Procedures for Radioactive User Samples at Diamond Light Source

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1. SUMMARY OF IMPORTANT DEADLINES AND TIME CONSTRAINTS

The intention to use a radioactive sample must be initially declared on the Experimental Proposal. Detailed information requested by Diamond Health Physics Team (see Section 4) must be provided as soon as possible, and at least **1 month** before the experiment starts. If all required information has not been provided within **2 weeks** of the experiment start date, Diamond reserves the right to cancel the experiment.

Users bringing radioactive samples to Diamond in person must arrive **between 0830 and 1600 on Monday – Friday**. They should report to the User Office, who will contact the Health Physics Laboratory on their behalf (see Section 5.1). Users arriving outside these times cannot bring radioactive samples with them, samples can be sent separately to Diamond. (See section 5.1)

During Office Hours (0830 - 1700 Mondays – Friday) samples will be held in the Radio-chemical Laboratory (DR, G142) and transferred from there to the beamline**.** Outside of these times, keys to the secure stores in the Radio-chemical laboratory will be held by the Experimental Hall Co-ordinators who are authorised to supervise the transfer of the samples as required (see section 6).

Users who need to access their samples outside office hours must contact the Principal Beamline Scientist (PBS) in advance of their beamtime to let them know their access requirements, they will contact the Experimental Hall Co-ordinators if additional cover is required.

At the end of the experiment, at least one of the Users must remain at Diamond until the number and integrity of all samples has been checked by the Health Physics Team. This must take place **between 0830 and 1600 on Monday – Friday** (see Section 7).

2. DEFINITION OF RADIOACTIVE SAMPLES

Table 1 defines the minimum activity above which solid and liquid samples will be considered radioactive for the purposes of these procedures. These limits have been set by Diamond, taking into account safety considerations and regulatory requirements.

For the purposes of these procedures:
'Solid' samples include crystalline or amorphous samples and wet pastes.
'Liquid' samples include crystals suspended in a liquid.

The use of gaseous, powder or aerosol samples containing *any* quantity of radioactive isotopes is not permitted on Diamond beamlines or in the laboratories.

Sample Description	**Activity or quantity limit above which these procedures apply**
Samples containing artificial radionuclides	Sum of the activities of all samples brought to Diamond > 1 Bq
Solid samples containing natural or depleted uranium	Sum of the activities of all samples brought to Diamond > 5 Bq of ^{238}U
Liquid samples containing natural or depleted uranium	Specific activity > 20 Bq of ^{238}U per litre for at least one individual sample Or Total volume of all samples brought to Diamond > 250 ml
Solid samples containing natural thorium	Sum of the activities of all samples brought to Diamond > 1 Bq of ^{232}Th
Liquid samples containing natural thorium	Specific activity > 20 Bq of ^{232}Th per litre for at least one individual sample Or Total volume of all samples brought to Diamond > 50 ml

Table 1: Definition of samples which are considered 'radioactive' for the purpose of the present procedures.

Samples containing artificial radionuclides, natural or depleted uranium or thorium below the quantity or activity limits in Table 1 must be accompanied by a document confirming this, and stating the method used for estimation or measurement of activity (if appropriate).

3. EXPERIMENTS WITH RADIOACTIVE SAMPLES WHICH ARE ALLOWED AT DIAMOND

The following sections give guidelines on the types of experiment with radioactive samples which are authorised at Diamond.

3.1 Experiments using natural or depleted uranium or thorium

Solid or liquid samples of natural or depleted uranium or thorium can be used.

Solid samples containing natural or depleted uranium must have at least single sample containment (see Section 8) unless they are chemically stable in air, in which case no sample containment is required.

Liquid samples containing natural or depleted uranium always require double containment.

Solid samples containing natural thorium must have at least single sample containment.

Liquid samples containing natural thorium always require at least double containment.

Table 2 gives the allowed activity per sample, and the maximum total activity of all samples of a given type.

Type of sample	**Maximum activity per sample**	**Maximum total activity of all samples of a given type**
Solid samples containing natural or depleted uranium	50 kBq of ^{238}U	500 kBq of ^{238}U
Liquid samples containing natural or depleted uranium	500 Bq of ^{238}U	5 kBq of ^{238}U
Solid samples containing natural thorium	2 kBq of ^{232}Th	20 kBq of ^{232}Th
Liquid samples containing natural thorium	20 Bq of ^{232}Th	200 Bq of ^{232}Th

Table 2: Maximum activities of samples containing natural or depleted uranium or natural thorium.

3.2 Samples containing artificial radionuclides

All samples must have at least double containment (see Section 8)

3.3 Restrictions on sample modification

Experimental methods which involve modification of the physical and/or chemical state of radioactive samples are forbidden at Diamond.

Heating and pressurising of radioactive samples is normally forbidden.

Cooling can be authorised but must be detailed in the information provided to Diamond Health Physics Team prior to the start of the experiment.

4. INFORMATION REQUIRED PRIOR TO ARRIVAL AT DIAMOND

When a proposal has been accepted by Diamond, the Principal Investigator must provide the Diamond Health Physics Team with a document as part of the Experimental Risk Assessment, containing all the necessary information to allow a complete safety analysis. This must include:

- A detailed list of all samples (number of samples, isotopic composition, weight, activity of the individual isotopes and total activity, physico-chemical state). Examples of how the activity of a sample can be given are:
  - Direct measurement with calibrated gamma spectrometry
  - Estimation from measurement of mass or size
  - Estimation from sample preparation (liquid samples or wet pastes)
  - Estimation from count rate from survey monitor

- A detailed description of the sample holders / containment. This description must explain how the necessary level of containment is achieved.

- A detailed description of the experimental set-up, including how the sample holder is to be mounted on the beamline.

- A detailed description of the quantity (activity in Bq, mass, volume) and how any radioactive waste (contaminated gloves etc.) produced during the experiment will be dealt with. Diamond can dispose of a limited amount of radioactive waste under the Environment Agency's exemptions regime.

- In the case of liquid samples, a description of the decontamination procedure in case of spillage, taking into account the physico-chemical properties of the samples.

- A certificate from the Principal Investigator (PI) indicating competence of all users in the group to work with radioactive materials. To satisfy this requirement, form TDI-HP-PRC-0005 (see Appendix 3) must be completed and supplied to Diamond Health Physics before work commences. Diamond may request further written evidence of competence.

- A detailed description of the sample shielding, if required.

- If samples are to be sent to Diamond from another country within the European Community, the contact address for the competent authority in that country must be included with the information sent before the experiment starts. (see also Section 9.1)

Where possible the information above should be submitted via the UAS (User Administration System) so that it can be seen by the whole experimental team ***at least one month*** before the experiment is due to start.
Any information not entered on the UAS can be sent by e-mail to:
Healthphysics@diamond.ac.uk
or by post to:

Health Physics Team
Diamond Light Source Ltd
Harwell Science and Innovation Campus
Didcot
Oxfordshire
OX11 0DE

If the samples can only be prepared a short time before the experiment, and therefore their exact activity will only be known at that time, an approximate maximum activity estimate will be accepted in the first instance. The exact number of samples and their activities may be advised to Diamond Health Physics at a later stage, ***but in any case no later than two weeks before the experiment is due to start***. Once all details are agreed between the Principal Investigator and Diamond Health Physics Team, a document will be sent to the Principal Investigator covering specific safety rules and procedures for the experiment. All conditions set out in this document must be complied with. In particular, it is forbidden to bring any radioactive sample not specified in the document to Diamond.

The following documents must accompany the samples when they arrive at Diamond:

- An official document from the Principal Investigator's home institute, certifying the nature of the samples (mass, activity, isotopic composition, physical and chemical state). This document is required for all radioactive samples including natural and depleted uranium and thorium.

- A certificate of non-contamination for all samples requiring containment.

- Gamma spectrometry results

  - For uranium and thorium samples with activity > 1 kBq, a qualitative spectrum for every sample with identification of peaks to prove absence of other isotopes (It is understood daughters may be present).
  - For uranium and thorium samples with activity < 1 kBq, a spectrum for every sample measured at contact for at least 5 minutes, to prove low activity and absence of other isotopes (It is understood daughters may be present).

- For samples containing artificial radionuclides, a quantitative spectrum for each sample with peak identification and calculation of the activity of each isotope.
- For pure alpha or beta emitters, a spectrum measured for at least 5 minutes to prove absence of gamma emitters.

The above documents must be signed by a Radiation Protection Adviser or Radiation Protection Supervisor from the Principal Investigator's home institute.

Exceptionally, Diamond can accept that these spectrometry results are not provided. Requests for such an exception to be made must be sent, with justification, to Diamond Health Physics Team ***at least one month*** before the samples are brought to Diamond. The User will be informed by e-mail as to whether this is acceptable for their particular samples.

Failure to provide one or more of these documents within the timescales stated will result in cancellation of the experiment, unless prior permission has been obtained from Diamond Health Physics Team.

The Health Physics Team will complete the form in appendix 5 and send this to the Experimental Hall Co-ordinators once all the information required has been received.

5. BRINGING RADIOACTIVE SAMPLES TO AND FROM DIAMOND

5.1 General

Whether the samples are delivered to Diamond by the User in person, or they are sent by post, the Health Physics Team must be informed in advance (Healthphysics@diamond.ac.uk) of the expected date and time when they will arrive. Users are responsible for ensuring that samples are packaged and transported in accordance with the relevant UK (CDG 2009) and / or international transport regulations (ADR 2009) for Class 7 dangerous goods.

Users who are bringing samples with them to Diamond must report to the User Office, who will contact the Health Physics Team on their behalf. ***This must be done between 0830 and 1600 on weekdays only***. Users arriving at Diamond outside these hours cannot bring any radioactive samples with them. In this case, the samples must be sent separately to Diamond.

When the Health Physics Team log in any samples they will contact an EHC to witness the samples and their storage location.

The Health Physics Team will perform a contamination check on the outer containment of all the samples. It should be assumed that sample containment may have been damaged or breached during transport. Health Physics will confirm that they have the most up-to-date information on the samples. Photographs of the samples will also be taken and sent to the EHCs.

All radioactive samples which are posted to Diamond should be shipped to the following address:

Health Physics Team,
Diamond Light Source Ltd,
Harwell Science and Innovation Campus,
Didcot,
Oxfordshire,
OX11 0DE

Samples which have been sent by post will (unless informed otherwise) will be stored in the Radio-chemical Laboratory.

Shipping of radioactive material by international public mail is forbidden. In the UK, the Royal Mail will not accept radioactive materials and samples that are classified as radioactive in Table 2-12 of the latest edition of the International Civil Aviation Organisations' (ICAO) Technical Instructions. If you wish to send samples by Royal Mail, it is advised to first check with their Customer Service Centre on 03456 000 606.

If a User wishes to send samples back to their home institute at the end of the experiment, rather than taking them away in person, they must have made the necessary arrangements with an approved transport company beforehand. If no such arrangement has been made, Diamond will send the samples back using a company of Diamond's choice, and will charge the User's institute. If samples are to be transported by air, return transport must be arranged in advance, and evidence of the arrangements must be presented to Diamond Health Physics Team before the samples are sent to Diamond.

It is forbidden to take radioactive samples onto the beamline(s) or laboratories before they have been checked by the Diamond Health Physics Team. It is forbidden at all times to take radioactive samples to Ridgeway House or to any area other than the beamline(s) or laboratories which have been authorised for their use.

6. SAFETY PROCEDURES DURING THE EXPERIMENT

Solid Samples of natural or depleted Uranium or natural Thorium below 250 Bq can be accessed by I18 and B18 users unsupervised ***between 0830 and 1700 on weekdays.*** Outside these times access to samples can be arranged via the EHCs.

Access to all other samples must be supervised by a member of Diamond staff.

If samples from more than one user group are being stored in the radiochemical lab access must be supervised by Diamond staff.

All samples removed from the radiochemical lab and returned to it, must be recorded in the log book.

During all experiments graded HIGH risk rating with radioactive samples, at least one member of the User Group or beamline staff must be present on the beamline at all times, unless the hutch door can be physically locked or access restricted to specified individuals via access card system. If an experiment is left unattended, section 3 of TDI-HP-PRC-0004 must be completed (see Appendix 4). If a physical lock is used the key must be retained by the user, a spare key should held by the EHC office. For access to the Experiments Hutch, at least two people must be present, although the second person may be an Experimental Hall Coordinator.

Any work with radioactive samples in the lab must be done in accordance with TDI-HP-LR-0006 Radio-chemical Laboratory Local Rules.

All experiment-specific safety procedures specified to the Principal Investigator in advance by Diamond Health Physics Team (see Section 4) must be obeyed.

Additional requirements for experiments involving Neptunium are detailed in Appendix 2.

7. PROCEDURES AT THE END OF THE EXPERIMENT

At the end of the experiment, Users must report to the Health Physics Team who will confirm that all radioactive samples are present, perform a contamination check and check the samples have not been damaged during the experiment. ***At least one User must remain at Diamond until this procedure has been completed. This procedure must take place between 0830 and 1600 on Monday – Friday.***

If the samples are being transported as an excepted package Diamond Health Physics will supply the users with a transport document and act as consignor.

Users are responsible for removing all samples from Diamond premises.

8. SAMPLE HOLDERS AND CONTAINMENT

8.1 Sample Holders

Detailed information on sample holders must be provided to Diamond Health Physics Team before the experiment starts (see Section 4).

The sample holder must be compatible with the set-up of the beamline on which it is to be used, and must guarantee the stability of the sample, taking into account its physical and chemical properties. Due to the possible corrosive effects of certain acid or basic solutions, it is preferred that materials such as PTFE are used for liquid sample holders. Users should be aware that PTFE has a limited radiation resistance, and alternatives should be considered if possible for long exposures. The use of glass is forbidden because of the risk of breakage.

If sample holders do not conform with the description provided under the requirements of Section 4, the experiment will be cancelled.

8.2 Sample Containment

The requirements for sample containment are defined in Section 3. Whether the sample holder can be considered as a first level of containment will depend on its design details, e.g. liquid samples simply sealed with a kapton foil will not be considered as adequately confined. The Diamond Health Physics Team will decide whether a sample holder can be considered as a first level of containment, based on information supplied by the User.

If the sample holder cannot be considered as the first level of containment, or if double containment is required, heat-sealed plastic bag containment is compulsory.

8.3 Shielding against external irradiation from sample

If dose rates from the sample will lead to Diamond's corporate dose limit (1 $mSv.y^{-1}$ or 0.5 $\mu Sv.h^{-1}$) being exceeded in an area which is accessible to people, shielding must be provided. As a general rule, the dose rate at 30 cm must not exceed 0.5 $\mu Sv.h^{-1}$, and the dose rate at contact, except on the windows for incoming and outgoing beams, must not exceed 15 $\mu Sv.h^{-1}$. If a sample requires shielding, the details of the shielding must be provided in the information requested under Section 4.

9. REQUIREMENTS FOR SAMPLES ARRIVING FROM OVERSEAS

9.1 Additional requirements for samples from within the European Community

Movements of radioactive material between member states of the European Community are regulated by the Council Regulation 1493/93 Euratom of 8th June 1993. This places duties on the consignor of radioactive material (in this case, the User) and on the consignee (in this case, Diamond) to ensure that the competent authorities in both countries are informed of the intended movement of radioactive samples from the User's home country to Diamond, and back to the User's institute when the experiment is completed.

Initially, Diamond will contact the Environment Agency, which is the competent authority in England, with details of the samples to be received, as provided by the User in accordance with Section 4 of these procedures. The Environment Agency will acknowledge receipt of this form and return it to Diamond, who will forward it to the User. Once this form has been received by the user, the User may send the samples to Diamond.

When the samples are to be returned to the User's home institute, the institute must contact the competent authority in their own country and obtain permission to receive the samples. Confirmation that this permission has been given must be sent to Diamond before the samples can be shipped back to the User's home institute.

The institute which has sent the samples must, within 21 days of the end of each calendar quarter, provide the following information to the Environment Agency:

- The total activity per radionuclide delivered to Diamond and the number of such deliveries made during the quarter.

- The highest single quantity of each radionuclide delivered to Diamond
- The nature of the samples (e.g. sealed source, other type of source…)

The declaration to the Environment Agency must state clearly that the samples were delivered to:

Diamond Light Source Ltd,
Harwell Science and Innovation Campus,
Didcot,
Oxfordshire,
OX11 0DE

The declaration must be addressed to:

Environment Agency,
Permitting and Support Centre
Environmental Permitting Team
Quadrant 2
99 Parkway Avenue
Parkway Business Park
Sheffield
S9 4WF

Diamond has to make the corresponding declaration to the competent authority in the institute's home country. Therefore, as noted in Section 4, if samples are being brought or sent to Diamond from another country in the European Community, the contact address for that country's competent authority must be included with the information sent before the experiment starts.

9.2 Additional requirements for samples from outside the European Community

The User is responsible for ensuring that the relevant regulations in their home country and for international transport are complied with. Diamond will co-operate in this by completing any declarations which are required from the recipient of the radioactive material.

9.3 Sending samples to and from Diamond by air

When radioactive samples are shipped by air, the IATA transport regulations for dangerous goods must be obeyed. The User is responsible for ensuring that they have complied with these regulations, and customs requirements. They must present Diamond Health Physics Team with evidence that the necessary arrangements for return of the samples has been made, before the samples are sent to Diamond.

APPENDIX 1. ARTIFICIAL RADIONUCLIDES

1.1 Solid samples containing artificial radionuclides

All samples must have at least double containment (see Section 8).

Diamond has agreed activity limits with the Environment Agency, which apply to the total alpha and beta/gamma activity of samples on the premises at any time. In the unlikely event that samples being brought onto Diamond premises for a given experiment would cause these limits to be breached (this would only occur if multiple beamlines were using their full activity allowance), the experiment may be rescheduled.

The total allowed activity per sample will be defined by Diamond Health Physics Team, taking into account the experimental details. As a general indication the activity of the sample should not exceed the activity A_{max} defined as follows. We assume that in the event of an accidental loss of sample containment, a person could be present within the hutch for typically 1 hour. During this 1 hour period, we assume that this person will breathe 1.2 m^3 of air, and we assume that in this initial phase the sample is dispersed in a volume of 10 m^3. We impose the condition that in the case of such an accidental exposure, the person should not receive more than one fiftieth of Diamond's corporate annual dose limit, i.e. 0.02 mSv. The following expression is used to calculate A_{max}:

$$A_{\max}\,[Bq] = \frac{10[m^3]\times 0.02[mSv]}{e[mSv\,Bq^{-1}]\times 1.2[m^3]\times k} \qquad \text{Equation 1}$$

Where *e* is the committed effective dose per unit intake (from ICRP 68) and *k* is the volatility factor. $k = 1.10^{-5}$ for solids.

Table 3 gives the value of A_{max} for some solid radionuclides. For radionuclides where the calculated A_{max} exceeds the 3.7 MBq experiment activity limit, A_{max} is limited to 3.7 MBq (corresponding to an experiment with a single sample of that radionuclide) regardless of the calculated value. (Note some exceptions for Pu241, Tc99 and H3).

Radionuclide	Maximum allowed activity per solid sample, A_{max}
Np 237	790 kBq
Pu 238	380 kBq
Pu 239	350 kBq
Pu 240	350 kBq
Pu 241†	15 MBq
Pu 242	370 kBq
Am 241	420 kBq
Am 243	420 kBq
Cf 249	250 kBq
Cm 248	100 kBq
Eu 152*	3.7 MBq
Cs 137*	3.7 MBq
I 129*	3.7 MBq
Tc 99†	37 MBq
Zn 65*	3.7 MBq
Co 60*	3.7 MBq
Co 57*	3.7 MBq
Mn 54*	3.7 MBq
P 32*	3.7 MBq
Zr 95*	3.7 MBq
Na 22*	3.7 MBq
C 14*	3.7 MBq
H 3†	37 MBq
U enriched < 20 %	50 kBq

Table 3: Maximum allowed activity for solid samples of some typical radionuclides (Calculated using ICRP 68 effective dose coefficients for inhalation of 1 micron AMAD particles)

† Has been allowed an A_{max} above the experiment activity limit (3.7 MBq)
* A_{max} has been restricted to experiment activity limit (3.7 MBq)

If a sample contains a mixture of radionuclides, the activities of each radionuclide in the sample, A_i, must satisfy the expression:

$$\sum \frac{A_i}{A_{\max,i}} \leq 1 \qquad \text{Equation 2}$$

Where $A_{max,i}$ is the maximum allowed activity for the i^{th} isotope in the sample, as obtained from Equation 1 or Table 3.

For a given experiment, up to 10 samples containing artificial radionuclides may be brought to Diamond. However, only one sample can be brought to the beamline at a time.

If more than one sample is brought to Diamond, the other samples will be stored in the Radio-chemical Laboratory.

1.2 Liquid samples containing artificial radionuclides

All samples must have at least double containment.

The total allowed activity per sample will be defined by Diamond Health Physics Team, taking into account the experimental details. Under no circumstances can it exceed the maximum activity A_{max} defined by Equation 3:

$$A_{max}\,[Bq] = \frac{10[m^3] \times 0.02[mSv]}{e[mSv\,Bq^{-1}] \times 1.2[m^3] \times k} \qquad \text{Equation 3}$$

Where the terms are as defined for equation 1, and with the volatility factor k ≥ 0.001, depending on the radionuclide.

Table 4 gives the value of A_{max} for some liquid radionuclides:

Radionuclide	**Maximum allowed activity per liquid sample, A_{max}**
Np 237	1 kBq
Pu 238	1.5 kBq
Pu 239	1.4 kBq
Pu 240	1 kBq
Pu 241	70 kBq
Pu 242	1.5 kBq
Am 241	1.7 kBq
Am 243	1.7 kBq
Cf 249	1 kBq
Cm 248	460 kBq
Eu 152	395 kBq
Cs 137*	3.7 MBq
I 129*	3.7 MBq
Tc 99	1.3 MBq
Zn 65	800 kBq
Co 60	100 kBq
Co 57	1 MBq
Mn 54	1 MBq
P 32	100 kBq
Na 22	1 MBq
C 14	2.8 kBq
H 3†	37 MBq
U enriched < 20 %	500 Bq

Table 4: Maximum allowed activity for liquid samples of some typical radionuclides
† Has been allowed an A_{max} above the experiment activity limit (3.7 MBq)

* A_{max} has been restricted to experiment activity limit (3.7 MBq)

If a sample contains a mixture of radionuclides, the activities of each radionuclide in the sample, A_i, must satisfy the expression:

$$\sum \frac{A_i}{A_{\max,i}} \leq 1 \qquad \text{Equation 4}$$

Where $A_{max,i}$ is the maximum allowed activity for the ith isotope in the sample, as obtained from equation 3 or Table 4.

For a given experiment, up to 10 samples containing artificial radionuclides may be brought to Diamond. However, only one sample can be brought to the beamline at a time. If more than one sample is brought to Diamond, the other samples will be stored in the Radio-chemical Laboratory.

APPENDIX 2. HANDLING PROCEDURE FOR EXPERIMENTS INVOLVING NEPTUNIUM AT DIAMOND

Handling Procedure for experiments involving Neptunium at Diamond

This is a general guide for radionuclide sample handling. A separate, detailed written protocol (Experimental Method) must be specifically prepared prior to each experiment. By default, this should include or consider the standard handling procedures described herein plus any additional conditions for safe sample handling and contamination monitoring for specific samples.

All persons present in the Radiochemistry Laboratory or the Beamline must read and understand the 'Radio-chemical Laboratory Local Rules', 'Use of Radioactive Sources at Diamond' and 'Procedures for the use of radioactive samples on the Diamond beamlines' (TDI-HP-LR-0006, TDI-HP-PRC-0002 and TDI-HP-PRC-0006).

READING OF THIS MANUAL HANDLING PROTOCOL CANNOT BE CONSIDERED AS SUITABLE TRAINING FOR HANDLING RADIOACTIVE MATERIAL. ONLY SPECIALISED TRAINING IN A SUITABLY EQUIPPED LAB IS APPROPRIATE. IT SHOULD BE NOTED THAT DIFFERENT LEVELS OF TRAINING AND EXPERIENCE APPLY FOR HANDLING DIFFERENT RADIONUCLIDES AND DIFFERENT ACTIVITY LEVELS.

HANDLING OF SAMPLES EXCEEDING COMPETENCE LEVEL IS UNACCEPTABLE.

ANY SAFETY CONCERNS SHOULD BE RAISED IMMEDIATELY WITH DLS STAFF.

1. SOLIDS, LIQUIDS AND PASTES SAMPLES – SPECTROSCOPY BEAMLINES

1.1 Experiment Pre-Requisites (to be setup prior to any work)

Any Users handling samples or performing contamination checks must be declared competent by the Principal Investigator (PI) using form TDI-HP-PRC-0005.

The DLS pre-approved protocol(s) applicable to the experiment must be printed and displayed in the Radiochemistry Laboratory and on the beamline before starting the experiment.

The Logbook for recording background radiation and measured contamination must be present before starting the experiment.

The PI for the experiment should be present and, if not the PI, a User with detailed knowledge of the sample (chemical composition, radioisotope content and containment levels).

PPE must be donned upon entering the Radiochemistry Laboratory or Beamline (by ALL entering)

- Lab coat (Howie type)
- Safety glasses
- Gloves (if handling)

A suitable contamination monitor must be selected and checked for correct operation. Users must ensure they can operate properly.

Designated work areas with spill trays will be assigned by DLS. Add signage if required.

A suitable spill kit must be present and contents checked (DLS provides one in the radiochemistry laboratory with protocol for usage).

1.2 Check-Out of Sample from Radiochemical Laboratory to Bring to Beamline

An EHC (or HP staff member, if EHC is not available) must be called to supervise sample access in the Radiochemistry laboratory.

The work area must be monitored and background radiation level recorded before any work begins (there must be no assumption that the area has not been contaminated between visits to the laboratory).

A sample can be retrieved from storage and placed on work area.

Before returning any containers (e.g. storage jar) to locked storage outer surfaces and hands must be monitored for contamination. Swab materials are available if required.

Working over the work area: The surface of the outer level of containment of the sample must be checked for contamination.

All contamination measurements must be recorded (using a numerical value) on the DLS supplied form.

Following any sample manipulation, e.g. removal of any packaging, the outer surface of the sample and any other surfaces in contact must be re-checked for contamination.

If the sample is exposed to non-standard conditions or is disrupted (e.g. the sample is dropped, unexpectedly heated) the sample should not be moved from that location (to prevent potential spread of contamination) and the outer containment level checked for contamination. Results should be recorded.

Any sample showing visible indication of containment damage should be monitored and placed into additional containment. DLS HP, EHCs and the Beamline should be promptly informed. The sample should be clearly labelled as damaged and must not be used and should be returned to locked storage before being returned to the Users home institute. Additional containment is available from DLS HP.

Once a sample has been contamination checked and cleared, it can be placed in a sealed, leak-proof, plastic box suitable for sample transport to the beamline. Prior to placing the sample in the box, the box should be monitored for contamination.

The sample must be logged out of the Radiochemical laboratory and beamline destination recorded in the log book.

Gloves must be checked for contamination before leaving the laboratory.

1.3 Transport to Beamline

- Users must be prepared and sure of the actions to take if the sample is damaged or exposed during transport to the beamline
- The sample (in transport box) can be transported to the beamline
- A contamination monitor must accompany the sample during transport between the Radiochemistry laboratory and the Beamline
- 2 Users to accompany sample (1 to attend sample; 1 to summon help if required)
- PPE is not required during sample transport
- If the sample is dropped, and the transport boxed breached, the sample outer containment should be visually checked for breach, before contamination check (swab if necessary)
- Do not swab if obvious sample breach – instead follow emergency protocol
- If the transport box is not breached the sample (in box) should be returned to the Radiochemistry laboratory to be checked
- At lab, if a sample breach suspected or proved by swab, the Emergency contamination protocol should be followed. DLS HP, EHCs and Beamline staff should be informed immediately

1.4 At the Beamline

Pre-requisites: as per Section 1.1.

- Upon sample arrival, the sample plus box must only be placed on the work area
- The sample should be removed from transport box and both the box and outer containment level checked for contamination
- The contamination monitoring results must be recorded in the radiation logbook at the Beamline experiments hutch
- The sample can be mounted in the approved sample holder
- Beamline Experiment can be performed
- PPE should not be worn in the Beamline control room
- If sample handling is complete, following handling and monitoring of gloves and PPE, hand washing is recommended

1.5 Upon Completion of Individual Sample Spectra Collection

- PPE must be donned on entering Beamline experiments hutch

- The sample outer containment should be monitored for damage (beam damage is possible at some beamlines)
- Monitoring should be performed in-situ at the sample position or within the designated work area – whichever is considered the most appropriate to monitor effectively while reducing risk of spread of any contamination. Any other surfaces contacted by the sample, sample holder, gloves etc., should also be monitored
- The contamination monitoring results should be recorded in the Beamlines logbook

1.6 Transport and Check Back into Radiochemistry Laboratory

- DLS HP or EHC must be called to allow access and supervise sample logging in and out, and physical sample exchange
- The sample can be returned to the Radiochemical Laboratory, in a box suitable for transport, as per transport instructions in Section 1.4
- The sample should be recorded as logged back into the Radiochemistry Logbook
- The sample can be returned directly to the container intended for return transport of the sample to the User home institute; outer containment can be monitored if desired

1.7 Upon Completion of Entire Experiment (End of Visit)

- All samples should be returned to the Radiochemistry Laboratory, following monitoring and logging-in procedures outlined above
- DLS HP should be called to return all samples to secure storage
- When all active samples have been removed from the beamline, DLS HP, or EHCs should monitor the beamline to confirm no contamination
- The designated work area and any signage can be removed
- Samples are to be returned to User home institution
- Any waste generated during the experiment must be recorded
- DLS to confirm all samples have been removed from the store before leaving Diamond

2. SAMPLE ENVIRONMENTS (NON-GENERIC)

2.1 Sample Environments

Sample environments to be used with the samples must be cleared for use by DLS HP and Beamline Staff.

Protocols for environment use, operation, sample handling and emergency must be covered in a separate protocol, specific to the experiment (historical protocols must be cleared for use prior to beamtime to ensure no change or potential to improve).

Extra sample handling and contamination monitoring should be clearly described.

Temporal changes of the sample following sample insert or removal from the environment should be considered e.g. due to temperature change, pressure change, chemical reaction.

All levels of the sample containment must be assessed for suitability in the environment before, during and after removal.

EMERGENCIES

If at any time there is a suspected sample breach, or improper means to check for contamination the experiment must be stopped immediately and samples left in-situ. The experiment hutch shutter should be closed to stop exposure of the sample to the synchrotron beam. DLS HP, EHC and Beamline staff must be informed immediately.

Users should not enter the experiment hutch until DLS staff arrive. Users should only enter the hutch by themselves if required to stop further spread of contamination AND such action is safe AND has been described in the experimental risk assessment.

If the sample containment has been breached, DLS staff will transfer the sample to suitable containment before removing the sample from the beamline to the Radiochemical laboratory. DLS staff will then co-ordinate a check for any contamination and arrange for beamline decontamination if required.

If the contamination monitor has failed, alternative monitoring equipment will be issued and the experiment can continue.

HP Tel: 01235 77(8269), 8438, 8671, 07825 523391
EHC Tel: 01235 77(8787)

APPENDIX 3.

Declaration of Competence in use of radioactive materials
To be completed by the Principal Investigator

I certify that the following named individuals have received appropriate training and are competent in working with the radioactive materials listed.

FULL NAME (please print)	AFFILIATION

Please print details of radioactive materials to be used, listing isotopes, activities, sealed or open as appropriate:

Principal Investigator's signature:

Print name:

Date:

APPENDIX 4.

SOURCE LOAN FORM

Section 1 of this form must be completed for each source borrowed. It should be displayed at the location where the source is in use, along with 'Radioactive Source in Use' sign(s).

A temporary handover of responsibility during attended use of the source should be recorded under Section 2.

If it is necessary for the source to remain in use whilst unattended, the relevant details should be entered in Section 3.

Please refer to TDI-HP-PRC-0002 'Use of Radioactive Sources at Diamond' for further information.

SECTION 1 LOAN DETAILS

Source ID	Nuclide	Activity	Location of intended use	Expected return date/time

Loaned to (PRINT NAME)	Signature	Date / time

SECTION 2 TEMPORARY HANDOVER OF RESPONSIBILITY

From (Name / sign)	To (Name / sign)	Date / time

SECTION 3 SOURCE IN UNATTENDED USE

Person responsible	Contact number(s)	Date/time left	Expected return date/time

Persons (other then the person listed above) entering the room whilst the responsible person is absent must enter details below:

Name (PRINT)	Date	Time in	Time out

SKETCH DIAGRAM SHOWING LOCATION OF SOURCE IN UNATTENDED USE

APPENDIX 5. EXPERIMENT SAMPLE INFORMATION SHEET FOR RADIOACTIVE HIGH & MEDIUM GRADED SESSIONS

EXPERIMENT & SAMPLE INFORMATION SHEET (FOR HIGH & MEDIUM GRADED)

Experiment Number: **Beamline:**

Date:

User Contact:

Experiment/Sample description:

Hazard source:

Risks:

Likely points of failure to look out for:

Security Requirements

☐ Restricted access to beamline/lab is required for this experiment (detail below)
☐ Experiment/Samples must not be left unattended unless beamline/lab is locked
☐ A count in/out of samples is required for this experiment

Other/detail:

Supervision Requirements

☐ Experiment/Samples must not be left unattended on the beamline/lab
☐ Experiment/Samples must not be left unattended unless beamline/lab is locked
☐ Experiment/Sample set-up must be checked by EHC/Local Contact before experiment commences.
☐ Experiment/Sample must be checked by EHC/Local Contact throughout the experiment. Frequency:

☐ Samples require special storage/disposal/emergency arrangements.

Other/Detail: