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EUROPEAN ORGANIZATION FOR NUCLEAR RESEARCH

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

Upgrade of the UHV-system ASPIC for the investigation of surfaces and two-dimensional materials by ultra-low energy implantation and deposition of radioactive probe atoms

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Abstract

[The proposal is part of the Consortial Project "Ecomarl" comprising the groups of Hans Hofsäss from University Göttingen, Peter Schaaf from TU Ilmenau and Doru Lupascu from University Duisburg-Essen. It has proven that these three teams form a very powerful team to foster German and international solid state activity at ISOLDE. The interrelation between these three partners has enormously improved the working environment at ISOLDE. We feel that this core group of PIs should be able to continue to work together even though the Projektträger (BMBF) has asked us to submit separate proposals. We are one unit of closely related projects, including a coordination project, and in particular these people have been extremely good for the ongoing work of infrastructure improvement at ISOLDE.

Requested shifts: nothing within the next 2 years

1 AIM

1.1 Overall aim of the project

With the proposed project, we intend to refurbish and upgrade the ASPIC UHV system at ISOLDE in order to provide a unique spectrometer system for the investigation two-dimensional materials (such as graphene and transition metal di-chalcogenides), surfaces and interfaces using a variety of different radioactive probe atoms suitable for perturbed γ–γ angular correlation (PAC) and Mössbauer spectrometry (MS). In contrast to the previously established evaporation method of decorating surfaces with radionuclides, we intend to build an ion optical system for ultra-low energy ion implantation of isotopes from the ISOLDE beam line. This technique was developed in our group at the University Göttingen and is successfully applied to doping of graphene or MoS_2 monolayer sheets with a variety of dopant ions such as B, N, F, Ne, Se, Mn, Fe, W, Au and others with energies adjustable between 10 eV and 30 eV [24,25,26,27,29,30]. For comparison, in the semiconductor industry shallow implantation is done at energies of 100eV and higher. ASPIC will be equipped with such a novel implantation facility and upgraded with a second measurement position suitable for Mössbauer spectrometry.

The investigation of surfaces using radioactive probe atoms was first realized by the group of Prof. Günther Schatz, University Konstanz in 1990. Several seminal publications on the structure and dynamics of diluted impurities on metal surfaces as well as investigations of magnetic properties of surfaces demonstrated the applicability of nuclear solid state physics methods such as perturbed γ–γ angular correlation (PAC) to the investigation of two-dimensional systems [2,3,4,5,6,7,8]. In these studies, radioactive probe atoms (^{111}In) were chemically prepared from solutions and carefully evaporated on the surfaces in a two-step transfer process under UHV conditions.

In 1994, a procedure for depositing these radioactive atoms on surfaces has been successfully tested at ISOLDE-2 for Cd, In and Rb [9]. A set of differential pumping stations was used to feed the radioactive beam from ISOLDE into an ultrahigh vacuum chamber. For theses pilot experiments a somewhat improvised UHV setup was employed. The basic idea was to implant the radioactive atoms into a pre-cleaned oven (catcher) from where they can be evaporated in two steps onto a properly prepared sample. For the PAC measurements the sample was moved to a site with γ-detectors positioned outside the vacuum. In these studies the quadrupole coupling of In and Cd probe atoms on various metal surfaces were determined. In addition, the feasibility of Mössbauer experiments at surfaces was investigated.

Eventually, at the new ISOLDE facility at the CERN PS-Booster a beamline in UHV standard and a special surface physics chamber has been constructed and successfully tested. This Apparatus for Surface Physics at ISOLDE CERN (**"ASPIC"**), specially designed for operation at ISOLDE, is a versatile system for surface and interface measurements. Surface studies were now possible with a variety of radionuclides as probe atoms, such as ^{111}Cd , ^{77}Se and the short-lived ^{79}Rb. In the following years ASPIC was mainly used to investigate magnetism at surfaces and thin film interfaces [10,11,12,13]. Due to the transformation of the Hahn-Meitner-Institute to the Helmholtz Center Berlin, ASPIC had little or now manpower and financial support. Later on in 2014 ASPIC was proposed as UHV system at the beam-line VITO (versatile ion-polarized techniques online) [13,14], but only the construction of a UHV beamline was achieved. Since then ASPIC is stored at ISOLDE and is waiting for new challenges.

In a coordinated effort together with the cooperation partners in Duisburg-Essen (Prof. Lupasco) and TU Ilmenau (Prof. Schaaf), the ISOLDE physics coordinator and other solid state physics groups at ISOLDE (Prof. L. Pereira, KU Leuven) we found an agreement that the group in Göttingen will carry out an upgrade for ASPIC, in particular to realize ultra-low energy ion implantation in the energy regime 10-30 eV. Regarding magnetic raw materials, in particular hard magnetic materials we will collaborate with the group of Prof. Schaaf, TU Ilmenau. Regarding 2D materials an existing cooperation will be extended to the investigation of radioactive probes in graphene.

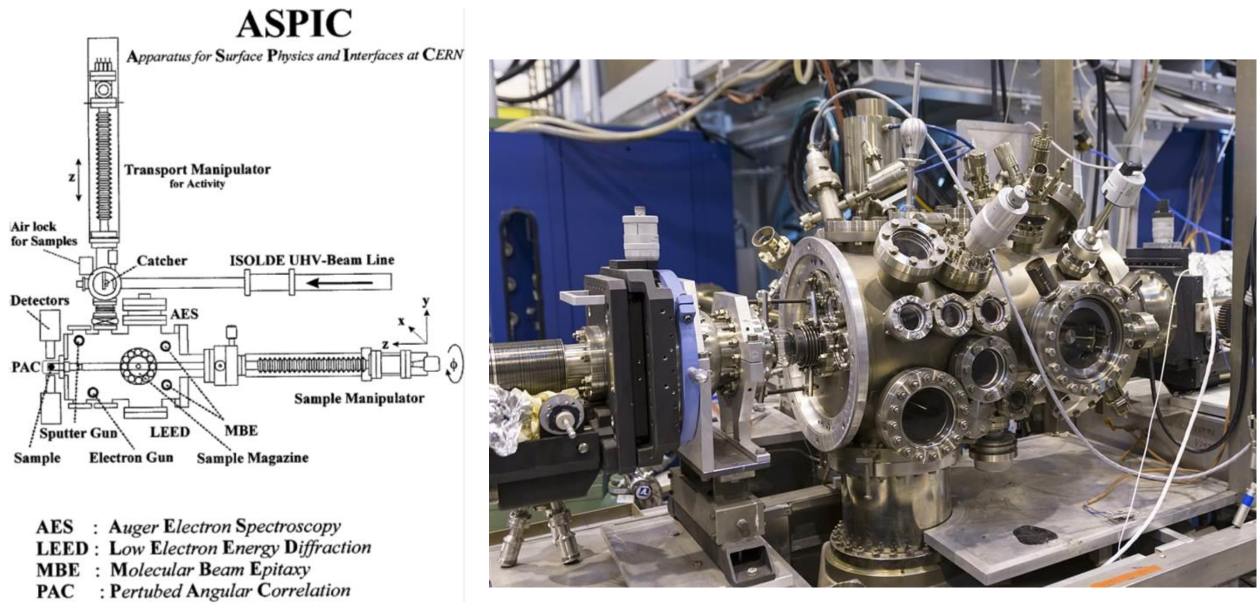

Fig.1 Left: Top view of ASPIC as it was used until 2007 (from ref. 11). The chamber named "catcher will be replaced by the ion deposition stage. Right: ASPIC UHV system as it was installed at the beamline for the VITO project. Sample transfer linear stages are connected to both sides. The sample position for PAC measurements is the glas tube near the left linear stage.

1.2 Referencing the project to the funding political aims

With the unique instrumentation proposed in this project we significantly broaden the possibilities to use ISOLDE for solid state physics and materials science research, in particular with regard to the investigation of low dimensional systems, two-dimensional materials and surfaces. The previously existing features of the ASPIC surface science UHV system will be extended and allows versatile investigations of state-of-the-art low dimensional materials systems. The ultra-low energy implantation feature will broaden the spectrum of materials systems which can be investigated with the help of radioactive prove atoms. The instrumentation complements the existing spectroscopic methods available at ISOLDE and opens again the door to surface science studies using radioactive probe atoms. The project is therefore in accord with the aims in the call for proposals regarding instrumentation for ion beams and nuclear probes.

1.3 Scientific and/or technical goals of the project

1.3.1 Scientific goals

Our group is successfully using ultra-low energy ion implantation since several years to dope 2D materials like graphene or MoS_2 with various dopant atoms (see section II.2 and Fig.2 and 3). With this project we want to enable the investigation of two-dimensional (2D) materials, surfaces and interfaces using radioactive probe atoms and hyperfine interaction methods like γ–γ angular correlation (PAC) and Mössbauer spectroscopy (MS). In particular local structural and magnetic properties of graphene and transition metal di-chalcogenides like MoS_2 shall be studied using probe atoms as already proposed in the review of Potzger et al [13], for example atoms with partially filled 3d, 4d and 4f shells, such as ^{61}Ni (3d8), ^{99}Ru (4d7), ^{181}Ta (5d3), ^{140}Ce (4f1), and ^{143}Pr (4f5) in addition to the standard PAC probes ^{111}Cd, ^{111}In. Adatoms and dopant atoms in graphene are known to modify the electronic properties, enable adatom mediated etching [15], or may exhibit possible ferromagnetic behavior [16]. These probe atoms are especially promising to investigate magneto-crystalline anisotropies magneto-transport in doped 2D-materials in graphene and also for possible spintronics applications. The probe atoms ^{56}Mn and ^{119}In, commonly used at ISOLDE would also be of interest for Mössbauer studies of local electronic and magnetic properties of these material systems. A support letter from Prof. Pereira, TU Leuven, Belgium, regarding his interest for using ASPIC to investigate graphene is attached. The groups of Prof. van Haesendonck and Prof. Bael express interest to investigate topological insulators such as $(Bi,Sb)_2Te_3$ and (Pb,Sn)Te; ferromagnetic Fe and Ni, and Sn in superconducting Nb_3Sn. The group of Prof. Vantomme expresses interest in using ASPIC to investigate Pb in Pb-based ferroelectrics, and transition metals and rare earths in ferromagnets.

Up to date hyperfine techniques were mainly applied to surface studies of metallic surfaces and magnetic interfaces [13]. The reloaded ASPIC system is expected to contribute to novel surface physics experiments carried out on semiconductor surfaces, topological insulators, superconductors and magnetic raw materials (in cooperation with the group of Prof. Schaaf, TU Ilmenau)

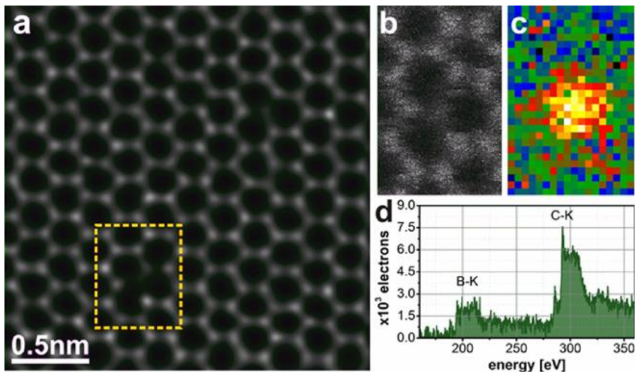

Fig.2: a) High resolution HAADF TEM image of graphene doped with ^{11}B by 25 eV ion implantation. The dark atom marks the substitutional B atom due to its lower Z contrast. b) raw data of the marked region in a). c) Intensity distribution of the B-K electron energy loss region, identifying B as dopant atoms. d) Electron energy loss spectrum at the position of the B atom. (from ref. 29).

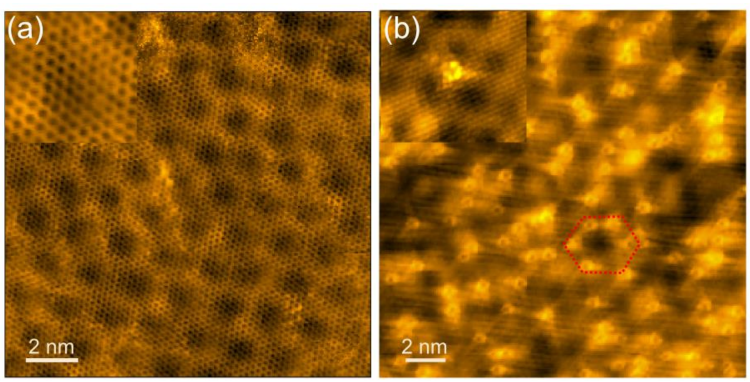

Fig.3: Scanning tunneling microscopy (STM) images of single layer graphene on SiC. a: STM of a pristine undoped sample showing the 6x6 corrugation and the graphene lattice magnified in the inset. b: STM image of a sample doped with ^{14}N atoms by ion implantation at 25 eV. Most N atom is incorporated substitutionally, and modify the local electronic structure due to scattering in a typical pattern with 3-fold symmetry. (from ref. 28)

1.3.2 Technical goals

The ASPIC UHV system for investigation of surfaces using hyperfine interaction methods is a worldwide unique systems which was developed in 1994 and used until about 2007. An attempt to reactivate ASPIC in connection to the planned VITO beamline in 2014 was not successful due to lack of funding. Based on the experience and expertise of the Göttingen group with ultra-low energy ion implantation of 2D materials it is planned to refurbish the UHV system and connect it to a new ion deceleration stage, which allows direct deposition of probe atoms on the surface or implant into 2D materials (see Fig.4). Compared to the deceleration system designed for the recently installed ISOLDE ion implantation chamber for implantations down to about 100eV, the deceleration for the ASPIC system will allow implantation or deposition at energies down to 10 eV. Technical goal is to design an optimum ion deceleration system, which is synchronized to the ISOLDE ion source potential with a sufficiently low energy spread. Typically ISOLDE uses a 30-40 keV acceleration voltage. The implantation stage will therefore designed to decelerate a 40 keV ion beam. Prior to the implantation stage a dedicated beamline segment with differential pumping, neutral rejector, beam sweep and lenses are necessary. The design of the different components is very similar to the system used in Göttingen and is schematically shown in Fig.5. The control of the deceleration unit requires some components to be operated at high potential. This is achieved through insulation transformers, a faraday cage and optical signal transmission. In addition, the existing sample transfer system has to be modified to replace the two stage evaporation process by direct ion deposition. In addition, the UHV chamber, although in a very good shape, has to be refurbished and the process control has to be upgraded. A new ion getter pump has to be installed.

Typical radioactive fluences will not exeed $10^{11}/cm^2$. Therefore, an activity measurement and only for adjustment puropses a beam current and charge measurement will be used. A sensitive current meter, developed in a previous BMBF project will be used as picoamperemeter and current integrator.

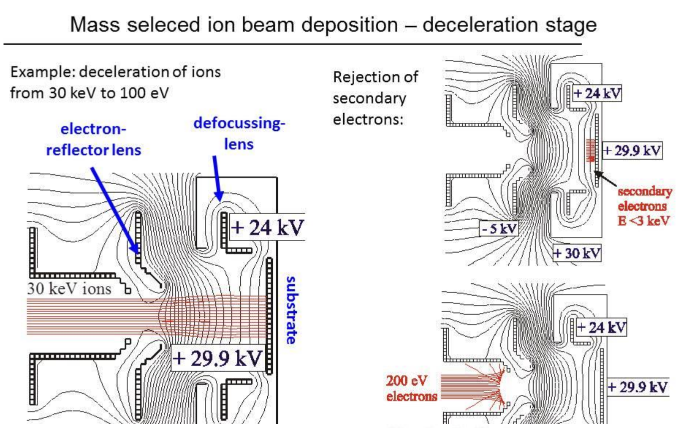

Fig.4: Schematics of the ion optics of the decelerations stage at the ultra-low energy ion beam system in Göttingen. The system is capable of deceration ions down to 10 eV

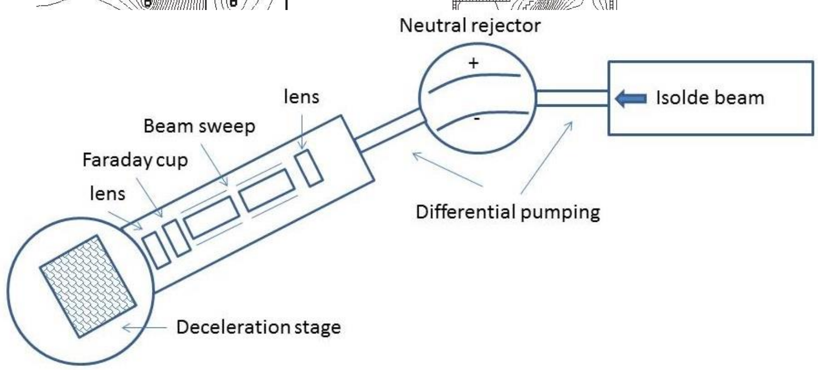

Fig.5 Schematic of the differentially pumped beam line segment conneting the ISOLDE beam line and the deceleration stage

2 STATUS OF SCIENCE AND TECHNOLOGY, PREVIOUS WORK

2.1 Status of science and technology

The scientific focus of our project will be the investigation of two-dimensional materials, surfaces and interfaces using radioactive probe atoms and spectroscopic method of nuclear solid state physics. In the recent review of Potzger et al. on surface science using radioactive ions at ISOLDE [13], chapter 3 focuses on future physics at ASPIC, in particular concerning adatoms on 2D materials. Potzger et al give a brief summary of seminal work on graphene and other 2D materials and highlights the case of magnetic adatoms on graphene as a showcase of the unrivalled capabilities of ASPIC. Potzger et al. conclude that, "while promising theoretical work in this context has been rapidly accumulating, experimental progress has been hindered by limitations of currently available techniques. Indeed, adatom magnetism is not only a good example of the rich new physics underlying two-dimensional materials, but it is also a showcase of the tremendous challenges that experimentalists must face in the flatland".

Scanning transmission microscopy and spectroscopy requires knowledge about the elemental nature of the impurity, Magnetotransport measurements provide information averaged over a large area, high resolution TEM allows identification of impurity species and lattice sites, electron energy loss spectroscopy provides details of the electron transfer between impurity and 2D lattice, and angle resolved photo electron spectroscopy (ARPES) can image the effect of doping on the band structure of 2D materials. Microscopic Raman and PL provide averaged information on the optical and vibrational features of dopants in 2D materials. Despite this variety of powerful techniques, which are applied to characterize 2D materials, radioactive probes are very unique sensors for electronic, structural and magnetic properties on the atomic scale and have unrivalled capabilities in particular regarding the magnetic properties.

In their review, however, Potzger et al. were possibly not aware that our group in Göttingen has realized ultra-low energy doping of 2D materials since 2013 and carried out many experimental studies on graphene and other 2D systems in cooperation with a number of different groups throughout Europe.

2.2 Previous work of the applicant

The group in Göttingen has built up and used a unique low energy mass selected ion beam deposition since 1992 to investigate the growth of diamond-like materials like tetrahedral amorphous carbon, cubic boron nitride and related carbide and nitride materials [21,22] and self-organized multilayer growth by hyperthermal ion deposition [23]. In 2013 the group investigated ion beam doping of graphene for the first time [30] and achieved a breakthrough together with the group of Bangert and Ramasse [29], demonstrating the substitutional doping of graphene with B and N ions through high resolution TEM spectroscopy of free standing graphene monolayers. We then achieved successful doping of graphene grown on SiC with B and N using scanning tunneling spectroscopy [27,281] and a detailed analysis of the electronic structure around dopant atoms [24]. A careful analysis and simulation of electron energy loss spectra demonstrated the distorted off-plane substitutional incorporation of P in graphene [26]. Recently we achieved implantation of Se into MoS_2 monolayers for possible single photon sources [25]. Unpublished work is related to intercalation of graphene using Ne ions and implantation of Fe and Mn, which has been achieved with the setup in Göttingen.

3 DETAILED DESCRIPTION OF THE WORK PLAN

We will start with the transportation of the existing ASPIC UHV system from CERN to Göttingen where it is thoroughly inspected and refurbished. We will test the surface analysis techniques LEED and Auger, the functionality of the vacuum system and the sample handling system. At the same time we will carry out ion optics simulations of the beamline and the new deceleration unit including all ion optical components necessary for beam transport and purification (rejections of neutrals and secondary electrons). At Isolde we will evaluate the beam properties like focus diameters, max. beam width etc. in order to optimize the ion deceleration system and the differential pumping system. We will also analyze the stability of the acceleration voltage and evaluate possibilities to synchronize the deceleration voltage to the ISOLDE ion source potential. We will also evaluate the energy spread of the different ISOLDE ion sources. An energy spread of up to 10-15 V may occur at the Göttingen system, but it only causes a tail in the energy distribution towards lower ion energy. These ions are deflected or may end eventually as adatoms. For ASPIC this is considered as an acceptable situation.

Depending on the final beam line position for ASPIC we will construct the differentially pumped beam line segment and the ion deceleration chamber operating under UHV conditions. This beam line segment must contain a rejector for possible neutralized ions and also lens systems to suppress secondary electrons being accelerated to the substrate. It also requires a beam sweep system to achieve a homogeneous deceleration onto an area of 1-2 cm^2. A technically complex task is the modification of the existing sample transfer system to make it compatible for direct ion deposition. It is expected that we may have to design and build new sample holders and adapters. A final technical task is the upgrade for a measurement position suitable for Mössbauer spectroscopy.

For a successful accomplishment of the work plan we require an experienced post doc and a technician to build a variety of dedicated mechanical components

3.1 Project related resource planning

Year	2019		2020				2021				2022	
Quarter	3	4	1	2	3	4	1	2	3	4	1	2
Simulation oft the beamline and ion optics	■	■										
Construction of the beam line segment		■	■	■								
Refurbishment of the ASPIC UHV system	■	■	■	■	■							
Construction of deceleration ion optics			■	■	■	■						
Modification of the sample transfer system			■	■	■	■	■	■				
Ion optics tests at the Göttingen 30 keV beam line						■	■	■				
Development of process control and automatization						■	■	■	■			
Upgrade of ASPIC for Mössbauer spectroscopy								■	■	■		
Commissioning, test and first experiments at ISOLDE									■	■	■	■

4 DIVISION OF LABOR/COOPERATION WITH THIRD PARTIES

Within this project we will establish a close collaboration with the project partners form University Duisburg-Essen and TU Ilmenau, but also to other groups working on 2D-materials (Beata Kardynal (Jülich), Ursel Bangert (Limerick, Ireland), L.Preira (KU Leuven, Belgium), M. Wenderoth (University Göttingen). We will also collaborate with people previously involved to upgrade the ASPIC system (K.Potzger, HZDR) and users of the solid state physics infrastructure available at ISOLDE.

5 NECESSITY OF FUNDING BY BMBF

The proposed project will lead to a significant improvement of the experimental instrumentation at ISOLDE/CERN. It offers users of ISOLDE radioactive beams novel and unique possibilities to investigate structural dynamic and magnetic properties of isolated impurities and dopants in and on 2D materials and surfaces.

A successful upgrade of ASPIC requires a new construction of the differentially pumped beam line segment, including beam optics and beam manipulation components, construction of the deceleration stage, modification of the sample transfer systems and sample holders, and construction of a second sample position for Mössbauer spectroscopy.

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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
	☐ Existing	☐ To be used without any modification
ASPIC UHV Chamber	☐ 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
	☐ 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	*ASPIC*		
Thermodynamic and fluidic			
Pressure	[pressure][Bar], [volume][l]		
Vacuum	Ultrahigh vacuum		
Temperature	[600] [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	Various solids		
Beam particle type (e, p, ions, etc)	ions		

Beam intensity	<1E8/s		
Beam energy	10eV – 60 keV		
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)*

About 3-6 kW ; 16 A 380 V supply is sufficient