



ESRF procedures for experiments using radioactive samples on beamlines other than beamline BM20 (ROBL)

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1 Summary of important deadlines and time constraints

*All detailed information concerning the samples and the experimental set-up must be provided to the ESRF as soon as possible, normally **one month** before the experiment starts. If **two weeks** before the starting date all the required information is not available, the experiment will be cancelled (see § 4).*

*Users bringing radioactive samples with them to the ESRF must arrive on **weekdays, between 08h00 and 16h00** (see § 7.1).*

*During the experiment transfer of samples between the Radioactive Samples Laboratory and the beamline is only possible on **working days, between 08h30 and 16h00** (see §§ 3.2 and 3.3).*

*At the end of the experiment at least one of the users must remain at the ESRF until all samples have been returned to and verified by the ESRF Radiation Protection Group. Note that samples can only be returned to the ESRF Radiation Protection Group **during working days, between 08h30 and 16h00** (see § 6).*

2 Scope of the present procedures: definition of radioactive samples

Table 1 defines the samples for which the present procedures must be applied. The limits indicated in this table have been defined by the ESRF, taking into account several aspects: safety aspects (contamination in case of spillage of a liquid sample, loss of a sample, external radiation, ...) and obligations from French and international regulations (import/export rules, authorisations, declarations, inventory, ...). As indicated, these limits refer to the activity of one individual sample or to the total activity of all samples brought to the ESRF for a given experiment.

Gaseous or powder samples containing traces of radioactive isotopes, no matter how small, are forbidden on all beamlines.

Samples containing artificial radionuclide(s)	Sum of the activities of all samples brought to the ESRF > 1 Bq
Solid* samples containing natural or depleted uranium	Sum of the activities of all samples brought to the ESRF > 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 the ESRF > 250 millilitres
Solid* samples containing natural thorium	Sum of the activities of all samples brought to the ESRF > 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 the ESRF > 50 millilitres

* solid samples are crystalline or amorphous samples or wet pastes (e.g. clays). Crystals suspended in a liquid are liquid samples.

Table 1: Definition of samples for which the present procedures apply

3 Different types of experiments with radioactive samples that can be authorised at the ESRF

The following sections give general guidelines concerning the type of experiment with radioactive samples that can be authorised at the ESRF.

These are only guidelines, and it does not mean that any experiment on samples below these limits will be automatically authorised.

Note: the total equivalent activity on the ESRF site is at all times limited, and so is the import/export flow of nuclear material. The ESRF may therefore require the rescheduling of an experiment if these overall limits would otherwise temporarily be exceeded.

3.1 Experiments involving natural or depleted uranium or thorium

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

Solid samples containing natural or depleted uranium must have a single sample confinement (see § 8) unless they are chemically stable in air, in which case no sample confinement is required. Liquid samples containing natural or depleted uranium always require a double confinement.

Solid samples containing natural thorium must have a single sample confinement. Liquid samples containing natural thorium always require a double confinement.

Table 2 gives the allowed activity per sample, as well as the maximum sum activity of all samples of a given type.

Type of sample	Maximum activity per sample	Maximum sum 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 for samples containing natural or depleted uranium or natural thorium

3.2 Solid samples containing artificial radionuclides

All samples must have a double sample confinement (see § 8).

The total allowed activity per sample will be defined by the ESRF Safety Group, taking into account the experimental details. As a general indication the activity of a sample should not exceed the activity A_{max} defined as follows. We assume that in the event of an accidental sample confinement loss, a person could be present inside the hutch for typically one hour. During this one hour period, we assume that this person will breath 1.2 m^3 of air, and we

assume that in this initial phase the evaporation of the sample spreads out in a volume of 10 m³. In the case of such an accidental exposure, the person shall not receive more than the allowed effective dose for one week, for a non-exposed worker, i.e. 0.02 mSv. The following expression is therefore used to calculate A_{max}:

$$A_{\max}[\text{Bq}] = \frac{10[\text{m}^3]}{e[\text{mSv} \cdot \text{Bq}^{-1}] \times 1.2[\text{m}^3] \times k} \times \frac{1}{50}[\text{mSv}] \quad (3.1)$$

with e the committed effective dose per unit intake, and k, the volatility factor, = 1. 10⁻⁵ for solids.

Table 3 gives the value of A_{max} for a non-exhaustive list of radionuclides.

Radionuclide	Maximum activity per sample A _{max} (expression 3.1)
Np 237	640 kBq
Pu 238	300 kBq
Pu 239	280 kBq
Pu 240	280 kBq
Pu 241	15 MBq
Pu 242	300 kBq
Am 241	340 kBq
Am 243	340 kBq
Cf 249	480 kBq
Cm 248	90 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
Na 22	3.7 MBq
C14	3.7 MBq
H 3	37 MBq
U enriched < 20 %	50 kBq

Table 3: Maximum activity per solid sample

If a sample contains a mixture of radionuclides, the activities A_i per sample, for the different radionuclides, must fulfill the expression:

$$\sum_i \frac{A_i}{A_{\max,i}} \leq 1 \quad (3.2)$$

where A_{max,i} is the maximum allowed activity for the ith isotope, per sample, as obtained from expression (3.1) - see table 3.

For a given experiment, one can bring up to 10 samples containing artificial radionuclides to the ESRF. However, only one sample can be at the beamline at a time. If more than one sample is brought to the ESRF, the other samples will be stored in the Radioactive Samples Laboratory of the ESRF. ***Transfer of samples to and from the beamline will only be possible on working days, between 08h30 and 16h00.***

The maximum total activity of all the samples brought to the ESRF, related to one accepted experiment, shall not exceed 3.7 MBq.

3.3 Liquid samples containing artificial radionuclides

All samples must have a double sample confinement.

The total allowed activity per sample will be defined by the ESRF Safety Group, taking into account the experimental details. Under no conditions can it exceed the activity A_{\max} defined as (see paragraph 3.2):

$$A_{\max}[\text{Bq}] = \frac{10[\text{m}^3]}{e[\text{mSv} \cdot \text{Bq}^{-1}] \times 1.2[\text{m}^3] \times k} \times \frac{1}{50}[\text{mSv}] \quad (3.3)$$

with k , the volatility factor, ≥ 0.001 , depending on the radionuclide.

Table 4 gives this maximum allowed activity per liquid sample A_{\max} for a non-exhaustive list of radionuclides.

In case of a sample containing a mixture of radionuclides, the activities A_i per sample, for the different radionuclides, must fulfill the expression:

$$\sum_i \frac{A_i}{A_{\max,i}} \leq 1 \quad (3.4)$$

where $A_{\max,i}$ is the maximum activity for the i th isotope, per sample, as defined by expression (3.3) - see table 4.

One can bring up to 10 samples containing artificial radionuclides to the ESRF for a given experiment. However, only one sample can be at the beamline at a time. If more than one sample is brought to the ESRF, the other samples will be stored in the Radioactive Samples Laboratory of the ESRF. ***Transfer of samples to and from the beamline will only be possible on working days, between 08h30 and 16h00.***

Radionuclide	Maximal activity per liquid sample A_{\max} (expression 2.5)
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 Bq
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
C14	2,8 kBq
H 3	37 MBq
U enrichi < 20 %	500 Bq

Table 4: Maximum activity per liquid sample

4 What must be done before the experiment starts

When a proposal has been accepted by the ESRF, the main proposer must provide the ESRF Radiation Protection Group with a document providing all the necessary information to allow a complete safety analysis. This document must contain:

- A detailed list of all the samples (number of samples, isotopic composition, weight, activity of the individual isotopes and sum 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/confinement. This description must explain how the required degree of confinement is obtained (see § 8).
- A detailed description of the experimental set-up.
- In the case of liquid samples, a description of the decontamination procedure in case of spillage, taking into account the physico-chemical characteristics of the samples.
- A detailed description of the sample shielding if required (see § 8.3).

This document must be sent to the ESRF Radiation Protection Group, normally ***one month before the experiment starts***, either by e-mail to radiop@esrf.fr or by post to:

ESRF
Att.: P. Colomp
6, rue Jules Horowitz
BP 220
F-38043 Grenoble Cedex
France.

If the samples can only be prepared short time before the experiment, and therefore their exact activity will only be known at that time, one should not wait to send the requested information to the ESRF until the number of samples and their activity are exactly known. The document could in the first instance indicate an approximate, maximum activity and the exact number of samples and their precise activity could be communicated in a later stage. However ***all information should be communicated at the latest two weeks before the experiments starts***. Once all details are mutually agreed between the proposer and the ESRF Safety Group, the latter will return a document to the main proposer, mentioning all specific safety rules and procedures for the given experiment. All conditions specified in this document must then be strictly respected. In particular, it is forbidden to bring any radioactive sample, not specified in this document, to the ESRF.

The following documents must be joined to the samples when they arrive at the ESRF:

1. An official document from the proposer's home institute certifying the nature of the samples dispatched to the ESRF (mass, activity, isotopic composition, physical and chemical state). This document is required for all radioactive samples, i.e. including uranium and thorium samples.
2. A certificate of non-contamination for all samples requiring a sample confinement, as specified in § 3.
3. gamma spectrometry
 - for uranium or thorium samples: a qualitative spectrum for every sample with activity > 1 kBq, with identification of peaks to prove absence of other radioisotopes; a spectrum for every sample < 1 kBq, measured at contact for minimum 5 minutes, to prove low activity and absence of other radioisotopes.
 - for samples containing artificial radionuclides: for each sample, a quantitative spectrum, with peak identification and calculation of the activity of each isotope.
 - for pure α or pure β emitters: a spectrum measured for minimum 5 minutes to prove absence of γ emitters.

The above documents must be signed by an authorised Radiation Protection Officer from the proposer's home institute.

Exceptionally the ESRF can accept that these spectrometry reports are not provided. The user has to request such an exception, with sound justification, to the ESRF (radiop@esrf.fr) at least four weeks before the shipment of the samples. The ESRF will inform the user by return-mail of its decision.

Failure to provide one of these documents, unless explicitly accepted by the ESRF, will result in the refusal of the experiment.

5 Safety procedures during the experiment

All experiments using radioactive samples (except certain experiments using only natural or depleted uranium) will be "red" experiments; during the experiment at least one person must be present on the beamline 24h/24h. Access to the Experimental Hutch requires the presence of at least two people (the second person could e.g. be the Experimental Hall Operator).

Apart from this general rule, the specific safety rules and procedures mentioned in § 4 must be strictly respected.

6 Procedures at the end of the experiment

At the end of the experiment, all samples must be returned to the ESRF Radiation Protection Group, who will recheck the samples, prepare the sample package for the return shipment and fill out all the required documents. ***Samples can only be returned to the ESRF Radiation Protection Group during working days, between 08h30 and 16h00. At least one of the users must be present at the ESRF until all samples have been returned to and verified by the ESRF Radiation Protection Group.***

7 How to bring radioactive samples to the ESRF, and how to take radioactive samples from the ESRF

7.1 General rules

The international transport rules for dangerous goods class 7 must be respected when transporting samples to or from the ESRF, as well as the relevant French legislation.

Below certain limits, radioactive samples can be transported in so-called excepted packages. Transport rules are less stringent in this case. Table 5 gives the upper activity limits for excepted packages for a non-exhaustive list of radionuclides. These limits should only be used as a guideline, and one must be aware of specific rules e.g. when transporting samples containing different radioisotopes. Please refer to the international transport rules for all details.

Radionuclides	Solid samples	Liquid samples
^{249}Cf	0.8 MBq	0.08 MBq
^{244}Cm	2 MBq	0.2 MBq
^{248}Cm	0.3 MBq	0.03 MBq
^{241}Am	1 MBq	0.1 MBq
^{243}Am	1 MBq	0.1 MBq
^{238}Pu	1 MBq	0.1 MBq
^{239}Pu	1 MBq	0.1 MBq
^{240}Pu	1 MBq	0.1 MBq
^{241}Pu	60 MBq	6 MBq
^{242}Pu	1 MBq	0.1 MBq
^{237}Np	2 MBq	0.1 MBq
^{99}Tc	900 MBq	90 MBq
Natural or depleted uranium	No limit	No limit
Natural thorium	No limit	No limit

Table 5: Upper limits for excepted packages

Above the limits for an excepted package, type A containers must be used and the samples must be sent to the ESRF through an authorised carrier.

All radioactive samples that are brought by the user directly to the ESRF need to be presented to the ESRF Safety Group immediately upon arrival at the ESRF, at the following location:

Radiation Protection Service
Experimental Hall, sector 40, 1st floor, rooms 30-1-06 or 30-1-22.

It is forbidden to take the samples directly to the beamline without presenting them to the ESRF Safety Group. It is also forbidden to take the samples to the Guesthouse or to any office.

Radioactive samples can only be presented to the ESRF Safety Group on weekdays, between 08h00 and 16h00. A user arriving at the ESRF outside these hours cannot bring any radioactive samples with him. Therefore, in the latter case the samples must be sent separately to the ESRF.

All radioactive samples which are sent to the ESRF should be shipped to the following address:

ESRF
Att.: P. Colomp or P. Berkvens
6, rue Jules Horowitz
BP 220
F-38043 Grenoble Cedex
France

The international transport rules define the obligatory safety signs that must be put on the packages or containers.

Note: it is **mandatory** that any package containing radioactive samples – in the sense of § 2 in this document - that one sends to the ESRF must have on the **external** containment, clearly visible, a label saying “radioactive material”, accompanied by the radioactive hazard symbol. This will guarantee that radioactive samples delivered to the ESRF stores are immediately identified as such.

Independent of whether samples are sent to the ESRF or brought personally to the ESRF, the ESRF Radiation Protection group (radiop@esrf.fr) must be informed in advance of the foreseen date and time of the arrival of the samples.

The shipping of radioactive material via international public mail is forbidden. Shipping exempted packages of radioactive material inside France by public mail is allowed under certain conditions. Only duly authorised shippers are allowed to do so. For more details see the corresponding French legislation, “arrêté du 22 mars 2001 relatif aux envois postaux de matières radioactives”.

If a user foresees to send the samples back to his home institute at the end of the experiment by a transport company, he must have made the necessary arrangements with this company beforehand. If no such arrangements have been made the ESRF will send the samples back to the user using a company of ESRF’s choice, at the user’s expense. If samples are returned by air the necessary arrangements must be made beforehand with the ESRF’s designated transporter (see § 7.5).

7.2 Specific rules for samples from within France

The following procedure has been agreed between the French authorities (IRSN) and the ESRF, and must be applied for radioactive samples arriving from another institute within France. The official IRSN document “Formulaire de demande de radioéléments artificiels en sources non-scellées” has to be filled out. Once an experiment has been scheduled, the ESRF

Radiation Protection Group sends this document to the institute from which the samples are brought to the ESRF. The latter completes the form and returns it to the ESRF, who sends it to the IRSN. Only then can the radioactive samples be sent to the ESRF.

7.3 Specific rules for samples from within the European Community

Movements of radioactive samples within the European Community are regulated by the Council Regulation 1493/93/Euratom of 8 June 1993 on shipments of radioactive substances between Member States.

In particular, if an institute has sent or brought radioactive samples to the ESRF, they must, within 21 days of the end of the calendar quarter during which the shipment(s) took place, inform in writing the competent French authority (IRSN), providing the following information:

- The total activity per radionuclide delivered to the ESRF and the number of such deliveries made during the quarter
- The highest single quantity of each radionuclide delivered to the ESRF
- The nature of the samples

One must clearly indicate that the samples were delivered to:

*ESRF European Synchrotron Radiation Facility
6, rue Jules Horowitz
BP 220
38043 Grenoble Cedex*

The declaration must be addressed to

INSTITUT DE RADIOPROTECTION ET DE SURETE NUCLEAIRE

UES
BP 90
60,68 Avenue du Général Leclerc
92263 Fontenay-aux-roses Cedex
France

The ESRF has to make the corresponding declaration to the competent authority in the institute's home country. Therefore, the information requested in § 4 must include the name and address of the competent authority, as well as the details of the institute's corresponding authorisation.

7.4 Specific rules for samples from outside the European Community

For samples arriving from outside the European Community, the official IRSN document "Demande d'autorisation d'importer des radioéléments artificiels" has to be filled out. The ESRF fills out this document, based on the information provided by the user. As soon as the ESRF receives the document in return from the IRSN, a copy is forwarded to the user. Only then can the radioactive samples be sent to the ESRF. Note that this document, duly signed by the IRSN, is necessary to take the samples through French customs. At the same time the ESRF also takes care of the second document "Demande d'autorisation d'exporter des radioéléments artificiels", necessary for the return shipment of the samples.

Both documents must define a time slot (typically a few weeks) for the arrival and the re-expedition of the samples. It is mandatory that the mentioned planning be respected.

7.5 Specific procedures for sending samples to and from the ESRF by air

When radioactive samples are shipped by air, the IATA transport rules for dangerous goods must be strictly respected. In this framework, the ESRF has designated an authorised transporter to deal with all transports by air of radioactive material to and from the ESRF. Therefore, if a user decides to send radioactive samples to the ESRF by air, the samples must be addressed to this designated transporter. The latter will take care of all the necessary customs formalities in France, both for the shipment to and from the ESRF. When a user decides to send his radioactive samples by air to the ESRF, he must contact as soon as possible the ESRF Safety Group (radiop@esrf.fr), who will communicate the address and other details of the transporter and all necessary information.

8 Sample holders and confinement

8.1 Sample holders

Part of the information requested by the ESRF (see § 4) deals with the sample holders. It is mandatory that a detailed description of the sample holder be given here, clearly explaining the degree of confinement.

The sample holder must be adapted to the experimental set-up of the concerned beamline, and must guarantee the stability of the sample, taking into account its physical and chemical properties. In particular, due to the possible corrosive effects of certain acid or basic solutions, preferably materials such as teflon must be used for liquid sample holders. Although glass is chemically inert, its use is forbidden for sample holders and for sample confinement, because of the risk of breakage.

If the sample holders do not conform with what was described by the user, the experiment will be refused.

8.2 Confinement

The requirements for sample confinement are defined in § 3. Whether the sample holder can be considered as a first confinement barrier will depend on its design details, e.g. liquid samples simply sealed with a kapton foil will not be considered as adequately confined. The ESRF Radiation Protection Group will decide whether or not a sample holder can be considered as confinement barrier, based on the information provided by the user.

If the sample holder cannot be considered as the first confinement barrier, or if a double confinement is required, hot-sealed plastic bag confinement(s) is compulsory.

8.3 Shielding against external irradiation from sample

The dose limits for non-exposed workers (1 mSv.y^{-1} or $0.5 \text{ }\mu\text{Sv.h}^{-1}$) must be respected at the ESRF in all places accessible to people. Therefore shielding of the sample must be provided if its external radiation exceeds these limits. As a general rule, the dose rate at 30 cm must not exceed $0.5 \text{ }\mu\text{Sv.h}^{-1}$, and the dose rate at contact, except on the windows for the incoming and outgoing beams, shall not exceed $15 \text{ }\mu\text{Sv.h}^{-1}$. If a sample requires shielding, the details of the shielding must be provided in the information requested in § 4.

9 Interventions on samples at the ESRF

As a general rule all interventions modifying the physical and/or chemical state of the samples are forbidden at the ESRF.

Although heating of the sample is forbidden, cooling can be authorised, but must be described by the user as part of the experimental set-up.