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The 3rd International Workshop on Radiopure Scintillators RPScint 2013

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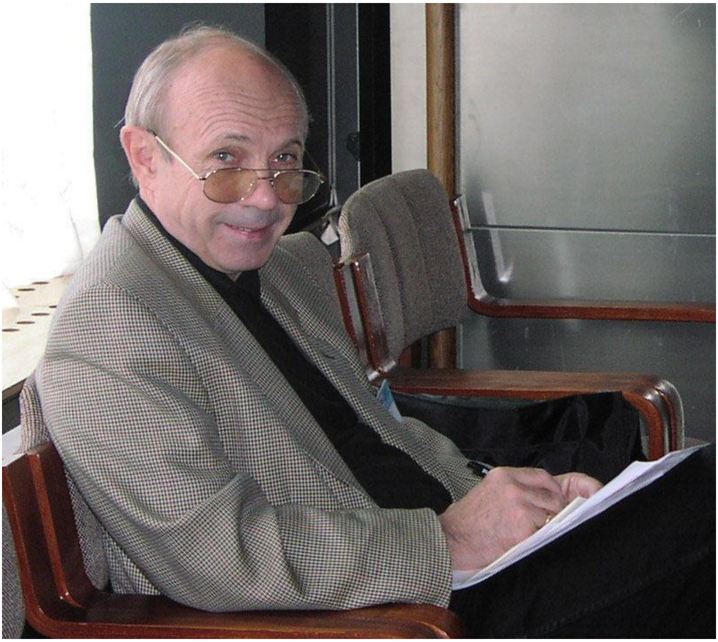

Dedicated to the 70th anniversary of Yuri Zdesenko (1943-2004)

Sponsored in part by the National Academy of Sciences of Ukraine

Edited by F.A. Danevich and V.I. Tretyak

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This talk will focus on inorganic scintillators applied to direct Dark Matter investigation and rare processes. Firstly, the important role played by inorganic crystal scintillators in the modern physics will be recalled, and the continuous innovation provided by physics, chemistry, and technology in the development of crystal scintillators with improved performances will be outlined. Some strategies towards the achievement of the best performances of inorganic scintillators (such as e.g. high detection efficiency, physical form, chemical and mechanical stability, luminescent characteristics, etc.) will be discussed. The comparative possibilities of highly radio-pure detectors will be addressed. Several detectors with new performances and/or materials will be discussed in the light of their further/future application to the investigation of rare processes.

$CaMoO_4$ crystal scintillator based $0\nu\beta\beta$ experiment: AMoRE

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The AMoRE (Advanced Mo-based Rare process Experiment) project is an international collaboration experiment search for neutrinoless double beta decay of Mo-100 using a cryogenic technique with $^{40}Ca^{100}MoO_4$ scintillation crystals at the underground laboratory in Korea. $CaMoO_4$ crystals show brightest scintillation light among variety of molybdate crystals at room and low temperature. The AMoRE will run at millikelvin temperature with $CaMoO_4$ crystals and metallic magnetic calorimeters (MMC) as temperature sensors. The MMC has relatively fast rise time that improve the rejection of random coincidence events of two neutrino double beta decay of ^{100}Mo. Pulse shape discrimination method will be applied with both scintillation and phonon signal readout to reject alpha induced backgrounds. Significant improvement of effective Majorana neutrino mass sensitivity at the level of inverted hierarchy, 20 – 50 meV could be achieved by AMoRE with 200 kg of $^{40}Ca^{100}MoO_4$ crystals. Current study of $CaMoO_4$ detectors at room and millikelvin temperatures as well as background reduction method will be presented.

Search for rare processes with $ZnWO_4$ crystal scintillators

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In the framework of the Collaboration between the DAMA project and the INR (Kyiv, Ukraine), radiopure $ZnWO_4$ crystal scintillators have been developed and measured in the Gran Sasso National Laboratories of the INFN [1,2]. In particular a search for the double beta decay of zinc and tungsten isotopes has been performed [3,4]. The new improved half-life limits on the double beta decay modes of ^{64}Zn, ^{70}Zn, ^{180}W and ^{186}W, established at the level of 10^{18}–10^{21} y, will be presented [4]. The new half-life limit on α transition of ^{183}W to the metastable excited level of ^{179}Hf will be also discussed [4]. Future perspectives of this crystal scintillator to search for double beta decay and for the investigation of dark matter by exploiting the directionality signature will be addressed [5].

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[1] P. Belli, Phys. Lett. B 658 (2008) 193.
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[4] P. Belli et al., J. Phys. G: Nucl. Part. Phys. 38 (2011) 115107.
[5] F. Cappella et al., Eur. Phys. J. C 73 (2013) 2276.

Neutrinoless double beta decay search with SNO+

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The SNO+ experiment [1] is the follow up of SNO. The active volume of the detector consists of 780 tons of Linear Alkyl Benzene (LAB) in an acrylic vessel of 12 m diameter, surrounded by about 9000 PMTs. LAB is a liquid scintillator chemically compatible with acrylic, that has a high light yield (about 10,000 photons/MeV), good optical transparency and low scattering. Moreover, high purity levels are easily available directly from the manufacturer and, due to the fast decay, it is possible to discriminate between beta and alpha particles, allowing a background rejection.

The main goal of the SNO+ experiment will be the search for neutrinoless double beta decay. The liquid scintillator will be initially loaded with 0.3% of natural Tellurium. ^{nat}Te contains, with an isotopic abundance of 34.08%, ^{130}Te, one of the candidates for the neutrinoless double beta decay.

Due to the rare signal searched for, the purity level of the liquid scintillator is very important. Great attention is paid to the purification of the liquid scintillator and selection of the materials that will be in direct contact with it or the acrylic vessel. The aim is to have a concentration of ^{238}U and ^{232}Th isotopes of the order of 1e-17 g/g.

In order to increase the detector sensitivity, coincidences studies and tagging techniques are also under investigation. Background sources in the energy window for the $0\nu2\beta$ are for instance ^{208}Tl, ^{214}Bi. Tagging techniques under development have shown that a >99.8% rejection on ^{214}Bi, and a >90% rejection on ^{208}Tl are achievable.

Complementing this double beta program, other exciting physical goals that can be explored are reactor neutrino oscillations, geo-neutrinos in a geologically-interesting location, supernova neutrino watch and solar neutrino studies.

In this talk, the physics program and the current status of the SNO+ experiment will be addressed.

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[1] M.C. Chen, Nucl. Phys. B (Proc. Suppl.) 145 (2005) 65.

First results of the experiment to search for double beta decay of ^{106}Cd with $^{106}CdWO_4$ crystal scintillator in coincidence with four crystals HPGe detector

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An experiment to search for double beta processes in ^{106}Cd by using cadmium tungstate crystal scintillator enriched in ^{106}Cd ($^{106}CdWO_4$ [1]) in coincidence with the four crystals HPGe detector GeMulti is in progress at the STELLA facility of the Gran Sasso underground laboratory of INFN (Italy). The $^{106}CdWO_4$ scintillator is viewed by a low-background photomultiplier tube through a lead tungstate crystal light-guide produced from deeply purified archaeological lead to suppress γ quanta from the photomultiplier tube. The surface contamination of the $^{106}CdWO_4$ crystal by ^{207}Bi [2] was removed thanks to the cleaning of the scintillator by potassium free detergent and ultra-pure nitric acid. Here we report the first results of the experiment after more than 3 thousands hours of the data taking.

References

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Search for 2β decay of ^{116}Cd with the help of enriched $^{116}CdWO_4$ crystal scintillators

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Cadmium tungstate crystal scintillators enriched in ^{116}Cd to 82% ($^{116}CdWO_4$, ≈ 1.2 kg) [1, 2] are used to search for 2β decay of ^{116}Cd deep underground at the Gran Sasso National Laboratory of the I.N.F.N. (Italy). The radioactive contamination of the $^{116}CdWO_4$ crystals is as following: ^{40}K < 0.5 mBq/kg, ^{113}Cd = 0.10(1) Bq/kg, ^{113m}Cd = 0.46(2) Bq/kg, ^{110m}Ag = 0.07(3) mBq/kg, ^{226}Ra < 0.005 mBq/kg, ^{228}Th = 0.031(3) – 0.054(5) mBq/kg, total α activity = 1.8(1) – 2.6(1) mBq/kg.

The measured half-life of ^{116}Cd relatively to 2ν2β decay is $T_{1/2} = (2.6 \pm 0.4) \times 10^{19}$ yr, in agreement with the results of previous experiments. The obtained limit on the 0ν2β decay of ^{116}Cd (considering the data of the last 5557 h run with an advanced background 0.14 counts/yr/keV/kg in the energy interval 2.7 – 2.9 MeV) is $T_{1/2} \geq 5.7 \times 10^{22}$ yr at 90% C.L. The sensitivity of the experiment to the 0ν2β process can be advanced (by further reduction of the background by a factor 2 – 10) to the level of lim $T_{1/2} = (0.5 – 1.5) \times 10^{24}$ yr over 5 years of the measurements.

References

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[2] D.V. Poda et al., Radiat. Meas., in press, DOI 10.1016/j.radmeas.2013.02.017.

Expected backgrounds in AMoRE experiment

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AMoRE is a planned large-scale experiment aimed to search for neutrinoless double beta decay of ^{100}Mo [1,2]. The detector will be assembled from single crystals of isotopically modified calcium molybdate $^{40}Ca^{100}MoO_4$. The crystals will be operated as cryogenic scintillation bolometers. The recording of shapes of heat and light pulses will allow to separate the events related to alpha and beta particles and the pile-up events. I will discuss the Monte-Carlo simulation of the expected backgrounds of the experiment. The contributions from the following sources to the total background are calculated: 1) contaminations of U and Th daughters in the detector crystals and in the construction materials; 2) cosmogenic radionuclides; 3) random coincidences of two-neutrino double beta decays of ^{100}Mo; 4) radio- and cosmogenic neutron flux in the YangYang Underground Laboratory; 5) cosmic muons. The possibilities of backround suppression by pulse shape analysis and by time-amplitude method will be discussed.

References

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Scintillator characterization

Influence of traps on the luminescent and scintillation properties of molybdates

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The scintillation properties of crystals are commonly dependent on the presence of traps in the material. Intermediate localization of charge carriers at shallow traps may result in the appearance of slow components in scintillation decay curves while the suppression of light yield may be expected in case of deep traps for conventional scintillators. Charge carrier trapping is a serious problem for cryogenic scintillators. Under the operating conditions, even shallow traps can have a strong impact on scintillation efficiency due to capturing separated charge carriers. Therefore, the study of the origin of traps in molybdates is of crucial importance for understanding their scintillation properties at low temperatures. Here we present data on the trap centers for the scheelite type crystals $CaMoO_4$, $SrMoO_4$, $PbMoO_4$ as well as for $ZnMoO_4$, which are considered for application as cryogenic scintillators. The origin of charge carrier traps in molybdates that are potential materials for application in cryogenic scintillation detectors is discussed and the influence of trapping phenomena on luminescence properties is demonstrated. Self-trapped electrons or self-trapped holes have been reported in all the studied crystals. The present study supplies arguments in favor of the presence of self-trapped holes in $CaMoO_4$ and $SrMoO_4$ and self-trapped electrons in $PbMoO_4$. It is shown that both self-trapped electrons and holes coexist in $ZnMoO_4$. The immobility of charge carriers at $T < 50$ K results in the substantial decrease of the probability of STE creation with consequent worsening of the luminescent properties and in an unusually low scintillation light yield of $ZnMoO_4$ at low temperatures.

Response of parylene-coated NaI(Tl) scintillators at low temperature

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Despite that it is widely used as a scintillator at room temperature, the hygroscopicity of NaI complicates its handling and limits its application for many purposes, for example as a cryogenic detector. To overcome this problem we study coating materials that can act as humidity barriers, in particular parylene, a polymer that can be deposited in very radiopure, thin and conformal layers. In this work, several NaI(Tl) samples coated with 2-5 μm parylene-C were tested at low temperature. Luminescence spectra are presented at several temperatures under X-ray excitation, as well as the light output vs temperature response at 1.5-300 K. Several thermoluminescence peaks were observed at around 60, 95 and 150 K during warm up to room temperature. The mechanical resistance of the coating under thermal cycles was also investigated, and a degradation of the optical appearance and the light output observed after cooling down to about 100 mK, which compromises the reusability of the samples.

Low-background setup for measurement of the intrinsic background of $CaMoO_4$ scintillation crystals

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The low-background setup for measurement of the intrinsic background of scintillation crystals $CaMoO_4$ in active mode is made. The results of measurements of background characteristics of low-background setup by the test detector made from plastic scintillator are given.

Monte-Carlo simulation of light collection efficiency of scintillation detectors using ZEMAX

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With recent advances in the technology of scintillation materials, pushing their intrinsic light yield towards the fundamental limit [1] a key factor that precludes further improvement in responsivity of scintillation detectors is the light collection efficiency. Typically fewer than half of the photons generated by a scintillator reach the photodetector and research is ongoing to investigate how this parameter can be improved by changing experiment geometry, scintillator shape, surface condition, wrapping, optical contact, etc. Monte-Carlo simulation is a powerful tool to study light transport in crystals and the currently most commonly used simulation software are Detect2000, Geant4, GATE and Litrani. In this talk we present the application of the commercial ray-tracing software ZEMAX to modelling the light collection properties of a scintillation detector. The aim is to devise an optical model for optimisation of a scintillation detector in a configuration typical for most cryogenic applications, i.e. where the crystal is kept in a vacuum and optical coupling by gels is not possible.

References

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Temperature dependent light output of scintillating crystals

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The **C**ryogenic **R**are **E**vent **S**earch with **S**uperconducting **T**hermometers (CRESST) is an experiment for direct detection of galactic dark matter particles measuring their deposited energy off scattering target nuclei. Besides passive shielding CRESST has an active event-by-event discrimination to distinguish possible weakly interacting massive particle (WIMP) induced recoil events from background. To achieve this discrimination CRESST uses scintillating $CaWO_4$ crystals as target material and measures not only the total deposited energy per event, but also the light produced by the interaction. Electron recoil events show different light yield than nuclear recoil, what allows to reject most background events, especially electron and γ particle interactions, which are the main part of background events. To study the properties of a potential WIMP, we aim to realize detectors with a larger variety of target nuclei. To achieve this we measure the relative light output of scintillating crystals down to 1.7K with an optical cryostat using the multiple photon counting coincidence (MPCC) technique [1]. One interesting material under study is NaI, also used in the DAMA experiment, which has measured an annual modulation signal in their data possibly induced by WIMP interactions. Measurements of the relative light output of pure NaI compared to Tl doped NaI crystals show completely different behaviour over the temperature range from 300K to 30K. For lower temperatures however the light output of both crystals become similar and at 2K both materials have a relative light yield of roughly 65% compared to room temperature values of NaI(Tl). This may be an indication that the scintillation mechanism of both crystals at low temperatures is dominated by intrinsic processes [2]. In addition to the light yield measurements, the phonon properties of NaI are investigated with an dilution refrigerator in an underground laboratory.

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Semi-empirical calculation of quenching factors for scintillators: new results

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Semi-empirical method of calculation of quenching factors for scintillators is presented [1] which is based on classical Birks formula. The *total* stopping powers for electrons and ions are used; they are calculated with the ESTAR and SRIM codes, respectively. Method has only one fitting parameter (the Birks factor *kB*) which can have different values for the same material in different conditions of measurements and data treatment. In [1], the hypothesis was used that, once obtained by fitting data for particles of one kind and in some energy region (e.g. for a few MeV α particles from internal contamination of a detector), the same *kB* value can be used to calculate quenching factors for particles of another kind and for another energies (e.g. for low energy nuclear recoils) – if all the data are measured in the same experimental conditions and are treated in the same way. Applicability of the method is demonstrated on many examples: old, already described in [1], but also new, obtained during last 3 years.

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Lithium-containing scintillating bolometers for low background physics

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We present the performances of Li-based compounds used as scintillating bolometer for rare decay studies such as double-beta decay and direct dark matter investigations. The compounds were tested in a dilution refrigerator installed in the underground laboratory of Laboratori Nazionali del Gran Sasso (Italy).

Low temperature scintillating properties were investigated by means of different radioactive sources, and the radio-purity levels for internal contaminations are estimated for possible employment for next generation experiments.

Scintillator development

Search for scintillating crystals for rare events physics application

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The production of scintillation materials to be used in rare events physics experiments, particularly in Neutrinoless Double Beta Decay (0νDBD) experiments, asks for dedicated methods to be applied for the entire production process from synthesis of raw materials up to the storage and transport of the finished product ready for use for the construction of the particle detector. Moreover, in the case of 0νDBD application, the crystal requires the presence of the nuclide of interest in a sufficient amount i.e. isotope enriched materials are employed. The current work will make a review of scientific and technological aspects related to the crystal production for bolometric application in 0νDBD experiments with a special concern for the obtainment of a maximum production yield. The presentation is based on the results obtained in two relevant experiments in the field: the Cryogenic Underground Observatory for Rare Events (CUORE) and the Low-background Underground Cryogenic Installation For Elusive Rates (LUCIFER).

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Growth and spectroscopic properties of ^{6}Li- and ^{10}B-enriched crystals for heat-scintillation cryogenic bolometers used in the rare events searches

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The dark matter content of the Universe accounts for 23% of its total energy [1]. Dark matter particles from the halo of our galaxy can be, in principle, detected by the nuclear recoils that they produce in a detector when scattered off nuclei, and great experimental efforts are nowadays dedicated to this subject [2]. In the past decade, improved sensitivity of this kind of experiments has come from the acquired capability of distinguishing nuclear recoils in the detector (like those presumably produced by WIMPs) from electronic recoils (produced by the most common radioactive background – gamma, electrons and alpha particles) [3-6]. Neutrons, which scatter off nuclei, are therefore the ultimate background of these searches. A bolometer consists of a crystal absorber strongly coupled to a thermometer, both weakly coupled to a heat sink maintained at 10-30 mK. When a particle of any kind interacts with the absorber, the energy released is seen as a temperature increase by the sensor. Scintillating bolometers are double readout (light and heat) detectors that measure simultaneously the heat and the light generated by a particle [7]. Particle discrimination is achieved by comparison of both signals, since the heat released by a particle is similar for all types of particles, and proportional to their energy, and the light yield strongly depends on the ionization power of the incident particle [3-7]. In this context, we are developing the growth of crystals with diameters of several centimeters and thicknesses several times the thermal neutron mean free path in ^{6}Li-based crystals and the range of neutron capture induced particles in crystals made of ^{6}Li or ^{10}B, typically

6 μm for α's and 34 μm for tritiums in lithium borates. As the ^{6}Li and ^{10}B isotopes, as well as several Gd isotopes, exhibit high neutron capture cross-sections ($\sigma^{10}_{B\text{-}n} \approx 4\times\sigma^{6}_{Li\text{-}n}$(10 keV) ~ 8×10^{-24} cm^2) [8,9], it soon turned out that crystals of the $Li_6Gd(BO_3)_3$-type would constitute ideal candidates for both tailoring HSBCs with high light yields over a wide neutron energy range and adapting them to powerful light detectors working at low temperature and available from UV to X-ray spectral ranges [3-7].

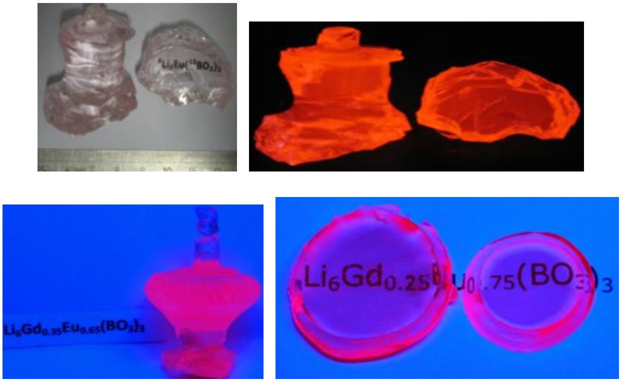

Figure 1. Top left: as-grown $^{6}Li_6Eu(^{10}BO_3)_3$ bulk crystals of 43.5 g and 17.7 g, respectively. Top right: the same crystals illuminated by 365 nm radiation. Bottom left: as-grown $Li_6Gd_{0.35}Eu_{0.65}(BO_3)_3$ bulk crystal of ~30 g. Bottom right : cut and polished $Li_6Gd_{0.25}Eu_{0.75}(BO_3)_3$ crystals 6 mm-thick and of diameters 30 mm and 23 mm, respectively. All crystals shown at the bottom are illuminated by 365 nm radiation.

Moreover, in contrast with the ^{6}LiF bulk crystals which we recently developed, $^{6}Li_6(Eu,Gd)(^{10}BO_3)_3$ crystals are much less likely to exhibit thermoluminescence properties detrimental to HSCB applications [10]. In this work, we present the crystal growth by combined Czochralski and Kyropoulos methods, initiated on specifically oriented seeds, of centimeter-sized $^{6}Li_6Eu(^{10}BO_3)_3$ and $Li_6(Eu,Gd)(BO_3)_3$ single crystals with an heretofore unexplored concentration range for HSBCs application. The crystal structure and lattice thermal expansion of the former, obtained for the first time by single crystal X-ray diffraction (XRD), and the spectroscopic characterizations together with a consistent set of related thermodynamical properties measurements (magnetic susceptibility, specific heat), also unknown to date, are discussed. Finally, preliminary scintillation measurements at low temperature between 300 and 1100 nm under X-ray excitation are shown.

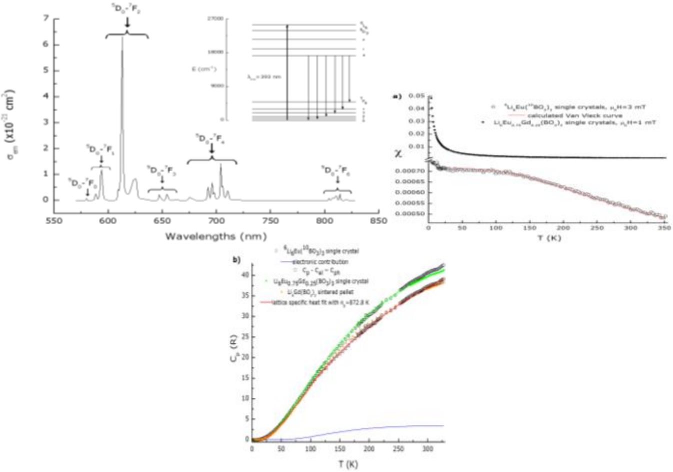

Figure 2. FL-calibrated stimulated emission cross section of $^{6}Li_6Eu(^{10}BO_3)_3$ single crystals at room temperature. The inset shows an experimental energy level diagram of Eu^{3+} ions in $^{6}Li_6Eu(^{10}BO_3)_3$ that was cross-checked by MKSA magnetic susceptibility measurements of unoriented $^{6}Li_6Eu(^{10}BO_3)_3$ and $Li_6Eu_{0.75}Gd_{0.25}(BO_3)_3$ single crystals, and specific heat measurements of $^{6}Li_6Eu(^{10}BO_3)_3$ and $Li_6Eu_{0.75}Gd_{0.25}(BO_3)_3$ single crystals and of a $Li_6Gd(BO_3)_3$ sintered pellet.

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Purification of molybdenum oxide and growth of medium size zinc molybdate crystals for the LUMINEU program

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The LUMINEU program aims at performing a pilot experiment on neutrinoless double beta decay of ^{100}Mo using radiopure $ZnMO_4$ (ZMO) crystals operated as scintillating bolometers. Growth of high quality single crystals with high level of radiopurity is a hard and complex task. There are no commercially available starting materials that are tested for the presence of radioactive contamination and have the required level of background radiation. Background test of the raw materials for crystal growth is extremely difficult and requires long measurement procedure. Typically such measurements can be only done after crystal growth. For this reason the solution of this problem requires a special approach to every stage of the work to obtain crystals with desired characteristics.

This paper discusses the different approaches to the realization of schemes of synthesis and purification of precursors for growth of ZMO crystals in the framework of the LUMINEU program. The first variant of the synthesis and purification of molybdenum oxide is based on the recrystallization in aqueous solutions of ammonium paramolybdate. The resulting level of content of impurities after single and double recrystallization of the raw materials are presented and discussed. As an alternative purification scheme a process of sublimation of MoO_3 is used. The sublimation process effectively removes tungsten, while tungsten separation is inefficient in aqueous solutions. The sublimation process also successfully removes metal oxides, which have a high vapor pressure at temperatures up to 1000^o C. For a more reliable and more complete removal of impurities a combination of a two-time sublimation process at high temperatures and crystallization in water solutions is used.

For charge preparation the synthesized and purified molybdenum oxide with HP grade ZnO produced by Umicore. Zinc molybdate crystals up to 1.5 kg were successfully grown by LTG CZ technique. In the future, crystals of increasing mass grown from purified precursors will be developed for the LUMINEU experiment, including also samples enriched in the isotope ^{100}Mo. A first batch of LUMINEU crystals have been grown, and their thermal and luminescence properties are under investigation.

Production and deep purification of isotopically-enriched materials for $^{40}Ca^{100}MoO_4$ crystal growing

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Report is devoted to deep purification of compounds of isotopically-enriched calcium and molybdenum and $^{40}Ca^{100}MoO_4$ raw materials of radioactive impurities of uranium, thorium and radium. $^{40}Ca^{100}MoO_4$ single crystals grown from this raw material will be used to build cryogenic scintillation detector AMoRE Collaboration for search of neutrinoless beta decay of ^{100}Mo isotope.

Sensitivity of AMoRE detector is defined by a level of the background in the ROI of Mo-100 isotope (E = 3034 keV). One of main sources of the background is a decay of ^{208}Tl (^{232}Th-chain) and ^{214}Bi (after decay of ^{226}Ra in ^{238}U-chain) [1]. Based on a decay scheme of ^{232}Th-chain and ^{238}U-chain, purification of ^{208}Tl and ^{214}Bi can be secured after removal of three chemical elements: uranium, thorium and radium. Special attention should be paid to purification of calcium-containing components of raw material, because calcium belongs to the same sub-group of chemical elements in the Periodic Table. Contents of radioactive isotopes of radium (^{228}Ra и ^{226}Ra) are measured by low background HPGe γ-spectrometry based on the detectors situated in underground laboratory [2].

Background after 2β2ν-decay of ^{48}Ca, contained in calcium (0.187%) can be eliminated due to use of calcium depleted on ^{48}Ca isotope [3].

Content of one of the most dangerous isotope in $^{40}Ca^{100}MoO_4$ raw material should be less than 2 mBq/kg (which corresponds to the specific activity $^{40}Ca^{100}MoO_4$, single crystal grown of this raw material, ≤ 0.05 mBq/kg, taking into account additional purification during by Czochralski crystal growing by double crystallization [4]).

Enriched molybdenum (with 96.1 % of enrichment on ^{100}Mo isotope) is produced by the JSC Production Association Electrochemical plant (Zelenogorsk, Krasnoyarsk region, Russia [5]) and supplied in the form of molybdenum oxide $^{100}MoO_3$. The results of ICP-MS measurements show that the enriched material is very pure. Concentrations of ^{238}U and ^{232}Th in the oxide do not exceed 0.07 and 0.1 ppb, respectively.

Calcium carbonate $^{40}CaCO_3$ enriched in ^{40}Ca (99.964 %) and depleted in ^{48}Ca (content is ≤ 0.001 %) is produced by the FSUE Electrochimpribor (Lesnoy, Sverdlovsk region, Russia [6]). The concentration of ^{238}U and ^{232}Th in the enriched powder measured by ICP-MS is below 0.2 and 0.8 ppb, respectively. However, HPGe measurements at the Baksan Neutrino Observatory showed that the activity of ^{226}Ra and its daughter isotopes in the decay chain was on a level of hundreds mBq/kg [7] (which corresponds to concentration equal ~20 ppb of ^{238}U in the assumption of secular equilibrium). For that reason the raw materials were subjected to additional purification. A new technique of purification of calcium carbonate in the form of calcium formate ^{40}Ca(HCOO)$_2$ [4] allowed to reduce a content of ^{40}K, ^{208}Tl, ^{228}Ac, ^{226}Ra (^{214}Bi) in 20, 8, 160 and 5 times respectively in comparison with the standard procedure of purification at the Electrochimpribor plant.

Table 1. Content of radioactive impurities in calcium carbonate ^{40}CaCO$_3$ (Sample #1) and in calcium formate ^{40}Ca(HCOO)$_2$. Measurements made by low background HPGe γ-spectrometry (Baksan Neutrino Observatory INS RAS).

Isotope	Specific activity (Bq/kg)	
	Sample #1 ^{40}CaCO$_3$ standard technology November 2009	Sample #2 ^{40}Ca(HCOO)$_2$ October 2011
^{40}K	$(7.3 \pm 3.1)\cdot10^{-2}$	$(3.6 \pm 2.7)\cdot10^{-3}$
^{208}Tl [^{228}Th]	$(4.4 \pm 3.6)\cdot10^{-3}$ [$(1.2 \pm 1.0)\cdot10^{-2}$]	$\leq 5.2\cdot10^{-4}$
^{214}Bi	$(2.6 \pm 0.2)\cdot10^{-1}$	$(5.1 \pm 0.2)\cdot10^{-2}$
^{228}Ac [^{228}Th]	$(1.6 \pm 0.2)\cdot10^{-1}$	$(1.0 \pm 0.8)\cdot10^{-3}$

Table 2. Content of radioactive impurities in different samples of initial isotopically-enriched materials and ^{40}Ca^{100}MoO$_4$ raw material. Measurements made by low background HPGe γ-spectrometry (Baksan Neutrino Observatory INS RAS).

Isotope	Specific activity (Bq/kg)		
	Molybdenum oxide, ^{100}MoO$_3$	Calcium formate ^{40}Ca(HCOO)$_2$ purified by JSC «NEOCHIM»	Raw material, ^{40}Ca^{100}MoO$_4$
^{40}K	$(5.3 \pm 0.8)\cdot10^{-2}$	$\leq 8.6\cdot10^{-3}$	$\leq9.4\cdot10^{-3}$
^{228}Ac (^{232}Th)	$\leq3.8\cdot10^{-3}$	$(1.3 \pm 1.1)\cdot10^{-3}$	$(1.9 \pm 1.3)\cdot10^{-3}$
^{208}Tl (^{232}Th)	$\leq1.0\cdot10^{-3}$ [$\leq2.8\cdot10^{-3}$]	$\leq 1.3\cdot10^{-3}$	$\leq 1.1\cdot10^{-3}$
^{214}Bi (^{226}Ra)	$\leq2.3\cdot10^{-3}$	$(1.4 \pm 0.9)\cdot10^{-3}$	$\leq 1.6\cdot10^{-3}$

Table 1 and 2 contain the results of the measurements by low background HPGe γ-spectrometry of compounds of isotopically-enriched calcium and molybdenum and $^{40}Ca^{100}MoO_4$ raw materials as an example of our efforts.

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Synthesis of ZnSe charge and growing methods of ZnSe single crystals

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For registration of neutrinoless double beta decay in the project "Lucifer", it is planned to apply the crystals ZnSe enriched in ^{82}Se. The use of isotopically enriched material in the form of a multi-element scintillating bolometer causes a number of requirements to the synthesis of the charge and the resulting crystals, namely, low-loss materials at each processing stage, high chemical purity and structural perfection of the crystals, the identical scintillating characteristics of the elements in the bolometric assembly.

We conducted a series of experiments on the synthesis of charge and obtaining crystals of ZnSe by melt crystallization and vapor deposition.

Synthesis of ZnSe from elemental Zn and Se was carried out in a quartz reactor. Thus the yield of the product is 95-99% with high purity.

It is shown that the crystallization from the melt does not provide the high structural perfection of the crystalline material. Because of the phase transition in wurtzite-sphalerite crystal ,defects are present in the form of twinning planes and variously blocks. Often, these crystals were strained and prone to cracking, which significantly reduces the yield of effective elements.

Significantly better observed in the crystal structure of crystals ZnSe, grown from the vapor deposition at a temperature below phase transition.

We demonstrated that the combination is a promising process of synthesis and crystal growth from vapor deposition in a single reaction volume. This allows you to avoid the loss of almost the initial charge, which is critical to the isotope-enriched crystals.

Scintillating $CaWO_4$ single crystals for CRESST-II and EURECA

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The CRESST-II experiment [1] for the direct detection of WIMP dark matter uses scintillating $CaWO_4$ crystals that are operated as low-temperature detectors. EURECA [2] is a joint collaboration of existing cryogenic direct dark matter searches to develop a future multi-material experiment with a target mass of up to one ton. For future runs of CRESST and the EURECA experiment it is important to ensure the availability of $CaWO_4$ crystals that meet the requirements of these experiments. While up to now the crystals were obtained from commercial suppliers, we recently started producing $CaWO_4$ single crystals with a dedicated Czochralski furnace at the Technische Universität München (TUM) to have a direct influence on the radiopurity and scintillation properties. We present here an overview of the growth process as well as measurements of the crystals' scintillation properties and radiopurity.

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Background radioactivity of construction materials, raw substance and ready-made $CaMoO_4$ crystals

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The results of measurements of natural radioactive element content in different source materials of natural and enriched composition used for $CaMoO_4$ scintillation crystal growing are presented. The crystals are to be used in the experiment to search for neutrinoless double beta decay of ^{100}Mo.

$Zn_xMg_{1-x}WO_4$ –A new crystal scintillator

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Scintillation detectors possess a range of important characteristics for a high sensitivity low background experiments. $ZnWO_4$ crystal is one of the most promising scintillators for such experiments. The radioactive contaminations of the best $ZnWO_4$ samples are estimated to be less than 0.002 mBq/kg (^{228}Th and ^{226}Ra), the total α activity is 0.18 mBq/kg [1]. Magnesium tungstate is of particular interest for such experiments due to the combination of heavy (W) and light (Mg and O) elements [2]. However, $MgWO_4$ crystals are grown from a flux prepared from sodium tungstate, which caused contamination by radioactive impurities.

The aim of this work was to develop continuous series of solid solutions zinc and magnesium tungstate with different ratio of Zn and Mg and to study their crystal structure, optical, luminescence, and scintillation properties.

The set of single crystals with general formula $Zn_xMg_{1-x}WO_4$ and value of x = 0.3, 0.4, 0.5, 0.6, 0.7, 0.8, 0.9, 1 were grown by the Czochralski method. XRD analysis showed that the obtained crystals have wolframite structure. Comparison of the lattice parameters of all samples was carried out. A transmittance of proposed single crystals was similar to $MgWO_4$ and $ZnWO_4$ crystals in the visible spectrum and over in the ultraviolet. The crystals $Zn_xMg_{1-x}WO_4$ were transparent in the range of their own luminescence. A single emission band was observed under X-ray excitation with maximum about 480 nm. The band was due to the emission of exciton, self-trapped on WO_6 complex. An increase of light output was detected in solid solutions $Zn_xMg_{1-x}WO_4$ for intermediate values of x. The light output of $Zn_{0.5}Mg_{0.5}WO_4$ was 145% of the value for $ZnWO_4$ under X-ray irradiation.

The first studies of the new scintillator properties have shown that it is promising for using in modern technology.

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Poster session

Purification of Ce, Nd and Gd for low background experiments

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Cerium, neodymium and gadolinium are potentially double beta (2β) active isotopes among lanthanide elements. The main problem of compounds containing lanthanide elements to use as a 2β decay source is their high radioactive contamination by uranium, radium, actinium and thorium. Even high purity grade (99.99% – 99.995%) lanthanide compounds contain uranium, radium and thorium typically on the level of ~ (0.1 – 1) Bq/kg. The new generation experiments to search for double beta decay of lanthanides require development of methods for a deep purification from radioactive elements.

In this work we use cerium, neodymium and gadolinium oxides as the simplest initial compounds of lanthanides. A combination of physical and chemical methods of purification was applied [1]. Liquid-liquid extraction technique was used to remove traces of Th and U from neodymium, gadolinium and for purification of cerium from Th, U, Ra and K. Co-precipitation and recrystallization methods were utilized for further reduction of the impurities. The radioactive contamination of the samples before and after the purification procedure was tested by using ultra-low-background HPGe gamma spectrometry at the underground Gran Sasso National Laboratories of the INFN (Italy). As a result of the purification procedure the radioactive contamination of gadolinium oxide (a similar purification efficiency was reached also with cerium and neodymium oxides) was decreased from 0.12 Bq/kg to 0.007 Bq/kg in ^{228}Th, from 0.04 Bq/kg to <0.006 Bq/kg in ^{226}Ra, and from 0.9 Bq/kg to 0.04 Bq/kg in ^{40}K. The purification methods are much less efficient for chemically very similar radioactive elements like actinium, lanthanum and lutetium. Further R&D of purification methods and preparation of experiments to search for 2β decay of several lanthanide isotopes are in progress.

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Rejection of randomly coinciding events in $ZnMoO_4$ scintillating bolometers

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Observation of neutrinoless double beta (0ν2β) decay would imply the violation of lepton number conservation and definitely new physics beyond the Standard Model, establishing the Majorana nature of neutrinos. Cryogenic scintillating bolometers look the most promising detectors to search for this extremely rare nuclear process in a few theoretically the most favorable nuclei. However, a serious disadvantage of the low temperature bolometers is a rather poor time resolution, which can lead to a non-negligible background at the energy $Q_{2\beta}$ due to random coincidences of signals, in particular those due to the unavoidable two-neutrino 2β decay events [1]. Counting rate of two randomly coincident 2ν2β events in cryogenic $Zn^{100}MoO_4$ detectors is expected to be on the level of 2.9×10^{-4} counts/(keV×kg×yr) at the $Q_{2\beta}$ energy, meaning that randomly coincident 2ν2β decays can be even a main source of background in a large scale 0ν2β experiment [2].

To develop pulse-shape discrimination of randomly coinciding events, a ten thousand of heat and light pulses (single and coincident pulses with the randomly distributed time difference) with two different time features (the rise-time 7.6 ms and 12.6 ms of the light pulses, and 13.6 ms of the heat pulses), and with a few values of the signal to noise ratios were generated. Pulse shapes and noise data were accumulated with a 0.3 kg $ZnMoO_4$ crystal operating as cryogenic scintillating bolometer in the Centre de Spectrométrie Nucléaire et de Spectrométrie de Masse (CSNSM, Orsay, France) and at the Modane underground laboratory (LSM, France).

We have utilized different approaches to discriminate randomly coinciding events by analyzing: (a) rise-time; by using (b) optimal filter, (c) mean time and (d) the χ^2 criteria methods. The rejection efficiency of pile-up pulses on the level of 85 – 95% was achieved (under a requirement to select 95% of single events), depending on the detector time properties and the signal to noise ratio.

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An experimental study of antireflective coatings in Ge light detectors for scintillating bolometers

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Luminescent bolometers are double-readout devices able to measure simultaneously the phonon and the light yields after a particle interaction in the detector. This operation allows in some cases to tag the type of the interacting quantum, crucial issue for background control in rare event experiments such as the search for neutrinoless double beta decay and for interactions of particle dark matter candidates.

The light detectors used in the LUCIFER and LUMINEU searches (projects aiming at the study of the double beta interesting candidates ^{82}Se and ^{100}Mo through scintillating bolometers of ZnSe and $ZnMoO_4$) consist of hyper-pure Ge thin slabs equipped with NTD thermistors. This structure is convenient and very well tested. However, due to the very low energy of the expected signals, an improvement of the light-detector sensitivity, which is crucial to achieve an adequate background rejection, is mandatory for the most challenging situations (dark matter application and detection of Cerenkov light in TeO_2 bolometers) and in any case welcome for the ZnSe and $ZnMoO_4$ cases.

A substantial sensitivity improvement of the Ge light detectors can be obtained through the use of proper anti-reflective coatings of the Ge side exposed to the luminescent bolometer. The present paper deals with the investigation of this aspect, proving and quantifying the positive effect of a SiO_2 and a SiO coating and setting the experimental bases for future tests of other coating materials.

The main idea is to fabricate thin detectors with a coating on only one side, and to make them as symmetric as possible with respect to the two sides in any other aspect. The same light source is then used to illuminate each light detector, in a first cryogenic run from one side and in a second cryogenic run from the other side. The detector-source geometrical coupling is kept identical in the two runs. The comparison of the light signals in the two runs allows extracting the improvement factor due to coating. In order to get a redundant confirmation of the results and to overcome inevitable small side asymmetries in the detector configuration, several bolometers for each coating configuration have been realized and studied.

The results confirm that an appropriate coating procedure helps in improving the sensitivity of bolometric light detectors and needs to be included in the recipe for the development of an optimized radiopure scintillating bolometer.

Optimization of light collection from crystal scintillators for cryogenic experiments

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Cryogenic scintillation bolometers are promising detectors to search for dark matter and neutrinoless double beta decay, and to investigate rare alpha decays thanks to very high energy resolution, low energy threshold and excellent particle discrimination ability. Increase of light collection and improvement of energy resolution in the light channel of scintillating bolometers are important tasks in such experiments. The purpose of our work was to study dependence of energy resolution and relative pulse amplitude of scintillation detector on crystal shape, shape and material of reflector, conditions of crystal surface, presence of optical contact between scintillator and photo detector. Two $ZnWO_4$ crystal scintillators (cut from one crystal boule) with similar optical properties were used. One crystal has cylindrical shape with sizes $\varnothing$ 20 × 20 mm, the other is a hexagonal prism (height 20 mm, diagonal 20 mm). In measurements with the crystal surrounded by a 3M reflector and without optical contact (the conditions of light collection closest matching normal operation of cryogenic scintillating bolometers) the best energy resolution and highest relative pulse amplitude was obtained for the hexagonal scintillator with diffuse surface. The light collection for the different conditions was simulated with a Monte Carlo method, using ZEMAX [1] and GEANT4 [2]. Results of the simulation confirm that the hexagonal shape and diffuse surface of a crystal improve the light collection ≈1.4 times (in comparison to a polished cylinder). As a next step we are going to carry out measurements and simulate light collection with $CaWO_4$ crystal scintillators of different shape with size of 40 mm (diameter, diagonal of prism) × 40 mm.

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Radioactive contamination of BaF_2 crystal scintillators

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Barium fluoride crystal scintillators (BaF_2) are promising detectors to search for double beta (2β) decay processes in ^{130}Ba ($Q_{2\beta}$ = 2619(3) keV) and ^{132}Ba ($Q_{2\beta}$ = 844(1) keV) [1, 2]. The ^{130}Ba isotope is of particular interest due to the reports on the observation of 2β decay in geochemical experiments [3, 4]. The radioactive contamination of BaF_2 scintillation crystal with the mass of 1.7 kg was measured over ~110 hours in a low-background set-up DAMA/R&D deep underground (3600 m w.e.) at the Gran Sasso National Laboratories INFN (LNGS, Italy). Also scintillation properties of BaF_2 detector were investigated (energy and time resolution); the methods to separate signals from α-particles, γ(β) and Bi-Po events by shape of the signal were developed. To carry out more sensitive experiments, one should develop crystal scintillators, enriched with ^{130}Ba, with low level of radioactive contamination. While radioactive contamination of materials by uranium and thorium can be the main source of background in the experiment, R&D of methods to purify barium chloride were performed at the Gran Sasso National Laboratories.

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Characterization of $Li_6RE(BO_3)_3:Ce^{3+}$ (RE = Lu, Y) neutron detection crystals

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The transparent $Li_6RE_{1-x}Ce_x(BO_3)_3$ (RE = Lu, Y, x = 0.03) crystals were grown with Czochralski method. Their densities are 3.54 g/cm^3 and 3.09 g/cm^3, the thicknesses of them are 1.4 mm and 0.78 mm. In order to study the luminescence properties of the scintillators, they were irradiated with gamma rays, alpha rays and thermal neutrons, which were from ^{241}Am, ^{239}Pu, ^{252}Cf, respectively. ^{241}Am source was wrapped in tyvek to shield its alpha rays. Since the samples are thin, the low energy gamma source is chosen to ensure the gamma rays energy can deposit in it. ^{239}Pu is used as alpha source. ^{252}Cf is surrounded by polyethylene and 5 cm Pb to give thermal neutron and shield gamma ray, these conditions were used to measure the properties of these scintillators for thermal neutron. The neutron shielding spectrum is also measured under the similar situation, but with 3 mm cadmium board placed before 5 cm Pb, it is calculated that 1 mm cadmium board can absorb thermal neutrons totally. Then, compared with the neutron shielding spectrum, the position of neutron peak can be evaluated from the pulse height distribution.

The study indicates that the transparent scintillator $Li_6Y(BO_3)_3$ shows light yield of 245% for thermal neutron irradiation, which is about twice that of $Li_6Lu(BO_3)_3$. Meanwhile both of them have short decay times, which are both less than 100 ns. The neutron detection efficiency is expected to be increased by replacing ^{nat}B, ^{nat}Li with ^{10}B, ^{6}Li and making the scintillator even thicker. If better gamma ray rejection is attainable, thinner crystal detectors should be demanded to decrease the deposit energy from gamma. These scintillators are promising candidates for detectors in neutron counter domain and the neutron imaging field.

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Characterization of a ZnSe scintillating bolometer prototype for neutrinoless double beta decay search

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As proposed in the LUCIFER project, ZnSe crystals are attractive materials to realize scintillating bolometers aiming at the search for neutrinoless double beta decay of the promising isotope ^{82}Se. However, the optimization of the ZnSe-based detectors is rather complex and requires a wide-range investigation of the crystal features: optical properties, crystalline quality, scintillation yields and bolometric behaviour. Samples tested up to now show problems in the reproducibility of crucial aspects of the detector performance. In this work, we present the results obtained with a scintillating bolometer operated aboveground at ~ 25 mK. The detector energy absorber was a single 1 cm^3 ZnSe crystal. The good energy resolution of the heat channel (~ 14 keV at 1460 keV) and the excellent alpha/beta discrimination capability are very encouraging for a successful realization of the LUCIFER program. The bolometric measurements were completed by optical tests on the crystal (optical transmission and luminescence measurements down to 10 K) and investigation of the crystalline structure. The work here described provides a set of parameters and procedures useful for a complete pre-characterization of ZnSe crystals in view of the realization of highly performing scintillating bolometers.

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