

# Molybdenum containing scintillating bolometers for double-beta decay search (LUMINEU program)

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## Abstract

A scintillating bolometer technology, promising to be used in a next-generation cryogenic experiment to search for neutrinoless double-beta decay, is currently under development within the LUMINEU (Luminescent Underground Molybdenum Investigation for NEUtrino mass and nature) program. The recent results about the R&D of high quality large volume molybdenum containing crystal scintillators (zinc and lithium molybdates), including ones produced from  $^{100}\text{Mo}$ -enriched powder, and aboveground / underground tests of cryogenic detectors based on these crystals are presented here.

Keywords: Double-beta decay, Scintillating bolometer,  $\text{ZnMoO}_4$ ,  $\text{Li}_2\text{MoO}_4$ , Low background experiment, Radiopurity

## 1 Introduction

The recent discovery of neutrino oscillations (the Nobel prize award in physics 2015), phenomena which demonstrate that neutrinos have mass [1], stirs up an additional great interest in

the searches for neutrinoless double-beta ( $0\nu 2\beta$ ) decay, a nuclear transformation of the type  $(A, Z) \rightarrow (A, Z + 2) + 2e^-$ . This never observed process is beyond the Standard Model (SM) and definitely requires finite value of neutrino mass, equivalence between neutrino and anti-neutrino (Majorana nature of neutrinos), and violation of the total lepton number by two units (see details in the recent review [2] and references herein).

The LUMINEU (Luminescent Underground Molybdenum Investigation for NEUtrino mass and nature) program [3, 4] is going to bridge the gap for high-sensitivity next-generation  $0\nu 2\beta$  studies by the development of a technology based on Mo-containing scintillating bolometers, heat-light double read-out cryogenic calorimeters, and the demonstration of its feasibility by the successful realization of a small-scale experiment to search for  $0\nu 2\beta$  decay of  $^{100}\text{Mo}$  with zero background in the range of interest (around  $Q_{2\beta} = 3034$  keV, the total energy of the two electrons emitted in  $0\nu 2\beta$  decay of  $^{100}\text{Mo}$ ). It will play an important role in the selection for the technology to be adopted for a tonne-scale cryogenic  $0\nu 2\beta$  experiment within the CUPID (CUORE Upgrade with Particle IDentification) project [5]. The main R&D results in the framework of the LUMINEU program are briefly presented here.

## 2 R&D of scintillating bolometers within LUMINEU

### 2.1 Tasks of the LUMINEU program

LUMINEU is performing an extensive R&D of Mo-containing scintillating bolometers which involves several general tasks related with: I) crystal scintillators; II) light detectors; III) temperature sensors; IV) prototypes of scintillating bolometers; V) final detectors for a pilot  $0\nu 2\beta$  experiment. Below we describe briefly the program and the main requirements of these tasks.

A high detection efficiency for  $0\nu 2\beta$  decay can be obtained in an experiment with the so-called “active-source” technique for which a  $0\nu 2\beta$  source (e.g.  $^{100}\text{Mo}$ ) is embedded into a detector. The baseline detector material for LUMINEU is a zinc molybdate ( $\text{ZnMoO}_4$ ) scintillator, however another promising Mo-based material, lithium molybdate ( $\text{Li}_2\text{MoO}_4$ ), is also under consideration. Therefore, task I is devoted to the development of purification and crystallization procedures for producing large volume, high optical quality, radiopure scintillators both from natural and  $^{100}\text{Mo}$ -enriched molybdenum. In view of the high cost of enriched  $^{100}\text{Mo}$  and the prospect to grow  $\sim 1000$  crystals (for a large-scale project), one needs to optimize a production line in order to get high throughput and low irrecoverable losses of the enriched material. Specifying these requirements, the main objectives are scintillators with a mass of  $\sim 1$  kg (scintillation elements up to 0.4 kg) produced with more than 70% crystal yield, a few % of irrecoverable losses, and with a level of internal radioactive contamination by  $^{228}\text{Th}$  and  $^{226}\text{Ra} \leq 0.01$  mBq/kg (the total alpha activity of radionuclides from U/Th, except that of  $^{210}\text{Po}$ , is below 1 mBq/kg).

The main background source above 2615 keV in a bolometric  $0\nu 2\beta$  experiment is caused by  $\alpha$  decays of natural radioactivity. Usually, a scintillator produces less light for  $\alpha$  particles with respect to  $\gamma(\beta)$ ’s of the same energy (this phenomenon is known as quenching). Therefore, by using a light detector complementary to a bolometer which scintillates at low temperature, one can efficiently discriminate an  $\alpha$ -induced background. It is convenient to make this light detector as a thin bolometer and mount it close to a scintillator-based bolometer, which all

together constitute a scintillating bolometer. Therefore, task II includes the fabrication of optical bolometers from high purity Ge wafers, widely used for this purpose. An important part of the activity within this task is dedicated to the optimization of light detectors with the aim to get an acceptable performance. Since both  $ZnMoO_4$  and  $Li_2MoO_4$  scintillators are characterized by very modest light yield (of the order of  $\sim 1$  keV per 1 MeV deposited energy), efficient light collection and absorption are important parameters. The energy resolution (FWHM) of the photodetector should be good enough to provide a 99.9% discrimination between light signals caused by  $\sim 3$  MeV  $\gamma(\beta)$  and  $\alpha$  particles impinging in the Mo-based scintillator. In particular, an  $\alpha/\gamma$  separation at the level of  $5\sigma$  at  $\sim 3$  MeV can be achieved with a light detector characterized by FWHM  $\sim 15\%$  at 5.9 keV X-ray of  $^{55}Fe$  (typically used for calibration of these devices) [6]. Finally, random coincidences of two neutrino double-beta decay of  $^{100}Mo$ , allowed in the SM process and registered with a half-life  $\sim 10^{18}$  yr [1], can constitute a major background for a bolometric  $0\nu2\beta$  experiment with Mo-containing scintillators, as it was pointed out for the first time in Ref. [7]. Therefore a light detector should have a fast response (e.g. a rise time  $\sim 1$  ms or less) and an as high as possible signal-to-noise ratio to provide an efficient discrimination of random coincidences.

A small temperature rise appeared after particle interaction inside a bolometer can be measured by a dedicated thermometer. Within task III, LUMINEU is developing three technologies of temperature sensors: Neutron Transmutation Doped (NTD) Ge thermistors [8], superconducting Transition-Edge Sensors (TES) [9], and Magnetic Metallic Calorimeters (MMC) [10]. The first two types exploit the dependence of resistivity on temperature, while the last one that of magnetization on temperature. All results given below were obtained with scintillating bolometers instrumented with NTD Ge-based thermometers.

Task IV involves the construction of small / middle size prototypes of scintillating bolometers and their low temperature test, below tens mK, both at aboveground (at CSNSM) and underground (at Modane in France and Gran Sasso in Italy) cryogenic facilities. The crucial point of this activity is achieving excellent performance of Mo-containing scintillating bolometers in terms of energy resolution (FWHM  $\leq 10$  keV at 3 MeV) and particle identification ( $\alpha/\gamma$  separation more than 99.9%), as well as proving low bulk radioactive contamination of crystals.

Task V requires the fabrication of at least two complete single modules based on massive  $ZnMoO_4$  crystals (with mass up to 0.4 kg; one produced from natural molybdenum and another one enriched in  $^{100}Mo$ ), and a realization of a pilot experiment with the aim to demonstrate the abovementioned key performance and radiopurity, which are associated with a zero-background  $0\nu2\beta$  experiment. Current status of the LUMINEU tasks are briefly summarized below (task III will not be highlighted here in more detail than it was done above).

## 2.2 R&D and preformance of Mo-containing scintillating bolometers

A technology of fabrication of large area light detectors (with diameter up to 50 mm and thickness around 250  $\mu m$ ) from high purity germanium wafers (Umicore, Belgium) was developed at CSNSM (Orsay, France) [6]. The adopted LUMINEU standard is a  $\odot 44$ -mm Ge slab coated by  $SiO_2$  to improve light absorption [11]. In addition to the batch of the LUMINEU NTD Ge-based light detectors, photodetectors produced within the LUCIFER  $0\nu2\beta$  project [12], as well as state-of-the-art extra thin ( $\sim 30$ – $50$   $\mu m$ ) germanium optical bolometers developed at IAS (Orsay, France) [13] were used to build and test LUMINEU scintillating bolometers. First test

of MMC-based light detectors developed within LUMINEU [14] demonstrate their potential to get  $\sim 100$  times faster response than that typical for NTD Ge-based photodetectors.

A dedicated protocol for molybdenum purification (double sublimation of molybdenum oxide in vacuum and double recrystallization of ammonium molybdate from aqueous solutions) has been adopted by LUMINEU to get high purity initial compound for crystal growth [4]. The chosen advanced directional solidification method developed at NIIC (Novosibirsk, Russia), low-thermal-gradient Czochralski (LTG Cz) technique, demonstrates the possibility to grow large (up to 1.5 kg)  $\text{ZnMoO}_4$  crystals with a high crystal yield ( $\sim 80\%$  of charge) [4]. The aboveground low temperature test of first LUMINEU crystals (scintillation elements with size of  $\varnothing 20 \times 40$  mm and  $\varnothing 35 \times 40$  mm and masses of 55 g and 160 g respectively), produced from  $\text{ZnMoO}_4$  boules grown from deeply purified materials, shows excellent signal-to-noise ratio, high signal amplitude, expected values of light-to-heat ratio and light quenching factor for  $\alpha$  particles [4]. Only internal  $^{210}\text{Po}$  contaminant was clearly observed over about two weeks of data taking which indicates high crystals' radiopurity and effectiveness of the purification procedure.

Advances in large volume  $\text{ZnMoO}_4$  growth have been achieved by applying double crystallization. In particular, by using this technique an improved quality  $\sim 1.0$  kg boule was grown from molybdenum purified by double recrystallization [15]. Two  $\text{ZnMoO}_4$  optical elements produced with a size expected for a pilot LUMINEU  $0\nu 2\beta$  experiment ( $\varnothing 50 \times 40$  mm; masses 336 and 334 g) were used to construct identical scintillating bolometers [16] according to a new special design compatible with the EDELWEISS set-up at the Modane underground laboratory (LSM, France). Results of long term (about 3000 h) low background measurements with these devices [15, 17] demonstrate an excellent energy resolution (e.g. FWHM  $\sim 9$  keV at 2615 keV) and an efficient  $\alpha/\gamma$  separation ( $15\sigma$  above 2.5 MeV) achieved with  $\text{ZnMoO}_4$ -based scintillating bolometers, as well as very high internal radiopurity of  $\text{ZnMoO}_4$  crystals (e.g. activity of  $^{228}\text{Th}$  and  $^{226}\text{Ra} \leq 0.004$  mBq/kg), which completely satisfy the LUMINEU requirements and those of a future large-scale experiment. An illustration of the main performance of a 334-g  $\text{ZnMoO}_4$  detector is shown in Fig. 1. Taking into account that the additional crystallization improves quality of a crystal boule and could also enhance bulk radiopurity thanks to segregation of radionuclides during growing process, double crystallization can be used for the production of large LUMINEU crystals.

A first  $^{100}\text{Mo}$ -enriched  $\text{Zn}^{100}\text{MoO}_4$  crystal with a mass of  $\sim 0.17$  kg has been successfully developed with 84% crystal yield and  $\sim 4\%$  irrecoverable losses of the enriched material [18]. The used  $^{100}\text{MoO}_3$  (99.5% enrichment in  $^{100}\text{Mo}$ ) was purified by sublimation in vacuum and double recrystallization from aqueous solutions. Molybdenum purification is related with the major part of losses (85%) and the minor part with the crystal growth. The  $\text{Zn}^{100}\text{MoO}_4$  boule had a non-regular shape and yellow coloration unlike similar size natural  $\text{ZnMoO}_4$  crystals. It is caused by still existing difficulties during the solidification, in particular by the effect of a second phase formation. A  $\text{Zn}^{100}\text{MoO}_4$  scintillating bolometer array, built from two produced small  $\text{Zn}^{100}\text{MoO}_4$  elements (59 g and 63 g) and tested aboveground, shows bolometric properties similar to non-enriched  $\text{ZnMoO}_4$  detectors [18]. The energy resolution of both bolometers was reasonably good taking into account a high counting rate due to the background conditions of the sea-level laboratory. In particular, 5 and 10 keV FWHM at 609 keV  $\gamma$ 's of  $^{214}\text{Bi}$  from environmental radioactivity was obtained with the 59-g and 63-g  $\text{Zn}^{100}\text{MoO}_4$  bolometers respectively [18]. After 42 h of data taking only a hint associated with  $^{210}\text{Po}$  was observed in the  $\alpha$  region, which implies encouraging radiopurity of the crystals [18]. During an underground

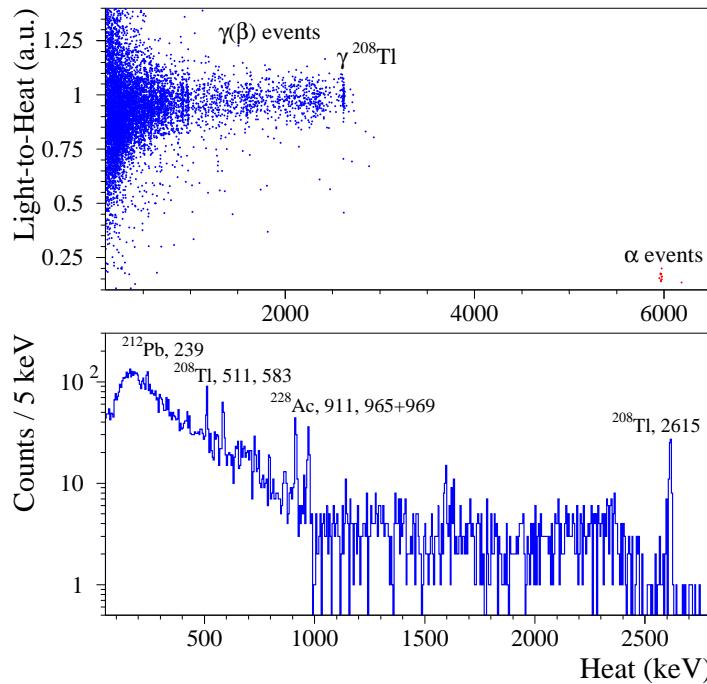


Figure 1: (Top) A 2-D histogram showing light-to-heat ratio versus heat for the data of the 14-h  $^{232}\text{Th}$  calibration measurements with the 334-g  $\text{ZnMoO}_4$ -based scintillating bolometer operated in the EDELWEISS set-up at LSM. A high potential of full separation between  $\alpha$  events and  $\gamma(\beta)$  band (red and blue dots respectively) is clearly evident. The region, where events caused by decays of  $\alpha$  radionuclides from U/Th (above 4 MeV) are expected, is mainly populated by  $^{210}\text{Po}$ , which demonstrates encouraging internal radiopurity. (Bottom) 1-D projection of the 2-D histogram shown at the figure top. Energies of  $\gamma$  quanta are given in keV.

test at LSM, the  $\text{Zn}^{100}\text{MoO}_4$  array which had a non-standard holder was strongly affected by a microphonic noise deteriorating the detector's energy resolution. It is caused by the absence of a suspension system in the EDELWEISS set-up (to be implemented soon), which can reduce effect of microphonics, in contrast to the cryostat [19] used for the aboveground measurements. Anyway, a full particle identification by  $\text{Zn}^{100}\text{MoO}_4$  detectors was clearly demonstrated in the underground test.

The  $\text{ZnMoO}_4$  solidification procedure was further improved by introducing a small amount of tungsten oxide (up to  $\sim 0.5$  wt%) to stabilize the melt and several high quality  $\sim 0.15$ -kg crystal boules were grown. No effect of W-doping on bolometric properties of  $\text{ZnMoO}_4$  was evidenced by the aboveground measurements with a scintillating bolometer array constructed from stoichiometric and W-doped  $\text{ZnMoO}_4$  crystals ( $\varnothing 20 \times 40$  mm each) [20].

Recently, a first large volume colorless  $\text{Zn}^{100}\text{MoO}_4$  crystal ( $\sim 1.4$  kg) has been developed [17, 21]. Two large scintillation elements were produced with a shape of hexagonal prism (with diagonal 60 mm and height 40 mm, masses 379 g and 382 g) in order to have an improved light output according to studies [22]. Massive  $\text{Zn}^{100}\text{MoO}_4$ -based scintillating bolometers have been tested in the CUORE R&D test cryostat at Gran Sasso National Laboratories (LNGS, Italy) and the data analysis is in progress. A new test in the EDELWEISS set-up at Modane is foreseen. Meanwhile, background conditions of a middle-scale  $0\nu 2\beta$  experiment with 48

$\text{Zn}^{100}\text{MoO}_4$  scintillating bolometers ( $\varnothing 60 \times 40$  mm, 495 g each) installed in the EDELWEISS set-up have been simulated with GEANT4-based code [21]. Considering the already achieved performance, radiopurity, and pulse-shape discrimination of random coincidences developed by LUMINEU [23], a background counting rate  $\approx 5 \times 10^{-4}$  counts/yr/kg/keV at  $Q_{2\beta}$  of  $^{100}\text{Mo}$ , corresponding to zero-background conditions, can be reached [21].

In spite of the achieved progress in the development of large  $\text{Zn}^{100}\text{MoO}_4$ , growing a regular cylindrical shape crystal boule with similar quality along its length is still a difficult task (e.g. see Fig. 1 in [17]). This problem can be resolved, without further R&D, just by pulling a short crystal boule. Another solution could be related with a choice of another Mo-based material,  $\text{Li}_2\text{MoO}_4$ , which is characterized by comparatively easy crystal growth process and has bolometric properties similar to  $\text{ZnMoO}_4$ . In particular, several perfect quality  $\text{Li}_2\text{MoO}_4$  crystal boules with masses 0.1–0.4 kg were grown by LTG Cz method from deeply purified Mo and commercial  $\text{Li}_2\text{CO}_3$  (99.99% purity grade) [24]. A first large  $\text{Li}_2\text{MoO}_4$ -based ( $\varnothing 40 \times 40$  mm, 151 g) scintillating bolometer was tested aboveground with quite encouraging results [24], which were reinforced by subsequent  $\sim 350$ -h underground measurements at LNGS [25]. In particular, an excellent spectrometric properties (FWHM  $\sim 4$  keV at 2615 keV), an efficient particle identification and high radiopurity for U/Th nuclides ( $^{228}\text{Th}$  and  $^{226}\text{Ra} \leq 0.02$  mBq/kg) were obtained [25]. There is only an issue caused by considerably high activity of  $^{40}\text{K}$  inside the tested crystal ( $\sim 60$  mBq/kg), which can be a problem due to random coincidences in a future  $0\nu 2\beta$  experiment. Fortunately, this issue can be easily resolved by selection of a Li-containing raw material with low concentration of  $^{40}\text{K}$  and by recrystallization of  $\text{Li}_2\text{MoO}_4$  crystals. It is worth to note that two scintillating bolometers based on large  $\text{Li}_2\text{MoO}_4$  crystals ( $\varnothing 50 \times 40$  mm, 242 g each) produced from different Li-based powder with low  $^{40}\text{K}$  content (tested by HPGe  $\gamma$  spectroscopy) are under investigation in order to prove low bulk activity of  $^{40}\text{K}$ . A Li-powder with the lowest  $^{40}\text{K}$  content will be used to produce a first  $\text{Li}_2^{100}\text{MoO}_4$  crystal. A dedicated low temperature test will be performed in order to completely demonstrate that  $\text{Li}_2\text{MoO}_4$ -based scintillating bolometer is a viable detector for a large-scale cryogenic  $0\nu 2\beta$  experiment.

### 3 Conclusions

A protocol for producing high quality large volume Mo-containing crystal scintillators from deeply purified molybdenum with both natural isotopic composition and enriched in  $^{100}\text{Mo}$  has been developed within the LUMINEU program. A technology for the development of high performance LUMINEU single modules based on radiopure large  $\text{ZnMoO}_4$  and  $\text{Li}_2\text{MoO}_4$  crystals is well established providing high energy resolution (better than 10 keV FWHM at 2615 keV), efficient  $\alpha/\gamma$  separation (more than  $5\sigma$ ) and required low level of bulk radioactivity (e.g.  $^{228}\text{Th}$  and  $^{226}\text{Ra} \sim 0.01$  mBq/kg). A first large  $\text{Zn}^{100}\text{MoO}_4$  crystal ( $\sim 1.4$  kg) was successfully grown and two 0.4-kg  $\text{Zn}^{100}\text{MoO}_4$ -based scintillating bolometers have been preliminary tested at LNGS (Italy). The development of a first large  $\text{Li}_2^{100}\text{MoO}_4$  crystal scintillator is in progress and a pilot LUMINEU test of massive  $\text{Zn}^{100}\text{MoO}_4$ - and  $\text{Li}_2^{100}\text{MoO}_4$ -based scintillating bolometers in the EDELWEISS set-up at LSM (France) is foreseen at the beginning of 2016. After completing this test we will make a final choice about the Mo-containing crystals to be produced from  $\sim 10$  kg of  $^{100}\text{Mo}$  (enriched isotope is already available) for a middle-scale  $0\nu 2\beta$  experiment (LUCINEU project) based on the LUMINEU technology. A Monte Carlo simulation of 48 0.5-kg  $\text{Zn}^{100}\text{MoO}_4$ -based scintillating bolometers installed in the EDELWEISS set-up demonstrates

the possibility to get a satisfactory low background counting rate in the range of interest (at  $\sim 3$  MeV). The LUMINEU activity is now part of the CUPID project, a proposed bolometric tonne-scale  $0\nu 2\beta$  experiment to be built as a follow-up to CUORE and exploiting as much as possible the CUORE infrastructures.

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