EUROPEAN ORGANIZATION FOR NUCLEAR RESEARCH

Letter of Intent to the ISOLDE and Neutron Time-of-Flight Committee

Long lived isotopes for emission Mössbauer spectroscopy during the long shutdown 2018-2020

[31. 5. 2017]

Roberto Mantovan[1], Alberta Bonanni[2], Jose Angel Garcia[3], Haraldur P. Gunnlaugsson[4], Ulli Köster[5], Doru C. Lupascu[6], Hilary Masenda[7], Yury Matveyev[8], Deena Naidoo[7], Sveinn Ólafsson[4], Fernando Plazaola[3], Iraultza Unzueta Solozabal[3], Juliana Schell[6,9], Andrei Zenkevich[8], Jianhua Zhao[10], Krish Bharuth-Ram[11], Karl Johnston[9]

[1]Laboratorio MDM, CNR-IMM, Unità di Agrate Brianza, Via Olivetti 2, 20864 Agrate Brianza (MB) Italia
[2]Institute for Semiconductor and Solid State Physics, Magnetic Spin Materials Group, Johannes Kepler University, Altenbergerstr. 69, Linz, Austria
[3]Dpto. Electricidad y Electronica, Universidad del Pais Vasco (UPV/EHU), CP. 644, 48080 Bilbao, Spain
[4]Science Institute, University of Iceland, Dunhaga 3, 107 Reykjavík, Iceland
[5]Institut Laue-Langevin, Grenoble, France
[6]Institute for Materials Science and Center for Nanointegration Duisburg-Essen (CENIDE), University of Duisburg-Essen, 45141 Essen, Germany
[7]School of Physics, University of the Witwatersrand, WITS 2050, South Africa
[8]Moscow Institute of Physics and Technology, 9 Institutskiy per., Dolgoprudny, Moscow Region, 141700, Russian Federation
[9]European Organization for Nuclear Research (CERN), CH-1211 Geneva, Switzerland
[10]State Key Laboratory of Superlattices and Microstructures, Institute of Semiconductors, Chinese Academy of Sciences, China
[11]Durban University of Technology, South Africa

Spokesperson(s): R. Mantovan (roberto.mantovan@mdm.imm.cnr.it), H. P. Gunnlaugsson (hpgunnlaugsson@cern.ch)
Local contact: Karl Johnston (karl.johnston@cern.ch), Juliana Schell (juliana.schell@cern.ch)

CERN-INTC-2017-061 / INTC-I-192
31/05/2017

Abstract

We propose using implantation of the long-lived isotopes ^{57}Co and ^{119m}Sn for off-line Mössbauer Spectroscopy (MS) investigations in solid state physics during the long shutdown 2018-2020. This will be an excellent addition to the existing experiments program using eMS at ISOLDE, and will benefit the physics program of the existing emission MS experiments. In this Letter of Intent, we outline some of the experiments that will benefit from the use of long lived isotopes and which we propose to do. We also give estimates of the implantation doses required. It is assumed that formal proposals will be requested at a later stage.

Requested shifts: ~7×10^{13} ^{57}Co and ~7×10^{13} ^{119m}Sn during the 2018-2020 long shutdown.

1 USE OF ^{57}Co AND ^{119m}Sn

1.1 Introduction

The relevant parts of the decay schemes of radioactive parent isotopes to the Mössbauer states of ^{57}Fe and ^{119}Sn are shown in Fig. 1.

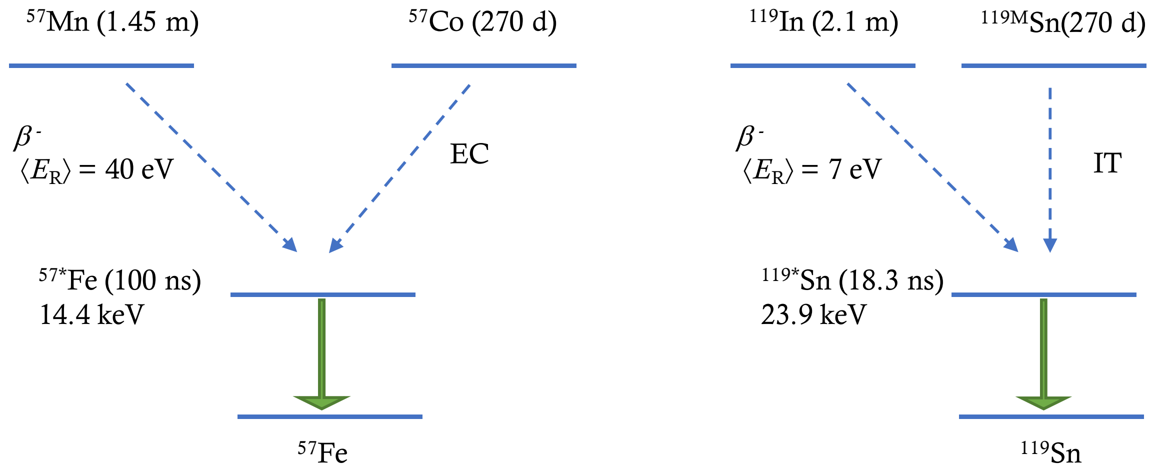


Fig. 1: Decays to the Mössbauer states of ^{57}Fe and ^{119m}Sn.

In experiments IS576, IS578 and IS630, we utilize (mostly) the short-lived isotopes ^{57}Mn and ^{119}In. The experimental conditions are limited to what can be obtained in an on-line setup. This limits the measurement temperature to 77 K (liquid nitrogen) and annealing is only possible at the measurement temperature during the lifetime of the probe atom.

The use of longer-lived pre-cursor isotopes opens up three new possibilities:

(1) Address the nature of implantation induced damage associated with short lived radioactive implantation through systematic and longer annealing.

(2) One can measure under different external experimental conditions and is not limited to what is possible in on-line setups. This includes (amongst others) external magnetic fields and low temperatures.

(3) Comparison with the results obtained with short lived pre-cursor isotopes, can give additional new information, such as the effect of recoil (no interstitials are created).

2 SCIENTIFIC MOTIVATION

In this LOI, several systems are described that are already a part of existing and/or recently concluded experiments at ISOLDE.

2.1 MnSi (part of IS578)

Mn-Si alloys exhibiting room temperature ferromagnetism (RTFM) are of high interest for establishing spin-based electronics with revolutionary potential in the modern information technology [1], the study of which is the focus of IS578. Within IS578 [2], we aim at identifying the role of defects and/or inclusions of additional phases in the origin and evolution of the internal magnetic field in Mn-rich Mn_xSi_{1-x}, as a function of composition x. Until now very promising data based on the short-lived ^{57}Mn isotope have been recorded but we encountered a major issue (common also to the other classes of Mn-based alloys): a huge contribution from implantation-induced damage which critically hinders some relevant features that could explain the atomic-scale origin of the macroscopic RTFM.

2.1.1 Current status

We have excellent data obtained with ^{57}Mn on the MnSi system. However, magnetic interactions seem to take place below ~110 K, which is difficult to measure on-line. Data obtained with ^{57}Co would allow us to make low temperature measurements, and strengthen the conclusions that can be drawn from the use of ^{57}Mn considerably.

2.1.2 Proposed experiments

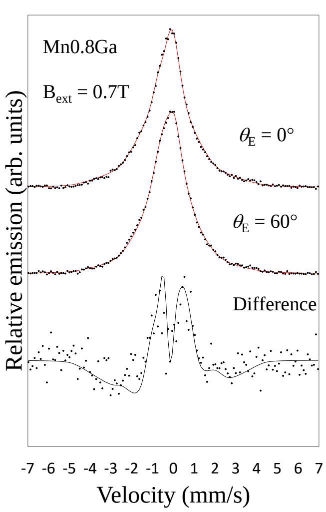
By implanting ^{57}Co (going to the metal (Mn) site), one can do eMS measurements as a function of annealing steps, to follow the damage annealing, and how this benefits the magnetic properties by measurements at low temperatures. This should be done for at least 3 samples. ^{119m}Sn (presumably going to the Si site) can be used.

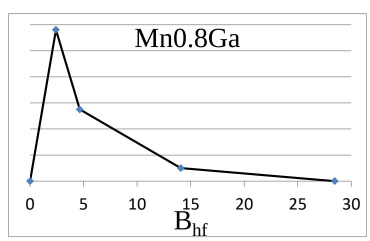
2.2 Ga$_x$Mn (part of IS578)

Rare-earth-free magnets such as Mn$_x$Ga have recently attracted much attention as new permanent magnets and materials for high-density perpendicular magnetic recording, magnetoresistive sensors, and spin-transfer-torque magnetic-RAM [2]. The samples that are used within the IS578 experiments are growth with state-of-the-art methods by the group headed by Prof. J. Zhao.The Mn$_x$Ga magnetic properties have little dependence on the annealing temperature but remarkable changes are observed by changing *annealing time* and/or by varying their stoichiometry *x* [2], for which ^{57}Co is an ideal probe.

2.2.1 Current status

The first experiments have concentrated on determining the basic properties of the magnetic order. The results obtained with ^{57}Mn (example in Fig. 2) indicate either (1) very low magnetic moment on the Mn sublattice (~0.5 μ_B/Mn) and/or (2) significant sensitivity to implantation.





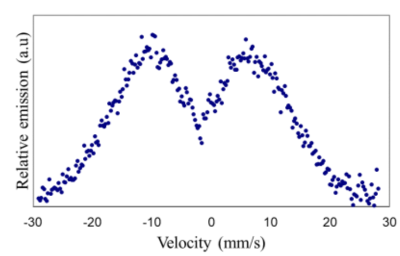


Fig. 2 Left: Field dependent ^{57}Fe eMS results obtained after implantation of ^{57}Mn, analysed in terms of magnetic hyperfine field distribution (middle). The distribution suggests magnetic moment ~0.5 μ_B/Fe. Right: ^{119}Sn eMS spectrum obtained after implantation of ^{119}In. The huge splitting corresponds to magnetic moment ~4 μ_B/Sn.

Preliminary results obtained with ^{119}In (which would occupy the Ga sublattice) suggest an order of magnitude higher magnetic moment, suggesting that the Ga atoms are responsible for the peculiar magnetic properties of the material.

2.2.2 Proposed experiments

At the moment, we have not been able to observe the details of the magnetic order of GaMn films, in particular, to verify the out-of plane magnetic properties. There are several possibilities:

(1) The magnetization is not on the Mn sublattice, but on the Ga sublattice. In this case, one should investigate the magnetic properties by ^{119m}Sn eMS, which would go on the Ga sublattice. Here it will be possible anneal and perform measurements at low temperatures. Some investigations are planned using ^{119}In during the 2017 Mn/In beam-times (June 2017) that may impact this plan.

(2) More careful annealing is needed to eliminate the implantation damage, and measurements at low temperatures. This is irrespective of whether the magnetization is on the Mn or the Ga sublattices.

It is proposed to investigate at least 4 samples should be investigated, two with ^{57}Co and two with ^{119m}Sn implantations. For ^{119m}Sn, a higher implantation dose is needed than usually due to the low resonance effect in the GaMn's.

Sample Ga_xMn	Isotope	Fluence
$x \sim 0.8$	^{57}Co	3×10^{12}
$x \sim 1.3$	^{57}Co	3×10^{12}
$x \sim 0.8$	^{119m}Sn	1×10^{13}
$x \sim 1.3$	^{119m}Sn	1×10^{13}

2.3 Heusler Alloys (part of IS578)

Ni-Mn-Z (Z = In, Sn, Sb) alloys are attracting considerable attention due to the multifunctional properties they exhibit (such as giant magnetoresistance, magnetic shape memory effect and large magnetocaloric effect) which arise as a result of the coupling between structure and magnetism [5,6,7]. The introduction of Co has been shown to induce an almost paramagnetic martenisite phase which enhances the magnetocaloric effect and the magnetic-field-induced transformation due to the high metamagnetic character. Additionally, the presence of Co increases the atomic scale magnetism compared with the ternary alloys. While some works point out that Co atoms preferably occupy Mn sites, others suggest that Co occupies Ni sites (among other existing interpretations) [9,10,11]. The study of the implantation of ^{57}Co would help in the understanding of the preferred sites of Co (<10^{-4} at%) as the local environment surrounding its daughter isotope (^{*57}Fe) can be easily monitored by ^{57}Fe MS. Additionally, the results obtained from ^{119m}Sn would add valuable information in Ni-Mn-In samples.

2.3.1 Current status

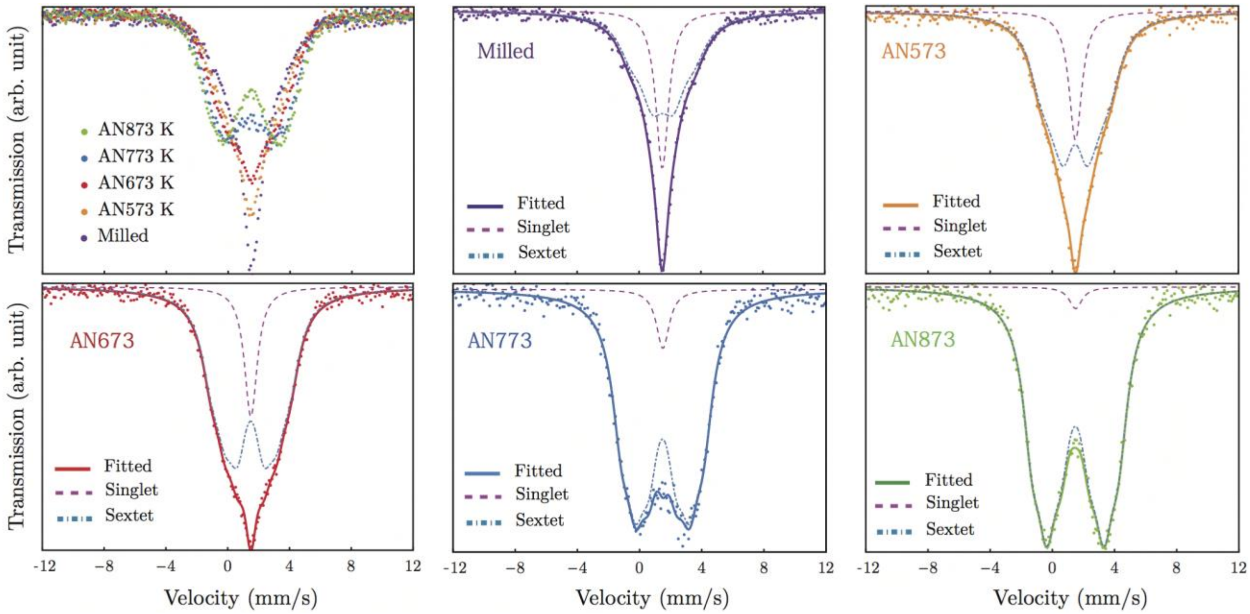


Fig. 3: ^{119}Sn Mössbauer spectra evolution of the milled $Ni_{50}Mn_{35}Sn_{15}$ sample annealed from 573 K up to 873 K.

2.3.2 Proposed experiments

In order to elucidate the preferred lattice position of the Co, Ni-Mn-Sn and Ni-Mn-In ternary samples will be implanted with ^{57}Co. In addition, ^{119m}Sn will also be implanted in both types of ternary samples. The interpretation of the obtained spectra will be supported by the existing data on ^{119}Sn MS, [12] which can be used as a reference.

Afterwards, the effect of the Co addition will be investigated in the NiMnSnCo samples and NiMnInCo samples by implanting both ^{57}Co and ^{119m}Sn. In this case, the origin of the different metamagnetic characters of the two samples will be obtained by comparing Ni-Mn-Sn and Ni-Mn-In alloys. Due to the long half-life of the implanted isotopes, annealing treatments could be also be employed in order to investigate the ordering/diffusion dynamics in these alloys.

Sample	Isotope	Fluence
NiMnIn	^{57}Co and ^{119m}Sn	5×10^{12}
NiMnSn	^{57}Co and ^{119m}Sn	5×10^{12}
NiMnInCo	^{57}Co and ^{119m}Sn	5×10^{12}
NiMnSnCo	^{57}Co and ^{119m}Sn	5×10^{12}

2.4 AlGaN's and InGaN's (Part of IS576 and IS630)

Ternary group III-nitrides have been the focus of intensive research over the past few decades due to the possibility of tuning their unique physical properties such as the energy band gap, effective masses of holes and electrons, and dielectric constants. This has opened a range of applications spanning over commercial full colour light emitting diodes, laser diodes, solar cells to high power field effect transistors. Experiments IS576 and IS630 are devoted to the investigation of AlGaN's and InGaN's, respectively.

2.4.1 Current status

The IS576 (Al,Ga)N's(:Mn) experiment [13] is nearing completion, and papers are being prepared. ^{57}Mn has been shown to occupy regular lattice sites, some of which are associated with point defects [14]. The stability of these defects has not been explored in detail. The IS630 (In,Ga)N's experiment has not yet started.

2.4.2 Proposed experiments

Interest in Sn doped III-nitrides has been motivated by the possibility to explore *p*-type doping in inherently *n*-type nitrides. This requires a detailed knowledge of the nature of defects (native and extrinsic), lattice sites and annealing behaviour which we hope to obtain from measurements following ^{119m}Sn implantation. ^{57}Co implantation, on the other hand, will allow for detailed investigations on the lattice location and annealing behaviour over longer periods of time. Coupled with measurements in an external magnetic field and as a function of emission angles the measurements will extend our understanding of the magnetic properties due to Fe incorporation into these materials.

A total of 14 samples will be studied, 7 with ^{57}Co (3×10^{12} at.cm^{-2}) and 7 with ^{119m}Sn (3×10^{12} at.cm^{-2}) implanted with the indicated fluences in each sample for the x = 0.0, 0.2, 0.6 and 1.0 for $Al_xGa_{1-x}N$ and x = 0.09, 0.13 and 1.0 for $In_xGa_{1-x}N$.

2.5 Oxides (extension of the IS501 experiment)

Previous results have indicated but not proven cage motion (fast jumping between nearby interstitial sites) observed in oxide materials such as α-Al_2O_3. [17]. These outstanding question cannot be resolved with ^{57}Mn, which would feasible with ^{57}Co and low temperature measurements. We suggest the study of ~3-4 oxides where single lines have been observed (α-Al_2O_3, MgO, …) and 3 to 4 metals (Si, Zr, …). This could suggest that cage motion of interstitial Fe is more common than hitherto believed. ~7 samples implanted with ~3×10^{12} ^{57}Co are needed.

3 TARGET/FLUENCES/YIELDS

Based on previous tests using ^{57}Co and ^{119m}Sn, useful samples contain $(3\text{-}5)\times10^{12}$ ^{57}Co atoms or $(3\text{-}10)\times10^{12}$ ^{119m}Sn depending on the complexity of the spectrum, the number of measurements needed, Mössbauer recoil free fraction etc.

^{119m}Sn can be produced on-line with an UC_x target and a Sn RILIS ion source. Yields of ^{119m}Sn have been measured to be $1.8\times10^9/\mu$C, accompanied by about $1.0\times10^9/\mu$C ^{119g}Sn [19]. A standard UC_x target that has been irradiated "cold" for one day with 1 μA proton beam and stored for half a year before reheating and ionizing with the RILIS would allow to implant about 10^{14} ions ^{119m}Sn off-line.

^{57}Co has been produced at ISOLDE with ZrO_2 targets and VADIS ion source. Yields during the 2010 beam time were ~$5\times10^7/\mu$C, but during the 2011 beam time, the yields with the same settings were at least one order of magnitude lower. Use of RILIS can be expected to increase the yield by a small factor. Assuming a RILIS efficiency of 10% and the measured spallation cross-section of 6 mb, a 6 g/cm^2 ZrO_2 felt target should provide a yield of $1.5\times10^8/\mu$C. If such a target were irradiated "cold" for four days with 2 μA proton beam and stored for 3 months, a total of 7×10^{13} ions ^{57}Co could be implanted.

Alternatively, ^{57}Co produced elsewhere (commercially available or produced e.g. by Fe(d,n) reactions at a cyclotron) could be introduced into an ISOLDE target and ion source unit, then ionized and implanted off-line. The intended quantity of 7×10^{13} ions corresponds to an activity of 2 MBq. Assuming a RILIS efficiency of about 10%, about 20 MBq ^{57}Co are required. This level of activity can be easily handled and the low-energy gamma rays of ^{57}Co are easily shielded, already by the 1 mm thick Ta target container

In addition, these experiments would require the presence of support from CERN's RP services during working hours.

4 SUMMARY OF REQUESTED SHIFTS:

Project	# ^{57}Co	# ^{119m}Sn
MnSi	9×10^{12}	9×10^{12}
Ga_xMn	6×10^{12}	20×10^{12}
Heusler Alloys	6×10^{12}	6×10^{12}
AlGaN's and InGaN's	21×10^{12}	21×10^{12}
Oxides	21×10^{12}	
Opportunistic/calibration (20%)	13×10^{12}	12×10^{12}
Total	7×10^{13}	6.8×10^{13}

For off-line implantations the ion current can be easily tailored to requirements by heating the target appropriately. Total currents of about 100 nA should not be exceeded in order to prevent jeopardizing the RILIS efficiency. Considering that several isotopes of the element of interest are contained in the target and will be ionized the current of the desired isotope/isomer (^{57}Co and ^{119m}Sn respectively) can reach several nA. Thus the actual implantation of all samples will not take more than few hours. Obviously outgassing the target, setting up the stable beam and tuning the RILIS will require more time.

In total, it is estimated that 3-4 days of implantation should be foreseen for each target would suffice.

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Appendix

DESCRIPTION OF THE PROPOSED EXPERIMENT

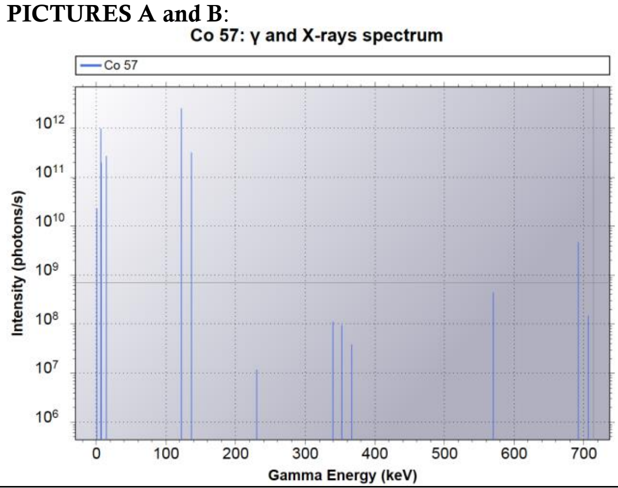
The experimental setup comprises: *(name the fixed-ISOLDE installations, as well as flexible elements of the experiment)*

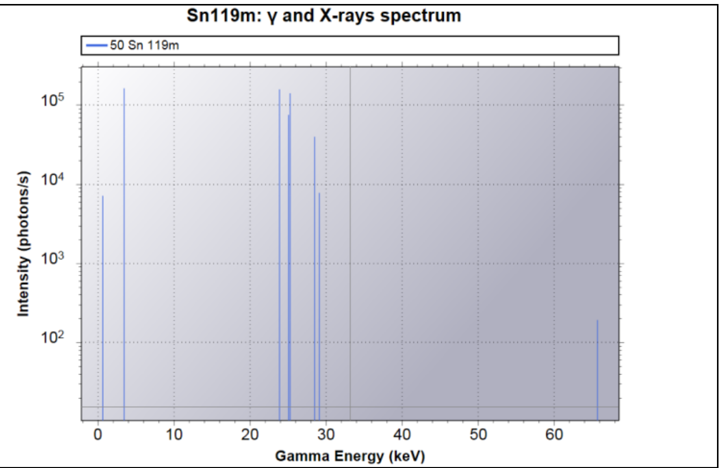
Part of the Choose an item.	Availability	Design and manufacturing
Part 1 of the experiment: SSP-GLM chamber located at GLM beam line in the ISOLDE hall	☒ Existing	☒ To be used without any modification

HAZARDS GENERATED BY THE EXPERIMENT

(if using fixed installation) Hazards named in the document relevant for the fixed [COLLAPS, CRIS, ISOLTRAP, MINIBALL + only CD, MINIBALL + T-REX, NICOLE, SSP-GLM chamber, SSP-GHM chamber, or WITCH] installation.

Additional hazards:

Hazards	*[Part 1 of the experiment]*
Pressure	Low pressure only
Vacuum	10^{-6} mbar during collection
Temperature	Room temperature
Heat transfer	
Thermal properties of materials	
Cryogenic fluid	
Electricity	230 V, max. 10 A single phase
Static electricity	
Magnetic field	[magnetic field] [T]
Batteries	☐
Capacitors	☐
Target material	Samples described in the proposal text
Beam particle type (e, p, ions, etc)	**PICTURES A and B**: 

	
Beam intensity	$<10^9\ s^{-1}$
Beam energy	30-50 keV
Cooling liquids	[liquid]
Gases	[gas]
Calibration sources:	☐
• Open source	☐
• Sealed source	☐ [ISO standard]
• Isotope	
• Activity	
Use of activated material:	
• Description	☒ Removal from chamber and shipping to home institution: The CERN Radiation Protection Group will assist the Logistic Services GS/IS in organising the shipping of all radioactive material from CERN to other institutes or external organisations, for the radiation protection issue. Our local contact will contact the Radiation Protection Service RP beforehand: Building 24/E-024 Email: service-rp-shipping@cern.ch
• Dose rate on contact and in 10 cm distance	**Information A** Dose equivalent near to the source: ^{57}Co: $\dot{H}'(0.07)$ = 8.86e-03 mSv/h in 10 cm Dose equivalent in contact with the source: $\dot{H}'(0.07)$ = 8.855 mSv/h in contact

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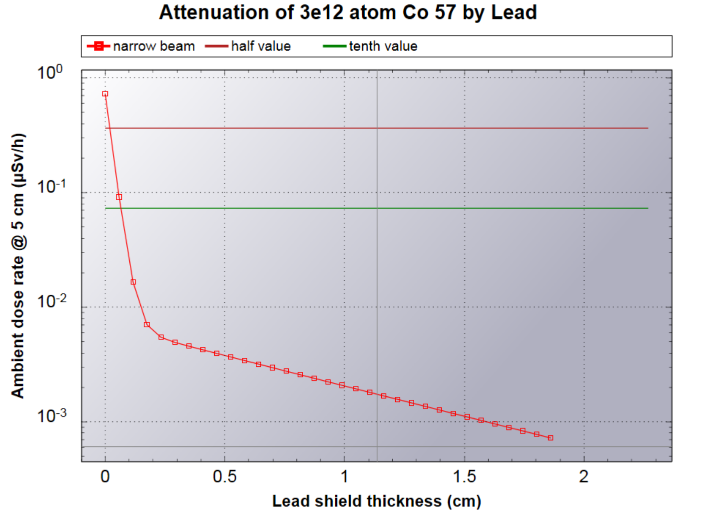
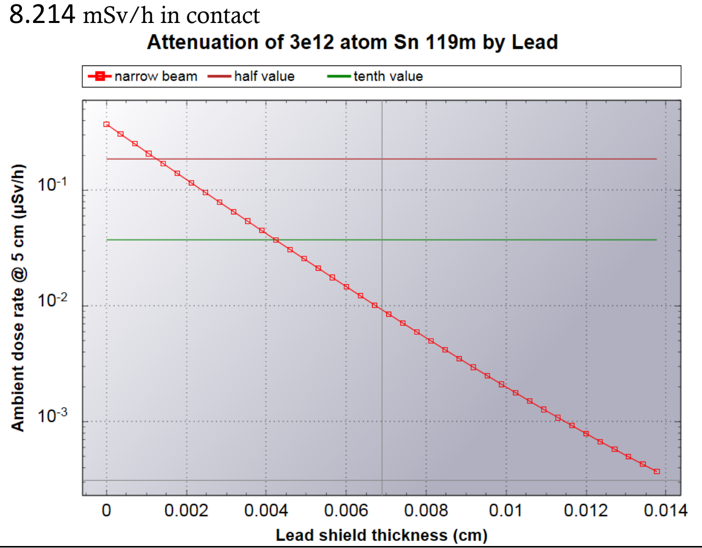


Dose equivalent near to the source:
^{119m}Sn:
$\dot{H}'(0.07)$ =
8.21e-05 mSv/h in 10 cm
Dose equivalent in contact with the source:
$\dot{H}'(0.07)$ =
8.214 mSv/h in contact


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<td>• Isotopes</td>
<td>Information B
^{57}Co
^{119m}Sn
Both are long-lived and require follow up by

RP. The document EDMS will apply accordingly.</td>
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<td>• Activities</td>
<td>Information C
Nucleonica data for 3e+12 atoms:
^{57}Co:
8.855e+4 Bq (LA = 8e+6 Bq)
^{119m}Sn:
8.214e+4 Bq (LA = 3e+6 Bq)</td>
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<tr>
<td>Laser</td>
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<td>UV light</td>
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Microwaves (300MHz-30 GHz)	
Radiofrequency (1-300MHz)	
Toxic	[chemical agent], [quantity]
Harmful	[chemical agent], [quantity]
CMR (carcinogens, mutagens and substances toxic to reproduction)	[chemical agent], [quantity]
Corrosive	[chemical agent], [quantity]
Irritant	[chemical agent], [quantity]
Flammable	[chemical agent], [quantity]
Oxidizing	[chemical agent], [quantity]
Explosiveness	[chemical agent], [quantity]
Asphyxiant	[chemical agent], [quantity]
Dangerous for the environment	[chemical agent], [quantity]
Physical impact or mechanical energy (moving parts)	[location]
Mechanical properties (Sharp, rough, slippery)	[location]
Vibration	[location]
Vehicles and Means of Transport	[location]
Frequency	ISOLDE hall background
Intensity	
Confined spaces	[location]
High workplaces	[location]
Access to high workplaces	[location]
Obstructions in passageways	[location]
Manual handling	[location]
Poor ergonomics	[location]

0.1 Hazard identification

3.2 Average electrical power requirements (excluding fixed ISOLDE-installation mentioned above): *(make a rough estimate of the total power consumption of the additional equipment used in the experiment)*

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The experimental setup comprises: *(name the fixed-ISOLDE installations, as well as flexible elements of the experiment)*

Part of the Choose an item.	Availability	Design and manufacturing
SSP-GLM chamber	☒ Existing	☒ To be used without any modification
[Part 1 of experiment/ equipment]	☐ Existing	☐ To be used without any modification ☐ To be modified
	☐ New	☐ Standard equipment supplied by a manufacturer ☐ CERN/collaboration responsible for the design and/or manufacturing
[Part 2 experiment/ equipment]	☐ Existing	☐ To be used without any modification ☐ To be modified
	☐ New	☐ Standard equipment supplied by a manufacturer ☐ CERN/collaboration responsible for the design and/or manufacturing
[insert lines if needed]		

HAZARDS GENERATED BY THE EXPERIMENT

(if using fixed installation) Hazards named in the document relevant for the fixed [COLLAPS, CRIS, ISOLTRAP, MINIBALL + only CD, MINIBALL + T-REX, NICOLE, SSP-GLM chamber, SSP-GHM chamber, or WITCH] installation.

Additional hazards:

Hazards	*[Part 1 of the experiment/equipment]*	*[Part 2 of the experiment/equipment]*	*[Part 3 of the experiment/equipment]*
Thermodynamic and fluidic			
Pressure	[pressure][Bar], [volume][l]		
Vacuum			
Temperature	[temperature] [K]		
Heat transfer			
Thermal properties of materials			
Cryogenic fluid	[fluid], [pressure][Bar], [volume][l]		
Electrical and electromagnetic			
Electricity	[voltage] [V], [current][A]		
Static electricity			
Magnetic field	[magnetic field] [T]		
Batteries	☐		
Capacitors	☐		
Ionizing radiation			
Target material	[material]		
Beam particle type (e, p, ions, etc)			
Beam intensity			

Beam energy			
Cooling liquids	[liquid]		
Gases	[gas]		
Calibration sources:	☐		
• Open source	☐		
• Sealed source	☐ [ISO standard]		
• Isotope			
• Activity			
Use of activated material:			
• Description	☐		
• Dose rate on contact and in 10 cm distance	[dose][mSV]		
• Isotope			
• Activity			
Non-ionizing radiation			
Laser			
UV light			
Microwaves (300MHz-30 GHz)			
Radiofrequency (1-300MHz)			
Chemical			
Toxic	[chemical agent], [quantity]		
Harmful	[chemical agent], [quantity]		
CMR (carcinogens, mutagens and substances toxic to reproduction)	[chemical agent], [quantity]		
Corrosive	[chemical agent], [quantity]		
Irritant	[chemical agent], [quantity]		
Flammable	[chemical agent], [quantity]		
Oxidizing	[chemical agent], [quantity]		
Explosiveness	[chemical agent], [quantity]		
Asphyxiant	[chemical agent], [quantity]		
Dangerous for the environment	[chemical agent], [quantity]		
Mechanical			
Physical impact or mechanical energy (moving parts)	[location]		
Mechanical properties (Sharp, rough, slippery)	[location]		
Vibration	[location]		
Vehicles and Means of Transport	[location]		
Noise			
Frequency	[frequency],[Hz]		
Intensity			
Physical			
Confined spaces	[location]		
High workplaces	[location]		
Access to high workplaces	[location]		
Obstructions in passageways	[location]		
Manual handling	[location]		
Poor ergonomics	[location]		

0.1 Hazard identification

3.2 Average electrical power requirements (excluding fixed ISOLDE-installation mentioned above): *(make a rough estimate of the total power consumption of the additional equipment used in the experiment)*