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A HIGH-CAPACITY NEUTRON ACTIVATION ANALYSIS FACILITY

R. C. HOCHEL, W. W. BOWMAN, AND C. W. ZEH

E. I. du Pont de Nemours & Co. (Inc.)
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Aiken, SC 29801

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ABSTRACT

A high-capacity neutron activation analysis facility, the Reactor Activation Facility, was designed and built and has been in operation for about a year at one of the Savannah River Plant's production reactors. The facility determines uranium and about 19 other elements in hydrogeochemical samples collected in the National Uranium Resource Evaluation program, which is sponsored and funded by the United States Department of Energy, Grand Junction Office. The facility has a demonstrated average analysis rate of over 10,000 samples per month, and a peak rate of over 16,000 samples per month.

Uranium is determined by cyclic activation and delayed neutron counting of the U-235 fission products; other elements are determined from gamma-ray spectra recorded in subsequent irradiation, decay, and counting steps. The method relies on the absolute activation technique and is highly automated for round-the-clock unattended operation.

CONTENTS

LIST OF FIGURES

LIST OF TABLES

A HIGH-CAPACITY NEUTRON ACTIVATION ANALYSIS FACILITY

INTRODUCTION

The Reactor Activation Facility (RAF) is a highly automated neutron activation analysis (NAA) system. The facility is installed at the Savannah River Plant's C-Area production reactor and has been in operation since September 1978. The RAF was built to provide NAA for the large number of hydrogeochemical samples collected in the National Uranium Resource Evaluation (NURE) program, which is sponsored and funded by the United States Department of Energy, Grand Junction Office. The Savannah River Laboratory (SRL) is one of three laboratories participating in the hydrogeochemical and stream sediment reconnaissance portion of the NURE program, and is responsible for sampling, analyzing, and reporting on an area of about 1,500,000 square miles in 30 eastern and 7 western states.

The RAF is intended to provide analyses for an estimated 400,000 samples to be collected in SRL's portion of the NURE program, and replaces a smaller pilot-scale facility, which was operated from September 1975 to November 1977.[1] The RAF has a demonstrated average analysis rate of over 10,000 samples per month, and a peak rate of over 16,000 samples per month. Uranium and up to 19 other elements are determined in sediment samples, and in ground and stream water samples after concentration on ion-exchange resin. Sediment and resin samples are prepackaged into special polyethylene irradiation capsules. Up to 2400 capsules can be loaded into the system for 72 hours of round-the-clock unattended operation.

Uranium is determined by cyclic activation and delayed neutron counting of the U-235 fission products; other elements are determined from gamma-ray spectra recorded in subsequent irradiation, decay, and counting steps. The method relies on the absolute activation technique. Details of the absolute activation technique are given by MacMurdo and Bowman,[2] and additional information about installation, checkout, and calibration of both the pilot facility and the RAF are documented in SRL-NURE quarterly and semiannual reports.[3]

DESCRIPTION OF THE REACTOR ACTIVATION FACILITY

Physical Layout

The RAF is located in the courtyard of C Reactor. Figure 1 shows the three trailer modules that constitute the RAF. The Control Module is in the foreground, the Storage Module is in the background, and the Counting Module is in the middle. A schematic diagram of the RAF system is shown in Figure 2.

Capsules are pneumatically transported to an irradiation assembly, which is inserted into Instrument Port D2 at the midplane of the reactor. The neutron spectrum at this position is very soft and provides a flux of about 5 x 10^{12} neutrons/(cm^2-sec). Six polyethylene transport tubes connect the irradiation assembly at the -20 foot level of the reactor to the Counting Module at the zero-foot level (a total distance of about 85 feet).

Control Module. The Control Module contains an SEL 32/55 computer (Systems Engineering Laboratory), pneumatic control components, and data acquisition electronics. The computer is used for both process control and data reduction. The Control Module also contains pulse height analyzers, scalers, associated nuclear instrument module electronics for the various gamma and neutron detectors, interfacing for all electronic and pneumatic equipment, and a switch panel for manual pneumatic control.

Storage Module. The Storage Module contains two identical sample storage stacks, each capable of handling about 6000 samples, and a four-way diverter (4W1). The stacks are used for loading, unloading, and storage of samples for radioactive decay before delayed counting.

Counting Module. The Counting Module was retained from the pilot-scale facility and uses the same external connections to the irradiation assembly at the -20 foot level of C Reactor (Figure 3). The interior of the module, however, contains an expanded pneumatic transport system and additional neutron and gamma detectors.

The Counting Module contains a portion of the pneumatic transport system, including the six connections (R1 to R6) that lead to the irradiation assembly at the -20 foot level. The two combination delayed-neutron and gamma counters (D1 and D2) are connected to reactor irradiation positions through four-way diverters (D1 to R1, R2, or R3 via 4W3; D2 to R4, R5, or R6 via 4W4). The four-way diverter 4W2 is used as a two-way diverter to direct samples from the stacks to either the D1 bank (detector and irradiation positions) or the D2 bank. Diverter 4W2 is connected via Diverter 4W1 to two entry/exit ports on each of the stacks (S1 and S2).

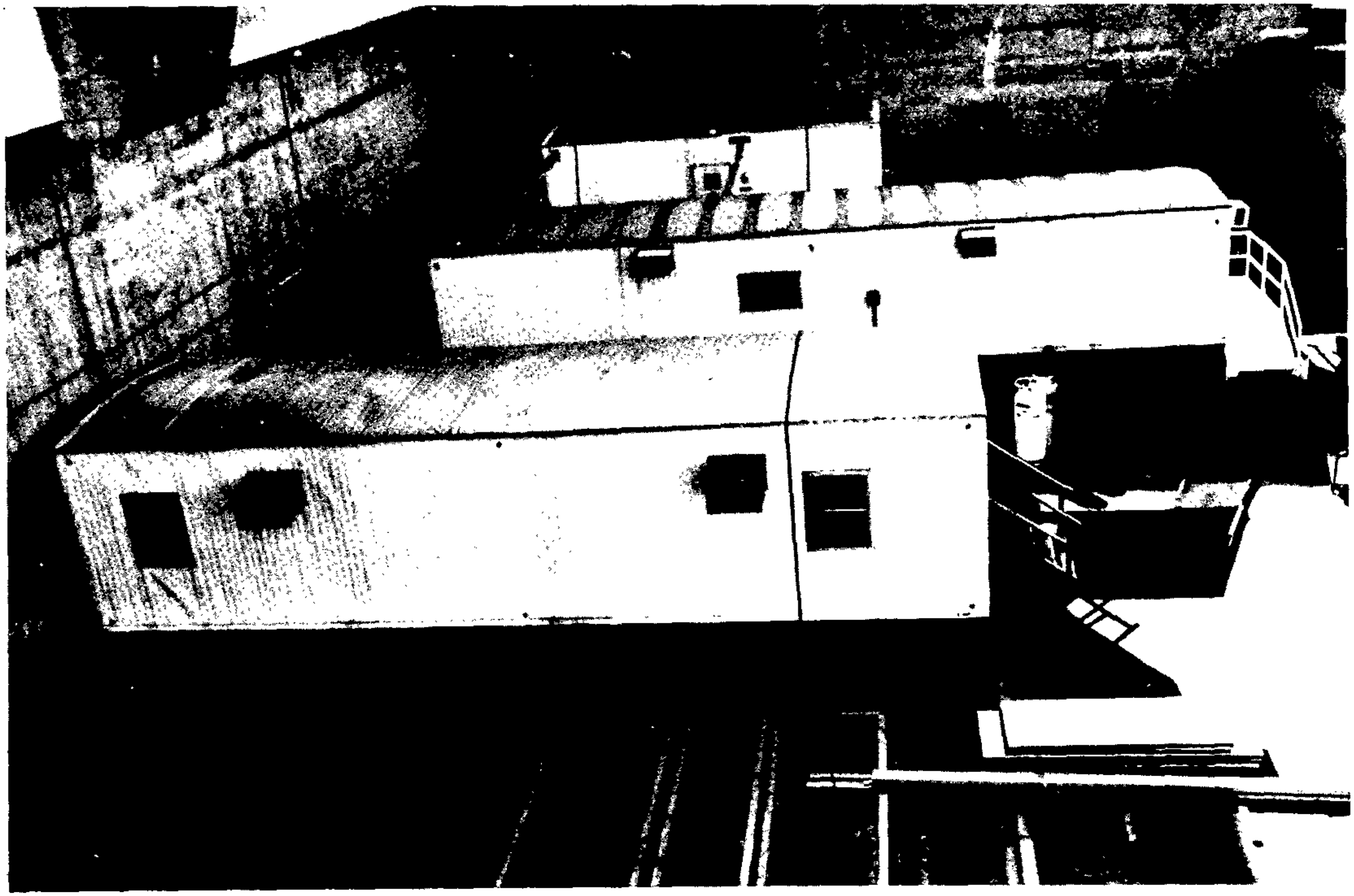
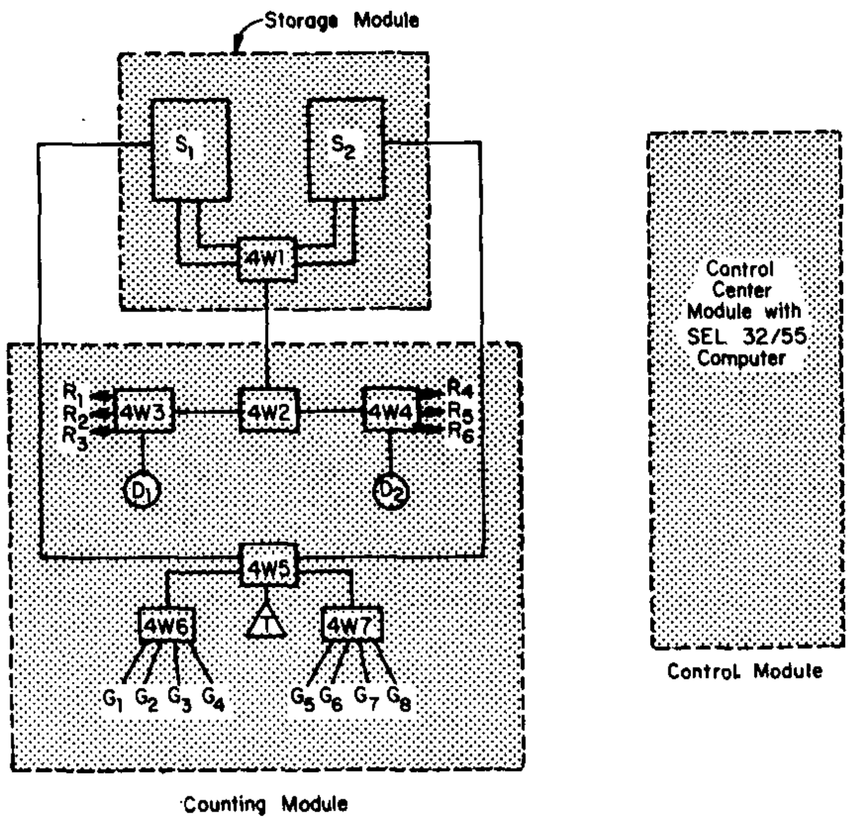


FIGURE 1. Modules of the Full-Scale Neutron Activation Facility at C Reactor



S Storage Stack
4W Four-way Diverter
R To Reactor Irradiation Position
— Pneumatic System
D Delayed-Neutron Detector
G Ge Detectors(1-4 Movable)
T Turnaround Station
--- Trailer Walls

FIGURE 2. Diagram of the RAF System

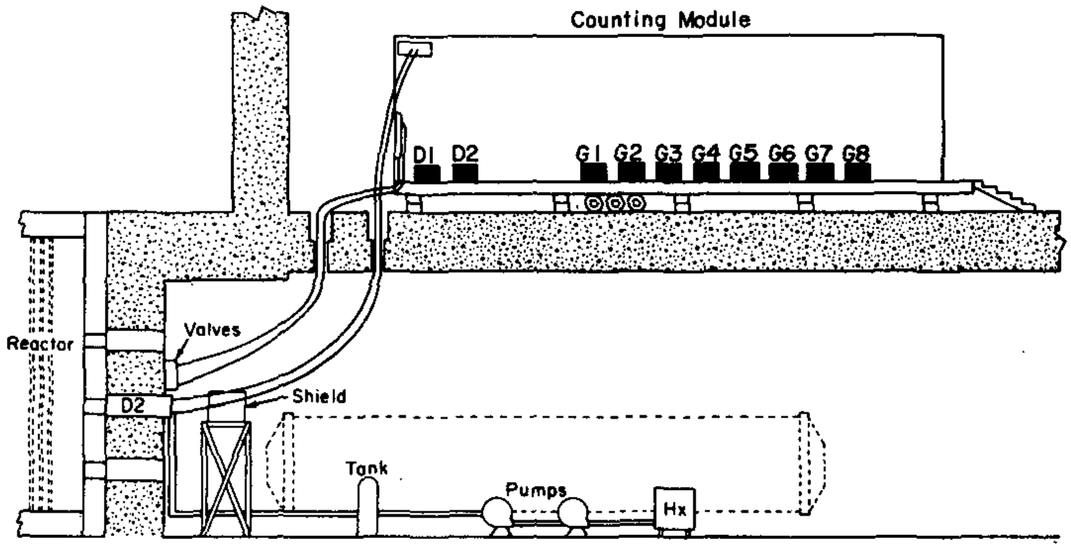


FIGURE 3. Diagram of the Counting Module Detectors and Transport System Connections to the Irradiation Assembly

A third port on each stack is used to access eight gamma detectors (G1 to G8) through Diverter 4W5 and the turnaround (T). The turnaround allows Diverter 4W5 to select either Diverter 4W6 or Diverter 4W7. The turnaround also turns the capsule sample-end down for counting by one of the gamma detectors; capsule orientation at Detectors D1 and D2 is sample-end up so that the sample is exposed to the maximum neutron flux in the irradiation assembly.

Irradiation Assembly

A diagram of the irradiation assembly (IRAS) is shown in Figure 4. It comprises an axial aluminum tube surrounded by six stainless steel subassemblies enclosed in an aluminum canister. The axial tube serves as a cooling water inlet and contains two self-powered rhodium flux monitors which provide continuous neutron flux data to the Control Module. Surrounding the axial tube are the six irradiation subassemblies (IRSAS). Each IRSAS contains a pair of concentric stainless steel tubes. The inner tubes are capsule transit tubes, which are slotted at the terminal ends (capsule irradiation sites) for flow of propulsion and exhaust air. The outer tube forms an annulus which channels propulsion and exhaust air to and from transit tubes. Sample capsule movement to and from the IRSAS, as well as all transport system terminus points at the zero-foot levels, are monitored by photodetectors interfaced to the SEL's interrupt logic. Passage of a capsule by a photodetector provides the computer with a vectored interrupt which determines the position and time of the movement.

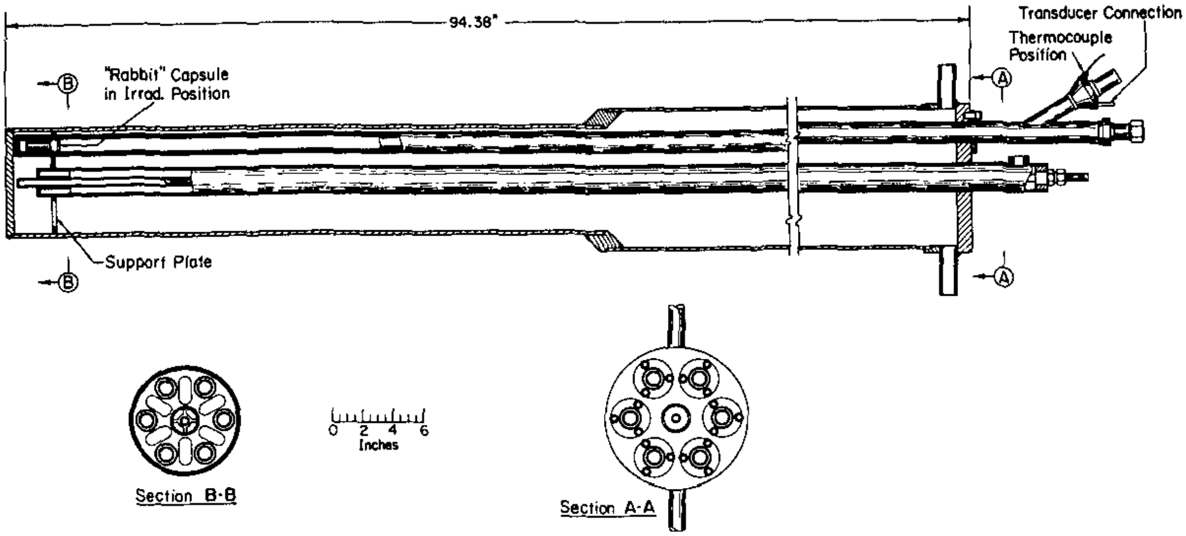


FIGURE 4. Schematic View of a Reactor Irradiation Assembly

Each IRSAS is monitored for temperature by a thermocouple attached to the reactor end of the assembly. Figure 5 shows the completed IRAS.

Control Module Components and Operation

SEL 32/55 Computer. Figure 6 shows a schematic diagram of the computer and associated peripheral components. Ten pulse height analyzers and 12 scalers, all designed and built at SRL,[4] record gamma spectra and monitor detector count rates. The computer handles all process control and data-reduction software. Analytical results are temporarily stored on discs and later written on magnetic tape for archiving and entry into the NURE Data Manager System[5] at SRL's IBM 360 computer facility. All vital process control information and sample analysis data are simultaneously written to two discs as a precaution against a severe disc "crash."

The SEL 32/55 computer is interfaced to the pneumatic components in the Counting and Storage Modules through a real-time peripheral (RTP) unit. The RTP unit is simply a bidirectional interface which allows the computer to communicate with peripheral analogs and digital devices such as pneumatic components. A switch panel simulator designed by SRL (Figure 7) is incorporated into the RTP unit. When the RAF is running under computer control, operations of the pneumatic transport system are visually displayed by lights on the panel of the simulator. The panel also allows simulation of pneumatic component responses to computer commands and manual operation of the entire transport system. The panel was also used to debug process control software by simulating the transport system before its actual installation.

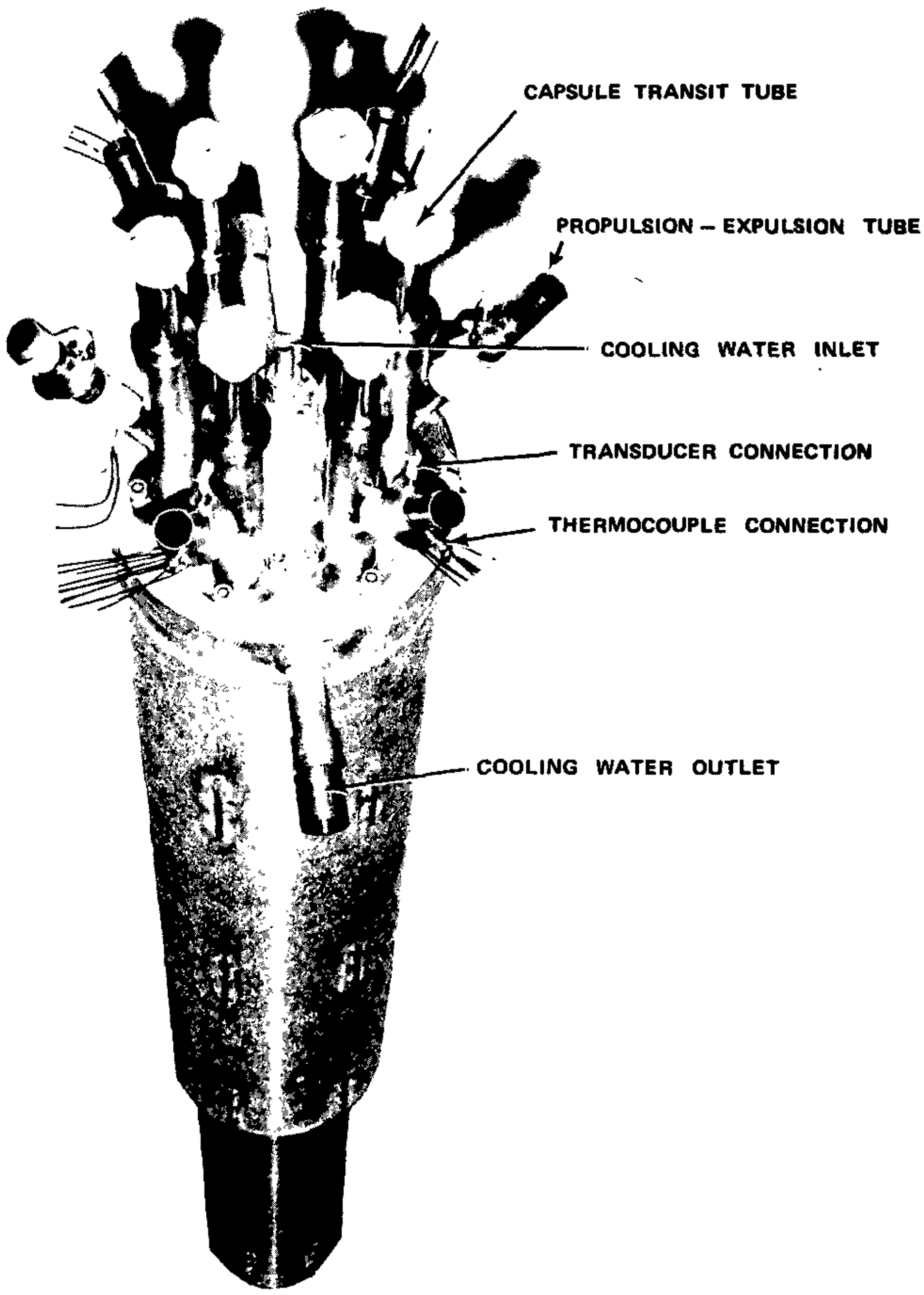


FIGURE 5. A Completed Reactor Irradiation Assembly

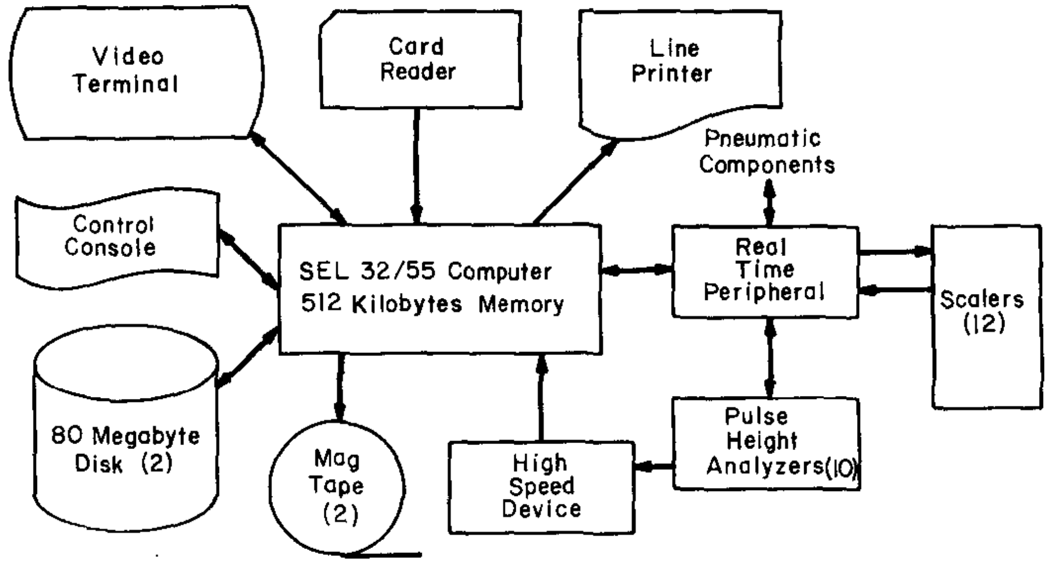


FIGURE 6. Relationship of the SEL 32/55 Computer and Associated Components

Software Design. The process control software (Figure 8) was designed to be flexible, to provide a high degree of automation, and to be resource-driven (resources are irradiation positions and the various neutron and gamma counting stations). A resource-driven system is one which continuously monitors the status of all resources and tries to keep each fully utilized. Because the RAF's resources are independent and duplicated, mechanical failure of any resource will usually only slow production, not stop it.

Software Functions. After samples are loaded into the stacks, the operator executes the program LOADSTAK, which reads card input to determine the number, type, and locations of the newly loaded samples. The output from LOADSTAK mirrors the input and allows the operator to verify input data before starting a run. Next, the operator executes STARTUP, which initializes all programs and requests the date and time. Finally, the operator executes the two task programs FINDRCAP and FINDDCAP, which drive the system by continuously looking for unused resources. If an unused resource is found, the stacks are scanned to see if any available samples can use that resource. If so, control is passed to a library of task programs responsible for carrying out all steps in the required analytical regime. Parameters governing the regime steps are contained in the DATAPOOL library stored in the computer memory; analytical regimes are easily altered by changing DATAPOOL.

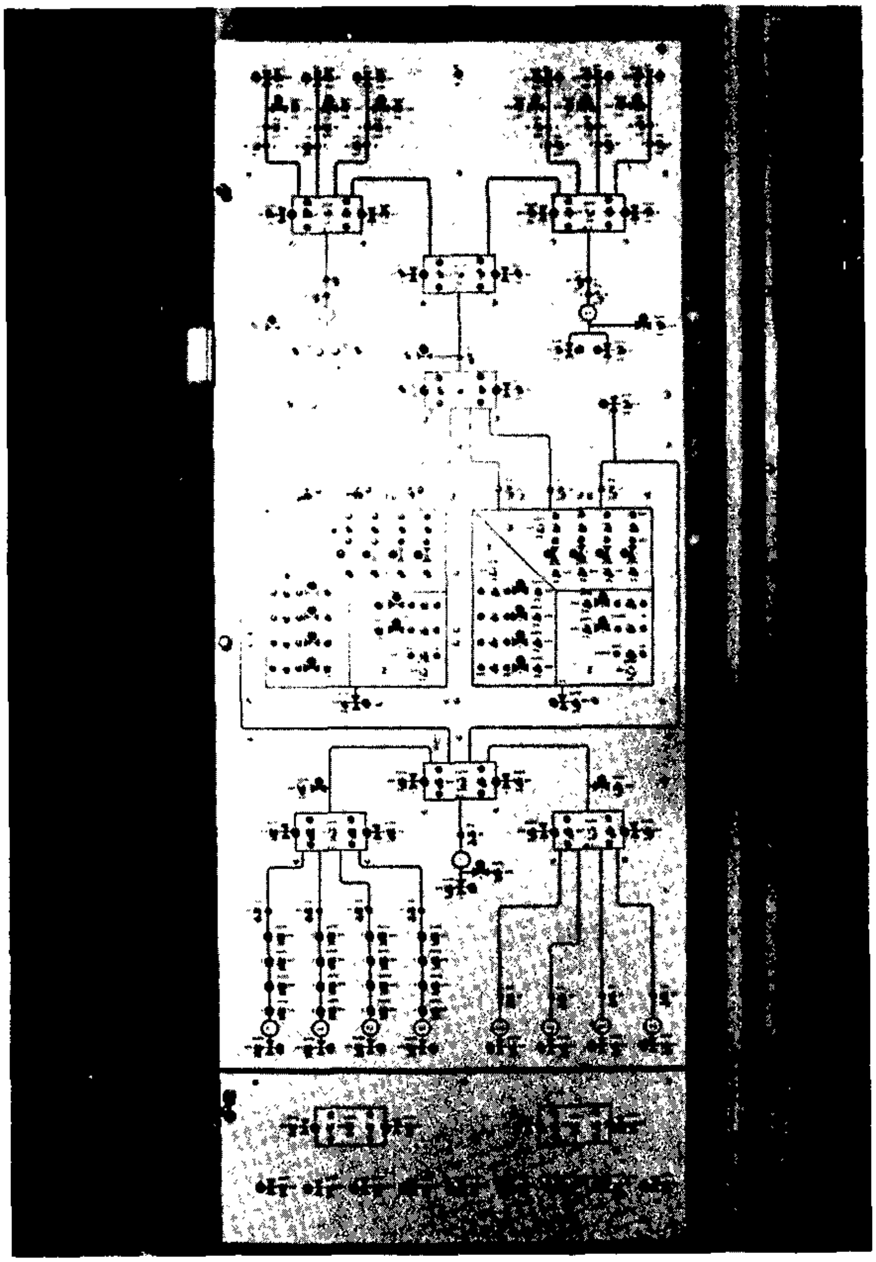


FIGURE 7. Switch Panel to Simulate Response of Pneumatic Components of the Sample Transport System

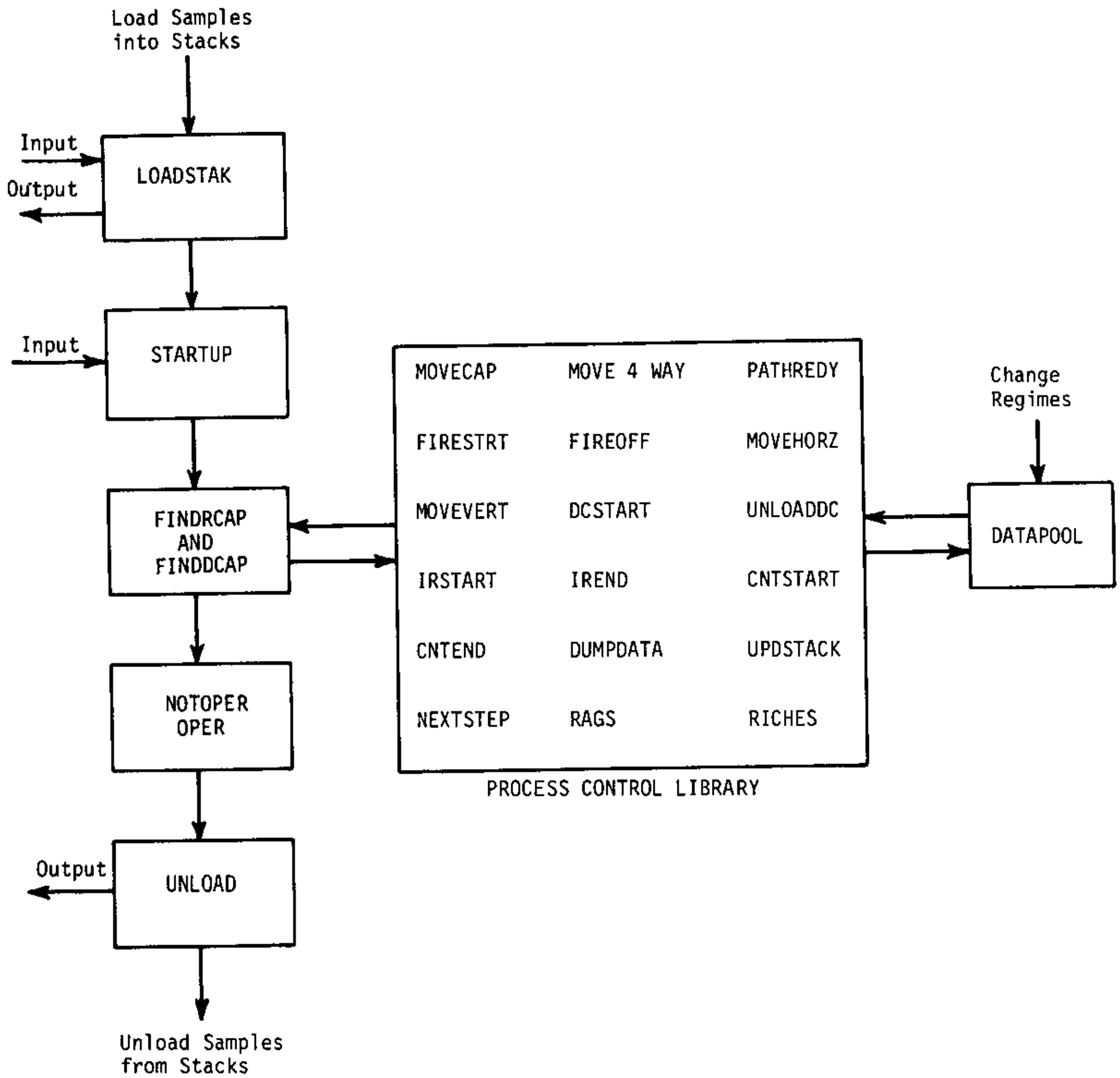


FIGURE 8. Diagram of the Process Control Flow System (Software)

Once the system is running, NOTOPER and OPER are used to disable and enable resources. Disabling one or more resources by NOTOPER provides a means of shutting down these resources in an orderly way; they are identified as "inoperable." However, processing of any samples already under way by any of these "inoperable" resources is completed before shutdown. No data are lost and the samples are returned to the stacks. The operator can then execute UNLOAD to obtain a listing of the identities and locations of samples ready to be unloaded. New samples can be loaded via LOADSTAK. Resources identified as "inoperable" remain unavailable to all subsequent samples until these resources are re-identified as "operable" (enabled) by execution of OPER. This allows the system to re-start where it left off.

The programs RAGS and RICHES in Figure 8 are data-reduction programs that are periodically activated by the process control software. RAGS[6] computes peak intensities and energies from the gamma spectral data from the pulse height analyzers. Output from RAGS is written to disc for access by RICHES and eventual archival storage. RICHES (Ref. 3, No. 14, pp 58-59) combines the neutron count, flux reading, and timing information with all the RAGS data for a sample. Identities and concentrations of elements in the sample are determined by non-linear least-squares analysis of these data combined with isotope and cross-section data in the RICHES library.

Both RAGS and RICHES data are periodically dumped from disc to tape. RAGS data are archived for further reference or corrections. RICHES data are incorporated into the NURE Data Manager System, which serves as a master data file for all NURE analyses. Disc space occupied by RAGS and RICHES data is freed only after the data have been archived or entered into the Data Manager.

Storage Module Components and Operation

Storage Stacks. Figure 9 shows one of the two storage stacks in the RAF. Both stacks are mechanically and operationally identical. A stack is a 16 x 16 array of vertical storage tubes held on 1.5-inch centers by a precision lattice plate. Each of the 256 tubes (save six used for transport, loader, and unloader) can hold up to 25 samples for an overall capacity of 6250 samples per stack. In the bottom of each tube is a grabber, a circular device of finger-like springs (Figure 10). Spreading the springs (fingers) allows a sample to fall from the bottom of the tube. Conversely, a sample can be pushed up into the device by a piston to a position where it is held by the fingers.

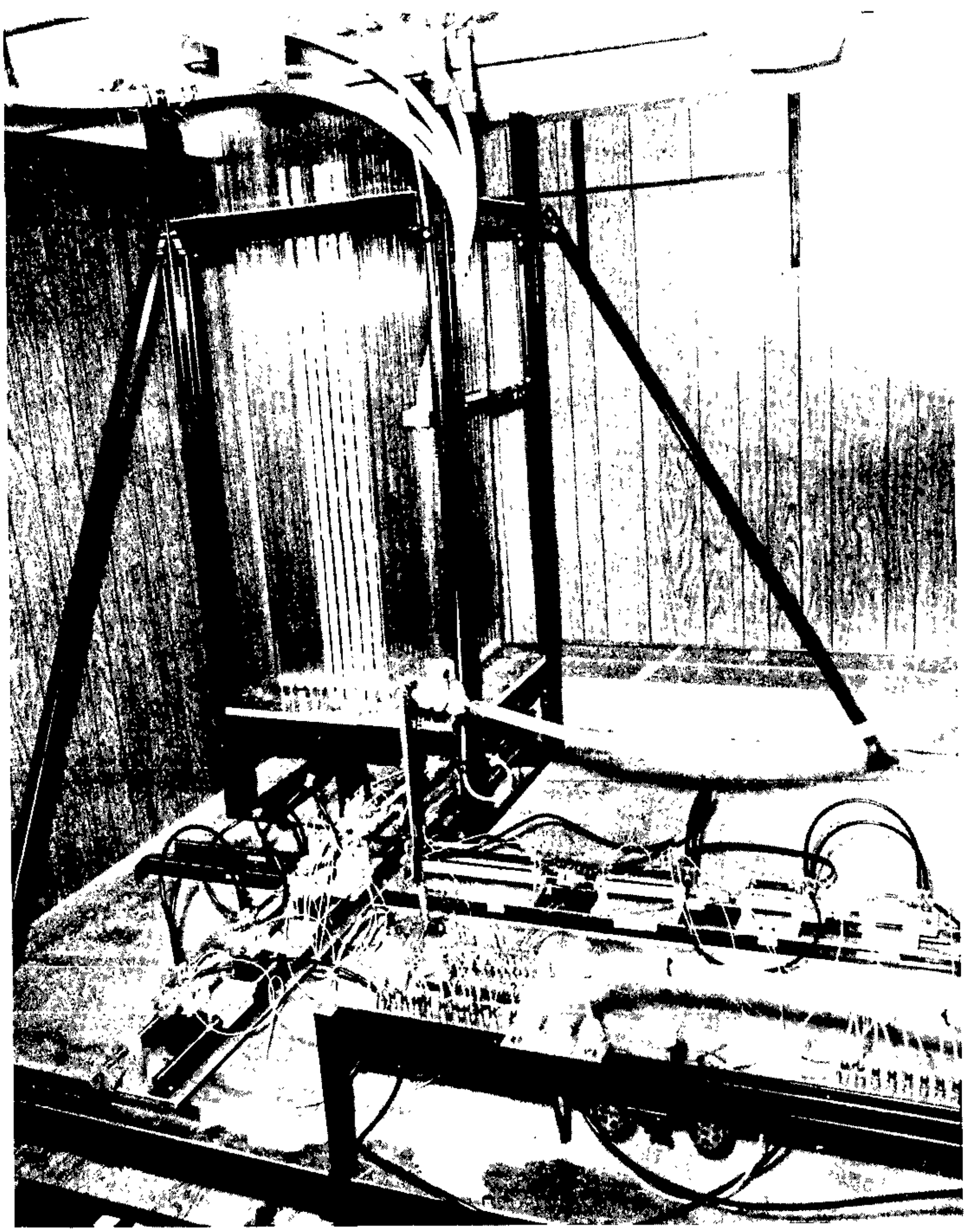


FIGURE 9. Sample Storage Stack

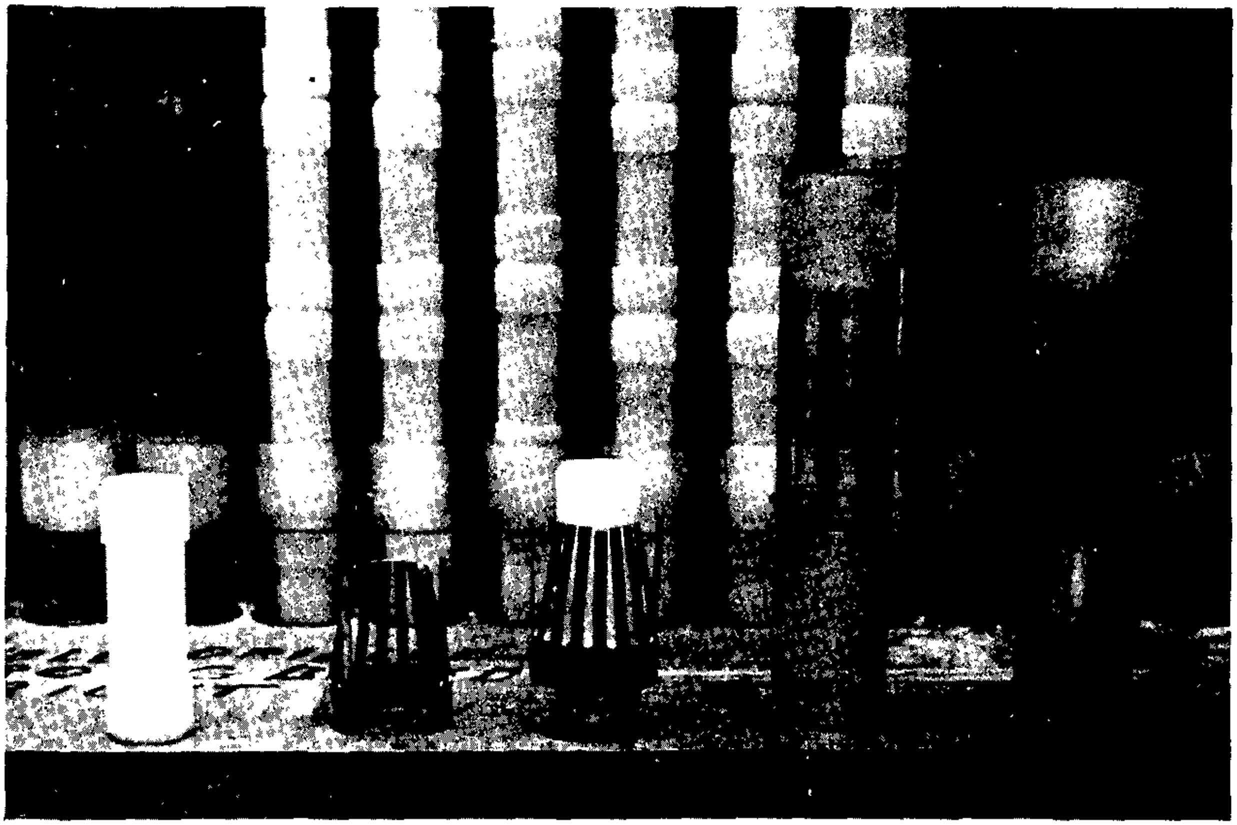


FIGURE 10. Device for Holding Sample Capsules in Stack

Store-Retrieve Device. A store/retrieve or Z-motion device under the lattice plate can either store or retrieve the bottom sample in any tube, or can eject samples from the storage stack by air pressure through any of the three entry/exit ports (Figure 2). The Z-mction device rides on the ways of a Y-axis frame, which in turn rides the ways of an X-axis frame. Motion along the X- and Y-axes is controlled by four binary-incremented air cylinders with strokes of 1-1/2, 3, 6, and 12 inches. By extending or retracting the appropriate air cylinders, the computer can position the Z-motion device under any one of the 256 tubes. Feedback from the air pressure that operates the cylinders signals their states to the computer through pressure switches. A photodetector senses the presence or absence of a sample in the Z-motion device.

Sample Management. Management of the storage stacks is under computer control. The computer keeps track of the location and status of all samples in the stacks; all samples in the stacks are awaiting either irradiation, intermediate counting, final counting, or unloading. Two tubes in each stack are designated as load tubes and are each fed by a 600-sample magazine (see section titled **Loading Station**). One tube in each stack is designated as an unloaded tube and feeds an automatic unloader (see section titled **Unloading Station**).

Unloading Station. The automatic unloader consists of four binary-incremented air cylinders identical to those used for the "X" and "Y" motions of the store-retrieve device in the Storage Module. Each storage stack has one tube designated for unloading. This tube is connected to a two-port head that moves with the strokes of the air cylinders. The four air cylinders allow 16 different positions to be selected as depositories for completed samples from each stack (Figure 11). Completed samples are deposited into 5-gallon removable cartons which serve as liners for each can in a 32-can cluster. The number and type (resin or sediment) of samples deposited in each can is selectable by computer software. Cartons containing completed samples are removed weekly. Unloading 3500 samples takes about 20 minutes.

Counting Module Components and Operation

The Counting Module contains all the neutron and gamma detectors and also the automatic loading device for samples stored in the stacks in the Storage Module.

Loading Station. The station for the automatic loader (Figure 12) consists of a bench-mounted V-shaped trough. A tube containing 25 samples is placed into the trough. A hand-operated lever (far left, Figure 12) actuates an air cylinder, which extends to engage and lock the tube at both ends. A length of polyethylene tubing connects the loader to a 4-way diverter (not shown in Figure 2) in the Storage Module. Each leg of the diverter is connected to a coil of tubing making two revolutions about the top perimeter of the Storage Module (upper right-hand corner of Figure 11). The coils terminate in four tubes (two in each stack) and provide a total capacity of 2400 samples. When a tube is locked into place in the loader, the operator selects one of the coils via the 4-way diverter and turns on an air valve. Air pressure (15 to 20 psig) moves the entire string of 25 samples into the selected coil. The operator can load and enter documentary data for 2400 samples in two hours.

Delayed Neutron/Gamma Detectors. Figure 13 shows the two delayed neutron/Ge detector stations (D_1 and D_2) in the RAF. One station was retained from the pilot-scale facility; the second station is identical, but newly fabricated. However, the Ge(Li) detector of the pilot-scale facility was replaced by a pure Ge detector; all gamma detectors in the RAF are intrinsic (pure) Ge detectors, not doped with Li. The neutron detectors are enclosed

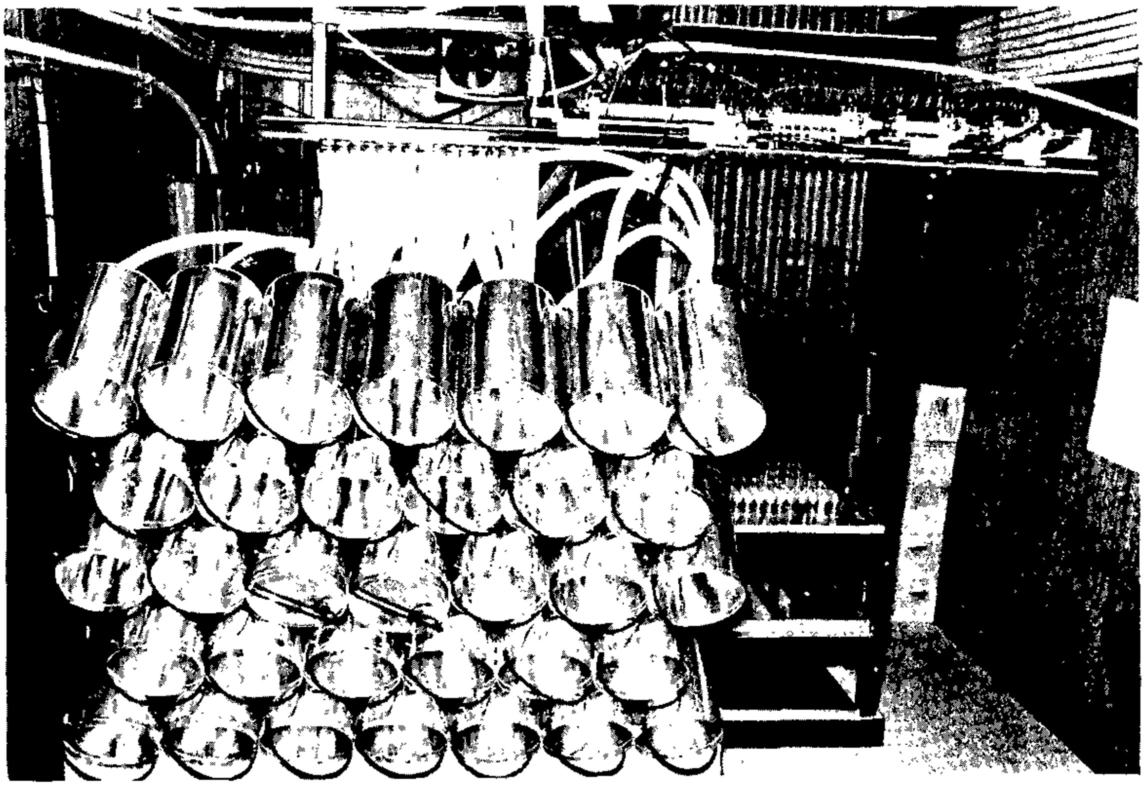


FIGURE 11. Automatic Sample Unloader

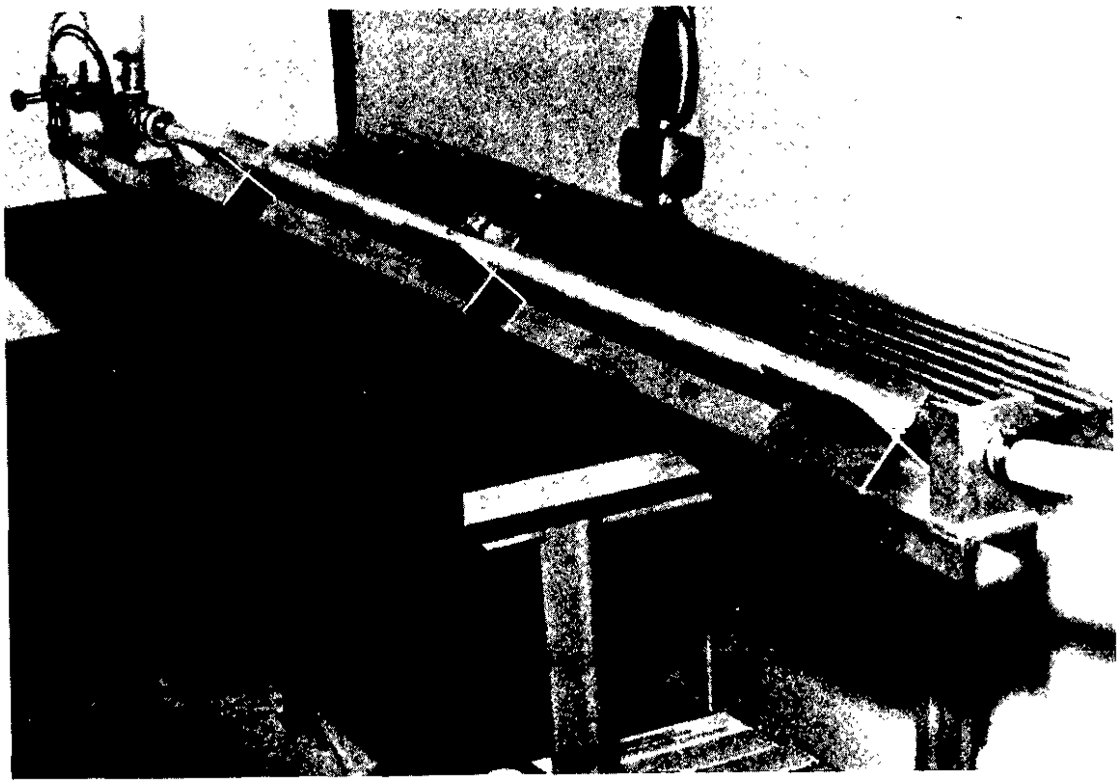


FIGURE 12. Station for Automatic Sample Loader

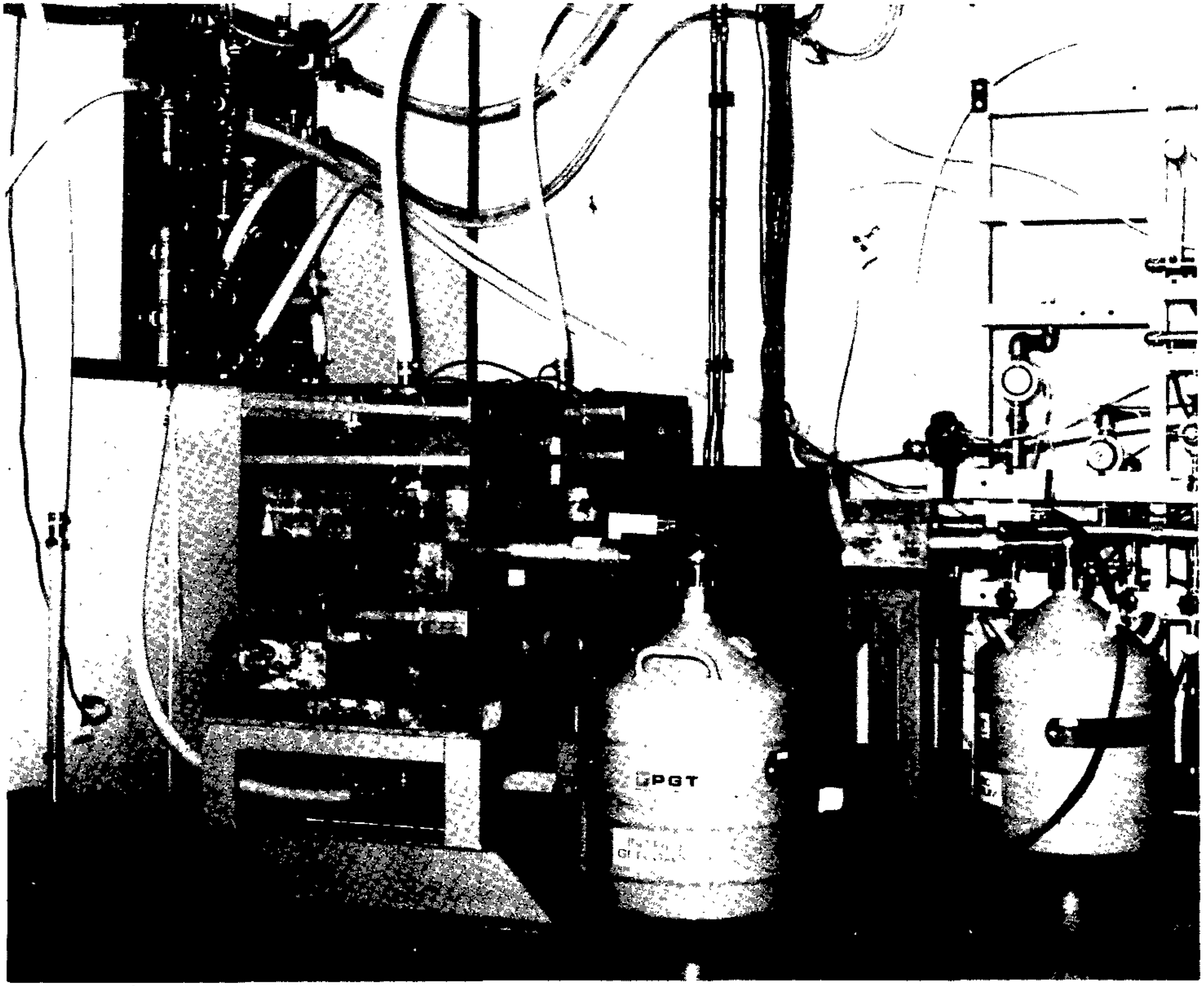


FIGURE 13. Delayed-Neutron/Gamma Detector Stations

by lead shielding. Each detector consists of an annulus of ten aluminum tubes containing boron trifluoride. The tubes are held in place in holes in a polyethylene moderator. The detectors have about 10% efficiency for measuring the delayed-neutron energy spectrum. A thin cadmium foil around each detector prevents neutrons emitted at one station from being detected at the other. Lead shielding reduces the gamma background in the detectors and collimates gamma rays from the samples to their respective Ge gamma detectors. The Ge detectors have efficiencies of about 10% and resolutions of about 2.1 keV at 1332 keV.

Variable-Geometry Gamma Detectors. Stations G_1 through G_4 are shown in Figure 14. Because samples vary widely in activity after irradiation, no single counting geometry is adequate. The drop tubes on top of each detector allow the computer to select the proper counting geometry (distance from sample to detector) for sediment samples containing activation products of intermediate (2 min to 24 hr) half-lives. Each drop tube has four small air cylinders positioned perpendicularly along the length of the tube; when extended, these cylinders serve as stops in the drop tube.

The computer selects the proper counting geometry by briefly scaling the detector output, starting at the top stop and working down if the count rate is less than a preset value. The lobes at the stop-points along the drop tube provide radial shielding. The air cylinder pistons penetrate the drop tube tangentially so as not to attenuate the gamma flux viewed by the Ge detector, which faces upward (Figure 15). Each of these intrinsic germanium detectors has an efficiency of about 8% and a resolution of about 1.8 keV at 1332 keV.

Fixed-Geometry Gamma Detectors. Stations G_5 through G_8 are shown in Figure 16. These stations are used to count the long-lived (>24 hr) gamma activity of sediment samples about seven days after irradiation. These stations are identical to Stations G_1 through G_4, except that drop tubes and stops have been eliminated. Only one counting geometry is available, that for Stations G_1 through G_4 with all stops retracted, so that the sample is close to the detector. Usually Stations G_5 through G_8 are used to count samples of low activity, while Stations G_1 through G_4 count samples of higher activity (stops inserted to increase sample-to-detector distance). However, Stations G_1 through G_4 can also count samples of low activity if all stops are retracted.

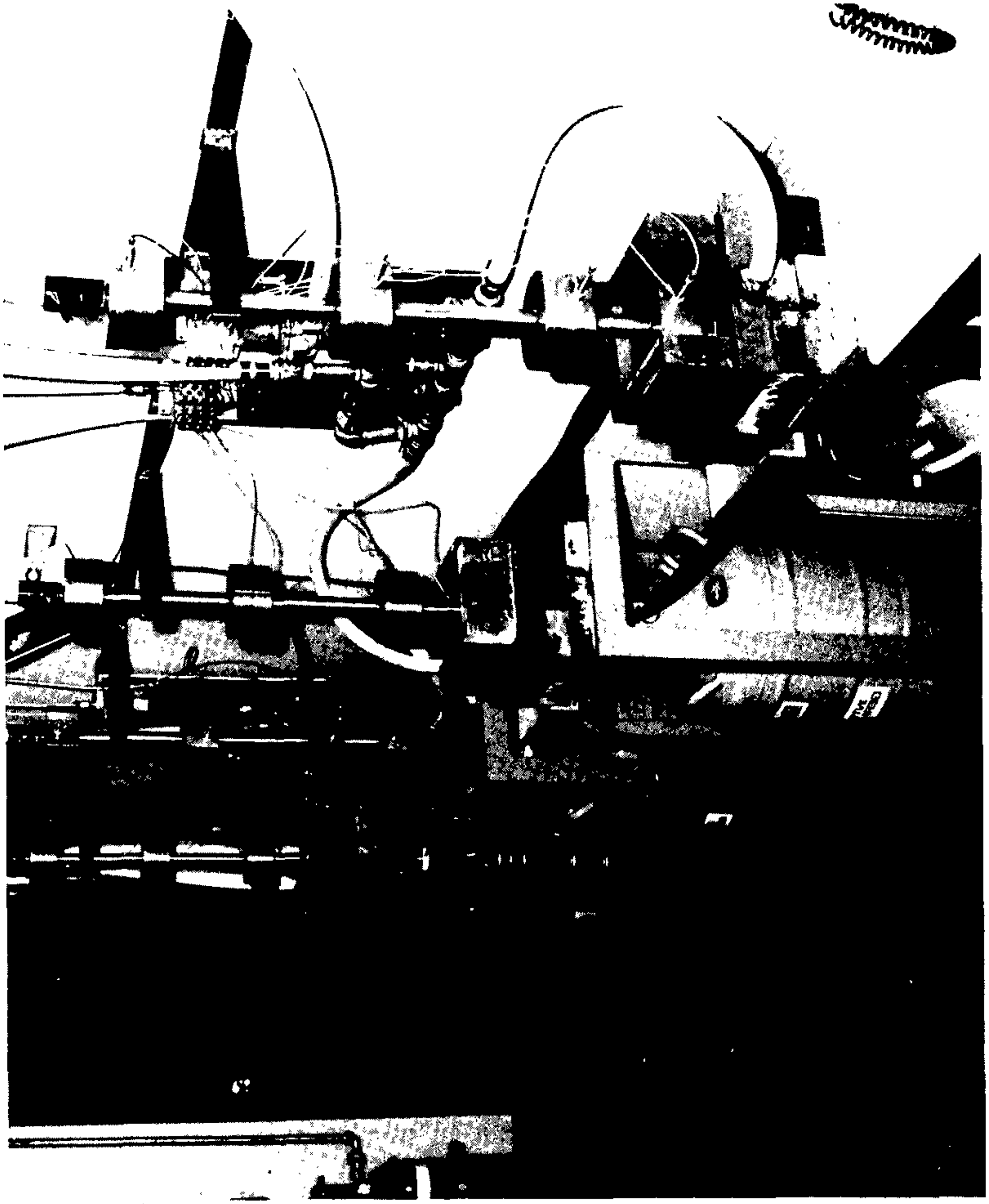


FIGURE 14. Variable-Geometry Gamma Detector Stations

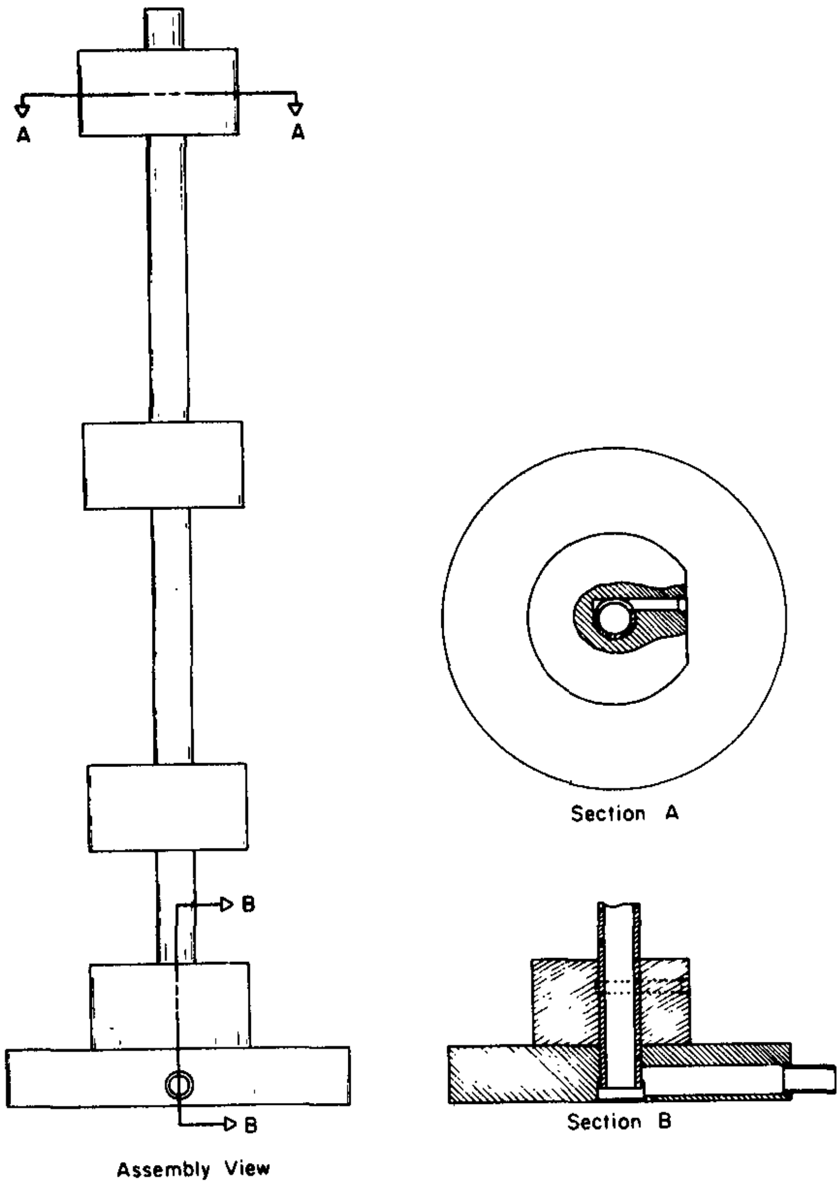


FIGURE 15. Diagram of a Variable-Geometry Gamma Detector Station

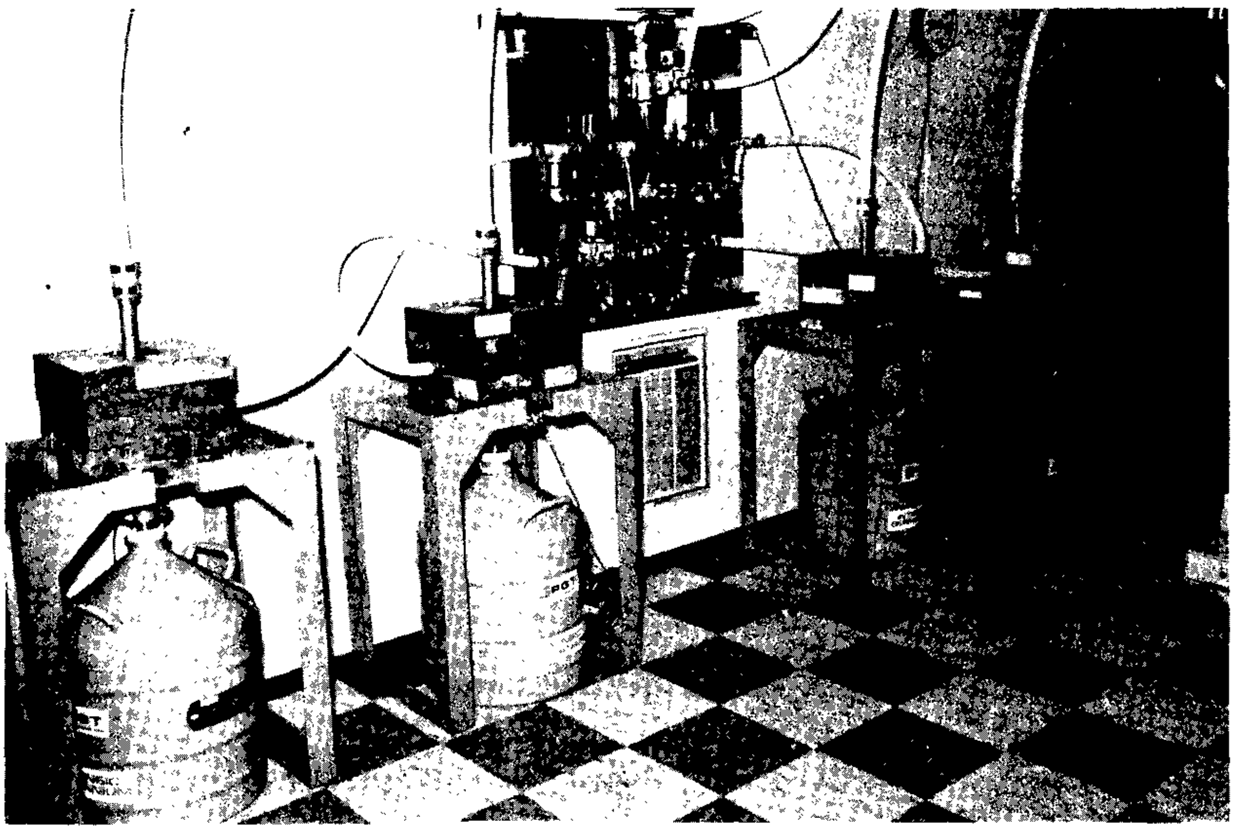


FIGURE 16. Fixed-Geometry Gamma Detector Stations

OPERATION OF THE REACTOR ACTIVATION FACILITY

Although the RAF can operate in several different modes, the number and types of samples and analytical requirements determine optimum use of the system. The RAF operates at maximum throughput when analyzing equal numbers of resin and sediment samples.

Table 1 outlines the analytical regimes in current use. Each sample receives a 2-second irradiation and 2-second combined gamma-neutron count to ensure that the sample can safely undergo further irradiation. This test is immediately followed by 10 cycles of 6-second irradiations and 6-second gamma-neutron counts. Uranium concentrations are determined from the neutron counts, and short-lived (<2 min) activation products are measured by the gamma counts. Following the recycle regime, resin samples are unloaded. Sediments receive an additional 400-second irradiation, followed by gamma counting after decay for 600 seconds (to measure intermediate-lived activation products) and 7 days (to measure long-lived activation products).

PERFORMANCE OF THE REACTOR ACTIVATION FACILITY

The throughput for all-resin runs averages about 38 samples/hr; all sediment runs average about 30 samples/hr. Steady-state throughput for mixed resin-sediment runs is about 20 resin samples and 16 sediment samples/hr. System throughput is governed by the number and length of cycles and by the sample turnover time.

Maintenance and overhead items (such as loading and unloading samples) and data retrieval limit production time to about 20 hours/day. Maximum RAF throughput for mixed sediment and resin runs is about 4000 samples per seven-day week assuming no system failures. In practice, 2500 to 3000 samples per week are normally activated.

For quality assurance, every 24th sample is a standard, and every 25th sample is a blank. Analytical results for uranium and other detectable elements are printed in real time for these samples as they are completed. These results are checked to ensure proper sample ordering and correct analyses. Table 2 shows typical results for a number of elements in one of the quality assurance standards as encountered in routine operation. Accuracies and precisions are quite good, considering that no comparision standards are used, and duplicates are prepared and packaged in production fashion with actual samples.

TABLE 1

Current RAF Analytical Regimes for NURE Samples

Sample Type	Irradiation Time, sec	Decay Time, sec	Neutron and Gamma Count Time, sec	Number of Cycles
Sediments	2	None	2	1
	6	None	6	10
	400	600	420	1
		7[a]	1320	1
Resins	2	None	2	1
	6	None	6	10

a. In days.

TABLE 2

Precision and Accuracy of the RAF System as Shown by Repeated Analyses of a Quality Assurance Sediment Standard

Element	Number of Determinations	Mean Value, ppm	Nominal Value, ppm	Coefficient of Variation, %
U	294	21.8	22.2	5.75
Th	295	115	125	15.2
Hf	296	146	156	18.1
Al	289	7,020	5,300	10.8
Ce	288	534	614	11.5
Fe	273	6,620	6,400	26.2
Mn	266	275	280	15.8
Na	278	150	119	36.3
Sc	297	3.41	3.96	26.8
Ti	271	12,400	12,900	20.6
V	282	33.7	34.7	16.9
Cl	101	22.2	20.0	62.4
La	276	279	301	17.5
Sm	280	47.7	49.6	38.2
Eu	175	2.74	2.24	35.4
Dy	275	27.3	26.7	38.0
Yb	294	15.9	18.2	21.3
Lu	253	2.55	2.9	13.3

ACKNOWLEDGMENTS

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No.	Period	SRL Doc. No.	DOE-GJO Doc. No.*
1	January-March 1975	DPST-75-138-1	GJBX-5(76)
2	April-June 1975	DPST-75-138-2	GJBX-6(76)
3	July-September 1975	DPST-75-138-3	GJBX-7(76)
4	October-December 1975	DPST-75-138-4	GJBX-8(76)
5	January-March 1976	DPST-76-138-1	GJBX-17(76)
6	April-June 1976	DPST-76-138-2	GJBX-27(76)
7	July-September 1976	DPST-76-138-3	GJBX-63(76)
8	October-December 1976	DPST-76-138-4	GJBX-6(77)
9	January-March 1977	DPST-77-138-1	GJBX-35(77)
10	April-June 1977	DPST-77-138-2	GJBX-55(77)
11	July-September 1977	DPST-77-138-3	GJBX-90(77)
12	October-December 1977	DPST-77-138-4	GJBX-37(78)
13	January-March 1978	DPST-78-138-1	GJBX-66(78)
14	April-September 1978	DPST-78-138-2	GJBX-13(79)
15	October 1978-March 1979	DPST-79-138-1	GJBX-86(79)
16	April-September 1979	DPST-79-138-2	(in process)

* DOE-GJO reports are available on microfiche from the Grand Junction Office, DOE, for $6.00. Prepaid orders should be sent to: Bendix Field Engineering Corporation, Technical Library, P. O. Box 1569, Grand Junction, CO 81501. Checks or money orders should be made out to Bendix Field Engineering Corporation, the operations contractor for DOE's Grand Junction Office.

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