#### PROPOSAL TO THE ISOLDE AND NEUTRON TIME-OF-FLIGHT EXPERIMENTS COMMITTEE (INTC)

#### CRYSTAL FIELD INVESTIGATIONS OF RARE EARTH DOPED WIDE BAND GAP SEMICONDUCTORS

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#### SUMMARY

Crystal field investigations play a central role in the studies of rare earth doped semiconductors. Optical stark level spectroscopy and lattice location studies of radioactive rare earth isotopes implanted at ISOLDE have provided important insight into these systems during the last years. It has been shown that despite a major site preference of the probe atoms in the lattice, several defect configurations do exist. These sites are visible in the optical spectra but their origin and nature aren't deducible from these spectra alone. Hyperfine measurements on the other hand should reveal these defect configurations and vield the parameters necessary for a description of the optical properties at the atomic scale. In order to study the crystal field with this alternative approach, we propose a new concept for perturbed  $\gamma\gamma$ -angular correlation (PAC) experiments at ISOLDE based on *digital signal* processing in contrast to earlier analog setups. The general functionality of the spectrometer is explained and its advantages are pointed to selected experiments with several rare earth isotopes implanted in wide band gap semiconductors, motivated by open questions important for a fundamental comprehension of the optical and magnetic properties of such systems. For a comprehensive picture of the physical situation the PAC measurements will be combined with photo-luminescence (PL) and emission channeling (EC) lattice location studies.

# 1) Introduction

In the last years there has been growing scientific and technological interest in rare earth (RE) doped semiconductors, not only justified due to already demonstrated solid state lasers [SPZ07] and light diodes [SHLG01], but also because of the potential for future systems and applications in the fields of quantum computers [OKMK02], spin tronics [AZH+04] and dilute magnetic semiconductors based on Gd in GaN [DBR+05], and GaN based light emitters on silicon substrates for fast on-chip communication [SPZ07]. This diversity in possible applications and devices can be regarded as one of the main motivations behind actual research.

Preceding decades of intensive studies of the optical properties of triply ionized RE in ionic crystals of oxides and fluorides, mostly absorption spectroscopy measurements [Die68], led to a comprehensive knowledge of the 4f energy levels and the observed fluorescence [GWB98]. The energetic positions of the 4f electron levels, electrons shielded by the outer 5s-and 5d-electrons and therefore only slightly affected by the outer crystal field, led to the knowledge of the 4f mean free ion energy levels as shown in Fig. 1. The characteristic luminescence arises from excitation of the 4f electron system followed by radiative intra-4f transitions. [GWB98]. Although the crystal field has only little influence on the energetic positions of the free ion energy levels, which is in contrast to transition metals, it is

responsible for the parity forbidden intra-4*f* electron transition through mixing of states of opposite parity. Without the crystal field only magnetic dipole transitions could occur, which are an order of magnitude less intense that the observed induced electric dipole transitions. The ions' ligands, which are the origin of the main part of the crystal field, are furthermore involved in excitation and deexcitation processes. Studying the crystal field does therefore directly get to the root of the problem.

Long ago people realized that embedding REs in semiconductor host matrices allows for an electronic excitation of the 4*f* electron system. [WUNT88], which opened a route for building specific solid state light emitting devices. First investigations were performed on REs in II-VIsemiconductors [ZKS81], followed by silicon [ESPA83]. It was only years later that the direct semiconductor GaN could be grown in sufficient purity and the luminescence of the REs reached the desired intensity at room temperature [LJB99b] [LJB99a] [LJB00] [LJB01] [JLB+01] [JL03]. Shortly after the wide band gap semiconductor *w*-AlN was available at sufficient purity, demonstrating once again the huge potential of RE doped wide band gap semiconductors [JLB+01] [VZH03] [LJBM05] [VGN+06]. Doping REs into semiconductors is either realized through ion implantation [LJB99b] [LJB00] [LJB01] [JL03] [VZH03] [VHT04] [LJBM05] [VGN+06] or during growth [VRR+03] [RMSW08] or synthesis [NTK+05]. Ion implantation has proven to be the most viable doping method since an incorporation of additional impurities (such as oxygen) ist avoided.

If the band gap of the semiconductor host matrix is sufficiently large, as is the case for e.g. GaN, AIN, ZnO, detailed crystal field analyses are feasible due to the large number of observed intra-4f electron transitions. These transitions are compared with calculated crystal field splittings. The measurements are typically performed at low temperatures where only the ground state of the excited levels is populated. Modeling the crystal field is based on the knowledge of the symmetry of the ions sites which cannot be gained from the luminescence measurements directly. In this respect emission channeling (EC) lattice locations studies [HL91] with radioactive probe atoms play a central role. [Vet03] [WAL+03] [WCR+04]. EC spectroscopy, on the other hand, does not reveal the symmetry of the site directly since it measures the position of the probe atoms in the host matrix unit cell. It is therefore an

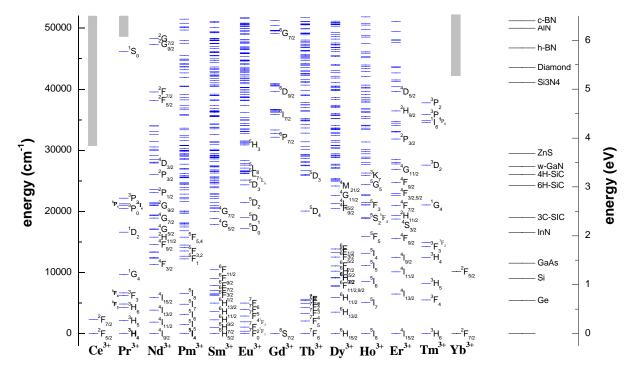


Fig.1: Calculated 4*f* energy level diagram of triply ionized rare earths, limited to energies of about 52.000 cm<sup>-1</sup>. Additionally the 5*d*-levels of Ce<sup>3+</sup>, Pr<sup>3+</sup> and Yb<sup>3+</sup>, are shown schematically, as well as the band gaps of some semiconductors (the top of the valence band has been adjusted to the position of the ions ground states for a clearer view).

important task to measure the symmetry of the site directly by measuring the gradient of the crystal field with PAC, which we will propose herewith. Combining PAC site symmetry with EC lattice location and optical Stark level spectrosocpy measurements, will provide a deep inside into the nature of the crystal field and of the luminescence it causes.

# 2) The new digital PAC spectrometer

Conventional PAC spectrometers typically process up to 10 kevents/s by fast, mostly analog electronics. For data reduction coincidences are detected by electronic logic operations. Since modern transient recorders now feature the necessary speed for data processing in the nanosecond range at affordable prices, the possibility to get rid of restricting electronics and replacing it by an all-digital approach when processing signals coming for usual  $\gamma$ -ray detectors.

The new digital PAC (DIGIPAC) spectrometer was proposed to the German BMBF ("Bundesministerium für Bildung und Forschung") some time ago as an additional instrumentation to ISOLDE and, after approval, was built and tested during the last months. Prior to this new setup, a first prototype was developed and tested [RHG+08]. Due to even faster digitizer cards and faster computers available in the meantime at an affordable price, we succeeded in significantly improving the first design, overcoming earlier dead time and synchronization problems.

In the new DIGIPAC spectrometer (a schematic drawing is shown in Fig. 2) signals coming from the  $\gamma$ -detectors (LYSO crystals + Hamamatsu XP2020 photomultipliers) are directly fed into Agilent Aquiris U1071A 8-bit digitizer cards without any preamplification. The digitizer cards, which are each hosted by a 2 x Xeon guad-core measurement computer (equivalent to 8 cores/computer), sample the incoming detector signal at a sampling rate of 2 GSamples/s. A configurable threshold trigger creates a time stamp for each event with an accuracy of 13 ps, possibly followed by a readout of the ring buffer where the sampled signals are stored. The trigger is rearmed after 350 ns. The main limitation in throughput and an additional contribution to the system's dead time is given by the data transfer rate from the Aguiris card to the host computer at the card's side. Our tests have shown that each detector channel is limited to 104,000 events/s at 1000 samples/event. Given that an external trigger signal of 2 GHz is chosen (this is the case in our current implementation) each detector signal is sampled for a time span of 500 ns, which is sufficient to determine the  $\gamma$ -ray energy from the signal's integral value. Further signal processing is done by software, where for each event time and energy information are generated. The main server (PAC-CONTROL) collects and sorts all events from the four measurement computers

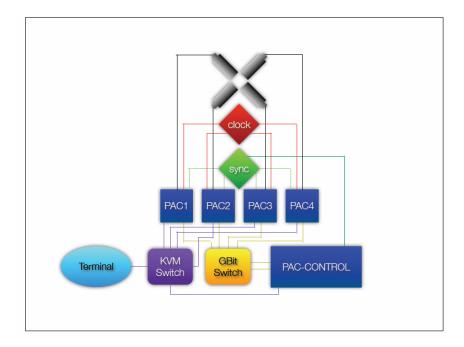


Fig. 2: Schematic drawing of the principal components of the new digital PAC spectrometer. For further details see text. (*PAC1..4*) and searches for correlations between the events on-the-fly to reduce the amount of data that needs to be stored. Integral rise time correction and energy calibration are done per software as well. The detector events, which then contain information on the detector number, the energy and time of the  $\gamma$ -rays are sorted regarding time, are finally written to a fast database in list mode. The maximum size of the database depends on the storage space available. Energy windows are set by an user interface for each channel and correlations between the channels are searched for automatically yielding the usual R(t) spectrum.

Due to the use of LYSO (Cerium-doped Lutetium Yttrium Orthosilicate) detector crystals the four channels can be calibrated automatically regarding energy as well as time because of the naturally abundant radioactive <sup>176</sup>Lu ( $t_{1/2}$ =3.6·10<sup>10</sup> y). The detector crystals we use feature an intrinsic count rate of 4 kHz, the energy spectrum showing the characteristic  $\beta$  - Spektrum as well as 88.34, 201.83 306.78 and 400.99 keV  $\gamma$ -rays and their sum peaks. This provides enough features for an automatic energy calibration. Moving the detectors close to each other the  $\gamma$ -rays are suitable for time calibration at different energies as well, since  $\gamma$ -rays from one of the detector crystals may penetrate another crystal. If both events are detected this will yield a prompt peak. LYSO detectors have been chosen because of their better energy and time resolution over Nal. BaF crystals are slightly faster but have a long-living decay in their signal.

Finally it is worth mentioning that the Aquiris cards feature a second detector input, which would eventually allow us to build an 8-detector PAC spectrometer, whereas each detector channel could be sampled at a maximum rate of 1 GHz. If we consider the maximum data transfer rate of the computers' 32-bit PCI busses clocked at 66 MHz, then one could alternatively add a second card to each computer, thus allowing to build an 8-detector setup with 8 cards or even a 16 detector setup. By adding more host computers there is in principle no limit for the number of detector channels. The principal design of the software takes this further possible extension already into account.

Since there is no loss of information regarding both time and energy of the incoming  $\gamma$ events, an important application of such a spectrometer lies in PAC spectroscopy where more than two energy windows are required. This can be the case in the decay of some PAC isotopes, which possess more than one useful  $\gamma\gamma$ -cascade, in the successive decay of shortlived mother and daughter isotopes, or in  $\gamma\gamma$ -cascades that are populated and/or depopulated through transitions with  $\gamma$ -rays with varying energies. These cases will be discussed in more detail in the next chapter.

#### 3) Rare earth PAC isotopes

PAC measurements with RE isotopes have been demonstrated in the past, even for host lattices like GaN and ZnO [NVT04] [NVC06], but in many cases the experiments had problems due to complicated gamma cascades as well as, quite often, due to the many beam contaminations with other isobars.

Since there is no need to adjust the energy windows for the PAC measurements prior to the measurement itself with the new setup, we expect that it will be much more efficient to optimize the energy window selection to apply, in particular, RE PAC probes in semiconductors systematically. In the following we will discuss cases where more than two energy windows are involved and may me fully utilised with the new PAC setup.

#### Level population and/or depopulation by multiple $\gamma$ -ray transitions

The most general form of the angular distribution  $W(\Theta)$  of the second  $\gamma$ -ray involved in  $\gamma\gamma$ -

transitions is given by  $W(\Theta) = \sum_{n=0}^{n_{max}} A_{nn} P_n(\cos\Theta)$ . The anisotropy coefficients  $A_{nn}$  depend on

nuclear spin as well as the multipolarity mixing of the  $\gamma$ -rays involved. A perturbation of the intermediate level is introduced through external electric field gradients and magentic fields.

In typical PAC measurements two energy windows per detector channel are set to select  $\gamma_1$  and  $\gamma_2$ . In many cases the intermediate level is populated by more than one transition with the  $\gamma$ -rays involved having different energies. The same holds for the depopulation of the intermediate level. In the case of conventional analog setups typically only two energy windows can be chosen, many viable events are discarded. Since we are able to monitor all events with the digital PAC spectrometer, we may significantly reduce the necessary number of probe atoms in a sample. Of course data analysis is more complex in case several  $\gamma$ -ray energies are involved and the different anisotropies must be balanced for the analysis. In the following table we list a number of isotopes for which the described scenario occurs.

Isotope		Mother isotope		Intermediate state		γ-rays	
	lifetime		lifetime(s)	E [keV]	t <sub>1/2</sub> [ns]	E [keV]	multipol.
$^{147}_{61}Pm$	2.62 y	$^{147}_{60}Nd$	10.98 d	91	2.50	91 (d)	M1+E2
						319 (p) 398 (p) 440 (p)	M1+E2 M1+E2 M1+E2
$^{147}_{63}Eu$	24.1 d	$^{147}_{64}Gd$	38.05 h	625	765	325 (d)	M2
						396 (d) 370 (p)	E3 M1+E2
						610 (p)	E2
1.40		1.40				619 (p)	M1+E2
$^{149}_{63}Eu$	93.1 d	$^{149}_{64}Gd$	9.28 d	496	2450	347 (d)	M2+E3
						496 (d)	E3
						299 (p)	M1+E2
$^{156}_{64}Gd$	stable	$^{156}_{63}Eu$	15.19 d	98	2.21	98 (d)	E2
						1154 (p) 1231 (p)	E2 E1
$^{172}_{70}Yb$	stable	$^{172}_{69}Tm$	63.6 h	79	1.65	79 (d)	E2
			lovel depend			182 (p) 1094 (p)	E2 M1+E2

(p): level-populating transition; (d): level-depopulating transition

# Isotopes with two useful intermediate levels

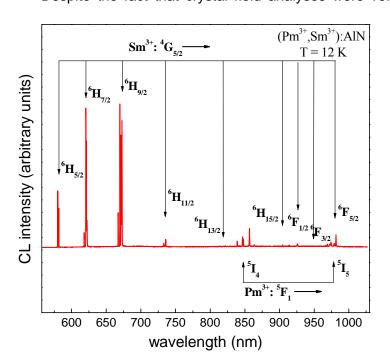
In certain cases the isotope has (at least) two long-lived intermediate states which are sufficiently populated. To use both levels in the PAC experiment which would allow for complementary measurements. Some examples are listed below:

Isotope		Mother isotope(s)		Intermediate states		γ-rays	
	lifetime		lifetime(s)	E [keV]	t <sub>1/2</sub> [ns]	E [keV]	multipol.
$^{169}_{69}Tm$	stable	$^{169}_{70}Yb$	32.03 d	379	52.6	93 (p)	M1+E2
					ĺ	63 (d)	E1
				316	660	63 (p)	E1
					ĺ	177 (d)	M1+E2
						198 (d)	M1+E2
						308 (d)	E2
$^{172}_{70}Yb$	stable	$^{172}_{71}Lu \setminus ^{172}_{69}Tm$	6.70 d \ 63.6 h	1172	8.3	1094 (d)	M1+E2
						912 (d)	M1+E2
					ĺ	91 (p)	M1+E2
						490 (p)	M1(+E2)
						528 (p)	M1(+E2)
				79	1.6	79 (d)	E2
						1094 (p)	M1+E2
						182 (p)	E2
						1523 (p)	E1+M1(+E0)

(p): level-populating transition; (d): level-depopulating transition

#### 4) Physics case

Studies on RE doped semiconductors with radioactive isotopes from ISOLDE have been a very successful story throughout the last years. Most experiments target a fundamental comprehension of the optical properties of such systems. Among the most important aspects are detailed crystal field analyses of the RE intra-4f electron transitions observed under light or electron excitation [GVH+04] [GZLJ02a], which in turn can explain the optical properties and open up a route for altering the luminescence properties as desired. Modeling such spectra is based upon knowledge of the ions' lattice sites or, in more detail, on the ligand configuration around the ions and the resulting crystal field [Vet03]. Very important contributions have been made with the help of lattice location studies applying the EC technique [HL91], which reveals the positions of the ions within the unit cell of the host semiconductor. Surprisingly and in contrast to the often quoted chemical identity of the REs, differences in lattice locations as well as crystal field investigations have shown that the RE series requires full exploration element by element [GZLJ02b]. In the last years several experiments have been performed to systematically explore the complete RE series in important wide band gap semiconductors such as w-GaN [GZLJ02a] and w-AIN [GVH+04] and others [VHWD03] [WAL+03]. In this context we were able to establish the combination of different methods such as EC and optical spectroscopy in the past [WAL+03] [VGN+06]. Among those studies are also the first ever-reported optical investigations of the radioactive element promethium [VGN+06], which was studied in w-AIN after implantation at ISOLDE (see Fig. 3). Such investigations represent very important contributions to crystal field investigations and mean free ion parameter sets of REs in general since optical studies are very scarce in the case of promethium. We will therefore put an additional focus on systematic investigations of promethium in varying wide band gap host matrices in the scope of this proposal.



Despite the fact that crystal field analyses were very successful, the derivation of the

Fig. 3: Cathodoluminescence spectrum of *w*-AlN, implanted at ISOLDE with  ${}^{147}_{60}Nd$  (t<sub>1/2</sub> = 10.98 d), taken several months after the implantation when only  ${}^{147}_{61}Pm$  (t<sub>1/2</sub> = 2.62 y) and  ${}^{147}_{62}Sm$  (stable) were left in the sample. Combined emission channeling investigations were applied to the same samples on the decay  ${}^{147}_{60}Nd \rightarrow {}^{147}_{61}Pm$ . ligands' position around the RE ions from lattice location studies only has been а maior uncertainty since such studies do directly measure not the symmetry of the ligands' crystal field. It was also observed through optical Stark level spectroscopy that the ions might occupy different sites in the host lattice [OH06]. It is difficult to address this site multiplicity with optical spectroscopy and lattice location studies alone, since the latter ones may not detect those sites, which aren't embedded regularly into the host lattice and indeed such 'random sites' often significantly contribute to the background in EC spectra. Optical laser spectroscopy may also miss such sites depending on the excitation wavelength. these Even if sites show luminescence it may overlap with the luminescence of other sites, which requires time-resolved optical spectroscopy. Therefore it is an important task to determine the magnitude (and possibly the direction) of the electric field gradient directly, which can be approached with PAC. Especially 'missing sites' should be detectable this way. The feasibility for RE PAC measurements in wide band gap semiconductors has already been demonstrated for  $\frac{172}{70}Yb$  in *w*-GaN and ZnO [NVT04], but more studies over the RE series including several host matrices and differently prepared samples are necessary.

Another important open question that needs to be addressed is the large *nephelauxetic* effect [Jor71] observed for RE doped III-nitrides. This red-shift in the luminescence spectra, attributed to an increase in the expectation value of the square of the 4*f* wavefuntion distance  $\langle r^2 \rangle$  from the nucleus is among the largest value ever observed for RE doped systems. Its origin in the case of RE doped wide band gap semiconductors is an open and much debated issue but might be due to the covalency of the bonding between the RE ion and the nitrogen ligand. Such bonding properties manifest themselves in the electric field gradient and might therefore be seen in the PAC measurements. The parameters obtained should be directly connectable to the ones drawn from crystal field analysis on the same isotope.

Finally we would like to study the RE crystal field in the dilute magnetic semiconductor GaN:Gd in more detail. It has been shown that ferromagnetism and a colossal magnetic moment can be achieved in Gd ion-implanted GaN, with variation of the magnetism depending on the concentration of the ions [DKN+06]. We propose to study this magnetism at the atomic level using PAC spectroscopy in combination with EC lattice location studies. Note that luminescence measurements on Gd<sup>3+</sup> in GaN are not useful since the band gap of GaN is lower that the energy difference between the ground state and the first excited state of Gd<sup>3+</sup>. The situation is however different for AIN with a much larger band gap. Many stable Gd isotopes feature excited states with sufficient long lifetimes and magnetic moments for PAC measurements. We suggest to start with  $\frac{156}{64}Gd$  first since there are no very long-lived contaminating isobars of mass 156.

Therefore we would like to use the  ${}^{147}_{61}Pm$ ,  ${}^{149}_{63}Eu$ ,  ${}^{156}_{64}Gd$ ,  ${}^{169}_{69}Tm$  and  ${}^{172}_{70}Yb$  PAC probing isotopes, which are respectively obtained onto decay of  ${}^{147}_{60}Nd$ ,  ${}^{149}_{64}Gd$ ,  ${}^{156}_{63}Eu$ ,  ${}^{169}_{70}Yb$  and  ${}^{172}_{71}Lu$  implanted into selected wide band gap semiconductors such as *w*-AIN, *w*-GaN, Si<sub>3</sub>N<sub>4</sub>, 6H-SiC, ZnS and diamond. Subsequent to implantations a series of investigations of hyperfine field, lattice location and cathodo- or photoluminescence spectroscopy will be carried out. It is worth mentioning that RE trifulorides were studied in a similar series of experiments, at that time combining integral PAC ( ${}^{154}_{64}Gd$ ), optical absorption spectroscopy and other methods to study the crystal field in detail [BGK+84].

To summarize the following experiments are considered:

# Promethium in semiconductors

PAC, EC and luminescence spectroscopy on  ${}^{147}_{60}Nd$  ( ${}^{147}_{61}Pm$ ) in GaN, ZnO, Si<sub>3</sub>N<sub>4</sub>, 6H-SIC and BN with a special emphasis on promethium Stark level luminescence spectroscopy and optical crystal field investigations. First EC lattice location and PAC site symmetry measurements will be performed which will yield the parameters necessary to model the Stark level splitting observed from preceding luminescence measurements. Note that  ${}^{147}_{61}Pm$  is the isotope with the longest lifetime among all other isotopes with mass 147, except the stable  ${}^{147}_{62}Sm$ . The luminescence measurements can be taken when all other long-lived isobars of mass 147, which will be present as contaminations, will have decayed completely.

# Site multiplicity and the nephelauxetic effect in RE doped wide band gap semiconductors

In order to study both effects systematically with PAC, EC and luminescence spectroscopy, several RE isotopes are chosen and implanted into different semiconductors which vary regarding the covalency of the bonding between the RE ion at the surrounding ligand. In semiconductors like BN, GaN and AIN the bonding between the RE ion and the nitrogen ligand has a more or less covalent/ionic character, while in SiC the bonding has a highly ionic character. In diamond the character of the bonding is extremely covalent. The following order of the experiments is suggested:

- a)  $\frac{172}{71}Lu(\frac{172}{70}Yb)$  in *w*-AIN, ZnO, 6H-SiC and diamond
- b)  ${}^{169}_{70}Yb$  ( ${}^{169}_{69}Tm$ ) in *w*-AIN, ZnO, 6H-SiC, ZnS and diamond
- c)  ${}^{149}_{64}Gd$  ( ${}^{149}_{63}Eu$ ) in *w*-AIN, ZnO, 6H-SiC

Note that in diamond there has been no observation of any RE luminescence so far, which is on its own an important issue that we will try to address with PAC alone first.

# The Dilute magnetic semiconductor GaN:Gd

PAC, EC and luminescence spectroscopy on  ${}^{156}_{64}Gd$  ( ${}^{156}_{63}Eu$ ) in GaN with a special emphasis on the magnetic properties of Gd<sup>3+</sup> in GaN. After obtaining sufficient insight we plan to study the system AIN:Gd in a similar manner.

# 5) Beam request and experimental requirements

Based on above considerations we would like to apply for beam time for the following isotopes during a period of 2 years:

isotope	number of shifts	target	ion source	min. yield [atoms/ μC]
$^{147}_{60}Nd$	3	UC <sub>2</sub>	W surface	5.4 · 10 <sup>6 (1)</sup>
$^{156}_{63}Eu$	2	UC <sub>2</sub>	W surface	1.3 · 10 <sup>6 (1)</sup>
$^{149}_{64}Gd$	3	Та	(2)	5 · 10 <sup>8 (2)</sup>
$^{169}_{70}Yb$	3	Та	Rilis (W)	1.9 · 10 <sup>8</sup>
$^{172}_{71}Lu$	3	Та	(3)	(3)

<sup>(1)</sup> SC yield, PSB yield not listed in database. Including the precursors we expect the yields to be nearly two orders of magnitude higher as it was the case in a run in the year 2000. <sup>(2)</sup> Isotope not listed in ISOLDE yield database, but implanted and used for emission channeling

<sup>(2)</sup> Isotope not listed in ISOLDE yield database, but implanted and used for emission channeling measurements in the past, see e.g. [VGN+06]. The yields for  $^{149}_{64}Gd$  should be in between the ones for

 $^{148}_{64}Gd$  (6  $\cdot$  10<sup>8</sup>) and  $^{150}_{64}Gd$  (4  $\cdot$  10<sup>8</sup>).

<sup>(3)</sup> Isotope not listed in ISOLDE yield database, but frequently implanted at ISOLDE and used for PAC measurements in the past, see e.g. [NVT04].

All implantations can be done at 60 keV at the solid-state physics implantation chamber, the PAC, EC and luminescence measurements will be performed off-line.

We expect to avail of the PL lab at ISOLDE to carry out optical measurements on our implanted samples, EC measurements can be done with the available new emission channeling spectrometers (for details see experiment no. IS453) at ISOLDE.

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