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N.M. Edelstein, D.K. Shuh, and J.B. Bucher

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Hazards Analysis for the E.O. Lawrence Berkeley National Laboratory X-Ray Absorption Experiments to be Performed at Stanford Synchrotron Radiation Laboratory

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1.0 OVERVIEW OF EXPERIMENT

The objective of this experiment is to determine the oxidation state(s) of neptunium (Np) in mouse skeleton and in soft tissue by X-ray Absorption Near Edge Structure (XANES). If Np is present in sufficient concentration, X-ray Absorption Fine Structure (XAFS) data will be obtained in order to further identify the Np species present. These data will be crucial in understanding the metabolic pathway of Np in mammals which will help in the design of reagents which can eliminate Np from mammals in the event of accidental exposure. It is proposed to run these experiments at the Stanford Synchrotron Radiation Laboratory (SSRL). This laboratory is a DOE national user facility located at the Stanford Linear Accelerator Center (SLAC).

Neptunium is a transuranium element with the following nuclear characteristics:

$$^{237}Np - t_{1/2} = 2.14x10^{6} \text{ years} \quad 7.05x10^{-4} \text{ Ci/gram.}$$

The ^{237}Np nucleus decays by the emission of an alpha particle and this particle emission is the principal hazard in handling Np samples. This hazard is mitigated by physical containment of the sample which stops the alpha particles within the containment.

The total amount of Np material that will be shipped to and be at SSRL at any one time will be **less than 1 gram.** This limit on the amount of Np will ensure that SLAC remains a low hazard, non-nuclear facility.

The Np samples will be solids or Np ions in aqueous solution. The total volume of the solution samples will be less than 0.5 ml. The solid or aqueous solution neptunium samples will contain approximately **5 mg** of Np per sample with an **absolute maximum limit of 10 mg per sample. All sample preparation will be done at LBNL.** The solution samples will be loaded in a cell containing a plastic spacer with a cutout of dimensions 2 x 20 x 3 mm. Two polyethylene windows will be sealed to the spacer to contain the solid or solution sample. Solution samples may also be loaded into primary sample cells consisting of: a) thermally sealed 0.25 ml polyethylene snap cap centrifuge vials, b) thermally sealed 0.5 ml polyethylene transfer pipette bulbs, c) 0.5 ml polyethylene screw cap vials in which the screw caps have been permanently immobilized with either heat shrink tubing or adhesive tape. These experimental arrangements have been used previously with solutions of uranium and no problems were found. For powdered, solid Np samples the Np solid will be diluted with an inert organic solid (*e.g.*, polystyrene (bio-beads)) or an inert inorganic solid (*e.g.*, boron nitride). The solid samples in cement matrices will be loaded into a polystyrene cuvette, capped, and taped to ensure that the cap will not come free. The loaded sample cell described above will be placed in a polyethylene (PE) bag which will be **heat sealed.** A second PE bag will be placed around the sample cell and the primary PE bag and will be **heat sealed.** Samples prepared this way will then be loaded into the sample containment cell for use on beamline 4-1 or 4-3.

The Np samples will be shipped to SSRL/SLAC OHP. SLAC OHP will inventory the samples and swipe the containers holding the triply contained samples, and then bring them to the SSRL Actinide trailer located outside building 131. The Np samples not in use at beamline will be stored in a 55 gallon drum in the locked trailer.

The following table gives the radioactivity for typical quantities of ^{237}Np:

Mass ^{237}Np (mg)	Radioactivity (μCi)
1	0.705
5	3.5
10	7.05
100	70.5
200	141.0
500	352.5

The QA counting records from the samples, as measured at LBNL, will be provided to SSRL and SLAC OHP prior to the arrival of the samples at SLAC OHP. A template of such a form is in Appendix 9.2. All samples will be prepared, sealed and inspected at LBNL and will be shipped to SLAC/SSRL in compliance with DOT regulations by LBNL EH&S shipping. All samples at SSRL will remain packaged as described above and will be handled and run under ambient conditions. After the runs are over they will be packaged and shipped back to LBNL by SLAC EH&S.

In addition, strict monitoring of the storage and experimental areas will be performed in accordance with SLAC/OHP radiation protection procedures to ensure against the release of contamination.

2.0 NON-NUCLEAR, LOW HAZARD CLASSIFICATION

2.1 Background

DOE Order 5481.1B, "Safety Analysis and Review System," states that hazards of a particular facility should be classified into one of the following three classes: low, moderate, or high hazard. Hazard classification is a measure of the inherent potential for materials or energy sources to cause harm to the worker, the public, or the environment.

DOE Order 5480.5, "Safety of Nuclear Facilities," defines a nuclear facility as "a facility whose operations involve radioactive materials in such a form and quantity that a significant hazard potentially exists to the employees or to the general public." Accelerators and their operations are not included as nuclear facilities. SLAC and SSRL are accelerator facilities and are not nuclear facilities.

2.2 Hazard Classification of XAFS Experiments, (per standard 1027-92)

The DOE has issued DOE-Standard-1027-92, "Guidance on Preliminary Hazard Classification and Accident Analysis Techniques for Compliance with DOE Order 5480.23, Nuclear Safety Analysis Reports," dated October, 1992. Hazard Category 3 under DOE Order 5480.23 is equivalent to a "low hazard" classification under DOE Order 5481.1B. Hazard Category 3 and "low hazard" are therefore synonymous, with the choice of terminology dictated by the DOE order to which they refer. The quantities presented in Section I for Np-237 are well below the threshold quantities for the Hazard Category 3. Operations or facilities with quantities below the threshold values may be categorized as "non-nuclear" and are potentially considered hazards generally accepted by the public (below a "low-hazard" classification) because the potential impact on people not involved with the experiment is low, along with a negligible chance of actually receiving that impact. According to these standards, this is a "low-hazard," "non-nuclear" experiment.

3.0 DETAILS OF NEPTUNIUM SPECIATION EXPERIMENTS

3.1 Samples, composition, and characteristics

The samples will be prepared in the Actinide Chemistry Group laboratories at the E. O. Lawrence Berkeley National Laboratory with the isotope ^{237}Np. The Np samples will be solids or Np ions in aqueous solution. The total volume of the solution samples will be less than 0.5 ml. The solid or aqueous solution neptunium samples will contain approximately 5 mg of Np per sample with an absolute maximum limit of 10 mg per sample. The solution samples will be loaded in a cell containing a plastic spacer with a cutout of dimensions 2 x 20 x 3 mm. Two polyethylene windows will be sealed to the spacer to contain the solid or solution sample. Solution samples may also be loaded into primary sample cells consisting of: a) thermally sealed 0.25 ml polyethylene snap cap centrifuge vials, b) thermally sealed 0.5 ml polyethylene transfer pipette bulbs, c) 0.5 ml polyethylene screw cap vials in which the screw caps have been permanently immobilized with either heat shrink tubing or adhesive tape. For powdered, solid Np samples the Np solid will be diluted with an inert organic solid such as polystyrene (bio-beads) or an inert inorganic solid such as boron nitride. The solid samples in cement matrices will be loaded into a polystyrene cuvette, capped, and taped to ensure that the cap will not come free. The loaded sample cell described above will be placed in a polyethylene bag which will be **heat sealed** and acts as **secondary** containment. A second polyethylene bag will be placed around the sample cell and the first polyethylene bag and **heat sealed.** This second polyethylene bag provides **tertiary** containment. Samples prepared this way will then be loaded into the sample containment cell (quaternary containment) for use on beamline 4-1 or 4-3.

3.2 Calculation of sample activity

The isotope ^{237}Np has a half-life of $2.14x10^6$ years and a specific activity of $7.05x10^{-4}$ Ci/gram. A 10 mg ^{237}Np sample then has an activity of

$$10x10^{-3}x7.05x10^{-4}\ Ci/gram = 7.05x10^{-6}\ Ci\ or\ 7.05\ \mu Ci$$

3.3 Description of sample cell

The samples will be loaded in a cell containing a polyethylene spacer with a cutout of dimensions 2 x 20 x 3 mm. Two polyethylene windows will be sealed to the spacer to contain the solid or solution sample. Solution samples may also be loaded into primary sample cells consisting of: a) thermally sealed 0.25 ml polyethylene snap cap centrifuge vials, b) thermally sealed 0.5 ml polyethylene transfer pipette bulbs, c) 0.5 ml polyethylene screw cap vials in which the screw caps have been permanently immobilized with either heat shrink tubing or adhesive tape. For powdered, solid Np samples the Np solid will be diluted with an inert organic solid such as polystyrene (bio-beads) or an inert inorganic solid such as boron nitride. The solid samples in cement matrices will be loaded into a polystyrene cuvette, capped, and taped to ensure that the cap will not come free.

The loaded sample cell will be placed in a PE bag which will be heat sealed (secondary containment). A second PE bag will be placed around the sample cell and the first PE bag and heat sealed (tertiary containment).

A schematic diagram of the sample cell based on the polyethylene space and barriers are shown in Fig. 1.

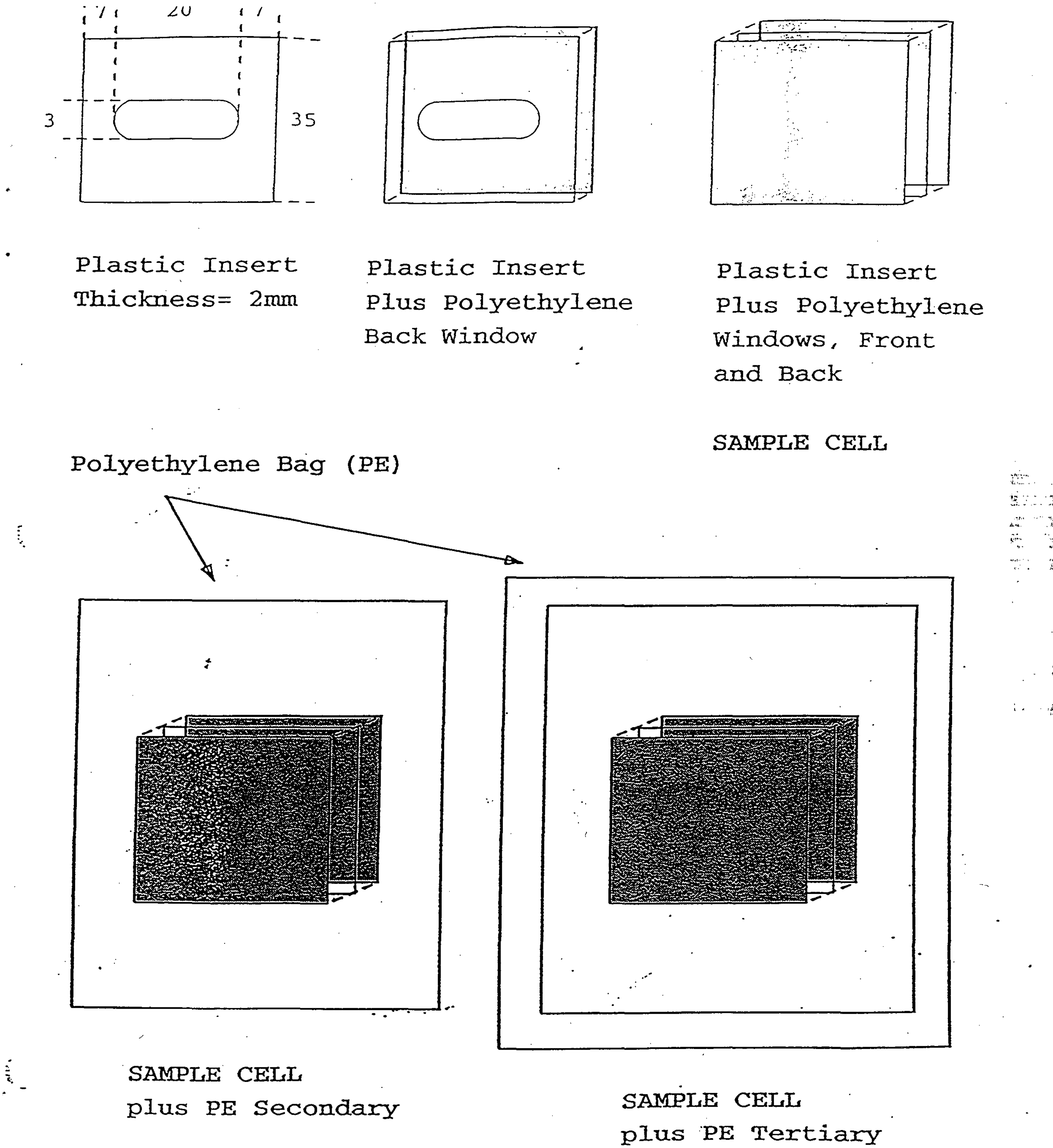

Figure 1. Schematic of Sample Cell Containment
(dimensions in mm, not to scale)

3.4 Description of the quaternary containment cell

The samples cells described above in Section 3.3 will be mounted on a slide mount which fits into fixed grooves in the quaternary sample containment cell. Up to 10 samples, each containing approximately 5 - 10 mg of ^{237}Np can be mounted with tape on this slide mount. The slide mount will be placed into the quaternary sample containment cell and this cell will be sealed with a gasket seal. This containment cell will be mounted on a support on the table in the hutch which contains vertical rails on which this cell will slide up and down. A linear actuator system will be used to lift the sample containment cell and to vertically align the samples contained inside with the x-ray beam. A containment cell containing only one Np sample and fixed in position will be prepared for the reference compartment. A schematic diagram of the quaternary sample containment cell is shown in Fig. 2. These loading operations of the Np samples into the quaternary containment cell will be performed in the hood in the storage/loading area of the actinide trailer.

The total amount of ^{237}Np contained in 20 samples in the two sample containment cells plus the one sample cell in the reference compartment will be less than 100 μCi. This amount will be the maximum in the experimental hutch at any one time.

3.5 Operating Conditions

All of the experiments will be carried out under ambient temperature and pressure.

3.6 Failure modes and effects

The failure mode is the unlikely possibility that the secondary and tertiary PE bags will be breached. In that case the sealed polyethylene windows of the sample cell will contain the Np sample. If the polyethylene windows are also breached, the quaternary containment cell on the beamline will contain the Np sample. If the quaternary containment cell was also breached, the sample would fall on the table in the hutch where it could be wiped up from the plastic sheeting covering the table. In this case, the emergency operating procedures of Section 6.4 of this document would be activated.

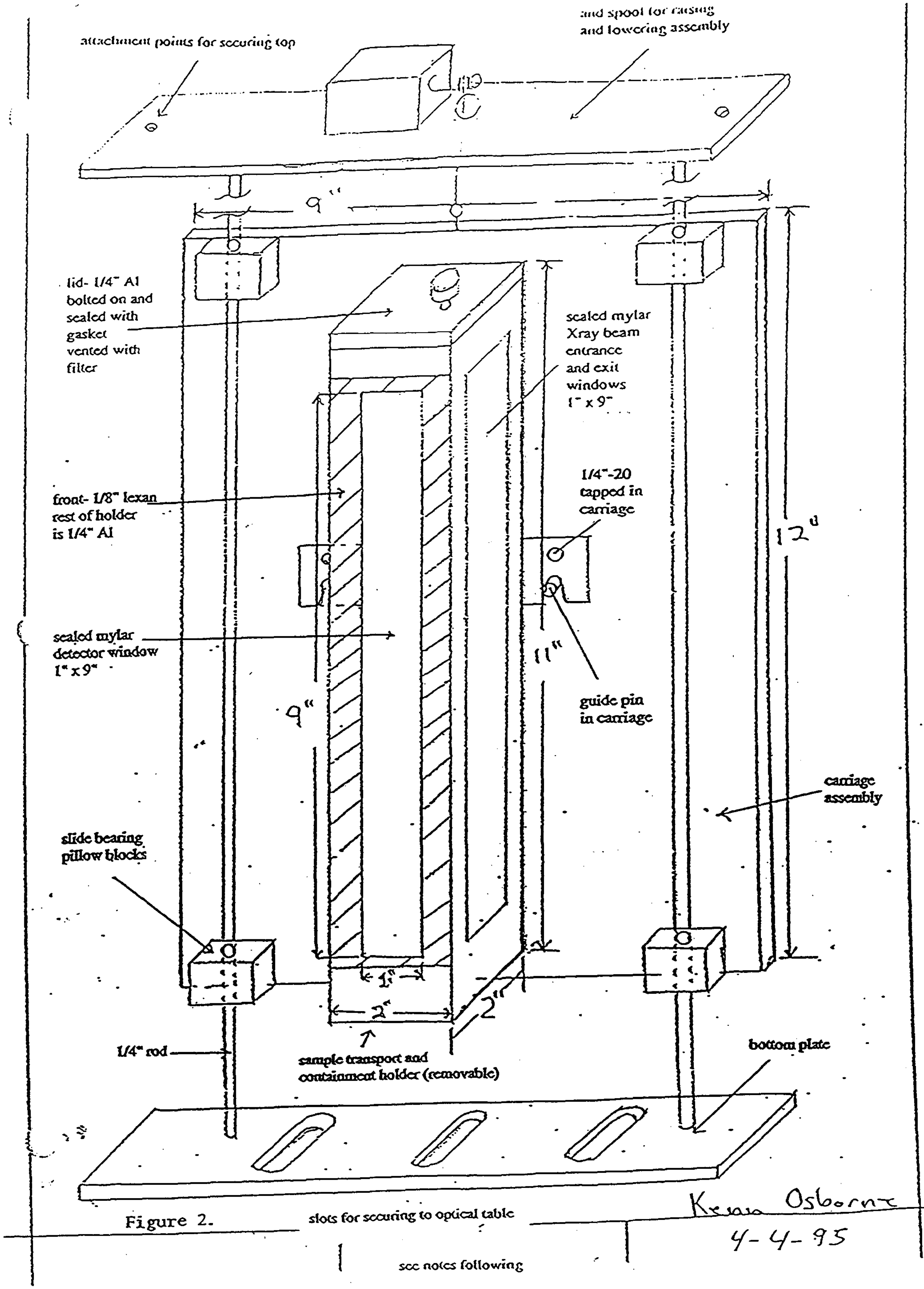
and spool for raising
and lowering assembly
attachment points for securing top
9"
lid- 1/4" Al
bolted on and
sealed with
gasket
vented with
filter
sealed mylar
Xray beam
entrance
and exit
windows
1" x 9"
front- 1/8" lexan
rest of holder
is 1/4" Al
1/4"-20
tapped in
carriage
12"
sealed mylar
detector window
1" x 9"
11"
guide pin
in carriage
9"
carriage
assembly
slide bearing
pillow blocks
1"
2"
2"
1/4" rod
sample transport and
containment holder (removable)
bottom plate
slots for securing to optical table
Kevin Osborne
4-4-95
see notes following

Figure 2.

Notes

1) The drawing is 1/2 scale. The distance from the base to the plate on top will be approximately 24 inches.

2) The system consists of two parts. The aluminum carriage will slide on two rods that are supported top and bottom. These will be 'permanently' mounted.

The sample transport and containment holder will be sealed and bolted to the carriage assembly with two 1/4" bolts. There are two guide pins on the carriage that will align the holder and support it while the bolts are installed.

3) The sample transport and containment holder will be constructed of 1/4" aluminum with an 1/8" lexan window on the front. The entrance, exit, and detector windows will be mylar. The aluminum pieces will be secured with #6 screws not more than 3 inches apart and sealed with RTV. The lexan will be secured to the aluminum with #4 screws not more than 2 inches apart and sealed with RTV. The top will be sealed with a gasket and vented through a 1/10 micron filter.

4) A cover for the mylar windows will be constructed to protect them during transport and mounting.

Figure 2. Notes

4.0 DESCRIPTION OF SHIPPING PROCEDURES

The sample materials for the proposed XANES and XAFS experiments will be shipped in compliance with all requirements of Title 49 of the Code of Federal Regulations (49 CFR) which addresses the use of proper shipping containers, correctly preparing shipping papers, and the proper marking, labeling, and transporting of radioactive material. In addition, all requirements of DOE Order 5480.3, "Safety Requirements for the Packaging and Transportation of Hazardous Materials, Hazardous Substances, and Hazardous Wastes," will be followed in shipping the sample materials. Each of the sample materials will be sealed within a sample cell (primary barrier), which, in turn, will be sealed within two additional PE bags which act as secondary and tertiary barriers. At LBNL, prior to placement into the shipping containers, these secondary and tertiary barriers will be loaded, sealed, checked, and surveyed for any surface contamination. The secured samples will be loaded into the DOT approved shipping containers. Thus, the sample materials will be contained within three barriers during shipment from LBNL to SSRL.

4.1 Description of the Shipping Containers

The shipping containers used will be those specified for shipping Type A samples. The Np samples will be placed in a metal can and the can will be sealed. This can will then be placed in a shipping carton which meets the specifications for a Type A container and will be labeled as required by Title 49 of the Code of Federal Regulations (49 CFR). For Np samples which have to be kept cold until used, the can containing the samples will be packed in dry ice, placed in a Styrofoam container, and then packaged in a Type A container.

4.2 Mode of Shipment from LBNL to SLAC

The packaged Np samples will be shipped by LBNL EH&S, Federal Express, or by an alternate express delivery company to the SLAC/SSRL OHP receiving office.

4.3 Receiving, Packing, and Unpacking Procedures

The Np samples will be received by SLAC/SSRL OHP where they will be checked for integrity and swiped. SLAC/SSRL OHP will deliver the samples to the designated storage/loading area. Further details of these procedures are described in Section 6.2.

4.4 Shipping Documentation and Accountability

All shipping papers will be completed in accordance with 49 CFR 172.202 and 172.203(d) which provide instructions on how to complete the documentation when shipping radioactive materials. All major common couriers (*i.e.*, Roadway, Federal Express, *etc.*) are permitted to transport radioactive materials in the state of California. LBNL will use one of these common carriers to transport the samples to SSRL within DOT-approved shipping containers or LBNL EH&S will deliver the sample materials.

All accountability procedures will be followed in compliance with DOE Order 5633.3, "Controls and Accountability of Nuclear Materials," including the documentation of the transfer of material from LBNL to SSRL. This process will involve interactions between the LBNL Shipping/Receiving Section, the Accountability Section of the Material Control, and the SLAC/SSRL nuclear material representative, who is currently in the Health Physics group at SLAC. When appropriate, LBNL will notify the California State Highway Patrol under the MOU with DOE concerning shipments of transuranics in California.

5.0 STORAGE OF SAMPLES

5.1 Description of storage

Samples will be stored in a 55 gallon drum except during the period when measurements are being performed on specific samples. These containers will be kept in the designated storage/loading area at SSRL. The current designated storage area is a modular building located immediately outside and between Bldgs. 120 and 131.

5.2 Security and documentation procedures

The storage/loading area will be kept locked, with a self-locking door, with the key controlled by designated SLAC/SSRL staff and the on-duty LBNL experimenter. Either designated SLAC/SSRL personnel or a LBNL experimenter must be present any time the storage area is not locked. These accesses and the status of the shipping containers (sealed/unsealed, samples within) will be recorded by either the LBNL experimenters or designated SLAC/SSRL personnel. One log book recording the location and disposition of each sample will be maintained. During the experiments, these log books will be kept by the LBNL group, and will be under the personal physical control of the LBNL experimenter on-duty.

5.3 Physical security measures

The self-locking ability of the door will be checked. The building containing the storage/loading area will be checked to ensure that unauthorized entry will be difficult and that it is in an area that is not too remote but routinely observed by site personnel. SLAC security will be notified of the presence of the materials.

6.0 OPERATIONS AND PROCEDURES

6.1 LBNL Experimental Team and SLAC RCT personnel.

The LBNL experimenters will be scientists or technicians whose principal responsibility will be sample handling and the performance of the XAFS measurements. At least one member of the team will have considerable synchrotron radiation experience and considerable experience with samples of the type being measured. These personnel need not be present at all times, but must be on call and available on short (*ca* 1 hour) notice 24 hours a day. At least one member of the team with current (and documented) Radiological Worker I training will be **present on the beamline at all times when a sample is in the experimental area with the following exception:** The Experimenter on Duty (EOD) may leave the beamline for ten minute periods for destinations within buildings 120 and 137. The EOD will log the destination, time left, time returned, and notify the Duty Operator prior to departure from the beamline. The EOD by definition will have the pager. The Duty Operator will be trained to respond to a CAM.

This will permit the performance of the experiment with a single person at the beamline with the following caveat: There must be two qualified individuals (both on the SSRL experimental form, one of which must have the proper Radiation Worker I certification, or a SLAC/OHP representative participating in the experiment) appropriately present whenever there is the manipulation of the sample containers within the experimental hutch.

SLAC will provide Radiation Control Technician (RCT) support. The designated RCT will be on call via a pager/beeper/telephone and available on short (*ca* 1 hour) notice 24 hours a day. The RCT will be present at all times when samples are being handled, including but not limited to, receipt and the initial surveys of the samples, transfer to and from the shipping/storage containers, and transfer of the samples to and from the experimental area. The SLAC RCT will be present at those times regardless of whether they or another member of the experimental team is performing the sample handling.

The LBNL experimenter on-duty will be the senior member of the experimental team present at SSRL. The LBNL experimenter on-duty will be responsible for ensuring that all procedures are followed and will be in charge of all operations involved in preparing the areas at SLAC/SSRL and in handling the samples from the time they are delivered to the loading/storage area until they have left the loading/storage area for shipment back to LBNL. Should the need arise, he (she) will direct emergency personnel. For certain types of non-routine procedures where it would be advantageous, he (she) may, with their concurrence, delegate this authority to someone more capable in terms of training or experience, *e.g.*, the SLAC Health Physics personnel.

6.2 Site Preparation at SLAC/SSRL

Prior to the arrival of the samples at SLAC/SSRL and the initiation of the experiments, the affected areas, including the storage/loading area, experimental area, and personnel access control area, must be established and prepared by the LBNL and SLAC/SSRL personnel. In addition, all radiation protection equipment and instrumentation will be on site prior to the unloading of the samples. Security and fire personnel will be notified of the arrival of the materials so that they can initiate specific procedures.

6.2.1 Monitoring

The following is a general overview of the types of monitoring controls that will be present in the storage/loading area and in the hutch area for these experiments.

SLAC/OHP will be in charge of the continuous air monitor (CAM) monitoring.

There are two types of monitors that will be used to detect alpha particles which would be present in the event of a release:

1) CAMs which are electronically controlled. Air is continuously sampled and counted by the use of an *in situ* detector along with a multichannel analyzer. One of the key parameters in using this type of monitor is determining the alarm set point (see below).

2) Swipe counters which rely on SLAC Health Physics personnel to accurately survey potential problem areas and insert the collected swipe into an alpha counter, record a reading and check against background.

Swipes of individual sample containers will be done as follows: a) before shipping from LBNL and these records will be provided to SSRL/SLAC OHP prior to their arrival at SLAC OHP, b) during unpacking from shipping container and prior to locating at beamline, and c) on removal from the hutch and before repackaging into the shipping container.

General swipes are needed as follows: a) of the storage trailer to gather a baseline and confirm that the area is clean before any sample arrives. b) of the hutch to gather a baseline before the experiment goes on line, c) of the hutch and the storage trailer after the experiment is completed.

Active monitoring of the hutch and the trailer using the CAMS is required while samples are present inside the hutch and when they are being loaded/unloaded in the trailer.

6.2.2 CAM Alarm set-point determination

Naturally occurring radon gas can result in a significant (relative to the desired minimum alarm level) background radiation level for the CAMs. The CAMs in use can discriminate radon from other alpha emitters on the basis of the different energies of their alpha emission. Prior to these experiments and the introduction to the site of any alpha emitter involved with these experiments, SLAC/OHP will determine the background levels at the experimental and other affected areas utilizing this energy resolving capability. The CAM alarm set point will then be determined in consultation with SLAC/SSRL Health Physics to maintain an adequate sensitivity level while minimizing the number of false alarms.

6.2.3 Storage area

Access to the storage area (actinide trailer) for the purpose of transferring samples will be coordinated in advance with SLAC/SSRL OHP.

The room will be doubly lined with plastic sheets. Work areas will be covered with additional plastic. This plastic will allow for containment and easy decontamination should a release occur and will, in conjunction with the filter system provide an additional barrier during sample transfer operations. A HEPA filtered blower (125 cfm) is attached so that the air flows into the room through the door at one end of the structure and out at the other end of the building. This will provide air flow from areas with the lowest potential for contamination to areas with a higher potential. The room air will be monitored using a Canberra Alpha Continuous Air Monitor

(Portable CAM) portable air sampler. The CAM will be operated during the times when a loading/unloading/transfer of TRU samples occurs or on a continuous basis while samples are stored depending on SLAC OHP procedures. The portable trailer will be posted as a controlled area and access will be controlled. The radiation detectors used to survey and monitor the storage and sample containers will be installed and checked.

A hood or containment area exhausting through the existing ventilation duct and HEPA filter should be provided in the storage area for loading and unloading the Np samples from the shipping containers.

The Np samples will be loaded into the beamline sample containment cell in this hood or containment area. The beamline containment cell will be checked for activity after loading the samples in the hood or containment area.

6.2.4 Hutch area

The experimental area, consisting of the inside of the hutch, will be monitored for alpha contamination initially and then prior to running each separate experiment/changing samples, using a Ludlum Model 139 alpha instrument and an Eberline SAC-4 alpha instrument.

Air sampling using a Canberra Alpha Continuous Air Monitors (Portable CAM) will be set up to monitor for the presence of alpha particles in the hutch containing the sample container positioner. The hutch will exhaust into a **HEPA filter** installed at the opening to the hutch ventilation system, passing by inlets for an inline sampler and a CAM. The ventilation will be controlled by a variable inlet and checked to ensure that all air is exhausting past the CAM and through the HEPA filter. The CAM intakes should be placed at the top of the experimental containment shroud prior to the entrance to the HEPA filter, in the general tabletop area inside of the hutch.

The experimental area (the lift table and the area on the lift table under the sample) will be lined with plastic as a contamination barrier. The use of plastic as a protective liner in the experimental area will minimize the spread of contamination and facilitate cleanup efforts with damp cloths or tissues if any contamination is detected. The optical bench assembly used to support the experimental apparatus will be fixed to the top of the hutch lift table. Open penetrations in the hutch will be closed off.

6.2.5 Personnel access control area

A personnel access control area will be established by SLAC/SSRL in consultation with the LBNL staff as part of the scheduling process. It will include the experimental hutch, the loading/storage area, and any adjacent area where personnel not directly associated with the experiment should be excluded. This area will be posted with warning signs and temporary barriers where needed. Only authorized and trained personnel, those from LBNL involved with the experiment and those designated by SLAC/SSRL, will be permitted in this area. Samples will only be allowed into the personnel access control area when they are transferred from the storage area to the experimental area in a closed rigid steel transfer container. Only two beamline sample container cell and one reference cell sample container (containing the Np samples) will be permitted in this area at any time.

6.3 Standard Operating Procedures at SLAC/SSRL

This section describes the step-by-step procedures at SLAC/SSRL involved in the actual handling of the samples, including receiving, performing the XANES and XAFS measurements, and shipping. The samples and the areas will be frequently (or continuously) checked for contamination and the results noted on the radiation survey logsheets at the storage site and the beamline. In the unlikely event of the simultaneous failure of the primary (sample cell), secondary and tertiary PE bags, and the quaternary sample containment container due to simultaneous puncture or concurrent failure of the seals of all four barriers, release of contamination could occur. If contamination is found, the decontamination procedure described later will be followed. In the case of a CAM alarm or airborne contamination, the specific procedure described in Sec. 6.4 will be followed.

6.3.1 Receipt of samples at SLAC

The shipping containers will be delivered to SLAC Health Physics only after swipe records have been received at SSRL/SLAC OHP. Prior to unloading, the outsides of the containers will be swiped to show < 20 dam/100 cm^2 removable contamination.

6.3.2 Transfer of samples to SSRL loading/storage area and verification

The samples inside the shipping containers will subsequently be transported to the loading/storage area at SSRL and remain there for the duration of the experiment. Security and documentation procedures will be implemented at this time. On receipt at the loading/storage area at SSRL, the SLAC/OHP RCT and other members of the experimental team will unpack the shipping container. LBNL personnel opening the shipping containers will wear gloves and lab coats. Surveys of the surfaces, *e.g.*, walls, floors, tables, *etc.*, will be obtained by wiping the surfaces with swipes which will subsequently be counted, and documented. This will be confirmed by checking each shipping container with a swipe which will be counted on the SAC-4 alpha instrument, verifying that they show < 20 cpm/100 cm^2 removable. For storage the samples will be placed into a resealable 55 gallon drum.

If alpha contamination is identified at any time during unpacking the process shall be stopped immediately. The secondary container will be decontaminated according to the LBNL decontamination procedures; the area will be checked and, if necessary, decontaminated according to the LBNL procedures. The sample will be placed back in the shipping container and the container will be sealed for return shipment to LBNL.

If the CAM alarms at any time during unpacking, the process shall be stopped immediately. The storage/loading building will be evacuated and if the alarm is found to be valid by SLAC/OHP, the LBNL emergency response team will be called according to the procedures of Section 6.4. The LBNL emergency response team will be responsible for checking for contamination and surveying the area and containers for a possible release of contamination. The CAM will be operated during the times when a loading/unloading/transfer of TRU samples occurs or on a continuous basis while samples are stored depending on SLAC OHP procedures.

6.3.3 Placement of samples in the quaternary container

A separately ventilated hood area or containment area with a HEPA filter will be provided in the storage area for loading and unloading the Np samples from the shipping containers. The Np samples will be loaded into the beamline containment cell in this hood or containment area. The beamline containment cell is designed to hold up to 10 Np samples at one time. The beamline containment cell will be checked for activity after loading the samples in the hood or containment area. A separate beamline containment cell will be used for the one sample (with surrounding primary, secondary and tertiary containment) which will be placed in the reference compartment at the beamline. The LBNL personnel performing these operations will wear lab coats and gloves and monitor the operations with a certified portable alpha counter.

The samples will be loaded into the quaternary sample container (the beamline containment cell). This quaternary sample container will be placed within a closed rigid steel transfer container for protection during the transfer to the experimental area. Only two beamline containment cells plus the reference containment cell with the reference Np sample may be outside of the storage area at any time. If the CAM alarms at any time during loading, the process shall be stopped immediately, and the storage/loading building will be evacuated. The building will be monitored by SLAC/OHP and reentered when safe. If necessary the LBNL emergency response team will be called for decontamination. The CAM will be operated during the times when a loading/unloading/transfer of TRU samples occurs or on a continuous basis while samples are stored depending on SLAC OHP procedures.

If the sample containers are subjected to any treatment outside of the standard handling procedure (i.e. dropped), the containers will be surveyed for any release of the sample. If there is an indication that the containment system may have been damaged, the sample will be returned to the storage container and will not be used. The event will be logged and reported to the SLAC/SSRL Evaluation Team. The SLAC/SSRL Evaluation Team will review all incidents and decide whether and how to proceed with the remaining samples. If the event is sufficiently minor so that the LBNL experimental team believes that no damage has occurred and this supposition is supported by the absence of any signs of damage during the subsequent inspection or by appropriate tests, then they may proceed with the experiment.

6.3.4 Transport of quaternary container to experimental area

The quaternary sample container within the rigid steel transfer container will be moved to the experimental area. A dolly or cart may be used to assist in transporting the transfer container. The transfer must follow the designated route. SLAC/OHP will escort the LBNL experimenter.

6.3.5 Mounting of quaternary container

The quaternary sample container will be removed from the transfer container and placed in the sample holding assembly on the table in the beam line hutch. A CAM located within the hutch will be turned on.

6.3.6 XAFS measurements

XAFS measurements will be made on the sample(s). At least one member of the LBNL experimental group must be in attendance at all times when a sample is present in the experimental area as described and defined in Section 6.1. In the case of a CAM alarm, the applicable procedures described in section 6.4 will be implemented. Please refer to section 6.4 for detailed information about emergency response to contamination and emergency response to CAM alarms.

If the sample containment cell or the samples within are dropped or any deviation from the standard procedure with regard to the containment vessel integrity occurs during this step, the procedure described in Sec. 6.3.3 will be followed. If the control and behavior of the sample does not follow the expected pattern for the specific experiment as described in Sec. 3, the experiment will be stopped until this event is reported to and evaluated by the SLAC/SSRL OHP, who will decide whether and how to proceed.

6.3.7 Completion of measurements, dismounting of quaternary container

Upon completion of the measurements for a sample or set of samples, and under the condition that the CAM monitoring the air around the beam line sample container shows no contamination, the quaternary sample container will be dismounted from inside the hutch and placed back in the transfer container.

6.3.8 Monitoring of experimental area

After the beamline containment cell is removed, the experimental apparatus and area will be monitored for alpha radiation and removable contamination. In the case of removable contamination, the area will be secured and the procedures of section 6.4 will be followed. In the case of a confirmed CAM alarm (see Section 6.4), the building will be evacuated and the applicable procedures described in section 6.4 will be implemented. In the case of a known or suspected release resulting from an accident or if contamination is detected during a survey, the procedures described in Sec. 6.4 will be implemented, as appropriate.

6.3.9 Return of samples to storage/loading area

The transfer container containing the quaternary sample container will be transferred back to the storage/loading area. It may not leave the designated path. The procedures outlined in 6.3.3 and 6.3.4 will be followed.

6.3.10 Monitoring of samples

Once within the storage area, the quaternary sample container will be opened and the sample slide mount will be removed. The container will be inspected visually. If there are any indications that the sample containment may have been breached, then the whole apparatus will be placed in a plastic bag for containment purposes and returned to LBNL. The security of the seals will be confirmed by checking the inside of the beamline sample container with a swipe which will be counted on the SAC-4 alpha instrument, verifying that they show < 20 cpm/100 cm^2 removable alpha contamination. The packaged samples will be placed back into the 55 gallon drum, which will be resealed. If the CAM alarms at any time during this procedure, the process shall be stopped immediately, the storage/loading building will be evacuated, and the area and containers surveyed by qualified personnel for a possible release of contamination. The CAM will be operated during the times when a loading/unloading/transfer of TRU samples occurs or on a continuous basis while samples are stored depending on SLAC OHP procedures.

6.3.11 Completion of experiments

Samples will be repacked into appropriate DOT shipping containers following the same procedures used for the original packing. The outside of these containers will be nucon swiped and the swipes counted on the SAC-4 alpha instrument to verify that they show < 20 dpm/100 cm^2 removable and no fixed alpha contamination. The health physics portion of the appropriate shipping documents will be completed. SLAC/SSRL will transport the sealed shipping containers from the storage/unpacking area to the SLAC OHP shipping area for shipment back to LBNL.

6.4 Emergency Operating Procedures

6.4.1 Response to an accidental release

Immediately following a release (or discovery of a release), the following list of procedures needs to be performed as appropriate to ensure the safety of those involved, to prevent spread of contamination, and finally clean up the contamination. The LBNL experimenter-on-duty will initially be the "person in charge" and have the responsibility for ensuring that these procedures are followed until such a time as he (she) may delegate this responsibility to someone more capable in terms of training and experience.

1. Provide for the safety of personnel involved:

   • first aid is given to serious injuries

   • contaminated personnel will be confined to the immediate area

   • help is summoned as needed.

2. Minimize the spread of contamination by maintaining or establishing controlled areas and post the areas as appropriate.

3. Call the LBNL emergency response team (see below).

4. Of less critical importance but also of immediate concern, the person in charge should:

   • Contact SLAC Fire department by calling 9-911 and the SLAC Main Control Center by calling extension 2151 (926-2151 if off-site).

   • Assure necessary equipment and materials for clean up are assembled, and

   • Assign a "technical reporter" to accurately record the accident response activities.

Decontamination Procedures

All decontamination procedures should be performed by qualified and certified personnel.

Personnel decontamination should be performed first followed by area decontamination. Waste generated during decontamination will be shipped back to LBNL in appropriate containers. Once the extent of the contamination is determined, a decontamination plan will be formulated using all available information. The decontamination procedures will be followed as appropriate.

Arrangements with LBNL

All waste (including liquid) generated during the accident response will be collected and sent back to LBNL for disposal. This will be performed by packing and shipping such waste as DOT type A, for which there is no restriction on the type of material. All samples are already type A, so that all waste generated during decontamination is also type A and would not require survey and division. Suitable shipping containers will be available at SLAC/SSRL during the experiments to be used in the event of a release. An LBNL Emergency Response Team will be on call in case of accidental release of radioactivity. Appendix 1 contains the names and telephone numbers of the LBNL Emergency Response Team.

6.4.2 Response to CAM alarms

Two types of CAM alarms may be distinguished; 1) CAM alarms from inside the hutch, and 2) CAM alarms within the loading/storage area. If the hutch CAM alarms, Ian Evans and SLAC/OHP will be called immediately (if not already present) and will decide how to proceed based on surveys of the area. If the loading/storage area CAM alarms or the hutch and plastic enclosure CAM alarm simultaneously, the following procedure will be followed:

1. The area will be evacuated immediately in response to a CAM alarm and personnel will assemble in a designated area. The SLAC Main Control Center (extension 2151, 926-2151 if calling off site), the Fire Department (9-911), and the SSRL Beamline Duty Operator will be notified of the alarm and the status of the neptunium.

2. SLAC/OHP will monitor all people who evacuated into designated areas after establishing controls to prevent inadvertent entry into the affected area. SLAC/OHP and the LBNL experimental team will determine if the LBNL emergency response team is to be called.

3. The LBNL emergency response team , if called, will evaluate the situation and perform appropriate decontamination procedures as needed.

6.4.3 Response to fire in the experimental area

Minor fire. Extinguish using available extinguishers. Close shutters, turn off high voltage power to ion chambers, fluorescence detector, and DC power to optical bench and AC sample positioner stepping motors and Lytle detector. Leave all radiation monitoring equipment on. Contact the SLAC Main Control Center (extension 2151, 926-2151 if off site), the Fire Department (9-911), the SSRL Beamline Duty Operator, and the Emergency Response Team and inform them of the fire and the status of the neptunium.

Major fire. Evacuate area immediately. Activate fire alarm. Report to the designated area. Contact the SLAC Main Control Center (extension 2151, 926-2151 if off site), the Fire Department (9-911), the SSRL Beamline Duty Operator, and the Emergency Response Team and inform them of the fire and the status of the neptunium.

6.4.4 Response to fire alarm outside of the experimental area.

Close shutters, turn off high voltage power to ion chambers, fluorescence detector, and DC power to optical bench and AC sample positioner stepping motors and Lytle detector. Leave all radiation monitoring equipment on. Report at the designated area. Inform the SLAC Main Control Center (extension 2151, 926-2151 if off site), the Fire Department (9-911), the SSRL Beamline Duty Operator, and the Emergency Response Team of the status of the neptunium.

6.4.5 Response to flooding or earthquake.

Turn off high voltage power to ion chambers, fluorescence detector, and DC power to optical bench and AC sample positioner stepping motors and Lytle detector. Leave all radiation monitoring equipment on. Inform the SLAC Main Control Center (extension 2151, 926-2151 if off site), the Fire Department (9-911), the SSRL Beamline Duty Operator, and the Emergency Response Team of the status of the neptunium. If necessary, evacuate the building and follow standard SLAC/SSRL procedures.

6.4.6 Response to theft and vandalism

In the event that a sample if taken or is found to be missing, or if it appears that unauthorized entry into the loading/storage area or the sample storage containers has been performed or attempted, SLAC Security will be notified immediately by calling extension 2551 (926-2551 if off site).

6.4.7 Response to a power failure

A unscheduled or scheduled power failure will not increase the probability of release. First consideration will be given to equipment that may be damaged upon restoration of power. During the power outage, the hutch will remain closed and the samples will not be handled while the CAM's are disabled. If the failure occurs during transfer of the samples, the samples will be returned to the location deemed safest. Once power has been restored and the CAM's are operational, experiments may proceed.

7.0 RADIATION PROTECTION AND TRAINING

The proposed XAFS experiments to be performed at the Stanford Synchrotron Radiation Laboratory will be subject to the requirements and controls of the SLAC/SSRL radiation protection program. The sample materials proposed for these experiments pose an additional set of radiological considerations. SLAC/SSRL and LBNL will provide programmatic, procedural, and personnel support to address any considerations unique to neptunium, plutonium, or actinides, as a class.

Certification of Training

Training records of the LBNL Experimental team certifying their expertise in handling radioactive materials will be provided to the health physics staff at SLAC prior to the arrival of the sample materials at SLAC/SSRL.

8.0 RESTRICTIONS AND LIMITATIONS

8.1 Material Quantity Limits

The quantities of material contained in any sample cell, as well as the total inventory of all sample materials for these experiments shall not exceed those quantities stated in Sec. 3, "Description of Experiments."

8.2 Storage Restrictions

Samples will be sealed into the sample cell, and the secondary and tertiary sample containers at LBNL, and these seals will be left intact at all times until their return to LBNL. Any samples not in use will be stored in a sealed 55 gallon drum (solution and liquid samples must be transferred from their type A shipping container). The area, or room, containing the shipping containers will be kept locked at all times except when samples are being handled in that area. This area, or room, will be selected jointly with SLAC Health Physics and SSRL staff, and will be prepared to serve as another barrier during loading or unloading operations. The samples that have been used in the previous run will be removed and placed back into the storage container before the new samples are loaded. Only those samples that will be loaded into the beamline containment cell to be used in the next run or in the reference containment cell will be removed from the shipping container. **One of the experimenters with Radiation Worker I certification will be in attendance at the experimental area at all times when samples are in place in accordance with the procedures defined in Section 6.1.**

8.3 Limit on Number of Samples in Use

Only two beamline sample containment cells (holding up to ten individual Np samples each) and one beamline reference sample containment cell (containing one Np reference sample) will be outside the actinide storage area and in use at any time. The number of samples and the total quantity of transuranic elements in the quaternary sample containment vessel and in the reference sample container are listed in Sec 3.

8.4 No Detectable Levels of Contamination

The baseline for no detectable levels of contamination will be established by surveying the storage and experimental areas prior to the arrival of the samples. This survey will be performed with SLAC Health Physics to certify and verify any existing contamination prior to the experiment.

8.5 Accountability and Ownership

Because the total amount of material is less than 50 g, the samples are Category 4 and will be classified as "LBNL Projects," so that LBNL will be accountable during the entire time they are off site. The materials will be logged in at SLAC and carried on SLAC's records indicating ownership as "LBNL Projects." SLAC will remove the materials from its inventory upon shipment back to LBNL. While at SLAC/SSRL, the materials will be owned by LBNL but SLAC/SSRL will be responsible for providing protection of the materials while they are on the SLAC/SSRL site. As part of the accountability procedures, the location and disposition of the samples while at SLAC/SSRL will be recorded in a log book.

LAWRENCE BERKELEY LABORATORY
Bldg.: 71 Room: 239 Ext.: 7609

APPENDIX

March 31, 1995
chron RA-95-83

MEMORANDUM

TO: Dr. Norman Edelstein

FROM: Glenn Garabedian

SUBJECT: SSRL Experiment - Emergency Response

The Hazards Analysis Document for your X-Ray Absorption Experiments, which will be conducted at Stanford Synchrotron Radiation Laboratory (SSRL), indicates the need for an LBL Emergency Response Team. In the event of an accidental release, LBL may be called upon to provide assistance in radioactive contamination control and decontamination as stated in Section 6.4 of the Hazards Analysis Document. Further, we will be required to collect and return to LBL all waste generated from such an incident.

Should it be necessary to request emergency assistance, use the following call list:

	LBL phone	Pac Tel Pager	Home phone
Gregory Buck	486-6213	448-3890	441-1029
Keith Heinzelman	486-6212	448-3891	559-8567
Cameron Huff	486-6214	448-3892	(707)588-9727
Glenn Garabedian	486-7609	840-6209	283-8706
David Anholm	486-5611	425-7971	(707)446-8015

Radiation Assessment's direct line is 486-7652. All telephone numbers are in the 510 area code unless noted otherwise.

GG:gg

cc: D. Anholm
M. Bushman
G. Buck
J. Chung
C. Donahue
J. Floyd
K. Heinzelman
C. Huff
R. Kloepping
K. Rivera
T. Sundsmo

CHECKLIST FOR ^{237}Np SOLIDS

Sample Description	______________________
Sample Identification	______________________
^{237}Np Quantity	______________________
Sample Containment	______________________

Primary Containment Vessel Assembly:

Seal integrity inspected visually ______
Fixed (direct) alpha contamination, <20 dpm/100cm^2 ______ / ______

Secondary Containment Vessel Assembly:

Seal integrity inspected visually ______
Fixed (direct) alpha contamination, <20 dpm/100cm^2 ______ / ______

Tertiary Containment Vessel Assembly:

Seal integrity inspected visually ______
Fixed (direct) alpha contamination, <20 dpm/100cm^2 ______ / ______

External Dose Rate:

Alpha	@ contact:	______ / ______
Beta	@ contact:	______ / ______
Gamma	@ contact:	______ / ______
Beta	@ 30 cm	______ / ______
Gamma	@ 30 cm	______ / ______

ALPHA	DOE/LBL Serial Number/manufacturer	
BETA	DOE/LBL Serial Number/manufacturer	
GAMMA	DOE/LBL Serial Number/manufacturer	

Loader:	______________	**Date:**	______________
Checker:	______________	**Date:**	______________
Surveyor:	______________	**Date:**	______________

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