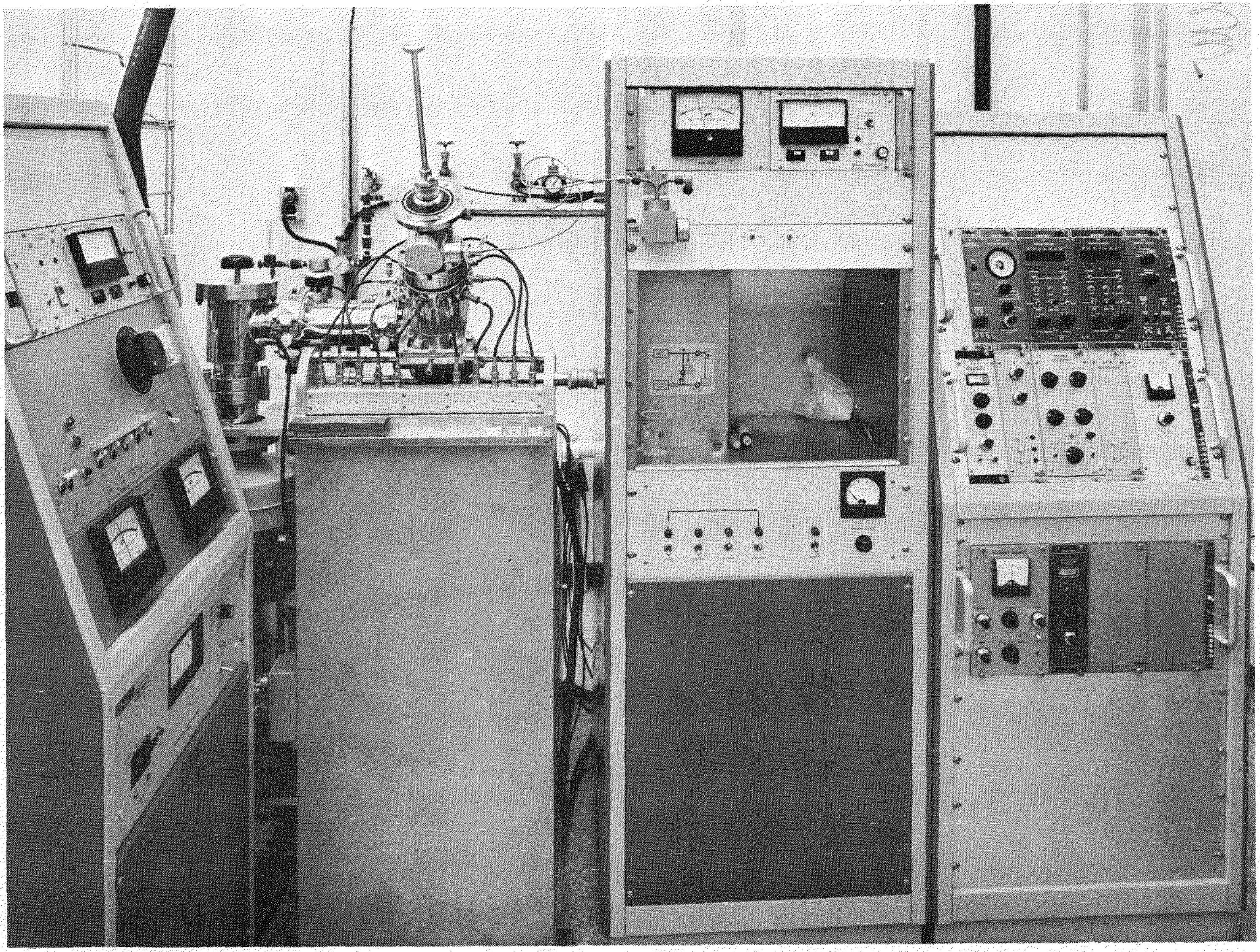

Figure 9

MASS SPECTROMETER USED FOR DETERMINATION OF URANIUM IN WATER

Table 3

DAY-TO-DAY PRECISION AND ACCURACY OF ANALYZING WATER STANDARDS BY IDTE MASS SPECTROMETRY

Standard ppb	Uncorrected ppb	Corrected for Blank	Standard Deviation	% Standard Deviation	No. Samples per Location
0.800	0.827	0.826	0.028	3.4	12
0.080	0.087	0.086	0.0038	4.4	12
0.008	0.009	0.008	0.0014	15.6	10
0.005	0.006	0.005	0.0011	18.3	8
Blank	0.0013	-	0.0005	38.5	11

Table 4

GROUND AND SURFACE WATER SAMPLES FROM MICHIGAN, MINNESOTA, AND WISCONSIN ANALYZED BY IDTE-MS

Sample ID Number	Uranium Concentration, μg/liter	Standard Deviation	% Standard Deviation	No. Samples per Location
520-533	0.37	0.005	1.4	4
350-363	0.42	0.020	4.8	14
452-465	0.42	0.020	4.8	14
401-404	0.38	0.050	13.2	4
497-510	0.43	0.080	18.6	14

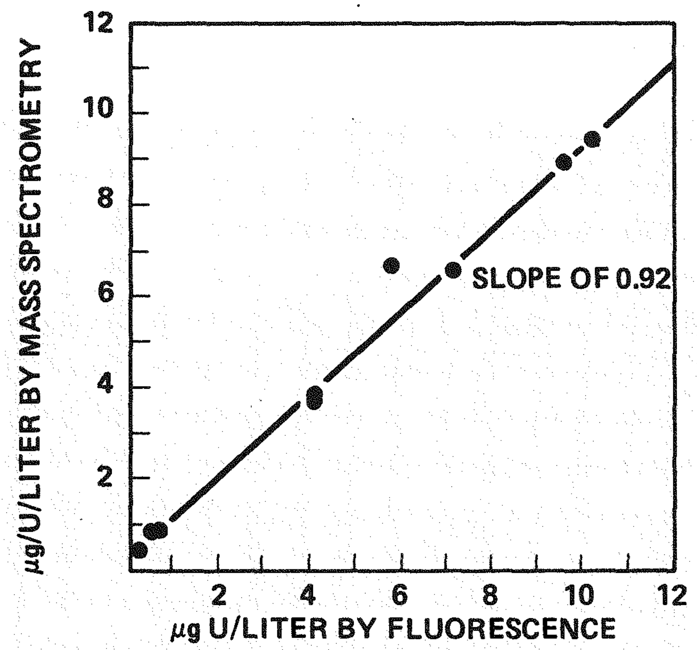

Figure 10

COMPARISON OF RESULTS FOR
URANIUM IN WATER BY ISOTOPE DILUTION
MASS SPECTROMETRY AND OPTICAL FLUORESCENCE SPECTROSCOPY

PLASMA-ARC EMISSION SPECTROGRAPH

The Jarrell-Ash AtomComp 750 Inductively Coupled Plasma (ICP)-dc Arc Emission Spectrograph has been installed in the URE Clean Room Laboratory. This instrument is shown in Figure 11. The power supply located at the extreme right in Figure 11 is used to excite the argon plasma torch located on the right side of the spectrograph. Mounted directly below is a 0.5-m scanning monochromator. The black box adjacent to the plasma houses the automatic electrode loaders for arc analyzis. The spectrograph, located on the left side of the instrument housing, has 33 analytical and 3 background channels mounted to receive radiation from both the plasma and dc arc sources. Control of the instrument and data processing is accomplished by the D.E.C. PDP 8 M minicomputer mounted in the instrument. A teletypewriter shown at the extreme left of the photograph is for communication with the instrument and for data output. A punched card reader is used to input sample identification to the computer.

Current efforts are being directed toward determination of instrument stability, interelement interferences, precision of results, and lower reporting limits. Once this is completed, analysis of water samples with manual sample changing will begin. The automatic sample changer will then be interfaced as soon as possible. Jarrell-Ash representatives are currently planning a training course on the plasma to be given in Oak Ridge during November or December.

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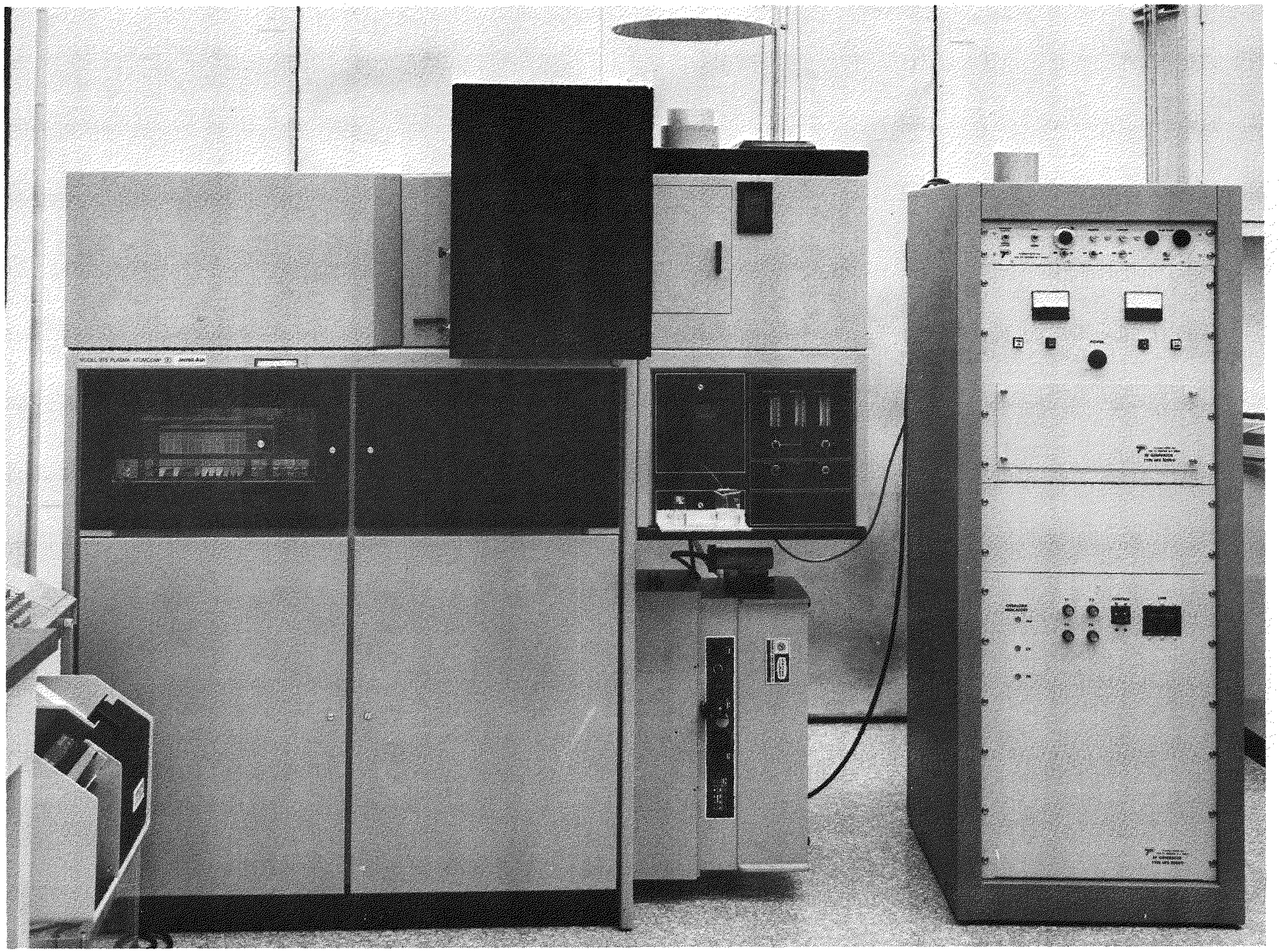

Figure 11

DUAL SOURCE EMISSION SPECTROGRAPH USED FOR MULTIELEMENT ANALYSIS OF SEDIMENT AND WATER SAMPLES

Work to bring the dc arc source into operation will start when operation with the plasma source becomes routine. The arc power supply was modified to make the grounding compatible with the automatic electrode loaders. A housing for the loaders has been fabricated and installed.

SEDIMENT PREPARATION LABORATORY

A chemical laboratory with a relatively low uranium background level and space enough to handle the anticipated sample flow for preparation of stream sediment samples has been set up in Building 9203 at the Y-12 Plant. Before any work was begun, all the equipment was removed and the entire laboratory cleaned, including revacuuming of the plenum above the ceiling and washing of the walls, hoods, cabinets, and floors. Both the room air, and bench tops were sampled after cleaning to determine the uranium background levels. Analysis by isotope dilution mass spectrometry show that a 24-hr. air sample had a total of 5 ng of uranium and the smear samples had an average of 0.3 ng/cm^2 uranium. All work surfaces are sampled on a periodic basis to make sure there has been no change in the uranium level.

Field samples collected and transported in small paper bags, all properly labeled and identified, are shipped to this laboratory for preparation. Included with each shipment of samples is a list of the samples, numbers, and a set of preprinted gummed labels. Each batch of sample bags is placed directly into a large (0.67 m^3) forced-air oven and dried at 80° C for approximately 16 hr. Each dried sample, contained in 1 to 3 bags, is transferred to a heavy polyethylene bag approximately 20 x 30 cm and shattered with a rubber mallet. The purpose of this step is to shatter the agglomerates into their primary particles without reducing their particle size. Particle size measurements have indicated that no significant difference exists between *as-received* samples and those same samples pulverized using the above technique.

The shattered samples are then mechanically sieved through a 100-mesh sieve with all the fines (<150 μm) saved for analysis. The bodies of the sieves are constructed of Plexiglas, which supports the 100-mesh nylon sieve cloth. Alternating sieves and collector pans are stackable so that up to 6 sets can be placed on the shaker for processing at one time. The sieves and pans are vacuum cleaned after each use and washed with soap and water at the end of each day. Data have been obtained which indicates that the vacuum cleaning of sieves between samples is a sufficient cleaning process: i.e., if a 48-mg residue from a previous sample (highest about 20 ppm) were transferred to the next 10-g sample it would represent approximately 0.1-ppm contamination, which is an insignificant amount.

The entire <150-μm portion of each sample is placed in a labeled vial and the entire batch (40 to 80 samples) is blended for approximately 2 hr to further ensure a homogeneous sample. At this point, a portion is taken from each sample and loaded in a polyethylene *rabbit* for neutron activation analysis. The remainder of the sample is then packaged for transfer to the URE chemical laboratory for analysis.

All aspects of this preparative procedure are periodically reviewed in an effort to improve and maximize the throughput of the laboratory. To date, approximately 600 samples have been processed through the laboratory. Fifty to 75 samples can be processed each day, depending largely on the amount of material in a given sample.

QUALITY CONTROL

The program for submission of anonymous samples to the analytical laboratory on a regular basis has been in continuous operation since February 1976. Charts have been generated showing plots of the analytical results for uranium and 13 to 17 other elements in water and sediment control batches. These are received twice a month, and sometimes weekly, to check for erroneous results.

At the end of the quarter, mean, bias, standard deviation, and coefficient of variation estimates were computed from the February through September 1976 results on all of the control batches. Three different batches of water were used, since they must be changed every 3 to 5 months. September data only are available for the natural water control batches A1 and B1. These batches were prepared by adding known amounts of uranium, arsenic, and selenium to Norris Lake water. The Batch 02 synthetic water controls were started in May. The two sediment controls have been in use since February. Results are presented in Tables 5-11.

Table 5

SUMMARY OF MEASUREMENT CONTROL RESULTS FOR NATURAL WATER, 0.8 PPB URANIUM, BATCH A1, SEPTEMBER 1976

Element and Method	Standard Values, ppb	Mean, ppb and No. of Samples	Bias-Mean -Standard Value ± 95% Confidence Limits, ppb	Standard Deviation, ppb	Coefficient of Variation
U (F)(a)	0.82	0.76 (6)	-0.06 ± 0.12	0.10	0.13
As (A)(b)	2.0	2.2 (6)	0.2 ± 1.0	0.6	0.27
Se (A)	2.0	1.8 (4)	-0.2 ± 0.8	0.5	0.28

(a)F = TOPO extraction-fluorometric.
(b)A = Atomic absorption.

Table 6

SUMMARY OF MEASUREMENT CONTROL RESULTS FOR NATURAL WATER, 7.7 PPB URANIUM, BATCH B1, SEPTEMBER 1976

Element and Method	Standard Values, ppb	Mean, ppb and No. of Samples	Bias-Mean -Standard Value ± 95% Confidence Limits, ppb	Standard Deviation, ppb	Coefficient of Variation
U (F)(a)	7.7	7.3 (6)	-0.4 ± 0.6	0.5	0.07
As (A)(b)	10.0	12.2 (4)	2.2 ± 0.7	0.4	0.03
Se (A)	1.0	1.1 (4)	0.1 ± 0.1	0.1	0.09

(a)F = TOPO extraction-fluorometric.
(b)A = Atomic absorption.

Table 7

SUMMARY OF MEASUREMENT CONTROL RESULTS FOR SYNTHETIC WATER, LOW URANIUM, BATCH 02, MAY THROUGH SEPTEMBER 1976

Element and Method	Standard Value, ppb	Mean, ppb and No. of Samples	Bias Mean - Standard Value ± 95% Confidence Limits	Standard Deviation, ppb	Coefficient of Variation
U (F)(a)	0.52	0.50 (26)	-0.02 ± 0.03	0.05	0.10
As (A)(b)	10	5.2 (21)	-4.8 ± 0.7	1.5	0.29
Se (A)	2	<0.2 (d)(21)			
Al (S)(c)	100	86 (18)	-14 ± 9	17	0.20
B (S)	3,000	2,000 (d)(19)			
Ba (S)	150	123 (19)	-27 ± 16	33	0.27
Co (S)	20	14 (19)	-6 ± 2	3	0.21
Cr (S)	100	82 (19)	-18 ± 6	11	0.13
Cu (S)	50	22 (19)	-28 ± 4	7	0.32
Fe (S)	100	94 (15)	-6 ± 9	17	0.18
Mn (S)	20	22 (19)	2 ± 3	6	0.27
Mo (S)	50	52 (19)	2 ± 6	12	0.23
Ni (S)	215	115 (18)	-100 ± 11	22	0.19
Ti (S)	102	109 (19)	7 ± 13	27	0.25
V (S)	10	12 (19)	2 ± 2	4	0.33
Zn (S)	500	696 (19)	196 ± 73	152	0.22

(a) F = TOPO extraction-fluorometric.
(b) A = Atomic absorption.
(c) S = Emission spectrographic.
(d) Insufficient measurements for valid standard deviation calculations. All measurements were identical.

Table 8

SUMMARY OF MEASUREMENT CONTROL RESULTS FOR SYNTHETIC WATER, HIGH URANIUM, BATCH 02, MAY THROUGH SEPTEMBER 1976

Element and Method	Standard Value, ppb	Mean, ppb and No. of Samples	Bias Mean - Standard Value ± 95% Confidence Limits	Standard Deviation, ppb	Coefficient of Variation
U (F)(a)	9.9	10.1 (29)	0.2 ± 0.4	0.7	0.07
As (A)(b)	2.0	1.3 (24)	0.7 ± 0.2	0.4	0.31
Se (A)	1.0	<0.2 (d)(24)			
Al (S)(c)	400	387 (20)	-13 ± 23	48	0.12
B (S)	200	192 (18)	- 8 ± 26	52	0.27
Ba (S)	30	26 (19)	- 4 ± 4	8	0.31
Co (S)	100	84 (20)	-16 ± 6	13	0.15
Cr (S)	20	20 (19)	0 ± 3	6	0.30
Cu (S)	200	114 (20)	-86 ± 30	64	0.56
Fe (S)	400	376 (19)	-24 ± 36	75	0.20
Mn (S)	100	96 (20)	- 4 ± 8	18	0.19
Mo (S)	10	8 (20)	- 2 ± 1	2	0.25
Ni (S)	43	34 (19)	- 9 ± 5	10	0.29
Ti (S)	41	38 (20)	- 3 ± 6	13	0.34
V (S)	50	62 (20)	12 ± 13	28	0.45
Zn (S)	50	<240 (d)(24)			

(a) F = TOPO extraction-fluorometric.

(b) A = Atomic absorption.

(c) S = Emission spectrographic.

(d) Insufficient measurements for valid standard deviation calculations. All measurements were identical.

Table 9

SUMMARY OF MEASUREMENT CONTROL RESULTS FOR SYNTHETIC WATER, LOW URANIUM, BATCH 01, FEBRUARY THROUGH SEPTEMBER 1976

Element and Method	Standard Value, ppb	Mean, ppb and No. of Samples	Bias Mean - Standard Value ± 95% Confidence Limits	Standard Deviation, ppb	Coefficient of Variation
U (F)(a)	0.80	0.81 (37)	0.01 ± 0.05	0.12	0.15
As (A)(b)	2	1.6 (37)	-0.4 ± 0.2	0.6	0.38
Se (A)	1	0.9 (37)	-0.1 ± 0.1	0.3	0.33
Al (S)(c)		140 (38)		125	0.89
B (S)		34 (38)		32	0.94
Ba (S)	19	20 (38)	1 ± 2	7	0.35
Co (S)	100	102 (38)	2 ± 8	23	0.22
Cr (S)	20	28 (38)	8 ± 5	14	0.50
Cu (S)	100	64 (36)	-36 ± 7	22	0.34
Fe (S)		97 (37)		57	0.59
Mn (S)		7 (37)		3	0.44
Mo (S)	10	9 (37)	-1 ± 1	3	0.33
Ni (S)	40	27 (36)	-13 ± 3	9	0.33
Pb (S)	20	27 (37)	7 ± 3	10	0.37
Ti (S)	38	30 (37)	-8 ± 2	7	0.23
V (S)	10	12 (38)	2 ± 2	5	0.42

(a) F = TOPO extraction-fluorometric.
(b) A = Atomic absorption.
(c) S = Emission spectrographic.

Table 10

SUMMARY OF MEASUREMENT CONTROL RESULTS FOR SYNTHETIC WATER, HIGH URANIUM, BATCH 01, FEBRUARY THROUGH SEPTEMBER 1976

Element and Method	Standard Value, ppb	Mean, ppb and No. of Samples	Bias Mean - Standard Value ± 95% Confidence Limits	Standard Deviation, ppb	Coefficient of Variation
U (F)(a)	10.3	10.2 (40)	-0.1 ± 0.3	0.81	0.08
As (A)(b)	8.0	5.3 (39)	-2.7 ± 0.5	1.5	0.28
Se (A)	2.0	1.2 (39)	-0.8 ± 0.2	0.6	0.50
Al (S)(c)		88 (34)		29	0.33
B (S)		48 (38)		48	1.0
Ba (S)	750	878 (38)	128 ± 73	221	0.25
Co (S)	250	261 (38)	11 ± 23	69	0.26
Cr (S)	150	148 (38)	-2 ± 15	46	0.31
Cu (S)	400	325 (38)	-75 ± 33	101	0.31
Fe (S)		93 (37)		25	0.27
Mn (S)		9 (36)		3	0.33
Mo (S)	50	61 (36)	11 ± 3	10	0.16
Ni (S)	537	408 (37)	-129 ± 37	112	0.27
Pb (S)	50	65 (38)	15 ± 7	22	0.34
Ti (S)	102	88 (38)	-14 ± 14	42	0.48
V (S)	50	62 (38)	12 ± 6	19	0.31

(a) F = TOPO extraction-fluorometric.
(b) S = Emission spectrographic.
(c) A = Atomic absorption.

Table 11

SUMMARY OF MEASUREMENT CONTROL RESULTS FOR SEDIMENTS, FEBRUARY THROUGH SEPTEMBER 1976

	Approx 5 ppm U Batch No. R1 (Standard Value Unknown)			Approx 10 ppm Batch No. S1 (Standard Value Unknown)		
Element and Method	Mean, ppm and No. of Samples	Standard Deviation, ppm	Coefficient of Variation	Mean, ppm and No. of Samples	Standard Deviation, ppm	Coefficient of Variation
U (F)(a)	3.3 (52)	0.31	0.09	9.0 (52)	0.75	0.08
U (D)(b)	5.2 (58)	0.18	0.03	10.6 (60)	0.20	0.02
As (A)(c)	6.6 (50)	1.5	0.23	12.6 (48)	2.2	0.17
Se (A)	0.4 (46)	0.26	0.65	0.6 (48)	0.30	0.50
B (S)(d)	62 (48)	32	0.52	92 (49)	50	0.54
Ba (S)	342 (48)	106	0.31	339 (49)	119	0.35
Co (S)	12 (49)	8	0.67	18 (49)	8	0.53
Cr (S)	52 (48)	18	0.35	58 (49)	19	0.33
Cu (S)	12 (49)	5	0.42	39 (49)	13	0.33
Li (S)	38 (43)	25	0.66	44 (47)	29	0.66
Mn (S)	676 (48)	232	0.34	532 (49)	189	0.36
Mo (S)	<2 (49)			18 (49)	6	0.33
Ni (S)	27 (49)	8	0.30	48 (48)	9	0.19
Pb (S)	14 (48)	2	0.14	19 (49)	9	0.47
Sc (S)	11 (49)	5	0.45	14 (49)	5	0.36
Ti (S)	5,439 (49)	1,638	0.30	5,408 (49)	1,574	0.29
V (S)	54 (48)	15	0.28	124 (49)	51	0.41
Y (S)	24 (49)	9	0.38	28 (49)	9	0.32
Zr (S)	232 (48)	88	0.38	156 (49)	46	0.29

(a) F = TOPO extraction-fluorometric.
(b) D = Delayed neutron counting.
(c) A = Atomic absorption.
(d) S = Emission spectrograph.

The fluorometric analyses for uranium in water and sediments and the delayed neutron counting analyses for uranium in sediments are in good control.

Measurements which have biases greater than 2 standard deviations of the analytical procedure are listed in Table 12. Only 4 results have been reported for Batch B1 and the arsenic bias has not yet been resolved. The low arsenic value for low uranium Batch 02 is attributed to interference from other elements in the control samples, since analysis of fresh standards and field samples spiked with known amounts of arsenic give satisfactory results. The negative bias for copper is unexplained, but is also thought to be due to interferences in the control. For nickel, analysis by atomic absorption indicated the true value to be lower than the reported standard value.

Table 12

MEASUREMENTS WITH BIAS GREATER THAN TWO STANDARD DEVIATIONS

Element	Batch	Table	Mean ppb	Bias	Standard Deviation
As	7.7 ppb U, Batch B1	2	12.2	2.2	0.4
As	Low U, Batch 02	3	5.2	-4.8	1.5
Cu	Low U, Batch 02	3	22	-28	7
Ni	Low U, Batch 02	3	115	-100	22

Some measurements have periods of low and high results. The standard deviations of these results are inflated, because the data for the low and high periods are included. Other cases with significant biases are arsenic, copper, and nickel in the high uranium batches, but these are not indicated because of the inflated standard deviations.

The atomic absorption analyses for selenium in the 02 Batches do not reflect the amounts added. The negative biases shown for arsenic, selenium, copper, and nickel are explained as being caused by interference from other elements in the sample.

GEOSTATISTICS

Major emphasis this quarter has involved improving procedures for computer verification of field data, error analysis for laboratory measurements, and upgrading plotting capabilities. Computerization of a principal component analysis to detect outliers in multivariate data was implemented. This procedure allows identification of samples which may have erroneous measurements for one or more of the variables. A principal component analysis was found to be very useful as a substitute for examination of numerous scatter plots, where 1 element versus another element is plotted. The principal component analysis should enable identification of samples which may require reanalysis in the laboratory.

The statistical analysis was completed on the Northwest Texas Pilot Survey. Field measurements for alkalinity and pH were compared. Laboratory data were analyzed using both principal component analysis and scatter plots, and cluster analysis procedures were used for interpretative analyses. Additionally, regression and correlation analyses were performed to assess interrelationships of variables in the different geologic units.

Three new statistical procedures were developed. A conversion procedure was implemented to enable URE data to be analyzed by the Statistical Analysis System (SAS) Computer Program studies. This system has the capability of a wide variety of statistical procedures and will enable considerable versatility. The cluster analysis used in the Llano Pilot Survey report has been modified to give summary statistics for the different clusters identified in the data. This will possibly enable identification of element concentration differences in the various clusters. A procedure is being developed to test two coefficients of variation. These statistics are obtained from the URE Quality Control Program. Evaluation of laboratory variation for the different elements will be an integral part of subsequent URE reports.

Preparation has begun for the writing of a paper *Clustering Problems for Geochemical Data* for presentation at the second ERDA Statistical Symposium in October 1976. This paper, to be presented by the URE Project statistician, will involve a wide range of data problems associated with geochemical sampling.

FIELD pH MEASUREMENTS

To assess field measurement methods of pH, paired pH readings were taken by a Corning Model 3 pH meter and pHydrion Lo-Ion pH paper. Table 13 gives the results of a statistical comparison of the two methods. The difference between the means for the 2 methods is significant for all 3 samplers. It should be noted that for 2 samplers, the paper gave a positive bias and the remaining sampler had a negative bias. Thus, the paper method can have a positive or negative bias depending upon the sampler. The variability of the meter and the paper is different. It is not possible to estimate the variation with these data unless the variability for one of the methods is assumed to be known. If it is assumed that the meter variability is ±0.15 (i.e., 2 standard deviations), then the paper variability ranges from ±0.36 to ±0.62. Hence, the paper is 2 to 4 times as variable as the meter. Subsequent analyses will compare the pH measurement from the Horiba Model U-7 Water Analyzer with the Lo-Ion pH paper. Currently, the Lo-Ion paper is used only as a backup method.

PLOTTING

A new Tektronix digitization system has been developed at the Oak Ridge National Laboratory and is currently being used to obtain the latitude/longitude of sampling sites. This system is remotely connected to the PDP-10 computer and includes a 4014-1 display unit, 4922 dual drive floppy disk, a 4923 tape cassette, a 4954 digital tablet (38.4 in. x 30.7 in.), 4952 joy stick, and a 4631 hard copy unit. With this system, the time required for digitization is reduced by 3 to 4 times.

Additional plotting changes for the general URE plotting routine, contour plotting, and cluster analysis plotting have been identified. The changes will improve the general quality of the plots used in the URE report. Additionally, modifications to the probability and frequency plotting programs have been made.

Table 13

COMPARISON OF TWO METHODS OF MEASURING pH USING THE WILCOXON SIGNED RANK TEST AND THE PAIRED t-TEST

		Mean									
Sampler(a)	N(b)	pH Meter(c)	pH LIP(c)	Wilcoxon Statistic	Significant Level(d)	t-Test Statistic	Significant Level(d)	Standard Deviation of Mean Difference	Estimated Standard Deviation for an LIP Method(e)	95% Confidence Interval for Standard Deviation(e)	Approximate Variability for LIP Method(e)
I (2/4, 3/19)	134	7.32	7.40	2,616	P<0.001	-4.80	P<0.001	0.017	0.18	(0.16, 0.22)	±0.36
II (1/29, 3/13)	131	7.14	7.05	5,848	P<0.001	3.52	P=0.001	0.024	0.25	(0.23, 0.31)	±0.50
III (1/29, 3/18)	112	7.25	7.42	1,309	P<0.001	-5.58	P<0.001	0.030	0.31	(0.27, 0.36)	±0.62

(a) Sampling dates in 1976 are in parentheses.
(b) Includes all well and stream water samples having paired pH measurements.
(c) Corning Model 3 pH meter and a pHydrion Lo-Ion pH paper.
(d) Small values of P (e.g., P<0.05) imply the two methods yield different means.
(e) Assumes meter variability is ±0.15.

DATA MANAGEMENT

The major data management activities this quarter were concerned with installing an updated version of the URE field form and the development of a computerized landowner's notification letter and generalized *retrospective* batch search program. The new field form necessitated modifications to the existing URE data record and batch file maintenance procedure. Additional data management effort was also expended in writing programs to provide sampling statistics, sample storage logs, and edit changes to various report programs.

UPDATED FIELD FORM

Data elements on the field form were added, deleted, and changed in an effort to ensure the more efficient collection of as much data as possible while in the field. The new field form that went into effect September 1976 is shown in Appendix A. The rearrangement of the field form and addition and deletion of field elements required modifications to the data record on the master file and the file maintenance procedure which processes the field form.

LANDOWNER'S NOTIFICATION LETTER

Landowners which request analytical results of samples collected on their property are provided the information after the data have been open filed. The landowner's name and address is recorded in the field form at the time the samples are collected and then stored in the URE data base. After the data have been open filed by the Grand Junction Office, the program pulls the landowner's name, address, and analytical results off the master file, and prints the notification letter, along with address labels for mailing. An example of the landowner's notification letter is presented in Appendix B.

GENERALIZED RETROSPECTIVE SEARCH

A new search program and procedure have been developed to facilitate multiple searches and a more comprehensive search strategy than has previously been possible. With the new procedure comes the ability to search on the following fields (search arguments) within a record: Map Code, Phase, Sample Type, Sampler's Initials, Stratigraphic Code, Producting Horizon Code, Sample Number Range, Latitude Range, or Longitude Range. The search employs full Boolean Logic, so search arguments can be combined in a manner to suit almost any need.

With slight modification of existing report-type programs, it will be possible to get multiple reports with 1 execution of the particular report program desired.

APPENDIX A

FIELD FORM

GENERAL SITE DATA

1	
1	Card Number

Attach Identical Sample Number Here

2	3	4	5	6	7

8	9	10	11	
				Site Number

12	13	14	15		16	17	
				-			Map Code

Sample Type

18		
M		Stream Sediment
H		Lake Sediment
S		Stream Water
W		Well Water
P		Spring Water
L		Lake Water
A		Bog Water
B		Plant
F		Soil (Use Remarks)
G		Rock
0		Other

19	
	Replicate Letter (A-Z)

Hr		Day		Mo		Yr	
20	21	22	23	24	25	26	27

28	29	30	
			Collector's Initials

31	
	Phase (P,1,or 2)

Field Sheet Status

32		
0		Original
C		Correction
V		Voiding

Control Sample

33		
A		Sediment, High U
B		Sediment, Low U
C		Water, High U
D		Water, Low U
0		Other

34	35	36	37	
			•	Air Temp (°C)

Location

Latitude						Longitude						
Deg		Min		Sec		Deg			Min		Sec	
38	39	40	41	42	43	44	45	46	47	48	49	50

51	52	53	54	
				Surface Geologic Unit Code

UCN-11992
(8-16-76)

Type of Veg.(1 Km Upstream)

55		
C		Conifer
&		Conifer & Deciduous
D		Deciduous
B		Brush
G		Grass
M		Moss
L		Lichen
0		Other

Density of Veg. (1 Km Upstream)

56		
B		Barren
S		Sparse
M		Moderate
D		Dense
V		Very Dense

Local Relief (1 Km Upstream)

57			
F		Flat	(<2m)
L		Low	(2-15m)
G		Gentle	(15-60m)
M		Moderate	(60-300m)
H		High	(>300m)
0		Other	

Weather

58			59		
C		Calm	C		Clear
P		Lt Wind	L		Pt Cldy
V		Windy	W		Overcst
R		V. Windy	V		Rainy
S		Gale	G		Snowy
0		Other			

Classes of Contaminants

60		
N		None
M		Mining (Use Remarks)
A		Agriculture
I		Industry
S		Sewage
P		Power Plant
U		Urban
0		Other

Stream Flow

61	62	63	
			Average Velocity (m/sec) N = No Visible Movement P = Stagnant Pool

64	65	66	
			Water Width (m)

67	68	69	
			Average Depth (m)

Water Level

70			70		
D		Dry	N		Normal
P		Pools	H		High
L		Low	F		Flood

Dominant Bed Material

71		
B		Boulder
C		Cobble
P		Pebble
S		Sand
T		Silt
Y		Clay
N		None (Use Remarks)

Sample Color (except Plants)

	Adj		Noun	
72	73	74	75	76

V	V Lt	PK	Pink
L	Light	RD	Red
M	Med	GN	Green
D	Dark	BU	Blue
		BN	Brown
CL	Clear	GY	Gray
WH	White	BK	Black
YL	Yellow	0T	Other
0R	Orange		

Odor of Sampled Material

77		
N		None
S		H_2S
0		Other

78		
R		Results Request (Use Remarks)

1	
2	Card Number

PLANT SAMPLE

18	19	
		No. of Plants Sampled (or No. of grabs for moss)

20	21	22	
•			Trunk Diameter (m) (1 m above ground)

23	24	25	
	•		Plant Height (m) (Avg. of plts. sam.)

Name of Tree, Deciduous

26			26		
R		Alto Verde	U		Locust
A		Ash	P		Maple
B		Beech	M		Mesquite
I		Birch	K		Oak, Other
D		Box Elder	V		Olive
N		Cottonwood	Y		Poplar
E		Elm	S		Sycamore
H		Hackberry	T		Salt Cedar
C		Hickory	X		Willow
W		Huisache	0		Other
L		Live Oak			

Name of Conifer

27			27		
A		N. Wh.Cedar	L		Larch
C		Cedar	P		Pine
F		Fir	S		Spruce
H		Hemlock	0		Other
J		Juniper			

Name of Bush

28			28		
A		Alder	W		Witch Hazel
B		Blueberry	Y		Yew
S		Sweet Gum	0		Other

Name of Moss

29		
P		Peat
S		Sphagnum
0		Other

Algae

30		
G		Blue-Green
B		Brown
0		Other

STREAM OR LAKE SEDIMENT

Sample Condition

31

D	Dry
W	Wet

Sample Treatment

32

N	None
S	Sieved
0	Other

33 34 No. of Grabs

35 36 % Organic Material

GENERAL WATER SAMPLES

Water Sample Treatment

37

N	None
F	Filtered Only
C	Acidified Only
A	Acidified and Filtered
0	Other

Depth of Visibility (m)

38 39 40 C = Clear

41 42 43 44 45 Conductivity (μmhos/cm)

46 47 48 Dissolved O_2 (ppm)

49 50 51 Temperature (°C)

52 53 54 pH

55 P pH by Lo-Ion Paper

56 57 58 59 Total Alkalinity(ppm)

60 61 62 63 P Alkalinity (ppm)

64 65 66 67 M Alkalinity (ppm)

Appearance of Water

68

C	Clear
M	Murky
A	Algal
0	Other

69 70 71 72 73 Discharge (liters/min)

74 75 76 77 Ident.of Prod.Horizon(s) (Geol. Unit Code)

Confid. of Unit Ident.

78

H	High Degree
R	Probably
S	Possible

Source of Unit Ident.

79

P	Publication
W	Owner
U	User
G	Geologic Inference
0	Other

1

3 Card Number

WELL WATER

Type of Well

18

D	Drilled
P	Drive Point
G	Dug
U	Unknown
0	Other

Power Classification

19

A	Artesian Flow
E	Electric
G	Gasoline
W	Wind
H	Hand
0	Other

Casing

20

N	None(Below Water Table)
S	Steel
G	Galvanized
P	Plastic
U	Unknown
0	Other

Pipe Composition

21

F	Steel
Z	Galvanized
C	Copper
P	Plastic
U	Unknown
0	Other

Where Sample Taken

22 23 24 Meters from Well Head

H=Holding Tank - Use Remarks

Where Sample Taken With Respect to Pressure Tank

25

B	Before
A	After
N	None

Use of Well

26

M	Municipal
H	Household
S	Stock
I	Irrigation
A	All of above
X	H and S
Y	H and I
Z	S and I
N	None
0	Other

Frequency of Pumping

27

C	Constant (hourly)
F	Frequent (daily)
I	Infrequent (weekly)
R	Rare (no recent use)

Depth to top of Prod.Horizon(s)

28 29 30 31 (Meters)

Confidence of Producing Depth

32

H	High
R	Probable
S	Possible

Source of Prod. Depth Info.

33

P	Publication
W	Owner
U	User
G	Geologic Inference
0	Other

Total Well Depth

34 35 36 37 (Meters)

Confidence of Total Depth

38

H	High
R	Probable
S	Possible

Source of Total Depth Info.

39

P	Publication
W	Owner
U	User
G	Geologic Inference
0	Other

LAKE WATER

Type of Lake

55

N	Natural
M	Manmade

56 57 58 59 Area (sq km)

Remarks: ____

APPENDIX B

LANDOWNER'S NOTIFICATION LETTER

URANIUM RESOURCE EVALUATION PROJECT
OAK RIDGE GASEOUS DIFFUSION PLANT
P.O. BOX P MAIL STOP 246
OAK RIDGE, TENNESSEE 37830

Date

Name
Address

DEAR SIR:

THESE ARE THE RESULTS OF ANALYSIS OF SAMPLES OBTAINED BY OUR GEOLOGIST FROM YOUR PROPERTY ON date IN CONNECTION WITH THE UNITED STATES ENERGY RESEARCH AND DEVELOPMENT ADMINISTRATION HYDROGEOCHEMICAL AND STREAM SEDIMENT SURVEY.

THANK YOU FOR YOUR COOPERATION.

J. W. ARENDT

PROJECT MANAGER

```
BOTANICAL SAMPLE        (SAMPLE NUMBER       )

STATION LOCATION : LATITUDE=           ; LONGITUDE=

     ELEMENT DETERMINATIONS(PPM)
          URANIUM    =        0.20
          SILVER     =   <      10
          ALUMINUM   =        1500
          GOLD       =   <      10
          BORON      =         400
          EARIUM     =         400
          COBALT     =   <      20
          CHROMIUM   =          20
          CCPPER     =          60
          IRCN       =        1000
          MAGNESIUM  =       25000
          MANGANESE  =         250
          MOLYBDENUM =   <      10
          NICBIUM    =   <      10
          NICKEL     =   <      40
          PHCSPHORUS =       10000
          LEAD       =          60
          SCANDIUM   =   <      10
          THCRIUM    =   <      40
          TITANIUM   =          60
          VANADIUM   =   <      10
          YTTRIUM    =   <      10
          ZINC       =   <     600
          ZIRCONIUM  =   <      20

PPM MEANS PARTS PER MILLION
< MEANS LESS THAN
```

```
     STREAM SEDIMENT SAMPLE (SAMPLE NUMBER        )

     STATION LOCATION : LATITUDE=          ; LONGITUDE=

          ELEMENT DETERMINATIONS(PPM)
               URANIUM     =        1.27
               ARSENIC     =         1.9
               SELENIUM    =         1.0
               SILVER      =   <       1
               GOLD        =   <      40
               BORON       =          30
               BARIUM      =         200
               COBALT      =   <       4
               CHROMIUM    =          15
               COPPER      =           8
               LITHIUM     =          30
               MANGANESE   =         100
               MOLYBDENUM  =   <       2
               NIOBIUM     =   <      20
               NICKEL      =           8
               PHOSPHORUS  =   <    1000
               LEAD        =           8
               PLATINUM    =   <      10
               SCANDIUM    =           4
               THORIUM     =   <     400
               TITANIUM    =        1500
               VANADIUM    =          15
               YTTRIUM     =   <      10
               ZINC        =   <     200
               ZIRCONIUM   =          80

PPM MEANS PARTS PER MILLION
< MEANS LESS THAN
```

```
  STREAM WATER SAMPLE    (SAMPLE NUMBER      )

  STATION LOCATION : LATITUDE=          ; LONGITUDE=

       WATER MEASUREMENTS
            TEMPERATURE ( C)         =     16.7
            PH                       =      8.0
            M ALKALINITY (PPM)       =      222
            TOTAL ALKALINITY (PPM)   =      238
            SULFATE (PPM)            =       35

       ELEMENT DETERMINATIONS(PPB)
            URANIUM     =        0.66
            ARSENIC     =         0.9
            SELENIUM    =         0.5
            SILVER      =   <       4
            ALUMINUM    =         100
            GOLD        =   <       4
            BORON       =         100
            BARIUM      =          60
            COBALT      =   <       8
            CHROMIUM    =   <       4
            COPPER      =   <      16
            IRON        =   <      40
            MANGANESE   =   <       4
            MOLYBDENUM  =   <       4
            NIOBIUM     =   <       4
            NICKEL      =   <      16
            PHOSPHORUS  =   <     400
            LEAD        =          80
            SCANDIUM    =   <       4
            THORIUM     =   <      16
            TITANIUM    =   <      24
            VANADIUM    =   <       4
            ZINC        =   <     240
            ZIRCONIUM   =   <       8

PPB MEANS PARTS PER BILLION
PPM MEANS PARTS PER MILLION
< MEANS LESS THAN
```

```
                                  *
     WELL WATER SAMPLE       (SAMPLE NUMBER       )

     STATION LOCATION : LATITUDE=          ; LONGITUDE=

          WATER MEASUREMENTS
               TEMPERATURE ( C)           =     20.1
               PH                         =      7.0
               M ALKALINITY (PPM)         =      345
               TOTAL ALKALINITY (PPM)     =      325
               SULFATE (PPM)              =       35

          ELEMENT DETERMINATIONS(PPB)
               URANIUM     =        0.87
               ARSENIC     =  <      0.5
               SELENIUM    =         0.4
               SILVER      =  <        4
               ALUMINUM    =          40
               GOLD        =  <        4
               BORON       =          40
               BARIUM      =          80
               COBALT      =  <        8
               CHROMIUM    =  <        4
               COPPER      =  <       16
               IRON        =  <       40
               MANGANESE   =  <        4
               MOLYBDENUM  =  <        4
               NIOBIUM     =  <        4
               NICKEL      =  <       16
               PHOSPHORUS  =  <      400
               LEAD        =          20
               SCANDIUM    =  <        4
               THORIUM     =  <       16
               TITANIUM    =  <       24
               VANADIUM    =  <        4
               ZINC        =  <      240
               ZIRCONIUM   =  <        8

          *
          THESE VALUES CAN NOT BE USED AS AN INDEX OF DRINKING WATER QUALITY

PPB MEANS PARTS PER BILLION
PPM MEANS PARTS PER MILLION
< MEANS LESS THAN
```

DISTRIBUTION

INTERNAL

1. Administrative Offices
Parks, C. J.

2- 6. Computer Sciences Division
Brooks, A. A.
Carter, H. P.
Green, R. O.
McLaren, R. A.
Westley, G. W.

7. Gaseous Diffusion Dev. Div.
Trammell, H. E.

8-19. K-25 Technical Services
Arendt, J. W. (10)
Levin, R. W.
Napolitan, D. S.

20-22. Library

23. Operations Analysis Div.
Bradbury, J. T.

24-26. ORGDP Records Department (RC)

27. Technical Director
Wilcox, W. J., Jr.

28-30. ORNL
Culler, F. L., Jr.
Postma, H.
Shults, W. D.

31-32. Paducah
Bewley, H. D.
Hopkins, C. C.

33-43. Y-12 Plant
Bernander, N. K. (2)
Case, J. M.
Cobert, H. I.
Gambill, E. F.
Hibbs, R. F.
Horde, G. W.
Vanstrum, P. R.
White, J. C.
Yaggi, W. J.
Zerby, C. D.

EXTERNAL

44-45. F. P. Baranowski, Director
Division of Nuclear Fuel Cycle and Production
U.S. Energy Research and Development Administration - Headquarters
Washington, D.C. 20545

46. W. A. Beetem
Water Resources Division
U.S. Geological Survey
National Center
Reston, Virginia 22092

47. Al Clebsch, Jr.
Water Resources Division
U.S. Geological Survey
Denver Federal Center
Denver, Colorado 80225

48. Warren I. Finch, Chief
Branch of Uranium-Thorium Resources
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Oak Ridge, Tennessee 37830

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70-71. Technical Information Center