EUROPEAN ORGANIZATION FOR NUCLEAR RESEARCH

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

Emission Mössbauer Spectroscopy of high energy transition isotopes

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Abstract

Emission Mössbauer spectroscopy of isotopes with transition energies above 50 keV require both source and absorber to be held at cryogenic temperatures. In 2015 we will set up facilities for these type of measurements at ISOLDE. We need several beams in order to test the setup, document calibration, absorbers, detectors performance etc. When this is accomplished, we propose using these beams in studies of biological systems and solid state physics.

Requested shifts: 4 shifts, (split into ~4 runs over ~2 years)

1 INTRODUCTION

The Mössbauer effect makes use of resonant absorption of gamma radiation from low energy nuclear states (E_0 < 150 keV). In some cases (e.g. ^{57}Fe and ^{119}Sn) the transition energy is low enough (14.4 keV and 23.9 keV, respectively) to allow for measurements at temperatures well above room temperature, while for most documented Mössbauer transitions, the transition energy is too high and both absorber and emitter have to be kept at cryogenic temperatures.

In emission Mössbauer Spectroscopy (eMS) it is possible to study dilute impurities (down to 10^{-4} at. %), which has obvious applications in studies of doping of materials (semiconductors in particular) or in studies intrinsic properties of materials. In some cases one can also make use of recoil of the daughter nucleus to study interstitial defects. When parent atoms are implanted, the low implantation fluence ensures that segregation and/or precipitation is avoided and that implantation damage cascades do not overlap.

For the last ~15 years, the Mössbauer collaboration at ISOLDE/CERN [MSColl] has used the Mössbauer states of ^{57}Fe and ^{119}Sn, but for a long time there has also been an interest to apply more isotopes for our eMS studies.

Each Mössbauer transition comes with its own challenges, such as finding a suitable single-line absorber material, suitable detector systems, suitable drive systems, suitable cryogenic facilities and in some cases development of data analysis tools.

Historically, the most used high energy Mössbauer transitions rely on neutron activation of sources and samples with the Mössbauer isotope. Emission Mössbauer Spectroscopy studies are scarce, due to the lack of facilities offering a wide range of intense clean beams. Therefore it can be argued that the only "correct" place for this facility is at ISOLDE/CERN.

With this letter of intent, we apply for beam time to start investigations using isotopes with high energy transitions for eMS studies. During the first trials, we intend to apply reasonably simple isotopes, where the chance of success is expected to be high, and where outstanding physics questions can be addressed.

2 SOME THEORETICAL BACKGROUND AND SETUP

The emission of recoil free gamma rays from a nuclear transition is governed by the Debye-Waller factor, which in the Debye approximation can be written as

$$f = \exp\left(-\frac{C \cdot E_0^2}{\theta_D M}\left(\frac{1}{4} + \left(\frac{T}{\theta_D} \right)^2 \int_0^{\theta_D/T} \frac{x}{\exp(x)-1} \right) \right),$$

where C = 37.272 Da·K/keV^2, E_0 the transition energy, θ_D the Debye temperature, M the mass of the nucleus, and T the temperature. The conditions for a useful Mössbauer experiment can be expressed in terms of E_0^2 / M and the Debye temperature, as illustrated in Fig. 1.

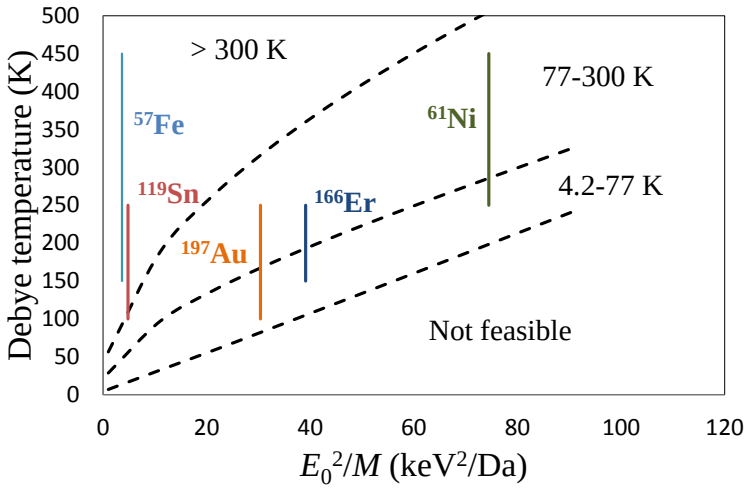

Fig. 1: Conditions for useful Mössbauer experiments. See explanation in the text.

For given values of E_0^2/M and Debye temperature one can calculate the maximum measurement temperature that gives a useful signal. This defines four regions in the plot separated by dashed lines in Fig. 1. Below the lowest dashed line, there is no possibility to perform useful Mössbauer experiments. The vertical lines in Fig. 1 show the value of E_0^2/M and typical spread of Debye temperatures for selected Mössbauer isotopes. For ^{57}Fe and ^{119}Sn, good data can be obtained above room temperature, while for the other isotopes shown in Fig. 1, measurements down to 4.2 K are required.

An illustration of the setup for the cold source/absorber eMS measurement is shown in Fig. 2.

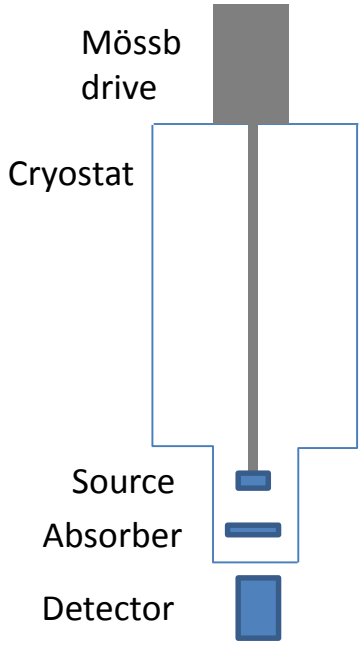

Fig. 2: Illustration of the setup for cold source/absorber Mössbauer measurements

The drive system is placed on top of the cryostat. The radiation from the sample passes through the absorber material and is measured with the detector mounted below the cryostat.

Suitable absorbers should be single-line, i.e. not show quadrupole and magnetic interactions (i.e. cubic non-magnetic materials). The thickness of an absorber is usually denoted as the unit-less quantity

$$t = n f_a \sigma_0 a ,$$

where n is the number of nuclei of the specific element per unit area, f_a the recoil free fraction, σ_0 the maximum Mössbauer cross section, and a the natural abundance of the Mössbauer isotope. Suitable absorbers have the value, t = 1.

3 BEAMS REQUESTED

From informal discussions between the proposers of this letter of intent, three Mössbauer transitions were identified as a good starting point and four radioactive ion beams (RIBs) that decay to the relevant Mössbauer states (cf. Table 1).

Table 1: Mössbauer transitions and beams proposed for this LOI. For more information see e.g. [MSData, Gütlich11].

Mössbauer Isotope	Energy (keV)	Beams	Lifetime	Decay to Mössbauer state (%)
^{61}Ni	67.4	^{61}Mn → ^{61}Fe → ^{61}Co	0.67 s → 6 m → 1.65 h	84.7
		^{61}Cu	3.33 h	4.2
^{166}Er	80.6	^{166}Ho	26.9 h	6.56
^{197}Au	77.35	^{197}Hg	65 h	18.7

The above Mössbauer transitions and RIBs were selected as being relatively "easy", where absorber material is commercially available, good beams at ISOLDE have been demonstrated, and specialized Mössbauer drive systems are not required. Of the requested four beams, at least one is expected to be available during 2015, and most/all of them (not necessarily clean) in the course of two years. This should be adequate to document the performance of the facility, solve potential problems and do some initial science.

In all cases, one needs to verify (and document)

- Mössbauer signal vs. beam intensity × implantation time
- Ensure that the absorber materials meet expectations
- Document potential drawbacks due to decay after-effects (i.e. metastable electronic states formed in the nuclear reaction), either by temperature dependent measurements or measurements in different types of systems
- Document changes in coordination geometries (biological systems) where applicable.

Although, this LOI is at this stage aimed at the technical development, the underlying motivation has a sound scientific basis, and we suggest that some of the testing takes place on systems of scientific interest. Some of the planned experiments are described in the next section, some of which describe only the systems that can be studied with these beams.

4 SCIENTIFIC MOTIVATIONS

4.1 Biological applications

In a project which has been running for several years, we (L. Hemmingsen in collaboration with V. L. Pecoraro, University of Michigan) aim to elucidate the origin of Hg^{2+} toxicity at the molecular level, most notably by exploring which coordination geometries of this metal ion may be observed in naturally occurring and synthetic proteins [Iranzo07, Luczkowski13]. As Hg^{2+} is assumed to bind to thiolate-rich binding sites in proteins, similar binding sites have been constructed for example in the *de novo* designed protein TRIL9C. In this context we have already carried out a variety of spectroscopic investigations using ^{199m}Hg perturbed angular correlation (PAC) spectroscopy and ^{199}Hg NMR spectroscopy [Iranzo07]. We are therefore able to design experiments where we know and can change the coordination of Hg^{2+} in a controlled manner, for example by changing pH, at protein binding sites. Specifically, we can change from a two-coordinated (HgS_2) to a three-coordinated (HgS_3^-) coordination geometry by changing pH in a solution containing Hg-TRIL9C from 6.5 to 8.7. This will serve as a test case in the current proposal, in order to establish if the two biologically important coordination geometries

(HgS_2 and HgS_3^-) can be discriminated by $^{197}Hg \rightarrow {}^{197}Au$ Mössbauer spectroscopy, and if so, to record the spectroscopic signatures of these species. This would establish $^{197}Hg \rightarrow {}^{197}Au$ Mössbauer spectroscopy as a useful technique in bioinorganic chemistry.

Cu^+ is difficult to observe using standard spectroscopic techniques due to its closed shell electronic structure. It is also one of the most important metal ions in biological systems, involved in both electron transport in photosynthesis and in redox chemistry. Thus, there is considerable interest in novel techniques able to elucidate the biochemistry of this metal ion. As a test system we will use simple systems like the crystalline Cu_2O, where the nuclear quadrupole interaction is known from nuclear quadrupole resonance experiments [Graham91] and eMS experiments [Bordovskii12]). For biomolecular proof-of-principle experiments, we aim to use the well characterized small blue copper proteins plastocyanin and azurin involved in electron transport, investigating and comparing the metal site structure in both the reduced (Cu^+) and oxidized (Cu^{2+}) state.

4.2 Solid state physics

The Mössbauer collaboration at ISOLDE/CERN has been involved for many years with the experiments IS443 & IS501 in the search of dilute room-temperature magnetism in semiconductors using Mn/Fe/Co implantations (see e.g. publications listed at [MSColl]) with success, but negative results. One possibility is that Fe is not a suitable impurity for long-range carrier mediated dilute magnetism. With ^{61}Ni eMS we could continue these studies where dilute Ni^{2+} in e.g. SnO_2 [archer05] has been suggested to give rise to dilute magnetism. As with ^{57}Fe eMS, such claims can be easily tested on the atomic scale with ^{61}Ni eMS, using both implantation of ^{61}Co and/or ^{61}Cu.

Many alloys, among them Heusler alloys (A_2BC, A= Cu, Ni, Co, Pd, Fe, Mn, B = Mn, Fe, V, C = Al, In, Sn, Sb, Ga, Si, Ge) are of current interest due to potential magnetocaloric effects, and there is an active experiment at ISOLDE (IS578) where the study of their magnetic properties is ongoing using implantation of ^{57}Mn (see references listed in the IS578 proposal). ^{61}Ni eMS would greatly add to the possibilities of studying this system with the possibilities of site selective doping and annealing of samples which is not possible with ^{57}Mn. Some data on Ni in Heusler alloys already exists [Kobayashi90] which will help with the interpretation of the new data.

Doping with rare earth metal ions, e.g. Er^{3+}, in various hosts to beneficial manipulate the optical properties of materials (such as upconversion) has been an increasing field within the last decade with applications like photovoltaics [Huang13, Ende09], sensors [Jacinto07], display technologies [Auzel04], luminescent labels for bio-imaging [Mader10, Wang06, Zhou12], fiber-optic communication [Xiaodan11] and others. ^{166}Er eMS can give direct information on the atomic configuration of the Er atom within its host material. In this proposal, several hosts can be explored, but initially we will investigate the two interesting host materials ZnO and TiO_2 which are both promising upconversion hosts. Detailed studies by ^{57}Fe eMS (IS443 & IS501) showed unusually temperature dependant slow spin-relaxations rates of Fe^{3+}, which is still to be investigated and understood. One hypothesis of this behaviour can be a link of upconverting properties of the host material related to phonon relaxation processes (radiative and/or non-radiative relaxation).

Multiferroic materials, i.e. showing at least 2 ferroic-type of ordering, are currently the subject of high interest for their potential application in spintronic devices. Among them, Er-Fe-O compounds have recently attracted attention as a new class of multiferroics (see [Mantovan14] and references therein). In order to plan an integration of those layers into practical devices, their synthesis in the thin film form is necessary, and the use of techniques such atomic layer-/chemical vapor deposition (ALD, CVD) is of interest being methods of choice in industrial contexts [Mantovan14]. To employ transmission MS is not possible due to limited thickness, and ^{57}Fe conversion electron Mossbauer spectroscopy (CEMS) can only be conducted by making use of the 2.2% natural abundance of ^{57}Fe [Mantovan14] since ^{57}Fe-enriched chemical precursors are not available. ^{166}Er eMS is therefore an attractive option to have access to

atomic-scale structure and magnetism of the synthesized Er-Fe-O thin films, also in order to optimize recipes for their synthesis in the thin film form through post-growth thermal annealing. It should be reasonably easy to make tests using this system.

5 SUMMARY OF REQUESTED SHIFTS:

Beam	Min. intensity (ions/s)	Target	Ion source	Shifts
^{61}Cu	2×10^{8}	Y_2O_3[a]	RILIS[b]	1
^{61}Mn	2×10^{7}	UCx	RILIS	1
^{166}Ho	1×10^{7}	Ta?	RILIS	1
^{197}Hg-g	3×10^{8}	Pb	RILIS[b] or VADIS	1

[a]Or other suitable target such as ZrO
[b]If available

The beam intensity is calculated from what is needed to obtain 2-4 samples where ~5 measurements of each sample could be made.

6 FEASIBILITY

The feasibility of this investigation should be described in some details. Some square meters of laboratory space are needed, and this should not be a major problem in the new building. It is assumed that ISOLDE/CERN delivers liquid Helium for these investigations (recuperations lines may be needed). The major parts of the investigation include 1) Cryostat, 2) Detector, 3) Electronics, 4) Pumps, 5) Radioactive samples, and 6) Suitable absorbers.

1. Cryostat will be supplied by IKS Leuven

2. Detector will be supplied by the former Mössbauer lab in Aarhus

3. Electronics will be supplied by the former Mössbauer lab in Aarhus, the on-line eMS setup and IKS Leuven

4. Pumps will be supplied by former Mössbauer lab in Aarhus and the on-line eMS setup

5. The table below lists suitable absorber materials and calibration (preliminary). All the absorber materials are commersially available.

Mössbauer isotope	Absorber	Non-resonant absorption (%)	Calibration
^{61}Ni	80 μm $Ni_{80}Cr_{20}$ foil	~6	$Ni_{80}Cr_{20}$ vs $Ni_{80}Cr_{20}$ and ^{57}Fe
^{166}Er	ErH_2 powder (in epoxy) ~2 mg/cm^2	1.3	ErH_2 vs Er metal or Er oxide
^{197}Au	25 μm Au foil	11	Au vs. Au and ^{57}Fe

6. From the ISOLDE Yield database, assuming 2 μA proton current, the expected yields in comparison with required beam intensity are:

Beam	Required	Yield database
^{61}Cu	2×10^{8}	8×10^{8}
^{61}Mn	2×10^{7}	3.4×10^{6}
^{166}Ho	1×10^{7}	N/A
^{197}Hg	3×10^{8}	2×10^{11}

The yields of ^{61}Mn may be too low. However, this could be sufficient for test experiments to document its usefulness.

7 CONCLUSIONS AND OUTLOOK

This proposal aims to establish and apply new isotopes in emission Mössbauer Spectroscopy (eMS). If successful it will allow for a broad spectrum of novel applications ranging from solid state physics to biochemistry, and thus allow for unprecedented characterization at the atomic level of electronic and molecular structure of such systems.

Upon completion of this project and when final documentation is in place, external users will be able to suggest the use of these new isotopes/beams for eMS. We envision further development of eMS of other isotope transitions that need cold source/absorber transitions.

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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
SSP-GLM chamber	☒ Existing	☒ To be used without any modification
Offline eMS cold source/sample setup	☐ 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
Annealing facilities	☒ 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.

HAZARD DUE TO SOURCE ACTIVITIES

Source	Activity (MBq)	Dose rate at 10 cm distance (only gamma considered) (mSv/h)
^{61}Co	10	0.012
^{61}Cu	95	1.1
^{166}Ho	1.2	$6 \cdot 10^{-4}$
^{197}Hg	1.4	$2.4 \cdot 10^{-6}$

The calculated activity of ^{61}Cu sources, demonstrate that extreme care has to be applied here, where all transport, sample preparation and mounting in cryostat will have to be done with appropriate shielding. Alternatively, one can us lesser implantation and repeated measurements.

Additional hazards:

Hazards			
	SSP-GLM chamber	*Offline eMS cold source/sample setup]*	*Annealing facilities*
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]	Liquid Helium	
Electrical and electromagnetic			
Electricity	[voltage] [V], [current][A]	Usual electronics, pumps etc.	
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	See information in table above.		
• 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)*