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Shielded Analytical Instruments For Characterization of Highly Radioactive Materials

**J. L. Daniel
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June 1986

**Prepared for the U.S. Department of Energy
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SHIELDED ANALYTICAL INSTRUMENTS
FOR CHARACTERIZATION OF HIGHLY
RADIOACTIVE MATERIALS

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Scanning Electron Microscope (SEM)

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J. L. Green, R. G. Sullivan, and L. C. Todd

Specimen Preparation Hot Cell

J. E. Coleman, J. L. Green, H. E. Kissinger, H. E. Kjarmo,
and R. G. Sullivan

SUMMARY

The Pacific Northwest Laboratory has developed a unique capability for analysis of high level radioactive material. This capability is achieved with a group of shielded research-quality instruments that can provide a multi-dimensional analysis of the same radioactive specimen. This instrument group includes an inductively coupled plasma (SICP) atomic emission spectrometer, an x-ray diffractometer (SXR), and a scanning electron microscope (SEM), as well as a scanning Auger analyzer (AES) modified for analysis of low-level radioactive specimens. A shielded microprobe (SEMP) and radiochemistry analytical facilities are also available in nearby laboratories, and a high-resolution fully shielded Auger instrument (SAES) is being added.

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INTRODUCTION

Scanning electron microscopes have been contained and/or shielded at other laboratories for examination of radioactive materials.⁽¹⁻⁶⁾ At the Pacific Northwest Laboratory (PNL)^(a), that concept has been extended to provide a special group of analytical instruments having unique capabilities for studies of highly radioactive material. These PNL instruments are especially applicable to microstructural characterization and bulk composition analysis of nuclear waste forms, spent fuel, and nuclear waste packages proposed for long-term underground disposal. Because of the high radiation levels associated with these materials, specially shielded analytical instruments must be used. Presently included in this group are: 1) a highly versatile shielded inductively coupled argon plasma atomic emission spectrometer (SICP), 2) a specially designed shielded x-ray diffractometer (SXR), and 3) a high-resolution shielded scanning electron microscope (SSEM). In addition, a fully shielded electron microprobe (SEMP) is in a nearby laboratory, an Auger electron spectrometer (AES) that is interfaced to an open front hood is available for examination of low-radiation specimens, and a fully shielded high-resolution scanning Auger electron spectrometer (SAES) is in the final design stage. By utilizing the full capability of these instruments, R&D staff members can take advantage of this one-stop facility to obtain a multidimensional analysis of radioactive materials. For example, overall bulk elemental composition is readily determined quantitatively with the SICP; identification of crystalline phases is achieved with the SXR; surface morphology, near-surface composition, and phase identification are obtained with the SSEM; systematic analysis of microphase constituents is possible with the SEMP; and elemental and molecular information from the outer-most surface will be provided by the SAES.

The three primary shielded instruments have been developed at PNL under the direction and sponsorship of the Department of Energy's (DOE) Nuclear Waste Materials Characterization Center (MCC) and the Commercial Spent Fuel Management Program. The MCC's responsibilities include basic and applied studies and

(a) Operated for DOE by Battelle Memorial Institute under Contract DE-AC06-76RLO 1830.

testing of radioactive nuclear waste materials and the production and distribution of carefully characterized standard reference materials such as radioactive and doped waste glasses and spent fuel. The new instruments are being used to characterize the properties and composition of test specimens and to analyze and evaluate the materials designed for use as standards. In addition, the instruments are available to other programs on a cost-recovery basis.

Three of the instruments--the SICP, the SXRD, and the SSEM--are described in detail in this paper. The other instruments are described briefly. In addition, the central specimen preparation hot cell facility, which services all of these "stand-alone" instruments, is described along with the specimen distribution methods. A summary of the instruments presently installed and those that are planned is listed in Table 1.

TABLE 1. Summary of Shielded Analytical Instruments at PNL

Instrument	Shielding	Equipment	Specimen Input	Limitations
ICP ^(a)	Plasma chamber glove box lined with 12 mm of lead.	26-element direct reader that requires <10 mL of sample; sequential spectrometer for most other elements.	Solutions; automatic sample changer.	Element concentrations >10 µg/mL in solution.
XRD ^(b)	Lead specimen chamber (10-cm walls) in a lead-lined instrument cabinet.	High-resolution diffractometer with graphite monochromator, Soller slit, and external detector.	30-mm mounts, metallographic or powder, in special specimen holder; tungsten alloy transfer cask.	Solid specimens (monoliths or powders).
SEM ^(c)	10 cm of lead in a retractable "clamshell" enclosing the column and specimen chamber.	High resolution research SEM with EDS spectrometer.	Three options for varying levels of radiation; 1) tungsten alloy cask, 2) evacuated transfer chamber, and 3) standard.	Solid specimens; EDS analysis limited to low radiation level specimens and elements of atomic number >10.
EMP ^(d)	Tungsten alloy specimen chamber (equivalent to 18 cm of lead).	Three spectrometers including light-element option; automatic specimen scan and spectrum readout.	30-mm mounts in special specimen holder; tungsten alloy transfer cask.	Solid specimens with polished surfaces.
AES ^(e)	10 cm of lead in a retractable clamshell enclosing column and specimen chamber.	High-resolution scanning Auger.	Two optional methods: 1) tungsten alloy transfer cask and 2) standard.	Solid specimens.

(a) Inductively coupled plasma atomic emission spectrometer, model ARL 3580.

(b) X-ray diffractometer of PNL design based on vertical mounting of a Diano XRD-6 goniometer; automation by Cal Scientific model L-11.

(c) Scanning electron microscope, model JSM-35C with TN-2000 EDS system.

(d) Electron microprobe, model MAC-450 with upgraded electronics.

(e) Auger electron spectrometer, model PHI 660 system; scheduled for installation by December 1986.

FACILITY CONCEPT AND SAMPLE PREPARATION/TRANSFER

The PNL/MCC shielded analytical facility is designed around the use of stand-alone fully shielded research-quality instruments that are served from a central hot cell for radioactive sample preparation (Figure 1). The arrangement of the hot cell and analytical instruments is shown in Figure 2. Several advantages are offered by this stand-alone design. Three of these advantages are: 1) all the instruments can be used readily on both nonradioactive specimens or standards and on highly radioactive materials; 2) routine maintenance and instrument adjustments for optimum performance are facilitated by easy hands-on accessibility; and 3) one hot cell can serve several instruments from a minimum of access ports.

Radioactive sample preparation can be conducted in the central hot cell, which is specially fitted with equipment for specimen cutting, mounting, polishing, and sample dissolution (Figure 3). Another dedicated metallography hot cell facility is also available for specialized metallographic sample preparation. Radioactive specimens prepared elsewhere are temporarily placed in the central hot cell and subsequently transferred to the shielded instruments. Transfers of samples to SXRD, SSEM, and SEMP are accomplished using a

FIGURE 1. A Central Sample Preparation Facility Supports All the Shielded Analytical Instruments

FIGURE 2. Configuration of the Sample Preparation Hot Cell and Analytical Instruments

FIGURE 3. Arrangement of Sample Preparation Equipment in the Hot Cell Facility

tungsten alloy cask that interfaces with the hot cell (Figure 4) as well as with each of these instruments. The use of similar casks and interfacing allows analysis of the same specimen with any or all of these instruments by redistribution via the hot cell. Radioactive solutions are transferred from the hot cell to the SICP with small lead-shielded containers.

The advantages of this philosophy of stand-alone instruments and a common specimen-preparation facility have been demonstrated during 15 years of SEMP use. This concept has resulted in a comprehensive one-stop facility for characterization of radioactive specimens, while retaining the flexibility to easily analyze nonradioactive specimens and perform necessary instrument maintenance.

FIGURE 4. Hot Cell Specimen Transfer Arrangement

SHIELDED INDUCTIVELY COUPLED PLASMA INSTRUMENT (SICP)

The MCC chose an inductively coupled argon plasma atomic emission spectrometer (ICP) to provide bulk composition analysis of waste forms and test solutions produced from testing waste forms. The SICP is a fully automated direct-reading instrument applicable to radioactive liquid specimens generated in leach testing of nuclear waste material immersed in water, brine, or other solution. Solid specimens of nuclear waste or other radioactive materials also can be analyzed after dissolution by high-temperature fusion methods. In conjunction with the analytical radiochemistry capability available in nearby laboratories, the SICP allows for complete compositional analysis of radioactive solutions.

A design team initially identified six key design features (Lautensleger and Hara 1984) that were subsequently incorporated into the instrument and the facility: 1) An ICP source stand located in a shielded glove box to control radioactivity; 2) a direct reader capable of analyzing up to 26 elements simultaneously; 3) a sequential analyzer module for analysis of additional elements as needed; 4) a simple, compact autosampler and software for remote control; 5) torch and sample aspiration systems that are easily replaced and maintained without affecting the optical alignment; and 6) electronic and gas controls located outside of the glovebox.

A modified Bausch and Lomb ARL model 3560 ICP with these design features was installed at PNL in 1983. The instrument was subsequently upgraded to a model 3580 ICP with the installation of a sequential analyzer. This sequential analyzer utilizes the same primary optical path as the direct reader, with a system of mirrors used to direct a portion of the light beam upward into the sequential analyzer. The sequential module with supporting control software allows detection and analysis for any element having a spectrum line of wavelength above 200 nanometers. This addition greatly enhances the analytical capability of the SICP instrument.

The glove box in which the source stand is mounted provides two functions: 1) containment of the radionuclides, of which plutonium has the most stringent requirements, and 2) shielding of radiation. Containment specifications were

based on design requirements for control of plutonium (ANSI 1981). Total dose-rate calculations based on the anticipated gamma-emitting radionuclides that would be present predicted that 12 mm of lead would provide adequate shielding to limit the operator dose rate to as low as reasonably achievable (ALARA).

A photograph of the installed SICP and glovebox is shown in Figure 5. Figure 6 is a schematic of the instrument and glovebox identifying the key features. The arrangement of the autosampler and source stand inside the glovebox is shown in Figure 7. The most critical feature of the complete ICP installation was the interface between the spectrometer and the lead-shielded glovebox, which weighs over 1-1/2 tons. This interface, shown in Figure 8, provides the optical path from the source stand to spectrometer. The spectrometer, glovebox, source stand, and interfacing plate are all bolted together. The interface plate with the sealed quartz window and Neoprene gasket is an integral part of the radionuclide containment; the gasket also provides some flexibility to reduce stress on the optical bench of the spectrometer.

The SICP is particularly useful in several areas of analysis. Compositional analysis of fabricated waste forms can be conducted on solutions of the materials. Dilute solutions that result from tests of glass durability in various aqueous media representing waste repository conditions also can be analyzed; the SICP is ideal for analysis of these radioactive solutions. In addition, this instrument can provide analyses of vitrification feed material and off-gas condensate samples taken during ceramic melting of waste material to produce a glass waste form. Table 2 shows an example of an analysis of a solid radioactive glass; ATM-9 is an Approved Testing Material produced by the MCC. Also included in the table is the radiochemistry analysis of the radioactive dopants present in the ATM.

FIGURE 5. Inductively Coupled Plasma Instrument Interfaced with the Glove Box

FIGURE 6. Schematic of the Shielded ICP Instrument with Shielded Glove Box Attached

FIGURE 7. Schematic of the Shielded ICP Sample Excitation Source with Necessary Components

FIGURE 8. Schematic of the Optical Path Interface through the Glove Box Wall

TABLE 2 Analyzed Composition of ATM-9 Glass

<u>Stable Oxides</u> ^(a)	<u>wt.%</u>	<u>Standard Deviation</u>
Al ₂ O ₃	4.944	0.125
B ₂ O ₃	7.149	0.231
BaO	0.118	0.008
CaO	1.616	0.028
Cs ₂ O	NA	---
Fe ₂ O ₃	11.18	0.56
K ₂ O	NA	---
Li ₂ O	4.807	0.312
MgO	0.768	0.035
MnO ₂	2.610	0.136
MoO ₃	0.093	0.028
Na ₂ O	11.02	0.40
Nd ₂ O ₃	0.344	0.497
NiO	NA	---
P ₂ O ₅	1.57	(b)
SiO ₂	50.43	2.42
TiO ₂	0.240	0.035
ZnO	0.075	0.010
ZrO ₂	0.847	0.017
<u>Radioactive Oxides</u> ^(c)		
Am ₂ O ₃	5.64E-4	3.61E-6
NpO ₂	1.29E-2	4.50E-4
PuO ₂	5.74E-3	2.85E-3
Tc ₂ O ₇	4.12E-3	2.28E-4
UO ₂	<u>1.96</u>	0.07
TOTAL	99.794	

(a) Based on SICP analysis of four specimens taken from three bars. Standard deviations are for an individual analysis.

(b) Single analysis, no standard deviation.

(c) Analyzed by radiochemistry methods.

NA = Not analyzed.

SHIELDED X-RAY DIFFRACTOMETER (SXR)

X-ray diffraction is an important method for determining the extent of devitrification of nuclear waste glass; for that reason the SXR was the first shielded instrument in this group to be applied to analysis of high-level radioactive samples. The PNL SXR was designed by a team of PNL design engineers and researchers and includes a composite of the best design features of several earlier instruments. The team identified six key features to be incorporated in the instrument: 1) a rugged monolithic design to avoid the necessity for frequent and difficult alignment; 2) all routinely adjustable or working parts accessible from outside the radiation field; 3) commercial off-the-shelf components for use wherever possible; 4) sufficient shielding to limit the radiation exposure of the operator to a safe level; 5) a simple and safe sample transfer mechanism; and 6) performance and resolution equivalent to conventional unshielded XRD instruments.

The instrument is a unique design in which the goniometer, x-ray source, and detectors operate in a vertical plane in order to provide a convenient orientation for horizontal input of specimens from a shielded transfer cask. The design utilizes a rigid fixed optical system for the diffracted x-ray beam (Figure 9). The beam path is a slot that has been cut in a massive stainless steel bar in which a receiving slit and Soller slit assembly are embedded. A curved pyrolytic graphite monochromator separates the diffracted characteristic radiation from the background radiation. This diffracting arrangement also helps reduce the effect of the decay radiation on the detected diffraction signal. Graphite, chosen as a monochromator material for its good diffracting intensity and for the absence of interfering fluorescence x-rays, is utilized in a manner widely used in conventional XRD instruments.

An interior view of the actual instrument is shown in Figure 10. The x-ray tube is mounted on a movable arm to provide the necessary angular motion. The sample rotates at half the angular velocity of the x-ray tube to provide the proper angular alignment. These motions are provided by a modified Diano (Rausch & Lomb) goniometer mounted on one face of the diffractometer cabinet as

FIGURE 9. Schematic of the X-Ray Tube, Sample Chamber, and Signal Detection System of the SXRD

FIGURE 10. View of the Interior of the SXRD Unit with Shielded Framework

shown in Figure 11. This goniometer, which would be mounted on a horizontal table in a conventional instrument, is mounted vertically with the theta and 2-theta angular-motion drive shafts extended through the side of the goniometer case and into the shielded frame. These extended shafts do not support any of the weight of the unit but only provide angular position motion. The x-ray tube mount and holder is carried on a large double-row ball bearing assembly and is counterbalanced to provide almost effortless rotation movement. The x-ray tube assembly is mounted over the sample chamber, which is a 25-cm lead cylinder into which a 12-mm slot has been cut to allow the x-ray beam to strike the specimen. The x-ray signal is detected with a scintillation detector. This complete assembly is enclosed in a steel-lined containment frame (Figure 10) with the goniometer mounted on one face and a cask interface for horizontal sample transfer mounted on the opposite face (Figure 12). The frame is designed to support up to 10 cm of lead shielding, but the lead sample chamber alone has provided sufficient shielding.

FIGURE 11. Shielded X-Ray Diffraction Instrument Operating Controls and Goniometer

FIGURE 12. Shielded XRD with Transfer Cask in Position for Specimen Transfer

The specimens are prepared in the previously described hot cell using remote handling techniques. Specimens may be in the form of polished metallographic surfaces, fracture fragments, or powders. The specimens are mounted in a specimen holder, coated with a spray Krylon, and then sealed with a Mylar window. These mounts are then inserted into the specimen holder and transferred from the hot cell into a tungsten transfer cask through a specially designed port on the rear face of the hot cell. The specimen holder is manipulated with a push rod through the rear end of the cask. Special guide pins hold the specimen holder firmly in place to provide correct alignment with the x-ray beam path when the transfer cask is interfaced to the SXRD and the specimen is inserted.

Figure 13 compares quartz crystal x-ray peak resolutions produced with an unshielded x-ray diffractometer and with PNL's shielded x-ray diffractometer to the ideal resolution. As can be seen by comparing the separation of the diffraction peaks in each example, the resolution of the SXRD is very good.

A test for x-ray diffractometer alignment:

The resolution of the three partially overlapping quartz lines between 67 and 69°

FIGURE 13. Examples of Ideal Peak Resolution Compared to the Resolution Attained with a Standard Diffractometer and the MCC Shielded Diffractometer

This instrument is being used to study high-level doped waste glass, several types of spent reactor fuel, and other special projects involving radioactive samples. Figure 14 is an example of test results produced when analyzing spent fuel.

a) UO_2

b) U_3O_8

FIGURE 14. Example of XRD Data from Analysis of Irradiated Uranium Oxide Fuel with the Shielded XRD

A test for x-ray diffractometer alignment:

The resolution of the three partially overlapping quartz lines between 67 and 69°

FIGURE 13. Examples of Ideal Peak Resolution Compared to the Resolution Attained with a Standard Diffractometer and the MCC Shielded Diffractometer

This instrument is being used to study high-level doped waste glass, several types of spent reactor fuel, and other special projects involving radioactive samples. Figure 14 is an example of test results produced when analyzing spent fuel.

FIGURE 14. Example of XRD Data from Analysis of Irradiated Uranium Oxide Fuel with the Shielded XRD

SHIELDED SCANNING ELECTRON MICROSCOPE (SSEM)

Scanning electron microscopy methods are well suited to examination and analysis of surface microstructures of radioactive materials. A research-grade SEM is capable of imaging surfaces from low magnification, approximately 20x, to very high magnifications, approximately 100,000x, rapidly and with minimal instrumental adjustments. This capability provides a method by which large or macro features can be identified in situ and then correlated with any fine microstructures present. Samples must be in solid form; they may be polished sections or as-received pieces such as fractures or corrosion products. SSEM analysis can provide unique microstructural information as well as a method of confirming XRD results by locating and identifying crystallites in a glass matrix. When an SEM is equipped with an energy dispersive x-ray fluorescence spectrometer (EDS), elemental information can be acquired from a specimen. This additional information is useful when identifying phases and areas of elemental segregation throughout a surface.

Six special design features are incorporated into this installation:

- 1) an exterior enclosure ("clamshell") type of radiation shielding with 10 cm of lead shielding to reduce operator exposure;
- 2) a vacuum column and specimen chamber separated from the control console;
- 3) specimen transfer from a tungsten alloy cask into the specimen chamber;
- 4) motorized x- and y-axis stage controls and remotely controlled stage tilt, rotation, and z-axis motion;
- 5) radionuclide containment during specimen transfer and examination; and
- 6) a back-up specimen retrieval system.

The retractable clamshell shielding method is used to achieve a free-standing independently shielded instrument (Figure 15). This clamshell is a two-piece rectangular structure made of 10-cm-thick lead mounted on a stainless steel frame. Each clamshell half is mounted on a track with a guide rod attached for alignment. Each section can be retracted to an open position to allow direct access to the SEM column, or it can be fully closed to provide total shielding around the SEM column.

A Japan Electronic Optic Laboratory (JEOL) model JSM-35C was easily modified to meet the required facility design. Figure 16 is a view of the SEM

FIGURE 15. Schematic Showing the Arrangement of the SSEM and Retractable Clamshell in the Open and Closed Positions

FIGURE 16. SEM Before Modification and Shielding

before any modifications. In addition to the separability of the column from the operating console, the spare access port located on the side of the sample chamber is an essential design feature of the SEM. This port (shown with the cover plate removed in Figure 17) is used in mounting an auxiliary specimen transfer tube on the column, and allows a horizontal transfer of a radioactive specimen from a transfer cask into the SEM sample chamber. The transfer is made using a push rod to move the sample from the transfer cask along a set of guides into the sample chamber and onto the sample stage dovetails. Figure 18 is a front view of the column with modifications and remote controls attached in the open-clamshell position. A pneumatically operated gate valve is interfaced to the sample transfer port located on the left side of the column. The sample chamber front door contains the motorized x and y drives as well as the remote control mechanisms (cables) that control the stage tilt and z motion. The spring-mounted table has four hydraulic clamps (one is visible in the lower right hand side of column) to hold the table rigidly in place during the sample

FIGURE 17. Side View of the SSEM Showing the Sample Chamber Port Used for Radioactive Sample Transfer

FIGURE 18. Front View of the Modified SSEM Column; Clamshell in the Open Position

transfer process. The interior of the clamshell and the sample transfer enclosure have a negative air flow system to prevent the escape of any airborne particles. This air flow passes through a high-efficiency particulate air (HEPA) filter and into the radioactive ventilation system for the building. Figure 19 is a view of the clamshell in the closed operating position with the sample transfer cask mounted to the shielding interface. The window located on the upper right side of the clamshell provides (through a system of mirrors) a view of the stage micrometer setting and the sample holder position on the stage.

The clamshell approach to shielding provides two additional features, both of which result from the fact that the shielding is easily retractable. First, with the open clamshell the SEM routine maintenance and filament changes continue to be a normal hands-on procedure requiring no remote operations. Optimum instrument performance is easier to maintain with this method. Second, the instrument can be operated without the shielding. Thus, nonradioactive or low-radiation specimens may be examined conveniently, with specimen transfer into

FIGURE 19. SSEM with Clamshell Closed and Sample Transfer Cask Interfaced with the Shielding

the specimen stage conducted by the standard method through an **airlock** on the specimen chamber door. Alternatively, a small, portable controlled-atmosphere transfer chamber can be used to transfer low-radiation specimens and actinide-doped material. This chamber interfaces directly with the specimen chamber **airlock** as shown in Figure 20. This system provides a safe method of transferring low-level radioactive samples into the **SSEM**. It can also be used to transfer specimens that require a special environment.

A **Tracor** Northern X-Ray Model **TX-10/144 669R** energy dispersive spectrometer system (EDS), including a **TN-2000** analyzer, is interfaced with the **SSEM** column. This spectrometer allows elemental analysis for elements of atomic numbers **>10** in the near-surface region of the specimen. Micro segregation and phases can be analyzed for elemental composition with this added capability unless the radiation **level** of the specimen interferes with elemental spectra.

FIGURE 20. Controlled Environment Transfer Cell Interfaced with the **SSEM** Sample Chamber Airlock

This shielded SEM design has proved to be very practical and versatile. Figure 21 is an example of high-level radioactive material characterization; surface morphology comparisons of spent fuel are shown. The fuel is an H. B. Robinson fuel that was discharged from Unit 2 in May 1974. The fuel burnup averaged approximately 28,000 MWD/ton.

FIGURE 21. Micrographs of PWR Spent Fuel from H. B. Robinson Reactor. After removal from cladding and exposure to air for: a and b, 1 year; c and d, 10 years.

OTHER INSTRUMENTS FOR RADIOACTIVE SAMPLE ANALYSIS

Two additional instruments for the analysis of radioactive samples are the shielded electron microprobe (SEMP) and the shielded Auger electron spectrometer (SAES). These instruments are discussed briefly in this section.

SHIELDED ELECTRON BEAM MICROPROBE (SEMP)

Electron beam instruments such as the SSEM can be equipped with EDS for use in obtaining elemental distribution information in areas of localized segregation, precipitates, and crystals. However, the EDS detection system on the SSEM is also sensitive to the decay radiation emitted from a radioactive specimen, and an alternative method for elemental distribution analysis is needed for analysis of highly radioactive materials. This method is provided by the shielded electron microprobe (SEMP). The SEMP, a MAC-450 microprobe with upgraded electronics (Figure 22), is located in the same facility as the other shielded analytical instruments and has demonstrated reliable and dependable analytical results for over 15 years. Specimens need to be solids with polished surfaces for quantitative SEMP analysis; elements down to atomic number five can be detected. The SEMP utilizes the same type of cask transfer system (Figure 23) used for the SSEM and SXRD, so the same specimen can be examined by each of these instruments. The SEMP has been used extensively to study the radial profile of spent nuclear fuel pellets. The use of the SEMP to study high-level radioactive waste can provide valuable elemental data that can be associated with the SSEM microstructural data and SXRD crystallinity data.

SHIELDED AUGER ELECTRON SPECTROMETER (SAES)

Another important materials characterization instrument is the high-resolution scanning Auger electron spectrometer (AES). Presently a nonshielded instrument, a Physical Sciences Model 550, has been interfaced to an open-front, negative-airflow hood to allow analysis of samples with low levels of radiation or actinide-doped specimens. A new high-resolution fully shielded AES is in the final stages of design. A set of design features has been

FIGURE 22. SEMP with Upgraded Electronic Control Console

FIGURE 23. Schematic Diagram of SEMP with Transfer Cask Interface

selected to define the requirements of the SAES system. These design requirements are: 1) a control console separate from the specimen chamber; 2) a retractable clamshell shielding system; 3) a reliable remote specimen transfer system for radioactive specimens; 4) a standard specimen input system for convenient use of the instrument for study of nonradioactive specimens; 5) a remote control option for all system functions and valves; 6) remote visual inspection of key instrument functions by TV. monitor; 7) an auxiliary chamber for special specimen preparation, such as ion etching or fracturing; and 8) a backup specimen retrieval system.

The instrument to be installed is a Physical Science Model 660. The fully shielded system is scheduled for completion by December 1986.

CONCLUSION

This group of specially shielded high-resolution analytical instruments has been in continuous use for more than 2 years. Several radioactive specimen types have been examined and analyzed, ranging from mildly radioactive actinide-doped materials to spent nuclear fuel and reactor structural components. The versatile stand-alone design has proven to be a valuable asset for all instruments, allowing convenient use of the instruments on both highly radioactive and nonradioactive materials and resulting in a higher use factor for all facilities. No radioactive contamination sufficient to prevent complete cleanup and subsequent use as a cold instrument has occurred with the SSEM and SXRD. The glovebox-mounted plasma stand of the SICP is maintained as an alpha-controlled system, still allowing easy application to nonradioactive specimens. The ongoing extension of the same operational approach to the shielded Auger instruments has required additional care in design, with special attention paid to the demands of a high-vacuum system.

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