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ESS SAMPLE HANDLING PROCEDURE

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1. PURPOSE

Based on the *NSS Concept of Operations* (ESS-0005817) [1], the *Neutron Scattering Systems* - System Design Description (ESS-0377817) [2], as well as the NSS radiological hazards described in *Neutron Scattering Systems* - *Radiological Hazards and Radiation Safety Provisions for* Operations and Maintenance (ESS-2972939) [3], this document addresses and describes the sample handling procedure for the ESS user program. The document is part of the *Sample Management Procedure* (ESS-0024109) [4] and follows the *Experiment Safety Review Procedure* (ESS-0024107) [5]. Moreover, this report describes the rules and regulations for sample handling in experimental areas of ESS, such as the instrument halls and laboratories considering the ESS Radiation Protection Rules [6] and provisions of the Swedish Authorities [7]. To combine the best practices, the procedures and processes from several (reactor- and accelerator-based) neutron sources were taken into account. In the appendix (chapter 7) four notional scenarios are discussed on how the Sample Management Procedure Will be carried out.

2. PROCEDURE APPLICABILITY

The procedure is applicable to the handling of all samples connected to proposed and accepted experiments within the ESS user program. It is part of the Sample Management Procedure [4] and follows the Experiment Safety Review [5].

3. SAMPLE HANDLING PROCEDURE

3.1. Procedure map



As soon as the Safety Review Committee (SRC) has performed the Experiment Safety Review resulting in the signed Experiment Safety Document (ESD), the ESD is available to the instrument team (ITE) that hosts the experiment. According to the ESD, the Instrument Team (ITE) plans the resources and materials needed from ESS to perform the experiment safely (Planning Step). Upon arrival of the user the Briefing Phase, including user training and pre-job briefing, starts. Thereafter the sample can be handled (start of the sample life-cycle) and the experiment is performed. The last part of the experiment is the retirement of the sample (end of sample life-cycle) in accordance with the ESD; sample retirement can be disposing the sample as (radioactive) waste, shipment of the sample to the users' home institute or storage of the sample for further use at ESS. When all the steps are complete, an Experiment Safety Report (ESR) is generated which includes the Experiment Safety Document (ESD) and information of the radiological survey and the retirement of the samples.



Figure 3-1: Flow chart of the Sample Handling Procedure. The starting point is the Experiment Safety Document (ESD) issued by the Safety Review Committee (SRC), followed by a Planning, Briefing and Experiment stage. Only after the sample retirement the Experiment Safety Report (ESR) is issued.

3.2.1. Input

The actual sample handling relies on the extensive review process that is performed before the experiment. The Sample Handling Procedure ensures that all safety requirements put upon the experiment by the reviewers of the Safety Review Committee (SRC) are fulfilled. The Experimental Safety Document (ESD) provides specific rules and regulations connected to the handling of the samples for each experiment, the handling of the sample environment equipment and the sample manipulation process in the instrument area and/or in ESS laboratories. The handling of the sample is done from cradle to grave; this time span is called sample life-cycle. Only after a sample is retired, the Experimental Safety Report (ESR) can be generated.

<i>3.2.2.</i>	Planning: Arrival of Sample			
Samp	les can arrive at ESS through several channels:	Respon	sible:	
•	Shipped to ESS	Instrume	ent Team	
•	Synthesised at ESS		Planning	
•	Brought to ESS by the user			
ESS ha rules a Experi Team arrival certain Instru requir proce not b briefir chemi as san	as to make sure the user is aware of the existence of transportation and regulations. The acknowledgement of these rules is part of the iment Safety Form (ESF), filled out by the user. The Instrument (ITE) assures that special requirements on sample storage upon are taken care of, for instance, requirement to be stored at a n temperature, in inert atmosphere or in a controlled place. The ment Team (ITE) also needs to make sure that if there is a tracking ement stated in the Experiment Safety Document (ESD), the ss will be initiated upon the arrival of the sample. The samples will e handed to the user until the required training and pre-job ng is done ("Experiment Step"). If the user brings in or ships cals to synthesise the sample at ESS, these chemicals are treated inples.			

Output/product	it/product The sample is stored in the correct place upon arrival. If tracking of the sample is necessary, it starts at this point in time. User will not perform work until briefed on rules and regulations.			
<i>3.2.3. Planning: Arrival of e</i>	pquipment			
It is the responsibility of the Instrument Team (ITE) to ensure that user- supplied equipment brought to ESS is checked for compliance with the current safety standard by the respective safety teams. For commercial products this can be as easy as to look for the safety certificate. For user- supplied equipment this might involve a check by an electrical engineer				
Output/product	Ensure that user-supplied equipment is safe to be	e used at ESS.		
3.2.4. Planning: Additional	controls required			
It is the responsibility of the Instrument Team (ITE) to fulfil additional requirements stated in the Experiment Safety Document (ESD). The role of the ITE is to coordinate with the respective teams to setup additional administrative and/or engineering controls for the experiment.				
Output/product	Output/product Ensure that required support through other teams with respect to administrative or engineering controls is guaranteed.			
3.2.5. Briefing: User Trainin	9			
Upon arrival the user(s) will undergo the general user training that informs them about hazards and controls in the experimental areas at ESS. If the Experiment Safety Document (ESD) requests additional training, e.g. for user laboratories or special equipment, it is done at this point. Responsible: Instrument Team / User Briefing				
Output/product	The user training stage ensures that users are familiand that they have all the required training before	iliar with the basic safety at ESS e the start of the experiment.		
3.2.6. Briefing: Pre-job brie	fing			
The Instrument Team (ITE) will review the Experiment Safety Document (ESD) with the user(s) in a pre-job briefing and explain hazards and requirements for the experiment as well as the controls that are put into place. The ITE also confirms that the user(s) has/have taken all the required training for the specific experiment. The user(s) and the ITE confirm the detailed experimental plan and clarify the chronology of the experiment. The user(s) confirm that they understand the rules and regulations and will obey them. The occurrence of this pre-iob meeting				

is confirmed by the user(s) and the ITE by signatures on the ESD. This is

Released Internal a pre-requisite for any work done by the user(s) at ANY of the ESS

Experiment

experimental facilities (laboratories and experimental hall).		
Output/product	Ensures that users are familiar with hazards of their experiment and how th hazards are controlled. Do's and Don'ts for the user(s).	

3.2.7. Experiment: Sample Life starts

Responsible: After the pre-job meeting the user(s) obtain the sample(s), if not stated otherwise in the Experiment Safety Document (ESD) (e.g. for controlled User or traceable samples the rules can be different). The ESD states what the Instrument Team (ITE) users are allowed to do with the sample(s) during their stay at ESS and other teams as required by where they are allowed to take the samples. A copy of the ESD is posted ESD at the location of the experiment (instrument area and/or laboratories). There are clear instructions on how the samples have to be labelled during their lifetime at ESS. The users have to ensure that the samples and connected hazards in their assigned working area are clearly identifiable through labelling and ESD.

Ensure that sample treatment is clear before the start of sample handling. Use Output/product standard controls (ESD and labelling) to clearly identify the state of the experiment.

3.2.8. Experiment: experiment on instrument Responsible: Transfer of sample before it has been in the neutron beam: User Low-Hazard samples: Instrument Team (ITE) The user can carry samples freely between the experimental hall and other teams as required by the laboratories within the supervised zone, if no further restrictions ESD are stated in the Experiment Safety Document (ESD). Medium- and high-hazard samples: Experiment These samples can have restrictions on the sample transfer, as stated in the ESD. These can be: • restrictions connected to the potential hazard of the sample (active samples, explosive samples, controlled substances, ...) • restrictions referring to closed containment and/or secondary containment of sample during transfer (engineering control) • restrictions due to tracking of the sample (administrative control) Loading into / removal of sample from the sample area into the instrument area: Low-Hazard samples: The low-hazard samples are mounted in the sample area of the instrument by the user as allowed by local instrument procedures, if no

further restrictions are stated in the ESD. It can also be removed by the user as allowed by local instrument procedures, if not further restrictions are stated in the ESD.

Medium-hazard samples:

Medium-hazard samples can have restrictions stated in the ESD as to who can mount/remove the sample from the sample area of the instrument. Depending on the sample there are

• restrictions connected to the potential hazards of handling the sample environment equipment

• restrictions connected to the potential hazard of the sample (radioactive samples, explosive samples, controlled substances, and others)

• restrictions connected to necessary tracking of sample (administrative control)

High-hazard samples:

High-hazard samples will have restrictions stated in the ESD as to who is allowed to move the sample from the sample area on the instrument. This might include Radiation Protection (RP) surveys before mounting and/or before removal of the sample from the respective sample area. The restrictions for this hazard-category are mostly related to conventional (chemical, biological, physical) hazards of the samples; these samples require extensive tracking.

Handling of samples after they have been exposed to the neutron beam:

Low-Hazard samples:

After the sample is exposed to the neutron beam, low-hazard samples can be transferred freely within the supervised area, if no further restrictions are stated in the ESD. In case of a spill of a sample, the Radiation Protection (RP) needs to be called. The RP designee carries out a survey of the contaminated area and assists in or coordinates the clean-up.

Medium-hazard samples:

After the sample is exposed to the neutron beam, medium-hazard samples can be transferred within the supervised area, if the radiation hazards are low and no further restrictions are stated in the ESD. In case of a spill of a sample, RP has to called. The RP designee surveys the contaminated area and assists in or coordinates the clean-up.

High-hazard samples:

After the sample is exposed to the neutron beam, high-hazard samples cannot be freely transferred within the supervised area. The restrictions for this hazard-category are mostly related to conventional (chemical, biological, physical) hazards of the samples; these samples require extensive tracking. In case of a spill of a sample that has been in the neutron beam, the RP designee surveys the area and decides on the clean-up process.

General Remarks:

If a sample is surveyed by an RP technician or the operational RP team after the exposure to beam and has received a tag to be free released, it can be handled from the radiological hazard viewpoint as a "lowhazard sample".

Examples for requirements given by the ESD are:

• Can be removed from the sample area (only) of the instrument by user or instrument team without survey by the RP team, but has to stay in the instrument area.

• Needs to be surveyed by user/instrument team with hand-held radiation monitor before removal from sample area of the instrument, but has to stay in the instrument area.

• Needs to be surveyed by RP team for internal/external contamination before removal from sample area of the instrument.

• Can be brought into an ESS laboratory and treated further after a radiation survey by the RP team.

• Needs to be in administrative control after removal from sample area of the instrument.

NO MATTER WHAT: Each sample that has been in the neutron beam has to be surveyed before leaving the supervised area, e.g. the experimental halls and surrounding laboratory buildings.

Output/product

Safe handling of samples within the supervised area.

3.2.9. Experiment: Use of laboratories before/during/after experiment

Access:	Respo	nsible:	
The access to the auxiliary laboratories is granted according to the	User		
Experiment Safety Document (ESD) and after the completion of the required training of the users. The users need an orientation on the rules	Instru	ment Team (ITE)	
and regulations of the respective laboratory during the "Briefing Step",	Labor	atory Team	
i.e. before the experiment is carried out. This orientation is given by the respective laboratory team. The laboratory team has to ensure that the	otner ESD	teams as require	а ру
experimental work done in the laboratories is performed according to the boundaries provided in the ESD. Depending on the hazards connected to the work, the work space might be a limited access area		Experiment	
and there might be further administrative and/or engineering controls in place. The ESD is posted in the working area to inform other workers about the performed work.			

Samples/Chemicals:

The laboratories contain chemicals and samples that have not been exposed to the neutron beam as well as samples that have been in the beam. Standard administrative and/or engineering controls ensure that mixing of non-irradiated chemicals/samples with irradiated samples does not occur. For high-hazard samples that have been exposed to the neutron beam and which are handled in a laboratory area additional controls are in place depending on the hazards connected to the samples as stated in the ESD. These restrictions are:

- keeping the sample in a separate area or in a laboratory that has restricted access for the time of processing (e.g. high explosives behind shield, biohazard level 3 samples in restricted areas, radioactive samples in the Radioactive Material Laboratory (RML)...).
- using dedicated equipment for each experiment with respective hazard signs.
- have the RP team survey the laboratory area for contamination during and after the experiment.
- have the RP team survey the sample container (can) for external radiation before transfer to the laboratory and unloading.

Output/product

Safe handling of samples in the auxiliary laboratories.

3.2.10. Experiment: Sample Retirement

All samples that have been in the neutron beam are surveyed by RP.		Responsible:	
Irradiated samples are transferred to the survey station within the	User		
supervised area and receive a tag with the survey results.	Instru	ment Team (ITE)	
• remain at FSS to cool down/decay (if reasonable) and are then	other teams as required by		
shipped to the home institution or disposed of.	ESD		
• be shipped to the home institution via a radioactive materials		Experiment	
transport should the home institution be able to receive a			
radioactive shipment.			
 be disposed as radioactive waste by ESS. 			
Should the samples not be activated:			
• the user can receive them after the RP survey outside of the			
supervised area			
• the samples can be shipped to the home institution by ESS			
• the samples can be stored at ESS for future experiments			
A sample is retired when it leaves ESS (sample life-cycle ends). Samples that are stored for future experiments or await cool-down are stored with the ESD and are the responsibility of the ITE. Samples that have			

been shipped off-site or have been added to the waste stream are retired and the Experiment Safety Report (ESR) is generated.			
Output/product	This is to ensure that samples will be surveyed before leaving the ESS supervised area and before they are retired. Samples that are stored have to be clearly identifiable and have to be accompanied by the ESD, so that hazards are known.		

3.2.11. Output

The final output of the sample management procedure is the Experiment Safety Report (ESR). The ESR includes the ESD, the RP survey tag from the radiological survey and a check-out form filled by the user to make sure no unknown materials are left at the ESS laboratories or ESS storage areas. The check-out form will also contain information related to the post-job briefing to learn from the experiment. The post-job briefing is information for the Safety Review Committee (SRC) for future experiments.

4. RULES, REGULATIONS, CONSTRAINTS, GUIDELINES

For some of the hazardous samples (e.g., for naturally radioactive samples or controlled substances), sample tracking is required. Dedicated ESS personnel make sure that the sample information is entered into a computer database, the ESD is approved and the "Briefing" has been performed before the sample is handled. There will be a separate Sample Tracking Procedure that will cover the way to track a sample during its life-cycle at ESS.

ESS will provide a database that will keep track of where the respective sample is located and who is handling it as well as other parameters that are required. Tracking might in some cases be performed by the users, but can be restricted to the instrument and laboratory teams or other ESS personnel. The controls on the sample depend on the statement in the ESD and can be:

- Administrative: computer-based tracking more or less detailed
- Engineering: samples are locked up with the key being more or less controlled
- Other options

5. GLOSSARY

Term	Definition
CLS	Chemistry Laboratory Support
ESD	Experiment Safety Document *
ESF	Experiment Safety Form (Information on materials, materials hazards and activities for proposed experiment *)
ESH	Environment, Safety & Health
ESR	Experiment Safety Report
ESS	European Spallation Source, ERIC
ESS Users	Scientists and engineers using ESS scientific services.
ITE	Instrument Team, ESS scientists and engineers hosting an experiment on a specific instrument.
NSS	Neutron Scattering Systems
OHS	Occupational Health and Safety
PIC	Person in Charge
PPE	Personal Protection Equipment
PI	Principal Investigator (PI) is the main proposer identified on the experiment proposal.
РТ	Proposal Team (PT) is everyone designated by the PI with the right of access to ESS scientific services.
RML	Radioactive Material Laboratory
RP	Radiation Protection
RPO	Radiation Protection Officer
SD	Science Directorate
SME	Subject Matter Expert
SRC	Safety Review Committee

(*) The described procedure, forms and documents can be supported in digital form as adequate.

6. **REFERENCES**

- [1] NSS Concept of Operations (ESS-0005817)
- [2] Neutron Scattering Systems System Design Description (ESS-0377817)
- [3] Neutron Scattering Systems Radiological Hazards and Radiation Safety Provisions for Operations and Maintenance (<u>ESS-2972939</u>)
- [4] Sample Management Procedure (<u>ESS-0024109</u>)
- [5] Experiment Safety Review Procedure (ESS-0024107)
- [6] ESS Handbook for Radiation Protection Chapter 2. General Radiation Protection Rules (ESS-0239718)
- [7] Swedish Authority Provision AFS 2014:43 (chemical risks); Swedish Authority Provision AFS 2014:1 (biological samples)

DOCUMENT REVISION HISTORY

Revision	Reason for and description of change	Author	Date
1	First issue	Monika Hartl	2015-09-17
2	Update of template, references and some amendments related to sample retirement and digitalisation. Changes to make text more consistent	Monika Hartl	2023-05-12

7. APPENDIX A - SCENARIOS

This appendix shows how the sample handling procedure might work for some typical user proposals. At this stage it is assumed that possible Experiment Safety Forms (ESFs) have been reviewed by the Safety Review Committee (SRC) and an Experiment Safety Document (ESD) has been generated (see document <u>ESS-0024107</u>, [5]). Four scenarios are provided to discuss the different steps of the Sample Management Procedure. All the statements are for demonstrating purposes only.

Experiment Safety Form (ESF)

- 1. General
- 2. Sample description
- 3. Experiment description
- 4. Equipment to be used
- 5. Laboratories to be used

Experiment Safety Document (ESD)

- 1. General (copied from ESF)
- 2. Sample hazards
- 3. Equipment hazards
- 4. Controls (engineering/admin.)
- 5. Experiment Hazard Level (EHL)/Tracking

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Scenario 1:

Experiment Safety Form (ESF)

- General: User X has approved beamtime on a neutron diffractometer with instrument team member Y being the PIC "Temperature dependent diffraction pattern of nano-Silica"
- 2. Sample Description:
 - a. Silica (SiO₂)
 - b. 4 samples with 1g/sample
 - c. Material is a powder.
 - d. Sample should be shipped back to home institution after the experiment.
 - e. No sample can. Sample will arrive in a glass vial.
 - f. A Vanadium can from ESS will be used as sample holder.
- 3. Experiment Description: They will measure 4 silica samples of varying particle size (from nanopower to crystalline silica) at 5 temperatures from 10K to 300K. Samples will be filled into the V cans at ESS under inert atmosphere. After the experiment they want to unload the sample in inert atmosphere.
- 4. Equipment: They are using the cryostat supplied by HEIMDAL (standard equipment) for the temperature dependant measurements. They will not bring equipment from home.
- 5. Aux. Laboratory usage: Sample will be loaded at ESS in inert atmosphere. One sample needs to be heated to 100°C in vacuum. No sample modification after it has been in the beam. No other labs needed. Sample will be unloaded in inert atmosphere. No other equipment needed.

Experiment Safety Document (ESD)

- 1. General information as in ESF
- 2. Samples are non-hazardous. Comment: If sample is nanosized, please always handle in hood or glove box.
- 3. Equipment: Follow rules and regulation supplied by instrument team for the use of the standard equipment.
- 4. No change in roles/responsibilities for the experiment. Chemistry laboratory training needed for use of laboratory and furnace.
- 5. EHL: low, no tracking needed.

Planning: Instrument team (ITE) will make sure there is a furnace in the chemistry laboratories available to the user and will coordinate for on the job training with Chemistry Laboratory Support (CLS). ITE receives the sample shipment from the user and will keep it in their cabinet until arrival of the user together with the ESD.

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<u>Briefing</u>: The user has completed the required general training. ITE meets with the user and talks about how to perform the experiment. According to the users' wishes, the plan is to load the samples into Vanadium cans in a glove box (inert atmosphere). Vanadium cans will be closed for the experiment. After the experiment the samples will be surveyed by the Radiation Protection Officer (RPO) before unloading in a glove box by the user. The ITE and the user talk to the CLS about the heating of the sample in a furnace. A workspace is assigned to the user and the user receives training on how to use the furnace and the glove box. The sample that needs heating in the furnace will be filled into a quartz tube through the valve on top in a hood. It will be heated under vacuum at 100°C. The valve will be closed when the heating is finished and sample will be transferred into Vanadium can inside the glove box.

<u>Experiment</u>: After signing the pre-job brief, the user starts the experiment. The user handles the Vanadium cans on the instrument and takes them to the RPO for survey afterward. Once the survey is done and release tags are on the samples, the user goes to the chemistry laboratory and unloads the sample into the glove box. The samples are filled into closed vials and will be shipped by ESS together with the release tag.

<u>OUTPUT:</u> An Experiment Safety Report (ESR) will be generated from the ESD and with copies of the release tags. The ITE signs a form that the user has checked out after removing their belongings from the support laboratories and instrument. Since the ITE is bringing the samples to the ESS mail station, the ITE is stating that the sample is retired by shipment to the home institution (end of sample life-cycle).

Scenario 2:

Experiment Safety Form (ESF):

- 1. General: User X has approved beamtime on a SANS machine with instrument team member Y being the PIC. Title: "Magnetic SANS on Iron soaps"
- 2. Sample Description:
 - a. Ferric Oleate, Fe(C₁₈O₂H₃₃)₃
 - b. One sample with 4g ferric oleate, powder but not nanoparticle
 - c. Material is a liquid
 - d. Sample can be disposed at ESS after the experiment.
 - e. No. Sample will arrive in a glass vial.
 - f. 11 quarz cuvettes from the users will be used as sample holder. Will need laboratory to weigh samples (0.2g/sample), mix them with 10ml H₂O/D₂O mixtures and load sample holders
- Experiment Description: They will mix 11 different samples containing 0.2g ferric oleate and 10ml H₂O/D₂O mixture. Each sample will contain a varying ratio of H₂O/D₂O: 0:10,1:9,2:8,3:7,4:6,5:5,6:4,7:3,8:2,9:1,10:0. They will also measure 11 respective H₂O/D₂O mixtures without ferric oleate for comparison. Samples will be filled into the quartz cuvettes (max volume=1 ml) in the chemistry laboratory. Cuvettes will be placed into magnet and measured at 10T. After one set of samples (11) they want to reuse their cuvettes.
- 4. Equipment: They are using the standard sample changer supplied by at room temperature. They will not bring equipment from home.
- 5. Aux. Laboratory usage: Sample will be mixed and loaded at ESS. After the measurement, they want to look at the IR signal from the sample in the spectroscopy lab at ESS. Will use spectrometer.

Experiment Safety Document (ESD):

- 1. Samples are non-hazardous.
- 2. Magnetic field poses a hazard.
- 3. Equipment: Follow rules and regulation supplied by instrument team for the use of the instrument. Administrative and engineering controls will be put into place for the use of the magnet. The magnet is interlocked with the door of the instrument cave. As soon as the door is opened the magnet is shut down. There will be signage on the door and on the instrument that there is a magnet in use. Users with pacemakers will be informed on this. Under these controls, the experiment can be classified as EHL: low
- 4. No change in roles/responsibilities for the experiment. Chemistry laboratory training needed for use of laboratory and furnace.
- 5. EHL: low, no tracking needed.

<u>Planning</u>: The Instrument Team (ITE) will coordinate with sample environment team to obtain the magnet and with systems engineering on implementing the control that the magnet shuts off upon

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entering the instrument cave. ITE will coordinate for on the job training with Chemistry Laboratory Support (CLS). ITE receives the sample shipment from the user and will keep it in their cabinet until arrival of the user, together with the ESD.

Briefing: The user has completed the required general training. ITE meets with the user and talks about how to perform the experiment. The plan is to load the samples into quartz cells on the instrument bench area since the samples are non-hazardous. ITE will supply the needed PPE, nitrile gloves and googles. After the experiment the samples in the guartz cuvettes will be emptied into a collection container that will be surveyed by the RPO before disposal. The cuvettes will be rinsed with H₂O and the wash liquid will be collected into the collection container. The ITE and the user talk to the CLS about using the pipettes from the chemistry laboratory for mixing H₂O/D₂O mixtures and the stirrer for dissolving the iron soaps in the mixtures. The user gets assigned to a work space and training.

Experiment: The user performs the experiment him/herself using the chemistry laboratory to make the solutions, transporting the solutions to the instrument area and filling the cuvettes on the instrument. After the first set of experiments the user empties the cuvettes into the collection container using nitrile gloves, googles and a lab coat. The user rinses the cuvettes twice using distilled water collecting the rinse in the collection container. The user does the same with the second set of samples. At the end of the experiment, the user brings the collection container to the RPO for survey. After the container has come back with a release tag, the user hands the container to CLS together with the ESD.

Output: CLS will sign that the sample is retired by disposal and an ESR is generated. ITE will sign the check-out for the user.

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Experiment Safety Form (ESF):

- 1. General: User X has approved beamtime on an inelastic spectrometer with instrument team member Y being the PIC. Titel: "Phononmodes of the high explosive TATP at 10-100K".
- 2. Sample Description:
 - a. Triacetonetriperoxide (C₉H₁₈O₆)
 - b. 1 sample with 1g/sample
 - c. Material is a powder.
 - d. Sample should be destroyed after experiment.
 - e. No sample can. Sample will arrive in a plastic vial.
 - f. An aluminium pouch in a large Al can from ESS will be used as sample holder.
- 3. Experiment Description: They will load the sample onto the Al foil at ESS. The foil will be rolled and placed into a large Al sample can with an open valve on the top (release of pressure should the sample decompose).
- 4. Equipment: They are using the cryostat supplied by VOR (standard equipment) for the temperature dependant measurements. They will not bring equipment from home.
- 5. Aux. Laboratory usage: Sample will be loaded at ESS. Sample needs to be destroyed at ESS. No other labs needed. No other equipment needed.

Experiment Safety Document (ESD):

- 1. General information as in ESF
- 2. Sample is high explosive, but amount used in experiment has very little energy stored in it. Sample will be shipped with special transport arranged by user to ESS and stored at temperatures below 10 °C in the refrigerator in laboratory XY to prevent decomposition. The refrigerator will not be used by other users during that time. The sample will be brought over to the experiment and immediately cooled down in the cryostat. After the experiment the sample will be removed while it is still cold. The sample will be put back into the refrigerator until a dedicated chemist can dissolved it in water (after dissolving it is no longer explosive) in a hood. The products will be brought to the RCT for survey before it will be further destroyed.
- 3. Equipment is standard. However, the sample vial will sit in a vacuum containment not in He gas, so in case of a decomposition of the sample, there will be a large volume for the gas to expand in.
- 4. Equipment: Follow rules and regulation supplied by instrument team for the use of the instrument. Only restricted personnel is allowed in the hutch for the sample change. There will be a chemist with experience available at the begin and end of the experiment for sample change. There will be a dedicated chemist taking part in the experiment. The responsibility for the experiment will be shared between instrument team and the ESS chemist. Chemistry laboratory training needed for use of laboratory.

5. EHL: medium, tracking needed.

<u>Planning</u>: The Instrument Team (ITE) will coordinate with the Chemistry Laboratory Support (CLS) to receive the high-explosive sample and have a dedicated refrigerator/freezer available. The CLS will provide an experienced chemist that will be part of the experiment and assist the user with the sample handling and sample storing before, during and after the experiment. ITE will coordinate for on the job training with CLS. CLS has a plan on how to destroy the sample after the beam time.

Briefing: The user has completed the required general training. ITE meets with the user and CLS to talk about how to perform the experiment. The plan is to freeze the samples in the -18°C freezer before loading to avoid the sample is warming up to more than +10°C during the loading process. The loading process will be performed behind a plastic shield in a laboratory that will have restricted access during the loading process. The required PPE consists of goggles, leather gloves and lab coat and will be supplied by CLS. The samples will be loaded into an open Teflon canister (soft material) behind a plastic shield. The open Teflon container will then be put into a larger aluminium container that can be closed and has a valve on top. This container will be evacuated while pulling vacuum on the sample container. After the loading process, the sample will be put back into the refrigerator keep its temperature below +10°C. When the instrument is ready to receive the sample, it will be brought over inside in a plastic container with ice. The sample will immediately be cooled down in the instrument cryostat. After the measurements the sample will be taken from the instrument and carried in a plastic container with ice to the laboratory and stored in the refrigerator until disposal. CLS is planning to dispose of the sample a week after the experiment by dissolving it in water and destroying it with chemicals. Since the explosion hazard is much higher than the radiation hazard the sample will be destroyed before being surveyed.

<u>Experiment:</u> The user performs the experiment with the chemist. They follow the experimental plan and the sample is stored after the experiment in the refrigerator. The chemist dissolves the compound in water in the hood in the chemistry laboratory and collects the solution. The explosive is destroyed by adding strong acid and the solution is brought to the RPO for survey in a closed container.

<u>Output</u>: CLS will sign that the sample has been retired by disposal (end of sample life-cycle) and an ESR is generated. ITE will sign the check-out for the user.

Procedure ESS-0024112

General: User X has approved beamtime on the diffractometer with instrument team member Y being the PIC "Measuring nanopowder of Plutonium at various temperatures between 10K and 300K".

- 1. Sample Description:
 - a. Plutonium Powder
 - b. 1 sample with 0.5g/sample
 - c. Material is a powder.
 - d. Sample should be sent to home institution after experiment.
 - e. Sample will arrive in a plastic vial in a lead drum.
 - f. A Vanadium can from ESS will be used as sample holder.
- 2. Experiment Description: They will load the sample into the V can at ESS.
- 3. Equipment: They are using the cryostat supplied by the diffractometer (standard equipment) for the temperature dependant measurements. They will not bring equipment from home.
- 4. Aux. Laboratory usage: Sample will be loaded at ESS. Sample needs to be sent back. No other labs needed. No other equipment needed.

Experiment Safety Document (ESD)

- 1. General information as in ESF
- 2. Sample is radioactive material AND nuclear material
- 3. Equipment: standard equipment
- 4. Follow rules and regulation supplied by instrument team for the use of the standard equipment. Put controls in place for radioactive materials/nuclear materials. change in roles/responsibilities for the experiment: 2 people responsible for sample tracking/handling
- 5. Tracking necessary. EHL: high

Planning: The Instrument Team (ITE) will correspond with the user on the type of sample container suitable for these measurements. ITE will send the user a suitable ESS sample canister. ITE will check with the nuclear materials custodian about storing the sample and inventory questions. ITE will coordinate with RP to have personnel ready to receive the Plutonium (Pu) sample and survey it upon arrival. ITE confirms that the sample is added to the tracking system upon arrival.

Briefing: The user has completed the required general training. ITE meets with the user and RP to talk about how to perform the experiment. The plan is to store the Pu sample in a locked cabinet on the instrument after RP releases it to the ITE and user. The keys to the cabinet are given to one member of ITE and one member of the user team. The user is aware that the movement of the sample

Date

State

ESS-0024112

has to be tracked by using the sample tracking system. The user is trained on the use of the sample tracking system by ITE. The user is aware that RP will survey the sample before removal from the instrument. After the experiment and survey, the sample will be sent back to the home institution. If the sample container can be decontaminated after removal of the Pu sample at the home institution, it will be sent back to ESS.

Experiment: The user performs the experiment according to the briefing. Radiation Protection (RP) surveys the sample after the measurement and finds no removable contamination. The sample is returned to the locked cabinet. The keys are given to the Radiation Protection Officer (RPO) and the RPO collects the sample and packs it before shipping it back to the home institution. RPO in collaboration with the ESS mail department will make sure the home institution is allowed to receive radioactive material and the transport is performed according to the international transport laws for radioactive goods.

Output: The RPO will sign that the sample has been retired by returning it to the home institution (end of sample life-cycle) and an ESR is generated. ITE will sign the check-out for the user.