



# ERNEST ORLANDO LAWRENCE BERKELEY NATIONAL LABORATORY

## Safety Procedures for the Electron Spectroscopy of Actinides at the ALS

D.K. Shuh, N.M. Edelstein, and J.J. Bucher  
Chemical Sciences Division

January 1996

MASTER

RECEIVED

MAR 3

C



DISTRIBUTION OF THIS DOCUMENT IS UNLIMITED

*J.B.*

#### **DISCLAIMER**

This document was prepared as an account of work sponsored by the United States Government. While this document is believed to contain correct information, neither the United States Government nor any agency thereof, nor The Regents of the University of California, nor any of their employees, makes any warranty, express or implied, or assumes any legal responsibility for the accuracy, completeness, or usefulness of any information, apparatus, product, or process disclosed, or represents that its use would not infringe privately owned rights. Reference herein to any specific commercial product, process, or service by its trade name, trademark, manufacturer, or otherwise, does not necessarily constitute or imply its endorsement, recommendation, or favoring by the United States Government or any agency thereof, or The Regents of the University of California. The views and opinions of authors expressed herein do not necessarily state or reflect those of the United States Government or any agency thereof, or The Regents of the University of California.

This report has been reproduced directly from the best available copy.

Ernest Orlando Lawrence Berkeley National Laboratory  
is an equal opportunity employer.

LBNL-39909  
UC-401

**Safety Procedures for the Electron Spectroscopy  
of Actinides at the ALS**

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

Chemical Sciences Division  
Ernest Orlando Lawrence Berkeley National Laboratory  
University of California  
Berkeley, California 94720

January 1996

This work was supported by the Director, Office of Energy Research, Office of Basic Energy Sciences,  
Chemical Sciences Division, of the U.S. Department of Energy under Contract No. DE-AC03-76SF00098.

**DISCLAIMER**

**Portions of this document may be illegible  
in electronic image products. Images are  
produced from the best available original  
document.**

# Safety Procedures for the Electron Spectroscopy of Actinides at the ALS

D. K. Shuh, N. M. Edelstein, and J. J. Bucher  
Chemical Sciences Division  
Berkeley Laboratory

## Contents

Summary	1
Experimental Procedures	
Sample Preparation	2
Procedures at the ALS	4
Emergency Procedures	8
List of Figures	9
List of Tables	10
Figures and Tables	11
ALS Experimental Forms	30
Safety Procedures (Nov.1994)	33

Prepared Jan. 1996

**Addendum to the ALS Experimental Safety Form  
Renewal for Actinide Microspot Experiments**

Jerry Bucher, Actinide Chemistry Group, CSD, LBNL, (510) 486-4484  
Norman Edelstein, Actinide Chemistry Group, CSD, LBNL, (510) 486-5624  
David Shuh, Actinide Chemistry Group, CSD, LBNL, (510) 486-6937

18 Jan. 1996

**SUMMARY**

This is an addendum to the ALS Experimental Safety Form Renewal for the continuation of actinide microspot experiments on beamlines 7.0. There are several modifications to the previously approved procedures. There is an increase in the amount of allowable material of the low activity isotopes  $^{238}\text{U}$ ,  $^{237}\text{Np}$ ,  $^{242}\text{Pu}$ , and  $^{248}\text{Cm}$ . There is also the addition of  $^{99}\text{Tc}$  and the low activity isotopes  $^{232}\text{Th}$  and  $^{243}\text{Am}$  to the list of permissible sample materials. All of the materials are alpha-emitters with negligible gamma fields with the exception of  $^{99}\text{Tc}$  which is a beta-emitter. There is a series of new experiments that requires the use of a crystal cleaver in the preparation chamber of the ultraESCA endstation. The beamline 7.0 ultraESCA endstation has been suitably modified to permit the safe cleave of YUPd alloy rectangular ingots. All of the sample materials are solids. The exact nature and composition of the samples are delineated in the sample preparation section that follows. A corresponding Radiological Work Authorization (RWA) must be issued for this work at ALS since the material amounts exceed those in the Low Activity Source (LAS) guidelines in Table I and those in the Values for Exemption of Sealed Source Inventory in Table II. The preliminary date for the next run of these sample materials has been tentatively scheduled in early Feb. 1996 and this will be with the uranium cleave alloys, not the transuranic materials.

## EXPERIMENTAL PROCEDURES

### *Sample Preparation*

Sample materials sent from institutions outside of LBNL will be shipped to:

Bette Muhammad  
EH & S Receiving  
LBNL  
1 Cyclotron Road  
Berkeley, CA 94720  
(510) 486-7602

C/O ALS Experimenter  
Local ALS contact phone #.

The materials will be forwarded to the Actinide Chemistry Laboratory in Building 70A-1145 for preparation, characterization, packaging, and subsequent transport to the ALS via EH&S personnel.

The sample preparation will follow the previously approved procedures and utilize some new preparation techniques that require slightly different experimental procedures. The initial experience gained from sample preparation and experimental work at beamline 7.0 will safely permit a slight increase in the amount of radioactive material employed compared to earlier experiments. Thus, this safety addendum reflects allowable material amounts that are 3-5 times greater than those previously used. Additionally, there are some new elemental isotopes on the list of those permitted. The new amounts and isotopes are shown on the ALS Experimental Modification form. All sample preparation and characterization of the activity of the samples will be done in Bldg. 70A-1129, 1145 under existing RWA procedures (Edelstein #1020). All mounting and physical handling of the samples, except for the loading and unloading into the beamline 7.0 endstation vacuum chamber, will be at this location. All samples will be characterized by alpha spectroscopy to ensure that the amount of activity is within work permit limits and to ensure that any part of the sample holder that comes in contact with the vacuum chamber sample manipulation apparatus is free from activity.

### *Aqueous Preparation*

The radionuclides used as sample materials will be prepared by dilution and delivered to the surface of a Pt counting disk or to a graphite disk (with a thin layer of Pt on the backside) using a microliter pipette. The resulting material will be primarily oxides of the particular radionuclide. The aqueous solvent will be removed by inductive heating. The radionuclide will be bonded to the substrate during this process as well. The samples will be observed under a microscope and the radioactivity characterized in a calibrated alpha spectrometer to determine the total activity. The amount of material will be within the limits specified on the ALS Modification form dated 12 Jan. 1996 that are informally derived from *Operational Health Physics: Laboratory Operations and Good Work Practices* that is attached as Table III. The adhesion of the radionuclide to the substrate will be determined by testing sample structures to ensure that there is no loose active material. The properties of the various isotopes to be used as sample materials are summarized in the Table IV attachments. The sample isotopes are never completely isotopically pure, thus a substantial portion of the total activity of the radionuclide sample may result from trace amounts of isotopic impurities.

### *Uranium Alloy Cleave Bar Preparation*

The cleave bar preparation will entail the examination of the integrity of the ingot mounting on the sample holder and the determination of the total activity of the sample. There may be some cleave bar handling in accordance with procedures in RWA #1020. The uranium alloy cleave bars are about 5 g, with a total uranium content of less than 0.5 g each and will be shipped from the Univ. of Michigan. The exact composition of the uranium alloy is  $Y_{1-x}U_xPd_3$

### *Special Samples*

Special samples will be handled on a case by case basis with reference to this document.

### *Thin Film Samples*

Thin film metallic samples of the radioactive materials will be prepared off site on suitable substrates and transported to LBNL. These samples will be



mounted to sample holders and the total activity characterized. Similar thin film samples may also be prepared at LBNL.

### *Sample Mounting and Packaging*

The radionuclide substrate and microsample will be affixed with spring-loaded clips, spot-welded clips, or bolted directly to sample holders used by beamline 7.0. The cleave ingots to be used in the uranium alloy experiments will be mounted as shown in Figure 1 and wired into place (this is common practice for cleaving). All samples will be appropriately labeled and packaged in ice cream cartons for safe transport to the ALS. The EH&S monitor will certify that the container is non-active and establish that the activity of the sample is within the limits of the approved work permits for the ALS. The sample container will be labeled "CAUTION-Radioactive Material" to warn personnel that a radioactive material is present. The samples will be transported to the ALS, with prior notification of the ALS EH&S monitor, in accordance with EH&S regulations. The samples will be transported, no more than two at a single time, by LBNL vehicle to the ALS.

### *Procedures at the ALS*

#### *General Procedures at Beamline 7.0*

There will be no handling of the sample on the experimental floor with exception of unpacking, loading, alpha characterization of, unloading, and re-packaging to transport back to the 70A-1145 laboratories. The ALS control room will be notified prior to the commencement of any experimental activities at beamline 7.0. and will be informed upon completion of the experimental program. Only two samples at a time will be brought to the ALS and there will be a maximum of four samples resident on the ALS floor at any time (during a full sample exchange at beamline 7.0) just prior to the removal of two samples from the ALS floor for transport back to radiochemistry lab in Building 70A-1145. All loading and unloading activities will be done with the ALS EH&S monitor present.

The samples will be brought to the ALS from the preparation laboratories in conjunction with EH&S, as per standard operating procedures. The samples will be loaded into the experimental chamber with a procedure utilizing laboratory coats, gloves, alpha meter, TLD/film badges, and beta-gamma meters that will be brought to the ALS by appropriately trained/supervised personnel from the Actinide Chemistry Group. A temporary Radiological Materials Area (RMA) will be

established, labeled, and casual access restricted. The sample/holders will be removed from the ice cream cartons one at a time, placed in the temporary RMA, and an alpha assay performed with an appropriately calibrated survey meter and geometric positioning equipment by the ALS EH&S monitor. The results will be recorded on an approved logsheet form and placed in the laboratory notebook. An example of this form is attached as Fig. 2 and a copy of the logsheets will be provided to the ALS EH&S monitor upon completion of the experiments. The samples will be examined under a microscope with a recording CCD camera. The samples will then be loaded into the sample load lock. A schematic of the beamline 7.0 ultraESCA endstation is shown in Fig. 3. The sample chamber or endstation will be labeled "CAUTION-Radioactive Material" to warn personnel that a radioactive sample is present. Once the radioactive samples are in the endstation, the temporary RMA will be surveyed and re-established as a non-RMA if no activity is found. The sample will be transferred under vacuum into the photoemission spectrometer on beamline 7.0 and the electron spectroscopy begun. Any other specialized in vacuum preparation or handling of the sample materials will be described in a following section that addresses procedures specific to certain sample types.

The beamline 7.0 endstation is an RMA when there are radioactive samples in the chamber, therefore RMA procedures must be employed regarding removal of samples or any other experimental equipment from within the vacuum envelope of the endstation until the endstation is declared a non-RMA. For example, this requires that a non-radioactive sample be handled in the same fashion as a radioactive sample if it is removed from the vacuum chamber while operating as an RMA.

Sample/holders will be removed from the beamline 7.0 endstation by re-establishing the temporary RMA work area and removing the samples/holders (one at a time) from the vacuum system. The respective samples will be characterized by alpha spectroscopy in the temporary RMA to ensure that no material has been lost. If no material has been lost, the experiments may proceed. The samples/holders will be placed in ice cream cartons for transport to the 70A-1145 laboratory. At this time, two new samples/holders may be removed (one at a time) from the ice cream cartons and loaded into the vacuum system as described in the loading procedures above. Thus, there will briefly be four samples on the ALS floor during a full sample exchange. After the new samples have been successfully loaded, the temporary RMA will be surveyed and declared a non-RMA. The samples/holders

removed from the endstation will be re-assayed by alpha spectroscopy in the Bldg. 70A-1145 laboratory.

Upon successful completion of the experiment and the documented removal of all samples as described above, swipe(s) will be taken of the accessible sample transfer apparatus. The beamline 7.0 endstation will be declared a non-RMA and the signs removed after successful swipe(s) results. Swipe(s) of the vacuum chamber will also be taken after the chamber is vented to atmosphere for the first convenient opportunity following the completion of these experiments. The swipes will be performed by the ALS EH&S monitor and recorded on a logsheet.

All of the EH&S assistance will be scheduled as far in advance as experimentally feasible and will be directed through Keith Heinzelman, LBNL ALS EH&S radiation safety monitor (Bldg. 80A, x6212) and Jim Hayes (Bldg. 70A).

#### *Procedures for the Experiments Requiring the Cleavage of Uranium Alloy Samples*

A series of new experiments on requires the use of a cleaver, shown in Fig. 4, to cleave a metallic, rectangular bar composed of an uranium alloy to expose a pristine surface for electron spectroscopy (sample/holder shown in Fig. ). This uranium alloy sample will contain ~0.5 g of  $^{238}\text{U}$ . This experiment poses additional complications since part of the cleave bar will drop to the bottom of the vacuum chamber upon a successful cleave and will have to be retrieved at some point. Additionally, there will be the generation of some small particles and dust under UHV conditions resulting from the cleave in the chamber. Furthermore, there is sometimes the need to slightly scrape the sample bar with cleaver to prepare the surface and this will result in some small particulates as well. The cleaver must also be made compatible with the existing sample transfer and the cleave must take place in a portion of the vacuum chamber that is remote from the parts that are normally used. The sample/holder will be cooled to 77K in the main spectrometer.

The endstation will be modified to accommodate the cleaver, provide a remote location for the cleave, interface to the sample transfer mechanism, and to provide a landing zone for the cleave bits. The chamber will be modified as shown in Figs. 3 and 5. The sample when cleaved will be held in a horizontal manipulator. A chip funnel will route the large cleave bits into an isolated catcher. The large chips can be removed by closing the 2.75" gate valve after the experiment and can be placed back in service by pumping through the right angle valve.

During the experiment the entire endstation will be labeled as an RMA but as result of the cleaving operation, not all of the active material will exit with the sample/holder assembly. Thus, the normal procedures for alpha counting will continue to be followed but there will not be complete recovery of the sample material or its associated activity. Thus, upon completion of the experimental program, the 2.75" gate valve will be closed and the preparation chamber labeled as a Radioactive Storage Area (RSA) and removed from the RMA designation. It is possible that there may be a extremely small amount of dust or a few tiny sample bits that do not fall into the catcher and remain at the lower portion of the cleaver cross. Designated as an RSA, experimental procedures may proceed without RMA constraints. The cleaver will not be used for any other experiments.

The beamline 7.0 endstation is a multi-purpose endstation and the researchers involved at beamline 7.0 would like to continue experimentation without having to vent the preparation section that includes the cleaver assembly until a later date when beam from the ALS is not available. There are no chemical operation or processes that would affect or mobilize the uranium material that does not fall into the catcher. Therefore, at the first convenient opportunity, the catcher will emptied and the cleaver section vented. The ALS EH&S monitor will swipe the cleaver chamber and remove any loose material therein. The cleaver cross (including the catcher assembly) will then be removed from the rest of the preparation chamber, sealed and bagged, and transported to the laboratory in Bldg. 70A-1145. The cleaver cross (including cleaver) will be thoroughly cleaned in preparation for re-use in the future and will be certified as non-radioactive before connection to the beamline 7.0 endstation. The designation of the chamber as a RMSA will be removed. All waste materials from the experiment will be disposed of by the Actinide Chemistry Group.

### *Thin Film Samples*

The thin film samples will be metallic, oxides, or an alloy that are permanently bonded to a substrate that will be affixed to the sample holder.

### *Special Samples*

There may be small particulates and other samples. The most important consideration is the mounting or affixing of the radioactive material to the sample holder such that none is lost during the operations. If significantly different from the work described in this document, each will be handled on a case by case basis.

### *Emergency Procedures*

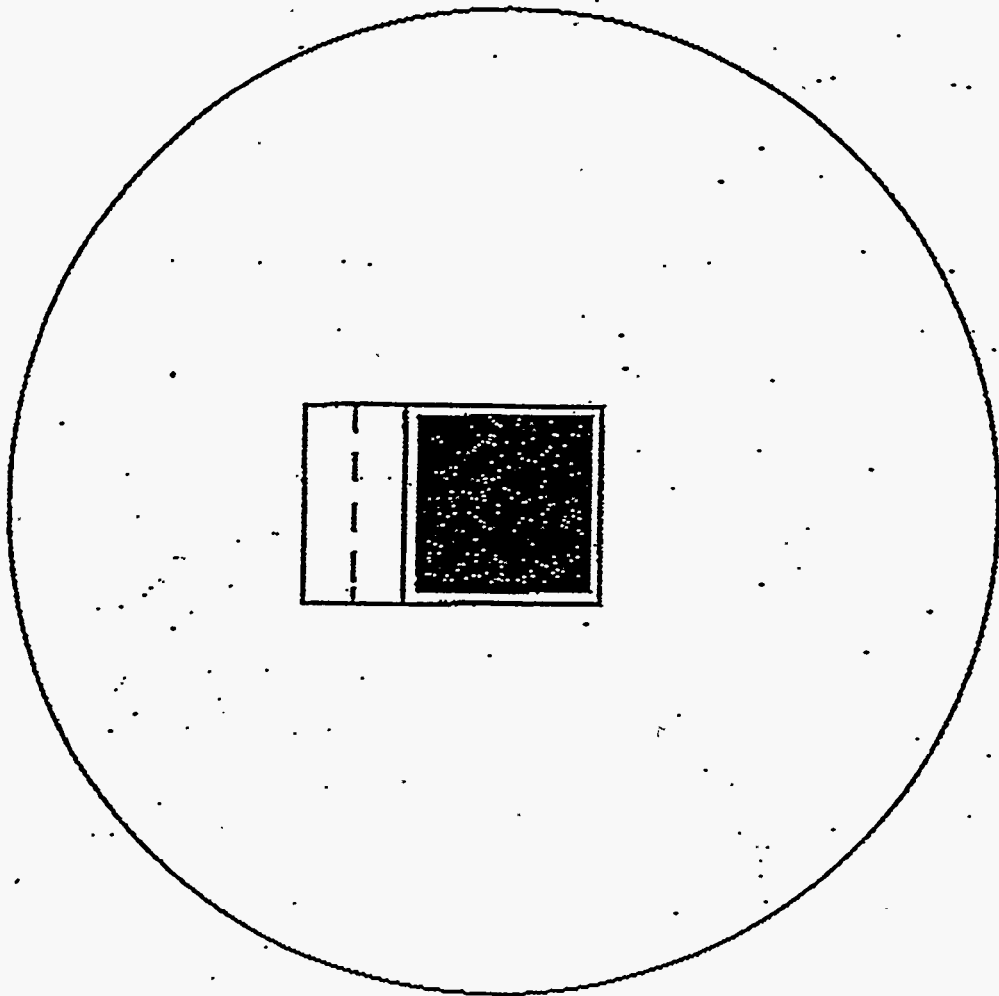
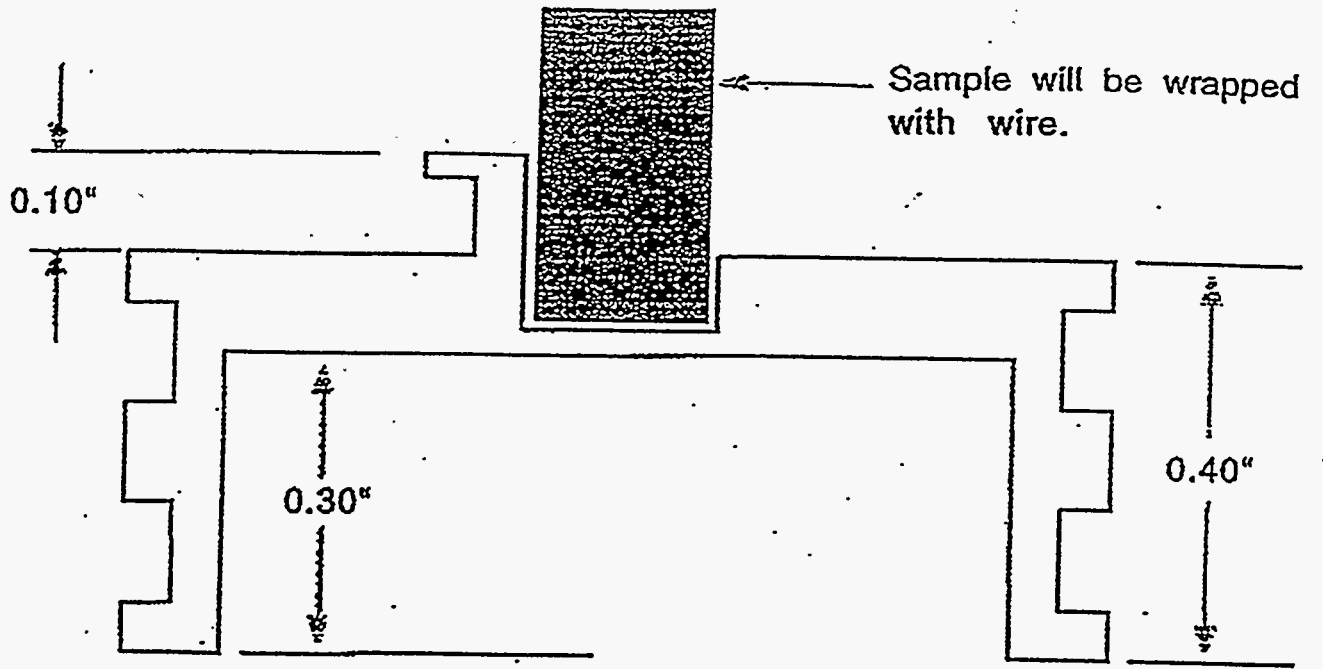
The RWA will be present on the beamline and in case of a spill or other accident involving the radioactive material, EH&S will be immediately notified. A small spill kit will be brought to the ALS.

## List of Figures

1. Diagram illustrating how the uranium ingot will be mounted on the sample holder for cleavage.
2. Example of the radiation survey form to be kept and placed in the experimental notebook.
3. General schematic of the ultraESCA endstation at Beamline 7.0 at the ALS.
4. Close-up sketch of the crystal cleaver.
5. Detailed schematic of cleaver cross used as a preparation chamber in the uranium ingot experiment.

## List of Tables

1. Summary of low activity source (LAS) parameters that determine the requirements for use of radioactive materials without an RWA.
2. Summary of the values for exemption of sealed source inventory.
3. Exerpts from a British Handbook of Laboratory Practices classifying the general hazard categories and required safety precautions for working with various radionuclides under specific laboratory conditions.
4. Table of Radioactive Isotopes information for the radionuclides to be use in the experiments.





RWA#1007 - CONTAMINATION SURVEY RESULTS - ALS BEAMLINE\_\_\_\_\_

DATE:\_\_\_\_\_ METER#:\_\_\_\_\_ SOURCE CHECK:\_\_\_\_\_ BKG:\_\_\_\_\_

LOCATION: \_\_\_\_\_ METER READING: \_\_\_\_\_

\_\_\_\_\_  
\_\_\_\_\_  
\_\_\_\_\_  
\_\_\_\_\_

SURVEYOR:\_\_\_\_\_

---

DATE:\_\_\_\_\_ METER#:\_\_\_\_\_ SOURCE CHECK:\_\_\_\_\_ BKG:\_\_\_\_\_

LOCATION: \_\_\_\_\_ METER READING: \_\_\_\_\_

\_\_\_\_\_  
\_\_\_\_\_  
\_\_\_\_\_  
\_\_\_\_\_

SURVEYOR:\_\_\_\_\_

---

DATE:\_\_\_\_\_ METER#:\_\_\_\_\_ SOURCE CHECK:\_\_\_\_\_ BKG:\_\_\_\_\_

LOCATION: \_\_\_\_\_ METER READING: \_\_\_\_\_

\_\_\_\_\_  
\_\_\_\_\_  
\_\_\_\_\_  
\_\_\_\_\_

SURVEYOR:\_\_\_\_\_

---

DATE:\_\_\_\_\_ METER#:\_\_\_\_\_ SOURCE CHECK:\_\_\_\_\_ BKG:\_\_\_\_\_

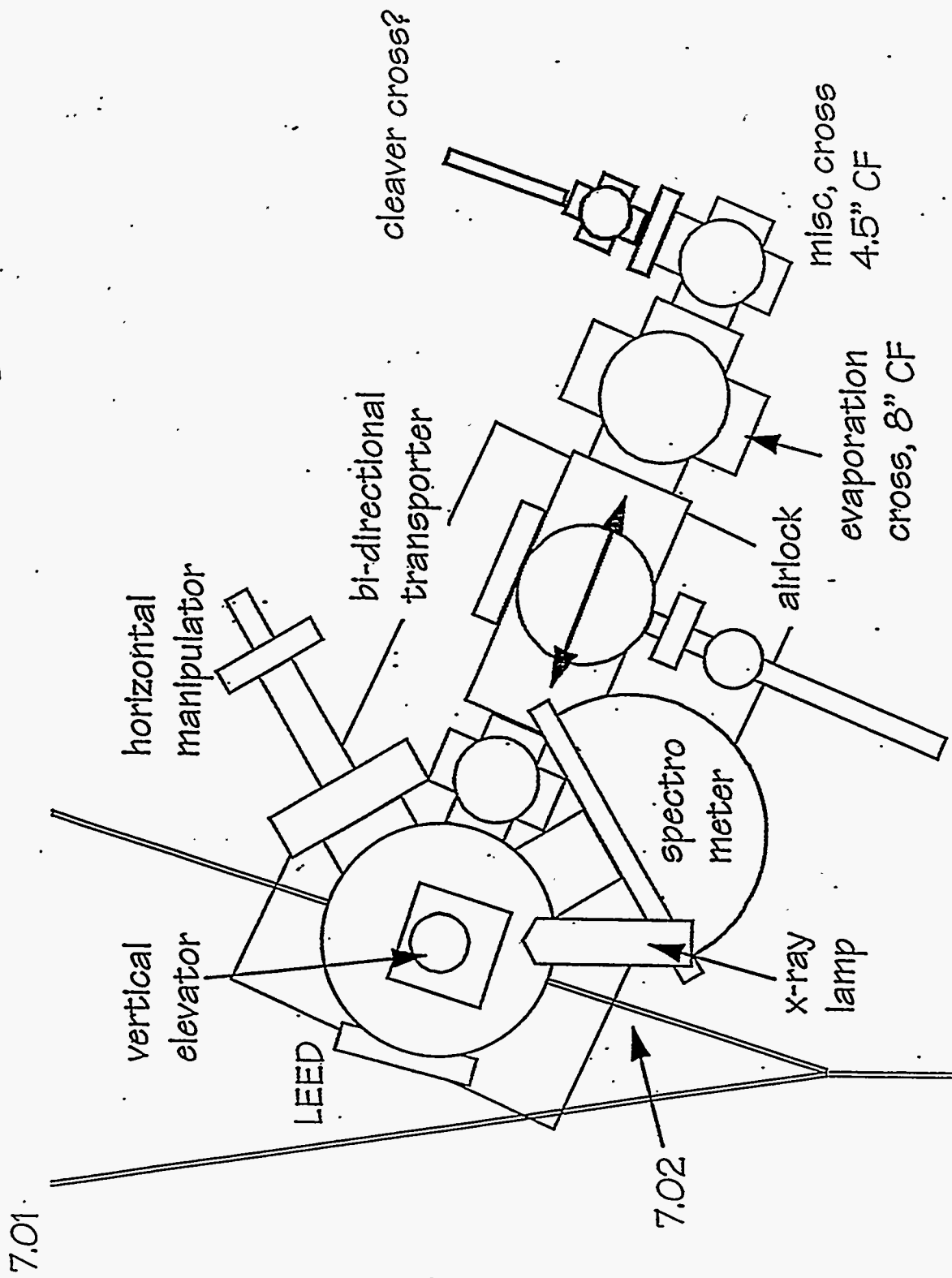
LOCATION: \_\_\_\_\_ METER READING: \_\_\_\_\_

\_\_\_\_\_  
\_\_\_\_\_  
\_\_\_\_\_  
\_\_\_\_\_

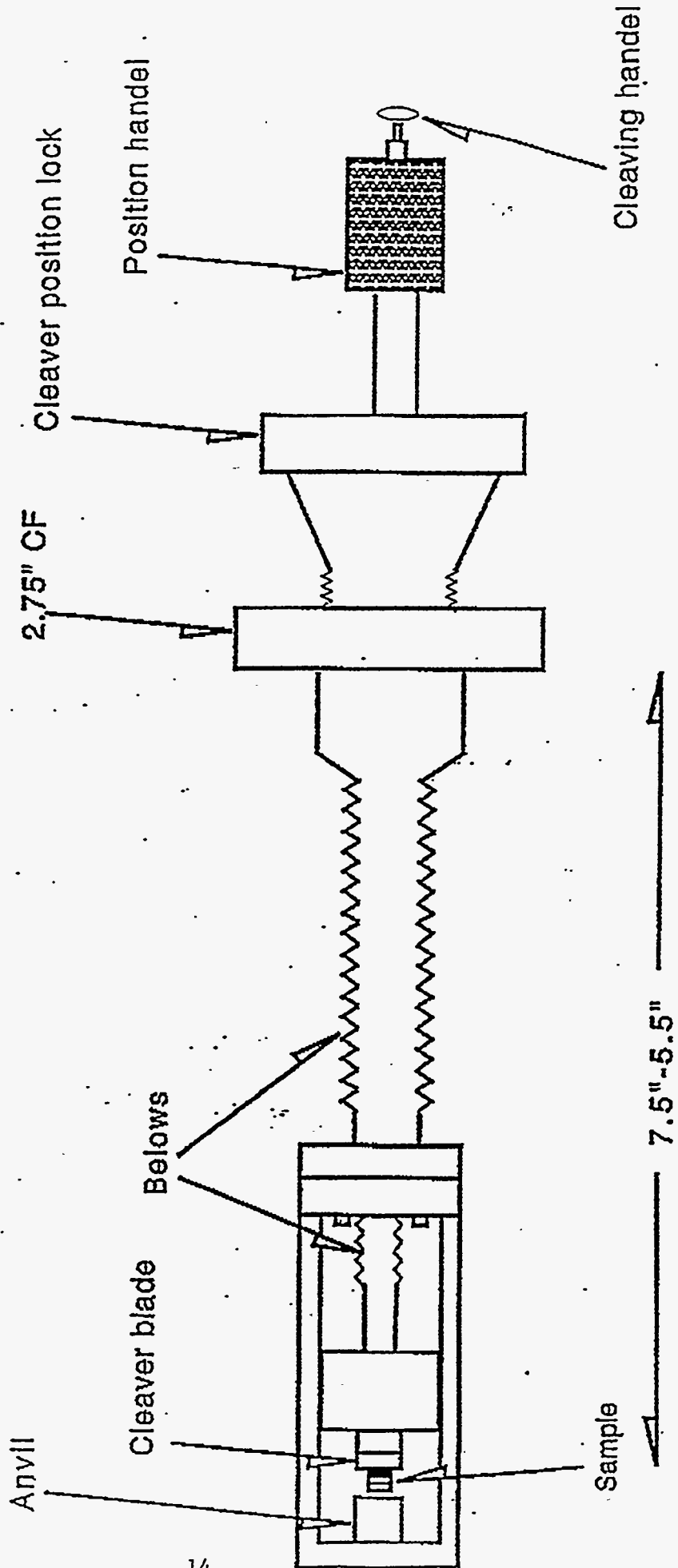
SURVEYOR:\_\_\_\_\_

---

# ultraESCA UHV Layout



# Sketch of the Cleaver



# J. Allen Cleaver Cross

11/27/95

Cleaver

2.75" x 1" DS flange

6"-2.75" reducer flange

Viewports

TOP

XYZ manipulator:

6-2.75" reducer nipple

MDC XY stage

2" linear motion feedthru

Viewports

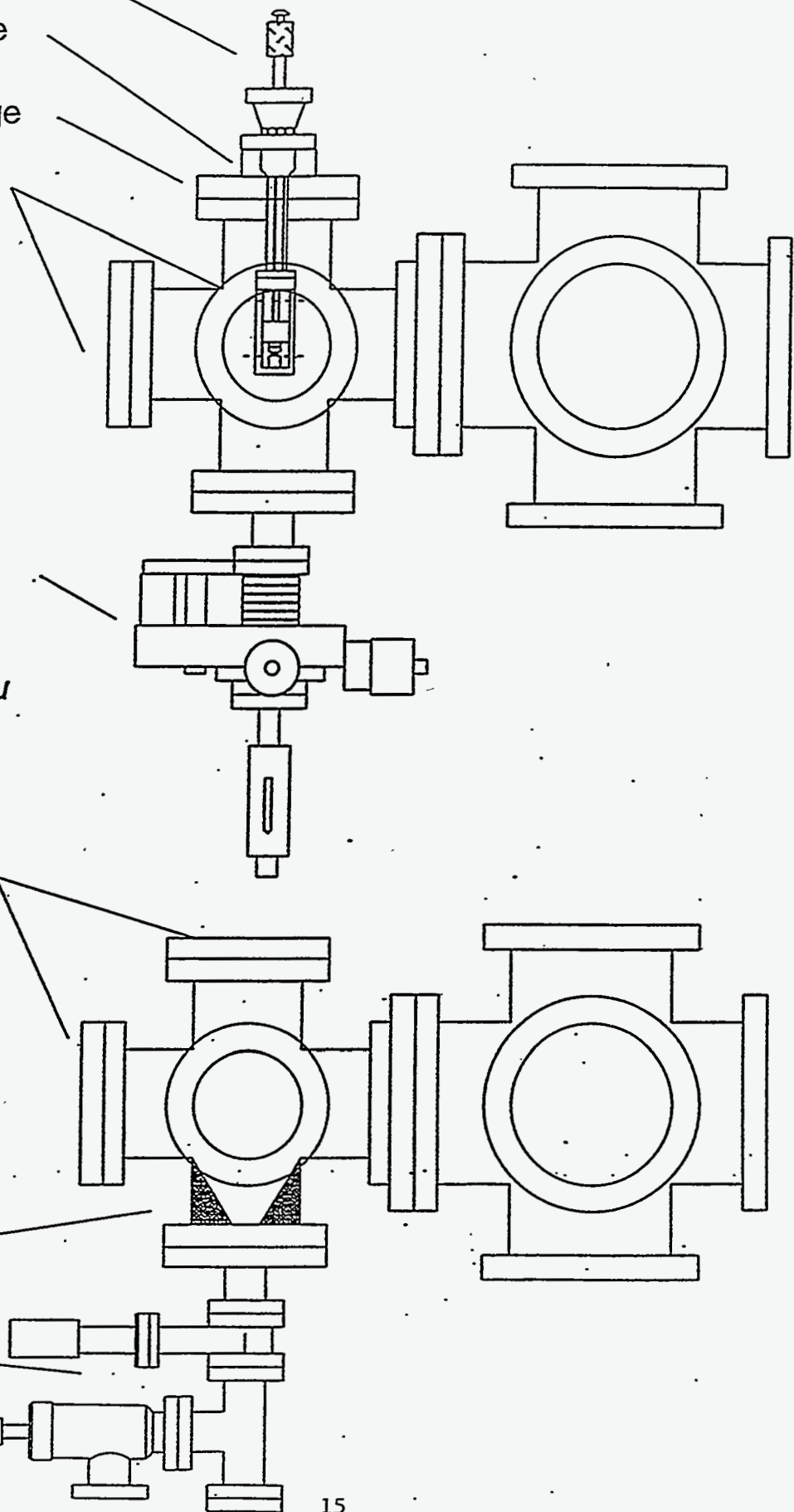
RIGHT SIDE

chip funnel

2.75" gate valve

chip catcher

angle valve



LESS THAN 10 samples in possession, SINGLE PROJECT  
REQUIRES "LAS" → NO RWA REQUIRED

Low Activity Source (LAS) Quantities

Less than 30  $\mu\text{Ci}$  ( $1 \times 10^7$  Bq)

H-3	Be-7	C-14	S-35	Ca-41	Ca-45	V-49	Mn-53
Ge-55	Ni-59	Ni-63	As-73	Se-79	Rb-87	Tc-99	Pd-107
Cd-113	In-115	Te-123	Cs-135	Ce-141	Gd-152	Tb-157	Ir-171
Ta-180	W-181	W-185	W-188	RE-187	Tl-204		

Less than 3  $\mu\text{Ci}$  ( $1 \times 10^5$  Bq)

P-32	P-33	Cl-36	K-40	Fe-59	Co-57	Se-75	Rb-84
Sr-85	Sr-89	Y-91	Zr-95	Nb-93m	Nb-95	Tc-97m	Ru-103
Ag-105	In-114m	Sn-113	Sn-119m	Sn-121m	Sn-123	Te-123m	Te-125m
Te-127m	Te-129m	I-125	La-137	Ce-139	Pm-143	Pm-145	Pm-147
Sm-145	Sm-151	Eu-149	Eu-155	Gd-151	Gd-153	Dy-159	Tm-170
Yb-169	Lu-173	Lu-174	Lu-174m	Hf-175	Hf-181	Ta-179	Re-184
Re-186m	Ir-192	Pt-193	Au-195	Hg-203	Pb-205	Np-235	Pu-237

Less than 300 nCi ( $1 \times 10^4$  Bq)

Be-10	Na-22	Al-26	Si-32	Sc-46	Ti-44	Mn-54	Fe-60
Co-56	Co-58	Co-60	Zn-65	Ge-68	Rb-83	Y-88	Zr-88
Zr-93	Nb-94	Mq-93	Tc-95m	Tc-97	Tc-98	Ru-106	Rh-101
Rh-102	Rh-102m	Ag-108m	Ag-110m	Cd-109	Sn-126	Sb-124	Sb-125
Te-121m	I-129	Cs-134	Cs-137	Ba-133	Ce-144	Pm-144	Pm-146
Pm-148m	Eu-148	Eu-150	Eu-152	Eu-154	Gd-146	Tb-158	Tb-160
Ho-166m	Lu-176	Lu-177	Hf-172	Ta-182	Re-184m	Os-185	Os-194
Ir-192m	Ir-194m	Hg-194	Pb-202	Bi-207	Bi-210m	Cm-241	

Less than 30 nCi ( $1 \times 10^3$  Bq)

Sr-90	Cd-113m	La-138	Hf-178m	Hf-182	Po-210	Ra-226	Ra-228
Pu-241	Bk-249	Es-254					

Less than 3 nCi ( $1 \times 10^2$  Bq)

Sm-146	Sm-147	Pb-210	Np-236	Cm-242	Cf-248	Fm-257	Md-258
--------	--------	--------	--------	--------	--------	--------	--------

Less than .3 nCi ( $1 \times 10^1$  Bq)

Gd-148	Th-228	Th-230	U-232	U-233	U-234	U-235	U-236
U-238	Np-237	Pu-236	Pu-238	Pu-239	Pu-240	Pu-242	Pu-244
Am-241	Am-242m	Am-243	Cm-243	Cm-244	Cm-245	Cm-246	Cm-247
	Bk-247	Cf-249	Cf-250	Cf-251	Cf-252	Cf-254	

Less than 30 pCi (1 Bq)

Ac-227	Th-229	Th-232	Pa-231	Cm-248	Cm-250
--------	--------	--------	--------	--------	--------

TABLE 1.

Values for exemption of sealed sources from inventory\*

Less than 300 $\mu\text{Ci}$ ( $1 \times 10^7$ Bq)							
H-3	Be-7	C-14	S-35	Ca-41	Ca-45	V-49	Mn-53
Fe-55	Ni-59	Ni-63	As-73	Se-79	Rb-87	Tc-99	Pd-107
Cd-113	In-115	Te-123	Cs-135	Ce-141	Gd-152	Tb-157	Tm-171
Ta-180	W-181	W-185	W-188	Re-187	Tl-204		
Less than 30 $\mu\text{Ci}$ ( $1 \times 10^6$ Bq)							
Cl-36	K-40	Fe-59	Co-57	Se-75	Rb-84	Sr-85	Sr-89
Y-91	Zr-95	Nb-93m	Nb-95	Tc-97m	Ru-103	Ag-105	In-114m
Sn-113	Sn-119m	Sn-121m	Sn-123	Te-123m	Te-125m	Te-127m	Te-129m
I-125	La-137	Ce-139	Pm-143	Pm-145	Pm-147	Sm-145	Sm-151
Eu-149	Eu-155	Gd-151	Gd-153	Dy-159	Tm-170	Yb-169	Lu-173
Lu-174	Lu-174m	Hf-175	Hf-181	Ta-179	Re-184	Re-186m	Ir-192
Pt-193	Au-195	Hg-203	Pb-205	Np-235	Pu-237		
Less than 3 $\mu\text{Ci}$ ( $1 \times 10^5$ Bq)							
Be-10	Na-22	Al-26	Si-32	Sc-46	Ti-44	Mn-54	Fe-60
Co-56	Co-58	Co-60	Zn-65	Ge-68	Rb-83	Y-88	Zr-88
Zr-93	Nb-94	Mo-93	Tc-95m	Tc-97	Tc-98	Ru-106	Rh-101
Rh-102	Rh-102m	Ag-108m	Ag-110m	Cd-109	Sn-126	Sb-124	Sb-125
Te-121m	I-129	Cs-134	Cs-137	Ba-133	Ce-144	Pm-144	Pm-146
Pm-148m	Eu-148	Eu-150	Eu-152	Eu-154	Gd-146	Tb-158	Tb-160
Ho-166m	Lu-176	Lu-177m	Hf-172	Ta-182	Re-184m	Os-185	Os-194
Ir-192m	Ir-194m	Hg-194	Pb-202	Bi-207	Bi-210m	Cm-241	
Less than 0.3 $\mu\text{Ci}$ ( $1 \times 10^4$ Bq)							
Sr-90	Cd-113m	La-138	Hf-178m	Hf-182	Po-210	Ra-226	Ra-228
Pu-241	Bk-249	Es-254					
Less than 0.03 $\mu\text{Ci}$ ( $1 \times 10^3$ Bq)							
Sm-146	Sm-147	Pb-210	Np-236	Cm-242	Cf-248	Fm-257	Md-258
Less than 0.003 $\mu\text{Ci}$ ( $1 \times 10^2$ Bq)							
Gd-148	Th-228	Th-230	U-232	U-233	U-234	U-235	U-236
<del>U-238</del>	Np-237	Pu-236	Pu-238	Pu-239	Pu-240	Pu-242	Pu-244
Am-241	Am-242m	Am-243	Cm-243	Cm-244	Cm-245	Cm-246	Cm-247
	Bk-247	Cf-249	Cf-250	Cf-251	Cf-252	Cf-254	
Less than 0.0003 $\mu\text{Ci}$ ( $1 \times 10^1$ Bq)							
Ac-227	Th-229	Th-232	Pa-231	Cm-248	Cm-250		

\* These activities were selected to yield a committed effective dose equivalent of 10 mrem (100  $\mu\text{Sv}$ ) or less for a credible incident to a member of the general public.

Table II.1.1 Classification of Workplaces (Continued)

(From International Labor Office Guidelines for the Radiation Protection of Workers in Industry (Ionizing Radiations)  
Occupational Safety and Health Series 62 (International Labour Organization 1989))

---

*Type II Workplace*

1. A type II (type B) workplace should be specifically designed, constructed and equipped for work with radioisotopes.
2. The levels of airborne activity should be kept as low as reasonably achievable by the use of totally or partially ventilated fume hoods or glove boxes.
3. The workplace should have reduced air pressure relative to the surrounding areas. The ventilation exhaust should be via a fume hood. There should be a space for an absolute filter to be put between the fume hood and the ventilation duct allowing for easy change of the filter and for monitoring the negative pressure gradient. Special attention should be given to avoiding the recirculation of air and the dispersion of contamination to other occupied areas.
4. The surfaces of the fume hood and the ventilation duct should be smooth and made of non-absorbent material that can withstand the chemicals normally used in the hood.
5. The speed of the air flow should be regular, without eddies, and should be such that there can be no escape of air from the fume hood into the workplace under typical operating conditions, including the opening of windows and doors and the suction of other fume hoods. This should be checked using smoke tests. The gas, water and electrical outputs should be operated from outside the hood.
6. Fume hoods and glove boxes where "active" work is carried out should be properly marked with the radiation symbol and the appropriate explanatory text.
7. A waste bin with a foot-operated lid should be available for the collection of low activity waste. The bin should bear the radiation warning sign. A plastic bottle which could withstand the effects of the various solvents and the effects of radiation should be provided for the temporary retention of liquid waste.
8. Facilities for washing hands should be foot or elbow operated.
9. A special room should be provided for storing radioactive substances.

*Type III Workplace*

1. A type III (type A) workplace should be specifically designed, constructed and equipped for handling large quantities of radioactive material in accordance with the specifications and requirements laid down by the competent authority.
  2. Processes involving risks of air contamination should be carried out in completely enclosed glove boxes or hot cells under negative pressure and provided with filters and transfer boxes.
  3. Radioactive substances should be stored only in a special room equipped with suitable shielding and ventilation, and in accordance with the provisions as regards waste storage.
-

Table II.L.L.I Toxicity Classification of Radionuclides<sup>a</sup>  
(From International Labor Office Guidelines for the Radiation Protection of Workers in Industry (Ionizing Radiations)  
Occupational Safety and Health Series 62 ©International Labour Organization 1989)

Very High Radiotoxicity (Group 1)

210Pb	228Ra	229Th	232U	236Pu	241Pu	243Am	244Cm	248Cm	251Cf
210Po	227Ac	230Th	233U	238Pu	242Pu	240Cm	245Cm	248Cf	252Cf
223Ra	227Th	231Pa	234U	239Pu	241Am	242Cm	246Cm	249Cf	254Cf
225Ra	228Th	230U	237Np	240Pu	242mAm	243Cm	247Cm	250Cf	254Es
226Ra									

High Radiotoxicity (Group 2)

22Na	90Sr	110mAg	124I	140Ba	170Tm	212Pb	228Ac	244Pu	253Cf
36Cl	91Y	115mCd	125I	144Ce	181Hf	207Bi	232Th	242Am	253Es
45Ca	93Zr	114mIn	126I	152Eu	182Ta	210Bi	Th Nat <sup>b</sup>	241Cm	254mEs
46Sc	94Nb	124Sb	131I	154Eu	192Ir	211At	230Pa	249Bk	255Fm
60Co	106Ru	125Sb	134Cs	160Tb	204Tl	224Ra	236U	246Cf	256Fm

Moderate Radiotoxicity (Group 3)

7Be	52Fe	82Br	97Zr	105Ag	134Te	143Ce	171Tm	198Au	237U
14C	55Fe	74Kr	90Nb	111Ag	120I	142Pr	175Yb	199Au	240U
18F	59Fe	77Kr	93mNb	109Cd	123I	143Pr	177Lu	197Hg	240Np
24Na	55Co	81Kr	95Nb	115Cd	130I	147Nd	181W	197mHg	239Np
31Si	56Co	88Kr	96Nb	115mIn	132mI	149Nd	185W	203Hg	234Pu
32P	57Co	86Rb	90Mo	113Sn	133I	147Pm	187W	200Tl	237Pu
33P	58Co	83Sr	93Mo	125Sn	135I	149Pm	183Re	201Tl	245Pu
35S	63Ni	85Sr	99Mo	122Sb	135Xe	151Sm	186Re	202Tl	238Am
36Cl	65Ni	89Sr	96Tc	121Te	132Cs	153Sm	188Re	203Pb	240Am
41Ar	65Cu	91Sr	97mTc	121mTe	136Cs	152mEu	185Os	206Bi	244mAm
42K	67Zn	92Sr	97Tc	123mTe	137Cs	155Eu	191Os	212Bi	244Am
42K	69mZn	90Y	99Tc	125mTe	131Ba	153Gd	193Os	220Rn	238Cm
47Ca	72Ga	92Y	97Ru	127mTe	140La	159Gd	190Ir	222Rn	250Bk
48Sc	73As	93Y	103Ru	129mTe	134Ce	165Dy	194Ir	226Th	244Cf
48Sc	74As	86Zr	105Ru	131Te	135Ce	166Dy	191Pt	231Th	254Fm
48V	76As	88Zr	105Rh	131mTe	137mCe	166Ho	193Pt	234Th	
51Cr	77As	89Zr	103Pd	132Te	139Ce	169Er	197Pt	233Pa	
52Mn	75Se	95Zr	109Pd	133mTe	141Ce	171Er	196Au	231U	
54Mn									

Low Radiotoxicity (Group 4)

1H	60mCo	81Kr	91mY	99mTc	120mI	127Cs	138Cs	207Po	243Pu
16O	61Co	83mKr	88Nb	103mRh	121I	129Cs	137Ce	227Ra	237Am
37Ar	62mCo	85mKr	89Nb	113mIn	128I	130Cs	191mOs	235U	239Am
51Mn	59Ni	85Kr	97Nb	116Te	129I	131Cs	193mPt	238U	245Am
52mMn	69Zn	80Sr	98Nb	123Te	134I	134mCs	197mPt	239U	246mAm
53Mn	71Ge	81Sr	93mMo	127Te	131mXe	135Cs	203Po	U NAT	246Am
54Mn	76Kr	85mSr	101Mo	129Te	133Xe	135mCs	205Po	350Pu	249Cm
54mCo	79Kr	87mSr	96mTc	133Te	125Cs				

<sup>a</sup> Based on the classification published in the Official Journal of the European Communities, No. L246, Vol. 23; Luxembourg; 17 Sep. 1980.

<sup>b</sup> One becquerel of natural thorium corresponds to 1 alpha disintegration per second (dps) (0.5 dps of <sup>232</sup>Th and 0.5 dps of <sup>228</sup>Th). One curie of natural thorium corresponds to 3.7 x 10<sup>10</sup> alpha disintegrations per second (3.7 x 10<sup>10</sup> dps of <sup>232</sup>Th and 1.85 x 10<sup>10</sup> dps of <sup>228</sup>Th).



Table II.L.1.2 Activity Limits for Use of Radionuclides in Various Types of Workplace<sup>a</sup>  
 (From International Labor Office *Guidelines for the Radiation Protection of Workers in Industry (Ionizing Radiations)*  
 Occupational Safety and Health Series 62 ©International Labour Organization 1989)

Radionuclide Group	Type of Workplace		
	Type I	Type II	Type II
1. Very high	500 K Bq or less $< 15 \mu\text{Ci}$	500 K Bq-500 M Bq	500 M Bq or more
2. High	5 M Bq or less $< 150 \mu\text{Ci}$	5 M Bq-5 G Bq	5 G Bq or more
3. Moderate	50 M Bq or less $< 15 \text{mCi}$	50 M Bq-50 G Bq	50 G Bq or more
4. Low	500 M Bq or less $< 15 \text{mCi}$	500 M Bq-500 G Bq	500 G Bq or more

<sup>a</sup> The above table provides, as precisely as the complexity of the subject will allow, a basis for assessing the type of workplace required for normal operations. According to the nature of the operations, the following modifying factors should be applied:

Operation	Modifying Factor
Storage (stock solutions)	x 100
Very simple wet operations	x 10
Normal operations	x 1
Complex wet operations with risk of spills and simple dry operations	x 0.1
Dry and dusty operations	x 0.01

**Table 11.1.1 Classification of Workplaces**

(From International Labor Office *Guidelines for the Radiation Protection of Workers in Industry (Ionizing Radiations)*  
Occupational Safety and Health Series 62 (International Labour Organization 1989))

---

*Introduction*

1. In view of the extreme diversity of processes carried out with unsealed radioactive sources and the great variety of potential risks, working areas and workshops should be classified according to the relative radiotoxicity of the radionuclides taking into account the nature of the operations and the total amount used.
2. Specialized installations should be divided into three types of workplace depending, to the extent practicable, on the factors referred to in paragraph 1 and in accordance with Table 11.1.1.1 for radiotoxicity classification. The types of workplace are commonly referred to as:
  - (a) type I workplace or type C workplace;
  - (b) type II workplace or type B workplace;
  - (c) type III workplace or type A workplace.
3. The activity limits for use of radionuclides in the various types of workplace are given in Table 11.1.1.2.
4. Workplaces of all three types should be:
  - (a) reserved exclusively for work with radioactive substances and isolated from other workplaces as far as is practicable;
  - (b) subject to classification according to the potential risks involved: normally areas where radioactive substances are used will be classified as controlled areas; however, areas where workers are not likely to receive more than three tenths of the dose limits may be either included in a controlled area or defined as supervised areas, if this is duly justified and considered more convenient.
5. A changing area should be provided at the entrances of areas where radioactive substances are prepared or used, in order to prevent contamination from being transported by persons to outside areas. The changing area should contain a foot barrier. Places for clean clothes should be left outside the barrier and protective clothing, equipment and containers for discarded, contaminated clothing should be provided on the active side of the barrier.
6. Washing facilities should be set up appropriate to the level of radioactivity present in the workplace. The wash basins should be elbow, knee or foot operated.
7. Changing areas should contain monitoring and control equipment, appropriate to the levels of radioactive materials present, to monitor the hands, feet, shoes and clothes of workers leaving controlled or supervised areas. Additional check points should be established within controlled areas when necessary, depending on the type of work being carried out.
8. Separate rooms should be assigned to different types of work when such work involves widely varying levels of activity, and in accordance with the classification of workplaces as given in this chapter. Counting apparatus should normally be placed in a separate room. The design should take into account, as far as practicable, the transfer of radioactive materials from one workplace to another, where necessary, without passing through the surrounding area.

*Type I Workplace*

1. The design, construction and equipment of a type I (type C) workplace should be similar to those of a good quality modern chemical laboratory.
  2. Normal ventilation is usually sufficient, and could be complemented with continuous movement of air into a fume hood.
-





<sup>238</sup>Pu(2.3 I min)

Mode:  $\beta^-$   
 $\Delta$ : 51270.300 keV  
SpA: 3.43  $\times 10^8$  Ci/g  
Prod: <sup>238</sup>U(n,p)

Photons (<sup>238</sup>Pa)

Table with columns:  $\gamma_{mode}$ ,  $\gamma$ (keV),  $\gamma$ (rel). Lists various photon decay modes and their energies and relative intensities for <sup>238</sup>Pa.

Photons (<sup>238</sup>Pa)  
(continued)

Table with columns:  $\gamma_{mode}$ ,  $\gamma$ (keV),  $\gamma$ (rel). Continuation of the <sup>238</sup>Pa photon data from the previous table.

Photons (<sup>238</sup>Pa)  
(continued)

Table with columns:  $\gamma_{mode}$ ,  $\gamma$ (keV),  $\gamma$ (rel). Continuation of the <sup>238</sup>Pa photon data.

\* combined intensity for doublet  
† 373 $\gamma$  + 375 $\gamma$  + 377 $\gamma$

<sup>238</sup>U (4.468 5  $\times 10^9$  yr)

Mode:  $\alpha$   
 $\Delta$ : 47306.021 keV  
SpA: Ci/g  
Prod: natural source  
%: 99.2745 IS

Alpha Particles (<sup>238</sup>U)

$\langle\alpha\rangle$ =4194.5 keV

Table with columns:  $\alpha$ (keV),  $\alpha$ (%). Lists alpha particle energy and percentage for <sup>238</sup>U.

Photons (<sup>238</sup>U)  
 $\langle\gamma\rangle$ =1.30 IS keV

Table with columns:  $\gamma_{mode}$ ,  $\gamma$ (keV),  $\gamma$ (%). Lists photon decay modes and their energies and relative intensities for <sup>238</sup>U.

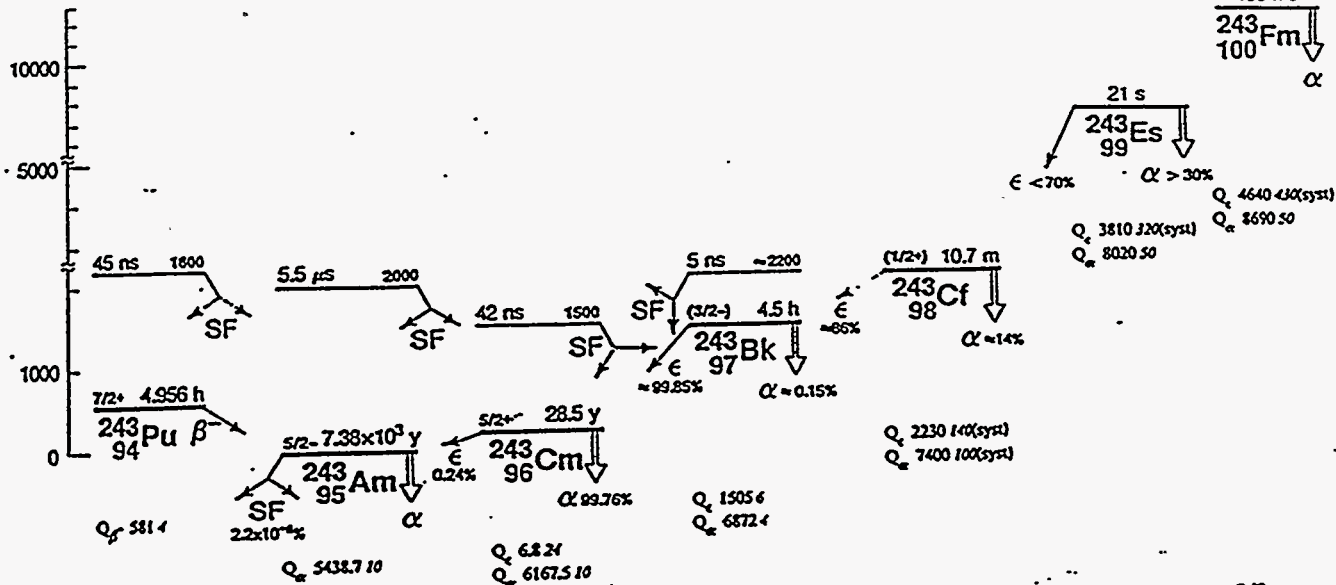
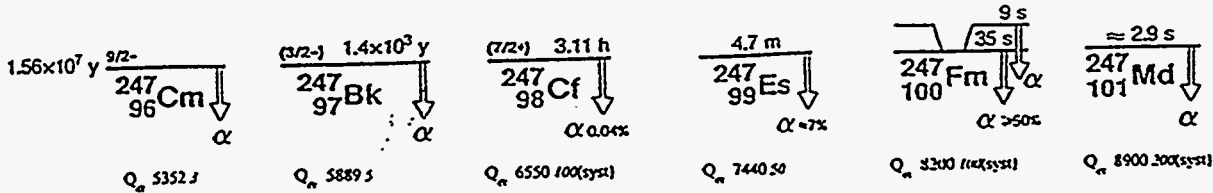
Atomic Electrons (<sup>238</sup>U)

$\langle e\rangle$ = 9.57 keV

Table with columns:  $e_{bin}$ (keV),  $\langle e\rangle$ (keV),  $e$ (%). Lists atomic electron binding energies and their contributions to the average energy for <sup>238</sup>U.







**$^{243}_{94}\text{Pu}$  (4.956 h)**

Mode:  $\beta^-$   
 $\Delta$ : 57751  $\pm$  keV  
 SpA: 2.602x10<sup>6</sup> Ci/g  
 Prod:  $^{242}\text{Pu}(n,\gamma)$

Photons ( $^{243}\text{Pu}$ )  
 $\langle \gamma \rangle = 26.4$  keV

$\gamma_{\text{mode}}$	$\gamma(\text{keV})$	$\gamma(\%)^{\dagger}$
Am L <sub>1</sub>	13.377	0.3715
Am L <sub>2</sub>	15.599	5.723
Am L <sub>3</sub>	16.819	0.094
Am L <sub>4</sub>	18.884	6.225
Am L <sub>5</sub>	22.290	1.54
$\gamma[\text{E1}]$	41.7514	0.767
$\gamma[\text{M1}+\sim 7.8\% \text{E2}]$	42.2022	$\sim$ 0.08
$\gamma[\text{M1+E2}]$	54.14	<0.023
$\gamma[\text{E1}]$	67.0625	$\sim$ 0.23
$\gamma[\text{E1}]$	83.9516	23.5
$\gamma[\text{E2}]$	96.34	0.013823
$\gamma[\text{E1}]$	101.33	<0.037
Am K <sub>1</sub>	102.026	0.11315
Am K <sub>2</sub>	105.472	0.17824
$\gamma[\text{E1}]$	109.2717	0.16116
Am K <sub>3</sub>	119.960	0.0669
Am K <sub>4</sub>	124.123	0.0223
$\gamma[\text{M1+E2}]$	322.1125	0.027623
$\gamma[\text{M1+E2}]$	343.25	$\sim$ 0.0014
$\gamma[\text{M1+E2}]$	356.3721	0.13112
$\gamma[\text{M1}]$	381.6821	0.553
$\gamma[\text{M1+E2}]$	388.9125	0.00467
$\gamma$	407.05	$\sim$ 0.0009
$\gamma[\text{M1+E2}]$	423.1725	0.012214
$\gamma[\text{E2}]$	448.53	$\sim$ 0.00023
$\gamma[\text{E1}]$	465.6421	<0.00023

**Atomic Electrons ( $^{243}\text{Pu}$ )**  
 $\langle e \rangle = 12.512$  keV

$e_{\text{bin}}(\text{keV})$	$\langle e \rangle(\text{keV})$	$e(\%)$
6-15	0.6	$\sim$ 6
18	0.8	4.321
19	0.6	3.215
20	1.0	$\sim$ 5
21-22	0.24	$\sim$ 1
23	1.2	5.524
24	0.40	$\sim$ 2
25-35	0.9	3.014
36	0.7	1.99
37-40	0.28	0.73
41	0.28	0.73
42-54	0.46	1.04
60	0.94	1.63
61	0.72	1.1824
62-63	0.0039	0.006225
64	0.69	1.0822
65-77	0.164	0.223
78	0.49	0.6313
79-119	0.61	0.747
197-231	0.14	$\sim$ 0.06
257	0.83	0.323
264-303	0.024	$\sim$ 0.008
316-365	0.29	0.08219
366-407	0.084	0.022214
417-466	0.0011	0.0002512

**Continuous Radiation ( $^{243}\text{Pu}$ )**  
 $\langle \beta^- \rangle = 161$  keV;  $\langle \text{IB} \rangle = 0.091$  keV

$E_{\text{bin}}(\text{keV})$	$\langle \beta^- \rangle(\text{keV})$	$\beta^-(\%)$	$\langle \text{IB} \rangle(\text{keV})$	$\text{IB}(\%)$
0-10	$\beta^-$	0.211	0.0082	4.22
10-20	$\beta^-$	0.62	0.0075	4.12
20-40	$\beta^-$	2.38	0.0131	8.0
40-100	$\beta^-$	15.0	0.028	0.046
100-300	$\beta^-$	92	0.031	21.7
300-581	$\beta^-$	51	0.0026	0.020
	IB			13.9
	IB			0.00076

**$^{243}_{95}\text{Am}$  (7380 yr)**

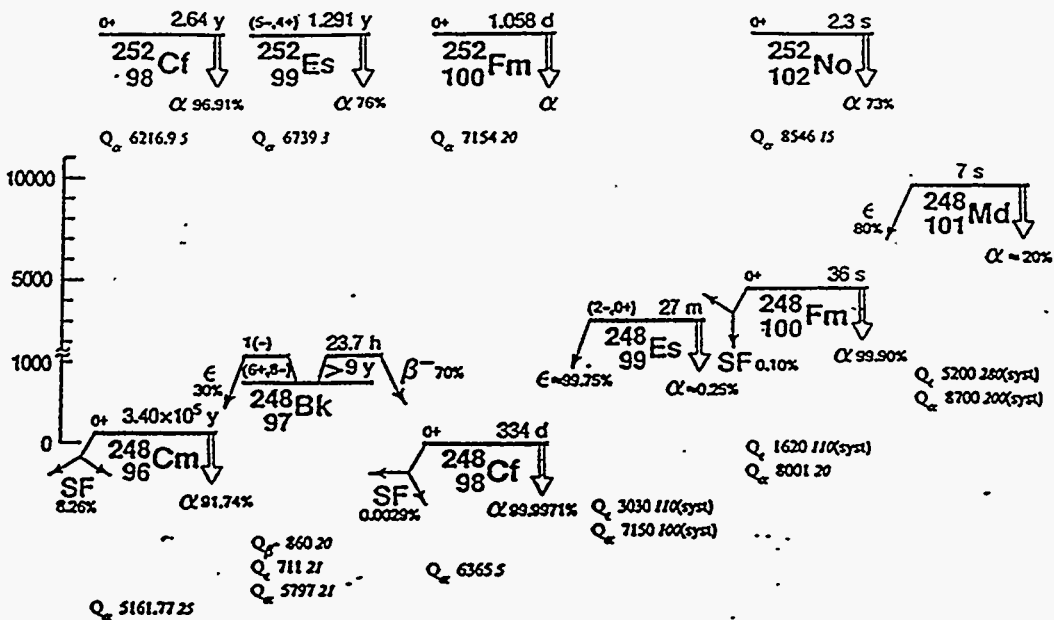
Mode:  $\alpha$ , SF (2.22x10<sup>-8</sup> %)  
 $\Delta$ : 57171  $\pm$  keV  
 SpA: 0.1993 Ci/g  
 Prod: multiple n-capture from  $^{238}\text{U}$ ;  
 multiple n-capture from  $^{239}\text{Pu}$

**Alpha Particles ( $^{243}\text{Am}$ )**

$\alpha(\text{keV})$	$\alpha(\%)$
4698.75	0.00175
4918.45	9x10 <sup>-5</sup>
4930.5	0.00018
4946.5	0.0003
4997.5	0.0016†
5008.5	







**<sup>248</sup><sub>96</sub>Cm (3.40 × 10<sup>5</sup> yr)**

Mode: α(91.74%), SF(8.26%)

Δ: 67388.5 keV

SpA: 0.00424 Ci/g

Prod: daughter <sup>252</sup>Cf  
multiple n-capture from <sup>238</sup>U;  
multiple n-capture from <sup>239</sup>Pu;  
multiple n-capture from <sup>244</sup>Cm

**Alpha Particles (<sup>248</sup>Cm)**

(α) = 4652.43 keV

α(keV)	α(%)
4776.0	<0.009
4931.1	0.070
5034.93	16.54
5078.45	75.1

**<sup>248</sup><sub>97</sub>Bk (>9 yr)**

decay not observed  
Δ: 68099.21 keV

SpA: <160 Ci/g

Prod: <sup>246</sup>Cm(α,pn)

**<sup>248</sup><sub>97</sub>Bk (23.7 h)**

Mode: β-(70%), α(30%)

Δ: 68099.21 keV

SpA: 5.33 × 10<sup>5</sup> Ci/g

Prod: <sup>247</sup>Bk(n,γ); <sup>245</sup>Cm(α,p)

**Photons (<sup>248</sup>Bk)**

(γ) = 55.6 keV

γ <sub>mode</sub>	γ(keV)	γ(%) <sup>†</sup>
Cm L <sub>γ</sub>	12.633	0.40
Cf L <sub>γ</sub>	13.146	~0.28
Cm L <sub>α</sub>	14.939	6.0
Cf L <sub>α</sub>	15.636	~4
Cm L <sub>γ</sub>	17.314	0.079
Cf L <sub>γ</sub>	18.347	~0.10
Cm L <sub>γ</sub>	19.083	5.7
Cf L <sub>γ</sub>	20.303	~5
Cm L <sub>γ</sub>	22.966	1.3
Cf L <sub>γ</sub>	24.273	~1
γ <sub>E2</sub> [E2]	41.3	~0.016
γ <sub>E2</sub>	43.399	~0.002
Cm K <sub>α2</sub>	104.586	6.2
Cm K <sub>α1</sub>	109.271	9.8
Cf K <sub>α2</sub>	109.826	0.0160
Cf K <sub>α1</sub>	115.032	0.0249
Cm K <sub>β1</sub>	123.059	3.67
Cm K <sub>β2</sub>	127.344	1.25
Cf K <sub>β1</sub>	129.436	0.0093
Cf K <sub>β2</sub>	133.949	0.00328
γ <sub>E1</sub>	550.71	5.0

<sup>†</sup> uncert(syst): 17% for α, 7.1% for β-

**Atomic Electrons (<sup>248</sup>Bk)**

(e) = 10.515 keV

e <sub>bin</sub> (keV)	(e)(keV)	e(%)
15	0.06	~0.37
16	1.5	~10
19	0.93	4.9
20	0.9	~4
21	1.7	~8
24	0.76	3.2
25	1.0	4.1
26	0.018	~0.07
35	1.0	~3
36	0.8	~2
37-39	0.20	0.52
40	0.6	~1
41-90	0.53	0.94
95-129	0.222	0.217
416	0.229	0.0552
525-551	0.0750	0.01415

**Continuous Radiation (<sup>248</sup>Bk)**

(β-) = 174 keV; (IB) = 0.52 keV

E <sub>bin</sub> (keV)	(β-)(keV)	(%)
0-10	β-	0.095
	IB	0.0085
10-20	β-	0.281
	IB	0.0095
20-40	β-	1.10
	IB	0.0151
40-100	β-	7.2
	IB	0.134
100-300	β-	53
	IB	0.32
300-600	β-	93
	IB	0.027
600-860	β-	20.4
	IB	0.00076

**EXPERIMENT RENEWAL FORM**  
(Please print or type)

**EXPERIMENT:**

Title of experiment:	Electron Spectroscopy of Actinides
LD. Number:	93-012
Beamline:	7.0
Date of Original Form/Experiment:	08 April 1994
Date of Completion of this form:	18 Jan. 1996

**EXPERIMENTER IN CHARGE:**

Name:	David Shuh
Affiliation:	LBL
Address:	70A-1147A, LBNL, Berkeley, CA
Phone:	(510) 486-6937
Local Address:	MS 70A-1150, LBNL, Berkeley, CA
Local Phone:	(510) 486-6937

**List Schedules Attached (from ALS Experiment Form):**

Schedule A and attachments

Check box if Renewal Request does not include changes: Check box if Renewal Request includes changes: 

Attach Experiment Modification Form if changes are included in renewal.

18 Jan. 1996

Signature/Experimenter-In-Charge

Date

## EXPERIMENT MODIFICATION FORM

(Please print or type)

**EXPERIMENT:**

Title of experiment:	Electron Spectroscopy of Actinides
LD. Number:	93-012
Beamline:	7.0
Date of completion of this form:	18 Jan. 1996

**EXPERIMENTER-IN-CHARGE:**

Name:	David Shuh
Office Phone:	(510) 486-6937

List modifications to the experiment (completed by Experimenter In Charge) (Operations Coordinator will determine type of change)	Type of Change																					
	Minor	Significant																				
Cleaver installed in special prep chamber to permit cleavage of sample ingot.																						
Four samples allowed on the ALS floor at any one time.																						
Two samples permitted in the vacuum chamber simultaneously.																						
Increased amount of sample materials permitted for experiments:																						
<table border="1" style="width: 100%; border-collapse: collapse; margin: 0 auto;"> <thead> <tr> <th style="width: 15%;"></th> <th style="width: 15%;">0.5% limit</th> <th style="width: 15%;">SpA</th> <th style="width: 15%;">Allowed Mass</th> </tr> </thead> <tbody> <tr> <td>238 uranium</td> <td>75 <math>\mu</math>Ci</td> <td><math>3.33 \times 10^{-7}</math> Ci/g</td> <td>~2 g</td> </tr> <tr> <td>237 neptunium</td> <td>75 nCi</td> <td><math>7.05 \times 10^{-4}</math> Ci/g</td> <td>100 <math>\mu</math>g</td> </tr> <tr> <td>242 plutonium</td> <td>75 nCi</td> <td><math>3.926 \times 10^{-3}</math> Ci/g</td> <td>19 <math>\mu</math>g</td> </tr> <tr> <td>248 curium</td> <td>75 nCi</td> <td><math>4.24 \times 10^{-3}</math> Ci/g</td> <td>17 <math>\mu</math>g</td> </tr> </tbody> </table>		0.5% limit	SpA	Allowed Mass	238 uranium	75 $\mu$ Ci	$3.33 \times 10^{-7}$ Ci/g	~2 g	237 neptunium	75 nCi	$7.05 \times 10^{-4}$ Ci/g	100 $\mu$ g	242 plutonium	75 nCi	$3.926 \times 10^{-3}$ Ci/g	19 $\mu$ g	248 curium	75 nCi	$4.24 \times 10^{-3}$ Ci/g	17 $\mu$ g		
	0.5% limit	SpA	Allowed Mass																			
238 uranium	75 $\mu$ Ci	$3.33 \times 10^{-7}$ Ci/g	~2 g																			
237 neptunium	75 nCi	$7.05 \times 10^{-4}$ Ci/g	100 $\mu$ g																			
242 plutonium	75 nCi	$3.926 \times 10^{-3}$ Ci/g	19 $\mu$ g																			
248 curium	75 nCi	$4.24 \times 10^{-3}$ Ci/g	17 $\mu$ g																			
Addition of three new elements:																						
<table border="1" style="width: 100%; border-collapse: collapse; margin: 0 auto;"> <thead> <tr> <th style="width: 15%;"></th> <th style="width: 15%;">0.5% limit</th> <th style="width: 15%;">SpA</th> <th style="width: 15%;">Allowed Mass</th> </tr> </thead> <tbody> <tr> <td>99 technetium</td> <td>15 <math>\mu</math>Ci</td> <td><math>1.7 \times 10^{-2}</math> Ci/g</td> <td>440 <math>\mu</math>g</td> </tr> <tr> <td>243 americium</td> <td>75 nCi</td> <td>0.199 Ci/g</td> <td>375 ng</td> </tr> <tr> <td>232 thorium</td> <td>750 nCi</td> <td><math>1.1 \times 10^{-1}</math> Ci/g</td> <td>145 mg</td> </tr> </tbody> </table>		0.5% limit	SpA	Allowed Mass	99 technetium	15 $\mu$ Ci	$1.7 \times 10^{-2}$ Ci/g	440 $\mu$ g	243 americium	75 nCi	0.199 Ci/g	375 ng	232 thorium	750 nCi	$1.1 \times 10^{-1}$ Ci/g	145 mg						
	0.5% limit	SpA	Allowed Mass																			
99 technetium	15 $\mu$ Ci	$1.7 \times 10^{-2}$ Ci/g	440 $\mu$ g																			
243 americium	75 nCi	0.199 Ci/g	375 ng																			
232 thorium	750 nCi	$1.1 \times 10^{-1}$ Ci/g	145 mg																			
Schedules from ALS Experiment Form Attached: Schedules A and attachment																						

18 Jan. 1996

\_\_\_\_\_  
Signature/Experimenter-in-Charge

\_\_\_\_\_  
Date

OR

\_\_\_\_\_  
Approval/Operations Coordinator

\_\_\_\_\_  
Date

\_\_\_\_\_  
Approval/ALS EH&S Program Manager  
or Designee

\_\_\_\_\_  
Date

Substance (Include samples and CAS No.—if applicable)	Radio Active	Cryogenic*	Flamm.	Corrosive	Carcinogenic	Total Volume	Quantity Required on Floor	State (gas, solid, or liquid)	Point of Discharge (air, water waste, none)
uranium -238	yes	NO	NO	NO	NO	214cc	25g	SOLID	NONE
neptunium -237	yes	NO	NO	NO	NO	140 <sup>5</sup> cc	200mg	SOLID	NONE
plutonium -242	yes	NO	NO	NO	NO	}	38mg	SOLID	NONE
cerium -248	yes	NO	NO	NO	NO		35mg	SOLID	NONE
americium -243	yes	NO	NO	NO	NO	↓	44mg	SOLID	NONE
technetium -99	yes	NO	NO	NO	NO		810 <sup>5</sup> cc	820mg	SOLID
thorium -232	yes	NO	NO	NO	NO	140 <sup>2</sup> cc	145mg	SOLID	NONE

\* Cryogenic systems can be potential pressure hazards. Therefore, the design of cryogenic systems must be reviewed by a qualified LBL mechanical engineer. Precautions when handling cryogenics are described in Chapter 7 of PUB-3000, with additional information found in Chapter 30.

[Schedule A: Materials (Cont.)]

LD Number:

**Addendum to the ALS Experimental Safety Form  
Renewal for Cm Microspot Experiments**

Jerry Bucher, Actinide Chemistry Group, CSD, LBL, (510) 486-4484  
Norman Edelstein, Actinide Chemistry Group, CSD, LBL, (510) 486-5624  
David Shuh, Actinide Chemistry Group, CSD, LBL, (510) 486-6937

02 Nov. 1994

This is an addendum to the ALS Experimental Safety Form Renewal for the continuation of curium microspot experiments on beamlines 7.0 and 10.3.1. There is the addition of the low activity isotopes  $^{238}\text{U}$ ,  $^{237}\text{Np}$ , and  $^{242}\text{Pu}$  to the list of permissible sample materials. There will also be continued use of  $^{248}\text{Cm}$ . These materials are alpha-emitters with negligible gamma fields.

The samples to be examined immediately will be  $^{248}\text{Cm}$  to complete work on curium and the initial investigation of one or more of the other radionuclides, time permitting. The preliminary date for the next run of these sample materials has been tentatively scheduled on 15-18 Nov. 1994 with beamline 7.0 personnel. All procedures requiring EH&S assistance will be scheduled as far in advance as experimentally feasible.

The previous addendum to the original ALS Experimental Safety Form, an example of the radiation survey logsheet, the previously approved RWA (RWP), and copies of pertinent information relating to the radionuclides of interest are attached to this addendum.

## **PROCEDURES**

### ***Sample Preparation***

The sample preparation will follow the previously approved procedures. However, the initial experience gained from sample preparation and experimental work at beamline 7.0, will allow the use of even less radioactive material than before. This

safety addendum reflects material amounts used at the previous levels. All sample preparation and characterization of the activity of the samples will be done in Bldg. 70A-1129, 1145 under existing RWA procedures. The radionuclides used as sample materials will be prepared by dilution and delivered to the surface of a Pt counting disk or to a graphite disk (with a thin layer of Pt on the backside) using a microliter pipette. The resulting material will be primarily oxides of the particular radionuclide. The aqueous solvent will be removed by inductive heating. The radionuclide will be bonded to the substrate during this process as well. The samples will be observed under a microscope and the radioactivity characterized in a calibrated alpha spectrometer to determine the total activity. Each sample will be limited to a maximum of 20 nCi total activity and less material will be used when possible. The amount of material will be typically around 1µg or less. The adhesion of the radionuclide to the substrate will be determined by testing sample structures to ensure that there is no loose active material. The sample will be loaded onto the sample holder to be used on beamline 7.0 or 10.3.1 at this time. Thus, there will be no handling of the sample on the experimental floor with exception of unpacking, loading, unloading, and re-packaging to transport back to the 70A-1129,1145 laboratories. The properties of the various isotopes to be used as sample materials and the allowable (20 nCi) limits are summarized in Table I. The sample isotopes are never completely isotopically pure, thus a substantial portion of the total activity of the radionuclide sample may result from trace amounts of isotopic impurities.

## **ALS PROCEDURES**

Only one sample at a time will be brought to the ALS and there will be only one sample resident on the ALS floor at any time. Sample identification and the results of the alpha spectroscopy (total activity) will be provided to the ALS EH&S monitor, as well as to ALS control room and operations personnel when the sample is brought to the ALS.

The sample will be packaged and removed from the preparation laboratories in conjunction with EH&S, as per standard operating procedures. The sample container will be labeled "CAUTION-Radioactive Material" to warn personnel that a radioactive source is present. The samples will be transported to the ALS, with prior notification of the ALS EH&S monitor, in accordance with EH&S regulations.

Swipes of the sample will be taken by the EH&S monitor upon placement in the chamber and after each use of a sample. The sample will be loaded into the experimental chamber with a procedure utilizing laboratory coats, gloves, alpha meter, TLD/film badges, and beta-gamma meters that will be brought to the ALS by appropriately trained/supervised personnel from the Actinide Chemistry Group. The sample chamber or endstation will be labeled "CAUTION-Radioactive Material" to warn personnel that a radioactive sample is present.

### **Beamline 7.0**

The radionuclide substrate and microsample will be affixed with spring-loaded clips or spot-welded clips to the sample holder from beamline 7.0. The sample will be loaded into the sample load lock immediately. The sample will be transferred under vacuum into the photoemission spectrometer on beamline 7.0 (Eli Rotenberg and Jonathon Denlinger, local contacts). The sample may require a brief ion bombardment to clean the surface, then the electron spectroscopy measurements will be performed.

The sample will be removed from the chamber, swipes taken of the sample transfer apparatus, and returned to 70A-1129,1145 for assay. Swipes of the vacuum chamber will be taken after the chamber is vented to atmosphere for the first convenient opportunity following the completion of these experiments. The total activity of the sample will be determined to ensure that no material has been lost. The sample will be re-counted and the results given to the ALS Safety Officers. Radiation survey logsheets will also be given to EH&S personnel. At this time, another sample may be taken to the ALS by the aforementioned procedures.

### **Beamline 10.3.1**

These experiments are in the process of being scheduled. The procedures for the microprobe beamline experiments will be the same as detailed for beamline 7.0, with the exception that the samples do not have to be placed in a vacuum chamber. Thus, the same counting, transportation, and swiping protocols will be employed. The sample will be brought to the ALS in a closed container already mounted on the microprobe sample holder contained within multiply sealed 0.002" polyethylene bags or other multiply-contained sample holder.