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M. Mancuso, D.M. Chernyak, F.A. Danevich, L. Dumoulin, A. Giachero, A. Giuliani, Henri Godfrin, C. Gotti, I.M. Ivanov, M. Maino, et al.

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M. Mancuso<sup>1,2</sup> · D. M. Chernyak<sup>1,3</sup> · F. A. Danevich<sup>3</sup> · L. Dumoulin<sup>1</sup> · A. Giachero<sup>4,5</sup> · A. Giuliani<sup>1,2,5</sup> · H. Godfrin<sup>6</sup> · C. Gotti<sup>4,5</sup> · I.M. Ivanov<sup>7</sup> · M. Maino<sup>4,5</sup> · E.P. Makarov<sup>7</sup> · E. Olivieri<sup>1</sup> · G. Pessina<sup>4,5</sup> · V.N. Shlegel<sup>7</sup> · A. Sultan<sup>6</sup> · M. Tenconi<sup>1</sup> · Ya.V. Vasiliev<sup>7</sup>

# An aboveground pulse-tube-based bolometric test facility for the validation of the LUMINEU ZnMoO<sub>4</sub> crystals

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**Abstract** The LUMINEU project aims at developing a pilot double  $\beta$  decay experiment using scintillating bolometers based on ZnMoO<sub>4</sub> crystals enriched in <sup>100</sup>Mo. In the next months regular deliveries of large-mass ZnMoO<sub>4</sub> crystals are expected from the Nikolaev Institute of Inorganic Chemistry (Novosibirsk, Russia). It is therefore crucial for the LUMINEU program to test systematically and in real time these samples in terms of bolometric properties, light yield and internal radioactive contamination. In this paper we describe an aboveground cryogenic facility based on a dilution refrigerator coupled to a pulse-tube cooler capable performing these measurements. A 23.8 g ZnMoO<sub>4</sub> crystal was fully characterised in this setup. We show also that macro-bolometers can be operated with high signal-to-noise ratio in liquid-free dilution refrigerators.

**Keywords** Double beta decay, Neutrino mass, Low background, Bolometric technique, ZnMoO<sub>4</sub> crystals, Scintillation.

## 1 Introduction

Neutrinoless double  $\beta$  decay<sup>1,2</sup> ( $0\nu\beta\beta$ ) is a hypothetical rare nucleus transition, where two neutrons in an even-even nucleus decay into two protons simultaneously emitting two electrons and no neutrinos. The observation of  $0\nu\beta\beta$  would imply

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1: CSNSM/IN2P3/CNRS and Université Paris XI, Bâtiment 108, 91405 Orsay Campus, France

2: Università dell'Insubria, 22100 Como, Italy

3: Institute for Nuclear Research, MSP 03680 Kyiv, Ukraine

4: Università di Milano-Bicocca, I-20126 Milano, Italy

5: Sezione dell'INFN di Milano Bicocca, I-20126 Milano, Italy

6: Institut Néel CNRS/UJF, 38042 Grenoble, France

7: Nikolaev Institute of Inorganic Chemistry, 630090 Novosibirsk, Russia

E-mail: michele.mancuso@csnsm.in2p3.fr

the violation of lepton number and the Majorana nature of the neutrino: definitely new physics beyond the standard model. From an experimental point of view the  $0\nu\beta\beta$  signature is represented by a peak at the Q-value of the nuclear transition in the energy spectra of the two emitted electrons.

The LUMINEU program envisages the development and study of large  $\text{ZnMoO}_4$  scintillating crystals, containing the excellent  $0\nu\beta\beta$  candidate  $^{100}\text{Mo}$ , characterised by a Q-value at 3.034 MeV. The crystals will be operated as scintillating bolometers. The position of the signal, which is expected outside the bulk of the natural  $\gamma$  background, and the elimination of the  $\alpha$  background thanks to the simultaneous read-out of heat and light, promises to achieve almost zero background at the ton $\times$ year scale, allowing the exploration of the inverted hierarchy region of the neutrino mass pattern in a next-generation search<sup>3</sup>. LUMINEU will develop a prototype of this approach, consisting of an underground pilot experiment containing about 1 kg of enriched molybdenum.

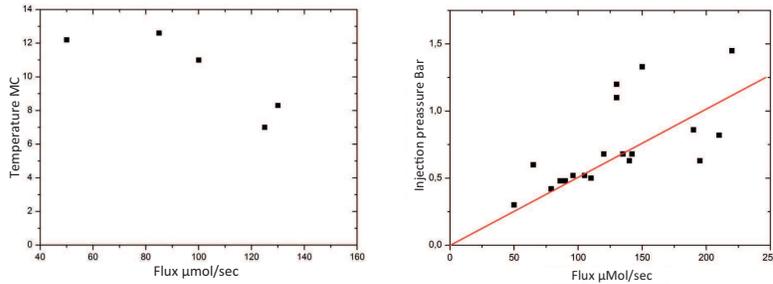
The LUMINEU  $\text{ZnMoO}_4$  crystals will be grown in the Nikolaev Institute of Inorganic Chemistry (Novosibirsk, Russia) using the low-thermal gradient Czochralski method<sup>4</sup>, which provides excellent crystal quality, extreme purity and negligible waste of the starting material. A mass of 0.4 kg for the single module is foreseen. In the next months, the raw material purification and the growth parameters will be adjusted in order to achieve samples with the desired properties. The crystal synthesis will be accompanied by a systematic test of the samples produced, in order to characterize them as scintillating bolometers in terms of bolometric response, light yield,  $\alpha$ -particle quenching factor, radiopurity (internal  $\alpha$  counting) and  $\alpha$  rejection factor. A dedicated facility, subject of the present paper, was developed in CSNSM (Orsay, France).

## 2 Setup for bolometer characterization

The CSNSM facility consists of a dry dilution refrigerator with an experimental space sufficient to house several large mass bolometers. The read out of the bolometer consists of a custom front-end electronics based on six low-noise voltage amplifiers and on a data acquisition system developed with commercial elements. The data is analysed with a dedicated software, optimised for bolometric signals.

Bolometers based on crystals with masses of tens or hundreds of grams need to be cooled to temperatures below 20 mK. This requires the use of dilution refrigerators (DR). The DR at the CSNSM facility consists of a classical dilution unit incorporating sintered silver heat exchangers and a large pumping system (40 m<sup>3</sup>/h rotary pump and 400 l/s turbo pump in He). It can achieve reasonably high <sup>3</sup>He flow rates, thus providing the cooling power needed by massive double- $\beta$ -decay detectors. In order to avoid the need for a constant supply of cryogenic fluids (typically  $\sim 15$  l of liquid helium daily), the dilution unit was coupled to a pulse-tube refrigerator, consisting of the commercial unit PT405 manufactured by Cryomech<sup>5</sup>. This model provides a cooling power of 25 W at 55 K at its first stage, and 0.3 W at 3.5 K at its second stage. Unlike other machines, characterised by high vibration levels, pulse tubes are rather quiet devices. However, since in macro-bolometers vibrations are very harmful for the detector sensitivity, their operation in a pulse-tube DR is not obvious.

The cryostat at the CSNSM facility is described in more detail elsewhere<sup>6</sup> with the acronym PT-DR1. With respect to the original PT-DR1 design, the number of the sintered silver heat exchangers was reduced from four to two, without affecting the base temperature (see Figure 1–left): the DR presently runs at 7 mK with a flow rate of 130  $\mu\text{mol/s}$ . In order to benefit from the cooling power available at the level of the regenerator, two copper heat exchangers were installed on the stainless steel tube connecting the two main stages of the pulse-tube cryocooler<sup>7</sup>, aiming at improving the effectiveness of the mixture condensation process. The result of this modification was spectacular: the mixture condensation time was reduced from  $\sim 20$  to only  $\sim 3$  hours.



**Fig. 1** Performance of the dilution refrigerator of the Orsay facility. Left panel: base temperature at different flow rates measured with CNM. Right panel: mixture injection pressure as a function of the flow rate with turbo pump on and  $^3\text{He}$  compressor off.

An interesting feature of the PT-DR1, particularly relevant in the present context, is that it is well optimized from the thermodynamical point of view. This enables to stop the  $^3\text{He}$  compressor, a major source of vibrations, during the normal operation, keeping a low injection pressure (of the order of 0.5 bar at a circulation rate of 130  $\mu\text{mol/s}$ ). The cooling power in this condition is of  $\sim 100 \mu\text{W}$  at 100 mK and  $\sim 0.5 \mu\text{W}$  at 7 mK. The experimental volume available for the bolometers corresponds to a cylindrical space with a diameter of 30 cm and a height of 20 cm. This allows to easily house arrays of four  $\text{ZnMoO}_4$  scintillating bolometers with a mass of 400 g each, target of the LUMINEU single module configuration.

In order to read out the bolometers, the refrigerator was equipped by a woven ribbon cable, manufactured by Tekdata<sup>8</sup>, carrying 12 twisted pairs of manganine leads in a polyamide resin. This arrangement enables the simultaneous operation of six scintillating bolometers with one heat and one light channel each.

The front-end electronics coupled to the scintillating bolometers consists of an array of six differential DC-coupled voltage-sensitive preamplifiers (followed by Bessel low-pass filters) designed to operate at room temperature with low series and parallel noise<sup>9</sup>. The differential configuration allows microphonism of the connecting wires and cross-talks of adjacent channels to be minimized. The series noise of the preamplifier is about 30  $\text{nV}/\sqrt{\text{Hz}}$  at 0.1 Hz and 7  $\text{nV}/\sqrt{\text{Hz}}$  at 1 Hz. The load resistors for the bolometer DC bias are also operated at room tem-

perature. To minimize the thermal noise contribution, their value can be chosen as  $2\text{ G}\Omega$  or  $60\text{ G}\Omega$ , depending on the bolometer resistance. Each readout/bias channel can be programmed individually in terms of gain, bessel-filter cut-off frequency, bias level and load resistance value remotely. The data acquisition is performed by a commercial National Instrument board (NI USB-6218)<sup>10</sup>, with 16 input channels, 16 bits resolution and 250 kHz sampling rate. The data is acquired in streaming and trigger is performed off line. The data analysis software is based on the optimal filter technique<sup>11</sup>. Besides extracting an optimised value for the pulse amplitude, related to the energy of the signal, the analysis program enables the correction of bolometric response drifts and the performance of a variety of pulse-shape selection.

### 3 Experimental Results

The CSNSM facility was tested with a  $\text{ZnMoO}_4$  crystal of mass 23.8 g and cylindrical shape ( $\varnothing 16 \times 28$  mm). This crystal was obtained by a crystalline boule of excellent optical quality synthesised at the NIIC. Details on the crystals properties can be found in Ref.<sup>12</sup>. The sample is inserted in a cylindrical copper holder, acting as a heat sink for the detector. The mechanical coupling to the holder is supplied by six PTFE elements. The two bases of the  $\text{ZnMoO}_4$  crystal are faced by two light-detecting square ultrapure Ge crystalline slabs with 15 mm side and 0.3 mm thickness. The light detectors are kept in position by two PTFE pieces, clamping the slabs in proximity of the two opposite edges. The light detector structure is similar to that described in details elsewhere<sup>13</sup>.

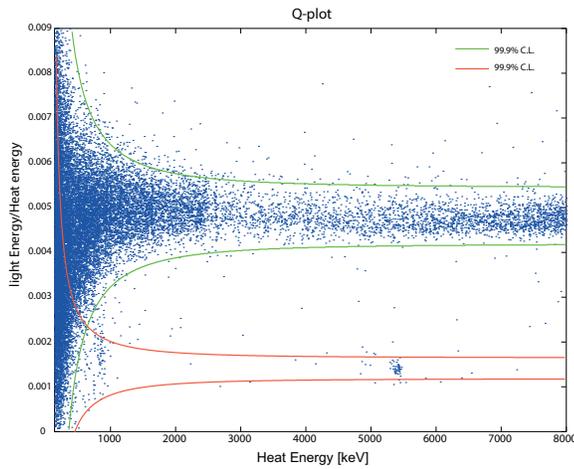
Heat signals from the  $\text{ZnMoO}_4$  crystal and from the Ge slabs were measured with NTD germanium thermistors thermally glued to the energy absorbers, chosen so as to have resistances of the order of a few  $\text{M}\Omega$  and a logarithmic sensitivity  $-d \log R / d \log T \sim 6$  in the 15 – 25 mK range. The thermistor mass is of the order of 20 mg.

The optimum operation point of the scintillating bolometer was chosen by injecting a fixed amount of energy in the  $\text{ZnMoO}_4$  crystal by means of a heater and of a pulser, and by registering the signal amplitude of the consequent thermal signal at several bias levels. At the selected point the thermistor resistance was of  $2.88\text{ M}\Omega$ , corresponding to a temperature of 17.3 mK. A similar procedure was adopted for the light detectors. The time structure of the  $\text{ZnMoO}_4$  thermal pulses is characterised by pulse rise-times (10%-90%) and decay-times (90%-30%) of 9 ms and 37 ms respectively. The light detectors, due to their smaller heat capacity, are faster, with rise and decay-times of the order of 2.5 ms and 14 ms respectively. The pulse amplitude at the operation point of the  $\text{ZnMoO}_4$  bolometer is  $81\ \mu\text{V}/\text{MeV}$ , compatible with the size and the operation temperature of the detector. The baseline width of the heat channel is  $\sim 4\text{ keV FWHM}$ , a factor 4 worse than that obtained with the same detector in a traditional helium bath cryostat<sup>12</sup>. This is mainly due to the fact that the overall noise of the setup, which is  $\sim 1.33\ \mu\text{V rms}$  for the heat channel in the signal bandwidth, is not optimized. The excess noise however is not related to pulse-tube effects, at least for the heat channel, but rather to electromagnetic interferences and a non-optimized grounding pattern. A large improvement is therefore expected in the next runs. The light channels

are much more sensitive to pulse-tube operation. WA mechanical decoupling system<sup>14</sup> is being installed inside the cryostat to reduce these effects.

**Table 1** Counting rate in the natural radioactivity region of the 23.8 g ZnMoO<sub>4</sub> detector in the Orsay test facility before and after the installation of a lead shield around the cryostat.

Energy range [keV]	Without Pb shield [counts/s]	With Pb shield [counts/s]	Reduction factor
100 – 500	1.84	0.084	0.046
500 – 1000	0.309	0.0156	0.050
1000 – 1500	0.114	0.0064	0.056
1500 – 2000	0.027	0.0034	0.126
2000 – 2500	0.014	0.0021	0.15
2500 – 3000	0.005	0.0016	0.32



**Fig. 2** Plots reporting the light-to-heat signal amplitude ratio as a function of the heat signal amplitude for the 23.8 g ZnMoO<sub>4</sub> scintillating bolometer tested in the Orsay facility. The two bands related to  $\beta$ /gamma events (which include also cosmic muon interactions) and  $\alpha$  events are well separated. The  $\alpha$  band is populated mainly by a contamination of  $^{210}\text{Po}$ .

The length of the signal in the heat channel poses the serious problem of pulse pile-up due to environmental radioactivity. In order to mitigate this effect, which is detrimental to the energy resolution on the  $\gamma$  lines, a shield of low-activity lead (less than 30 Bq/kg in  $^{210}\text{Pb}$  content) was placed around the cryostat, with a minimum thickness of 10 cm. The benefit of the shield can be appreciated in Table 1. The good performance of the scintillating bolometer allows the calibration of the heat channel with  $\gamma$  sources placed between the cryostat and the lead shield and the estimation of all the parameters discussed in Section 1. These parameters are listed in Table 2 for the 23.8 g ZnMoO<sub>4</sub> test detector.

An example of the  $\alpha/\beta$  separation that can be obtained in such a setup is shown in Figure 2. The only detectable internal  $\alpha$  contamination is due to  $^{210}\text{Pb}$ , of about

**Table 2** Performances of a 23.8 g ZnMoO<sub>4</sub> detector in the Orsay test facility. We report the sensitivity of the heat channel  $S_h$ , the sensitivities of the two light channels  $S_{l1}$  and  $S_{l2}$ , the FWHM intrinsic energy resolution of the heat channel  $E_R$ , the light yield LY for  $\beta$ -like particles and the light (heat) quenching factor  $Q_l$  ( $Q_h$ ) of  $\alpha$  particle signals with respect to  $\beta$  particle signals at  $\sim 5.4$  MeV.

$S_h$ [ $\mu$ V/MeV]	$S_{l1}$ [ $\mu$ V/keV]	$S_{l2}$ [ $\mu$ V/keV]	$E_R$ [keV]	LY [keV/MeV]	$Q_l$	$Q_h$
81	0.21	0.39	4.0	1.8	0.19	1.3

20 mBq/kg. Sensitivities better than 1 mBq/kg to crystal contamination of  $^{232}\text{Th}$ ,  $^{238}\text{U}$  and their daughters can be achieved in a few days of data taking. This is of course far from the demanded radiopurity in  $^{232}\text{Th}$  (less than  $10 \mu\text{Bq/kg}^3$ ), which can be checked only in long measurements in an underground environment, but it is enough for a real-time quality control of the crystals.

## 4 Conclusions

We have developed a setup in Orsay capable characterising systematically the foreseen LUMINEU ZnMoO<sub>4</sub> crystal production. We have also shown, for the first time in the literature to our knowledge, that macro-bolometers can be efficiently operated in a pulse-tube dilution refrigerator.

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