

Performance of ZnMoO_4 crystal as cryogenic scintillating bolometer to search for double beta decay of molybdenum

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ABSTRACT: Zinc molybdate (ZnMoO_4) single crystals were grown for the first time by the Czochralski method and their luminescence was measured under X-ray excitation in the temperature range 85–400 K. Properties of ZnMoO_4 crystal as cryogenic low temperature scintillator were checked for the first time. Radioactive contamination of the ZnMoO_4 crystal was estimated as ≤ 0.3 mBq/kg (^{228}Th) and 8 mBq/kg (^{226}Ra). Thanks to the simultaneous measurement of the scintillation light and the phonon signal, the α particles can be discriminated from the γ/β interactions, making this compound extremely promising for the search of neutrinoless Double Beta Decay of ^{100}Mo . We also report on the ability to discriminate the α -induced background without the light measurement, thanks to a different shape of the thermal signal that characterizes γ/β and α particle interactions.

KEYWORDS: Cryogenic detectors; Gamma detectors (scintillators, CZT, HPG, HgI etc); Particle identification methods

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1 Introduction

Neutrinoless Double Beta Decay ($0\nu 2\beta$) of atomic nuclei is a rare nuclear process able to give very important information about properties of neutrino and weak interaction.

Observations of neutrino oscillations [1–3] give a clear evidence that neutrino is a massive particle. While oscillation experiments are sensitive to the neutrinos squared-mass differences, only the measurement of a $0\nu 2\beta$ decay rate could establish the Majorana nature of the neutrino, participate in the determination of the absolute scale of neutrino masses and test lepton number conservation [4–6]. Moreover, this process can clarify the presence of right-handed currents in weak interaction, and prove the existence of Majorons [6]. Taking into account ambiguity of theoretical estimations [7–10], development of experimental methods for different 2β isotopes is highly requested.

^{100}Mo is one of the most promising 2β isotopes because of its large transition energy $Q_{2\beta} = 3035$ keV [11] and a considerable natural isotopic abundance $\delta = 9.67\%$ [12]. From the experimental point of view a large $Q_{2\beta}$ value simplifies the problem of background induced by natural radioactivity and cosmogenic activation.

Nowadays the best sensitivity to $0\nu 2\beta$ decay of ^{100}Mo is the one reached by the NEMO experiment [13] that, with $\simeq 7$ kg of enriched ^{100}Mo , has obtained a half-life limit $T_{1/2}^{0\nu} > 4.6 \times 10^{23}$ yr at 90% C.L. Despite the beautiful result, the NEMO technique presents two disadvantages that limit the achievable sensitivity: the low detection efficiency of $0\nu 2\beta$ events ($\approx 14\%$) and the poor energy resolution ($\approx 10\%$ at the energy of $Q_{2\beta}$ of ^{100}Mo). Both these limitations can be overcome by the use of a high resolution detector containing in its sensitive volume the DBD candidate, i.e. by the use of the so called source=detector technique.¹ In particular, the energy resolution (FWHM $< 1\%$) needed to investigate the normal hierarchy of the neutrino mass (half-life sensitivity on the level of $10^{28} - 10^{30}$ years) could be achieved only by bolometers or/and semiconductors providing an energy resolution of about few keV [16].

¹For instance the detection efficiency is $\approx 86\%$ in the CUORICINO experiment with 0.75 kg tellurium oxide crystals [14] and $\approx 93\%$ for Ge detectors [15].

Table 1. Properties of ZnMoO₄ crystal scintillators.

Density (g/cm ³)	4.3	[31]
Melting point (°C)	1003 ± 5	[31]
Structural type	Triclinic, <i>P1</i>	[31, 33]
Cleavage plane	Weak (001)	[31]
Wavelength of emission maximum* (nm)	544	Present work

*Under X-ray excitation at room temperature.

The main issue for the bolometric technique is the suppression -or active rejection- of the background induced by α emitters located close to the surface of a detector. Simulations show that this contribution will largely dominate the expected background of the CUORE Experiment [17, 18] in the region of interest. The natural way to discriminate this background is to use a scintillating bolometer [19]. In such a device the simultaneous and independent read out of the heat and the scintillation light permits to discriminate events due to γ/β , α and neutrons thanks to their different scintillation yield.

Several inorganic scintillators containing molybdenum were developed in the last years. The most promising of them are molybdates of Calcium (CaMoO₄) [20, 21], Cadmium (CdMoO₄) [19, 22], Lead (PbMoO₄) [19, 23, 24], Lithium-Zinc (Li₂Zn₂(MoO₄)₃) [25], and Lithium (Li₂MoO₄) [26, 27]. However CaMoO₄ contains the $2\nu 2\beta$ active isotope ⁴⁸Ca which, even if present in natural Ca with a very small abundance of $\delta = 0.187\%$ [12], creates background at $Q_{2\beta}$ energy of ¹⁰⁰Mo [21]. CdMoO₄ contains the β active ¹¹³Cd ($T_{1/2} = 8.04 \times 10^{15}$ yr [28], $\delta = 12.22\%$ [12]) which, besides being beta active, has a very high cross section to capture thermal neutrons. A potential disadvantage of PbMoO₄ (supposing crystals would be produced from low-radioactive ancient lead [29, 30]) is that ¹⁰⁰Mo would be only 27% of the total mass. Finally Li₂Zn₂(MoO₄)₃ and Li₂MoO₄ have low Light Yield (LY).

ZnMoO₄ crystals were developed only very recently [31, 32]. An important advantage of ZnMoO₄ is the absence of heavy elements and high concentration of molybdenum (43% in weight).

The purpose of our work is to investigate ZnMoO₄ crystals as scintillating bolometers to search for double beta decay of ¹⁰⁰Mo. Luminescence of material was studied under X-ray irradiation. The performances of the detector were measured through the use of the bolometric technique.

2 Growth of ZnMoO₄ crystals

The zinc molybdate (ZnMoO₄) charge was obtained by a solid-phase synthesis technique from MoO₃ and ZnO powders (both of 99.995% purity). Single crystals of up to dia=30 mm, h=40 mm were grown by the Czochralski technique with a drawing speed of 1.9 mm/h. The material is highly inert; the melting point is at (1003 ± 5) °C. The crystal density calculated from the X-ray data is 4.317 g/cm³, while the density measured by the pycnometric method is 4.19 g/cm³ [31]. Properties of ZnMoO₄ crystals are presented in table 1.

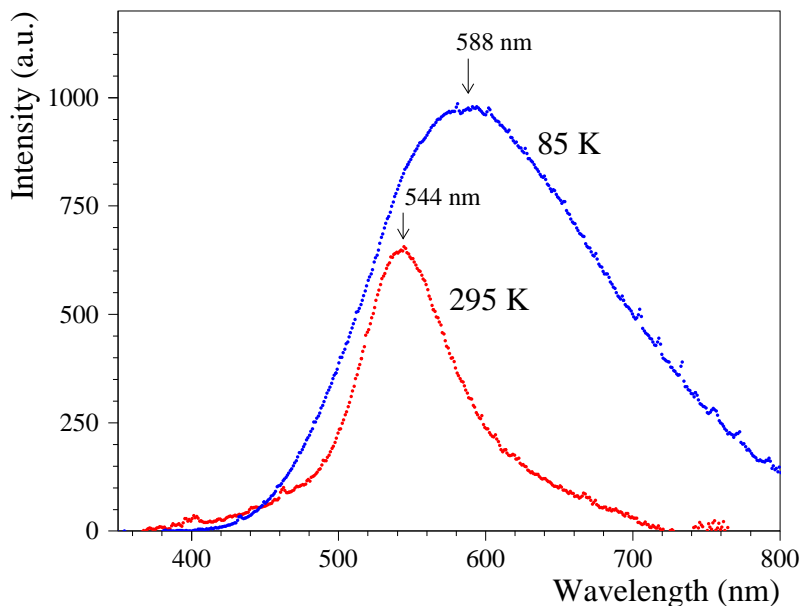


Figure 1. (Color online) Emission spectrum of ZnMoO_4 crystal under X-ray excitation at 85 K and 295 K.

3 Luminescence under X-ray excitation

We measured the luminescence of ZnMoO_4 crystal in the temperature interval 85–400 K under X-ray excitation. A sample of ZnMoO_4 crystal ($10 \times 10 \times 2 \text{ mm}^3$) was irradiated by X-ray from a BHV7 tube with a rhenium anode (20 kV, 20 mA). The light emitted by the crystal was detected in the visible region by a FEU-106 photomultiplier (sensitive in the wide wavelength region of 300–800 nm). Spectral measurements were carried out using a high-aperture MDP-2 monochromator.

One band in the visible region with the maximum at 544 nm was observed at room temperature (figure 1). The radioluminescence (RL) spectrum, measured at 85 K, is shifted to 588 nm. In addition, the spectrum measured at 85 K is much wider and distributed in the wavelength region above 700 nm.

The temperature dependence of luminescence is presented in figure 2. The intensity of luminescence slowly falls above 150 K, but it still remains observable even at ≈ 400 K temperature. A sharp peak of thermostimulated luminescence (TSL) was observed at ≈ 120 K and across the wide temperature range 220–320 K (see figure 3), with maxima at ≈ 230 , ≈ 260 , and ≈ 300 K. The TSL observed in our measurements indicates the presence of traps in the ZnMoO_4 sample. We assume it is due to defects and impurities in the crystal. Both can deteriorate the scintillation and/or the bolometric performances. Therefore, further R&D is necessary to improve the quality of ZnMoO_4 crystals.

4 ZnMoO_4 as a scintillating bolometer

We present in this section the results obtained with a small sample (19.8 g) of ZnMoO_4 operated as scintillating bolometer. The shape of our sample is a regular hexagon, with a diagonal of 25 mm and a height of 11 mm. The color of the sample is fairly orange, showing evident inclusions along

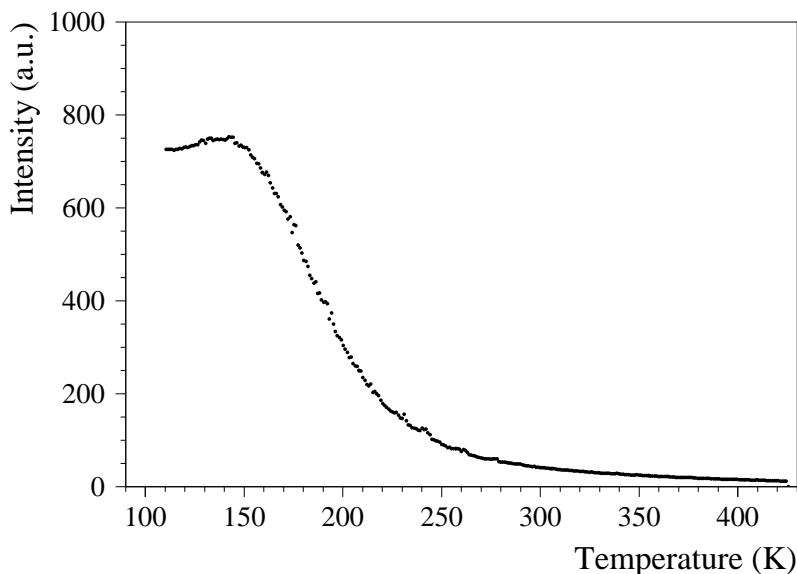


Figure 2. Temperature dependence of ZnMoO_4 luminescence intensity under X-ray excitation.

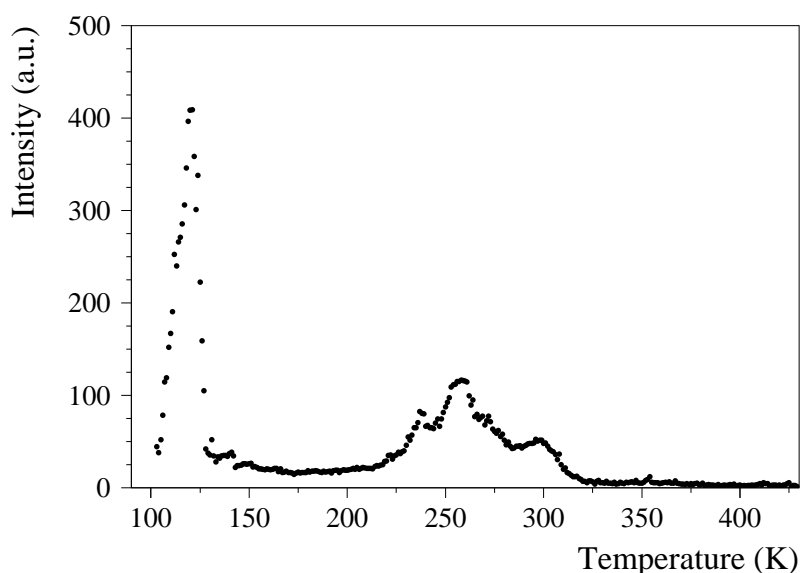


Figure 3. Thermoluminescence of ZnMoO_4 crystal after X-ray excitation at liquid nitrogen temperature.

the central axis of the growth. The composite device (bolometer + light detector) is schematized in figure 4. The crystal is held by means of two L-shaped Teflon (PTFE) pieces fixed to two cylindrical Cu frames; the PTFE forces the crystal to the base consisting of a Cu plate covered with an Al foil. The crystal is surrounded by a 25.1 mm diameter cylindrical reflecting foil (3M VM2002). At cryogenic temperatures ($10 \div 100$ mK) for which a detector can work as bolometer, no “standard” light detectors can work properly. The best way to overcome this problem is to use a second - very sensitive- “dark” bolometer that absorbs the scintillation light giving rise to a measurable increase of its temperature. Our Light Detector (LD) [34] consists of a 36 mm diameter, 1 mm

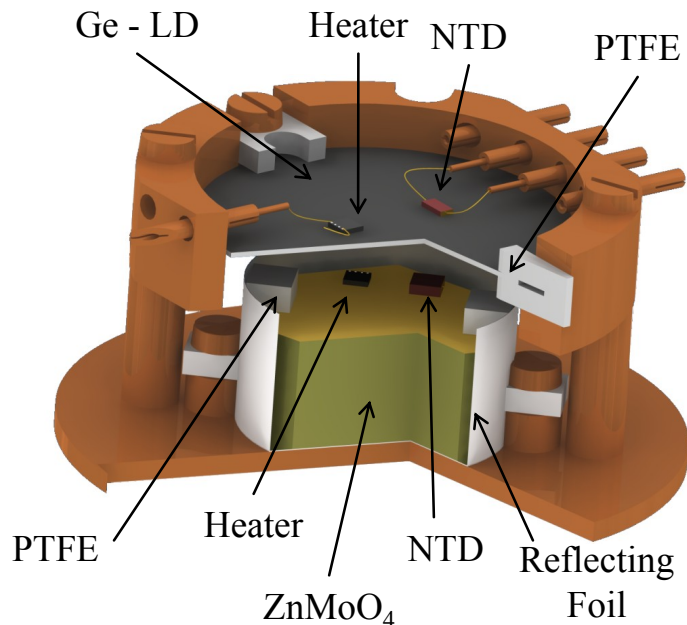


Figure 4. Setup of the detector.

thick pure Ge crystal absorber. The temperature sensor of the ZnMoO_4 crystal is a $3 \times 3 \times 1 \text{ mm}^3$ neutron transmutation doped Germanium thermistor, identical to the ones used in the CUORICINO experiment [14]. The temperature sensor of the LD has a smaller volume ($3 \times 1.5 \times 0.4 \text{ mm}^3$) in order to decrease its heat capacity, increasing therefore its thermal signal. A resistor of $\sim 300 \text{ k}\Omega$, realized with a heavily doped meander on a 3.5 mm^3 silicon chip, is attached to each absorber and acts as a heater to stabilize the gain of the bolometer [35, 36]. The detectors were operated deep underground in the Gran Sasso National Laboratories in the CUORE R&D test cryostat. The details of the electronics and the cryogenic facility can be found elsewhere [37–39].

The heat and light pulses, produced by a particle interacting in the ZnMoO_4 crystal and transduced in a voltage pulse by the NTD thermistors, are amplified and fed into a 16 bit NI 6225 USB ADC unit. The entire waveform (*raw pulse*) of each triggered voltage pulse is sampled and acquired. The amplitude V^{heat} and the shape of the voltage pulse is then determined by the off-line analysis that makes use of the Optimal Filter (OF) technique [17, 40]. The signal amplitudes are computed as the maximum of the optimally filtered pulse, while the signal shape is evaluated on the basis of four different parameters: τ_{rise} and τ_{decay} , TVL and TVR.

τ_{rise} (the rise time) and τ_{decay} (the decay time) are evaluated on the *raw pulse* as $(t_{90\%} - t_{10\%})$ and $(t_{30\%} - t_{90\%})$ respectively. The rise time is dominated by the time constant of the absorber-glue-sensor interface (as well as the heat capacity), while the decay time is determined by the crystal heat capacity and by its thermal conductance toward the heat sink.

TVR (Test Value Right) and TVL (Test Value Left) are computed on the optimally filtered pulse as the least square differences with respect to the filtered response function² of the detector:

²The response function of the detector, i.e. the shape of a pulse in absence of noise, is computed with a proper average over a large number of raw pulses. It is also used, together with the measured noise power spectrum, to construct the transfer function of the Optimal Filter.

Table 2. Main parameters of the ZnMoO₄ bolometer and light detector. The OF (Optimum Filter) FWHM represent the theoretical energy resolution as evaluated from the signal-to-noise ratio. The (absolute) Signal represents the voltage read across the thermistor for a unitary deposition of energy.

	Resistance [MΩ]	OF FWHM [keV]	τ_{rise} [ms]	τ_{decay} [ms]	Signal [μV/MeV]
ZnMoO ₄	2.5	1.6	12.2	59.5	107
LD	14.5	0.48	4.0	14.3	1490

TVR on the right side of the optimally filtered pulse maximum and TVL on the left. In fact TVR measures the shape difference of each sampled pulse with respect to the “average thermal pulse” (i.e. the response function), evaluating this difference within the “falling side” of the pulse. TVL makes use of the same algorithm, but is evaluated on the “rising side” of the pulse. These two parameters do not have a direct physical meaning, however they are extremely sensitive (even in noisy conditions) to any difference between the shape of the analyzed pulse and the response function. Consequently, they are used either to reject fake triggered signals or to identify variations in the pulse shape with respect to the response function (and this will be our case).

The thermal pulses are acquired within a 512 ms time window with a sampling rate of 2 kHz. The trigger of the ZnMoO₄ is software generated while the LD is automatically acquired in coincidence with the former.

The energy calibration of the ZnMoO₄ crystal is performed using γ sources (²³²Th) placed outside the cryostat. The energy calibration of the LD, on the contrary, is obtained thanks to a weak ⁵⁵Fe source placed close to the Ge that illuminates homogeneously the face opposed to the ZnMoO₄ crystal. During the LD calibration its trigger is set independent from the one of the ZnMoO₄. The LD is calibrated using a simple linear function. The main parameters of the two bolometers are listed in table 2.

Two sets of data have been collected with this device: a calibration using external ²³²Th sources (65 h) and a background measurement (66 h). The temperature of the detectors during our test was ~ 14 mK. The intensity of the calibration sources was not optimized for small crystals, so that the statistics collected within the calibration peaks of ZnMoO₄ is rather poor. The energy resolution evaluated at 911, 2615 and at 5407 keV (²²⁸Ac, ²⁰⁸Tl and internal contamination of ²¹⁰Po, respectively) are 3.6 ± 1 , 5.6 ± 2 and 6 ± 1 keV, respectively. The FWHM energy resolution of the LD, evaluated on the X doublet at 5.90 and 6.49 keV, is 470 ± 20 eV.

In this field the usual way to present the results is to draw the light vs. heat scatter plot [41]. Here each event is identified by a point with abscissa equal to the heat signal (recorded by the ZnMoO₄ bolometer), and ordinate equal to the light signal (simultaneously recorded by the LD). In the scatter plot, γ/β and α give rise to separate bands, in virtue of their characteristic light to heat ratio. In figure 5 we present the scatter plot light vs. heat of the sum of the two above mentioned measurements (Calibration + Background). The LY of this crystal (measured on the 2615 keV ²⁰⁸Tl line) can be evaluated as 1.1 keV/MeV. This value is rather small. Using the same LD we evaluated the LY of a large (510 g) CdWO₄ crystal to be 17.4 keV/MeV [41]. Nonetheless it is evident from

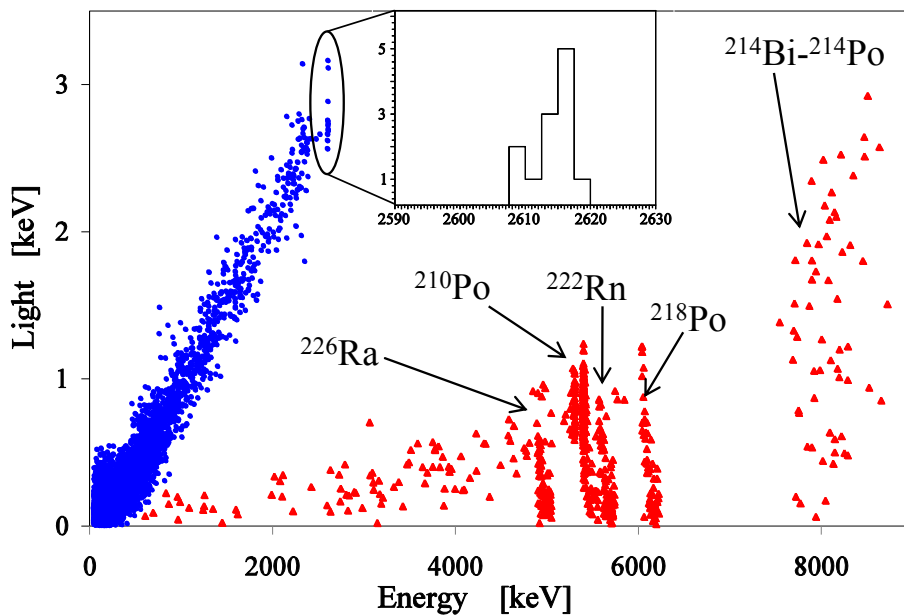


Figure 5. Scatter plot light vs. heat obtained with a ^{232}Th calibration (65 h) plus a background measurement (66 h). The main observed lines are pointed out. In the inset the 2615 keV γ -line of ^{208}Tl . The dark (blue) points are identified as γ/β events, while the light (red) triangles are identified as α -particles. The BiPo points are due to mixed events induced by a γ/β decay “immediately” followed by an α decay, with $\tau=164 \mu\text{s}$, too fast for both our detectors to be distinguished from the parent γ/β .

figure 5 that the continuous background induced by α -emitters located close to the surfaces is very efficiently recognized with respect to the γ/β events. The γ/β band is mainly due to the interaction of γ rays produced from the calibration source or from radioactive contamination of the detector and its set-up. The events in the α band are due to intrinsic radioactive contamination of ^{226}Ra , of the ^{238}U chain. The continuum below the ^{226}Ra peak can be ascribed to an alpha contamination in the surfaces of the mounting structure, producing degraded alpha particles with a continuous distribution of energies. ^{214}Bi - ^{214}Po events are due to the fast decay chain of these two isotopes that produce a beta followed by an alpha, with a time separation below the integration time of the detector (i.e. they are recorded as a single event with an energy corresponding to the sum of α and β energies).

A strange feature has however to be pointed out: all the α -lines are characterized by a tail that draws negative slopes “bands”, as it is apparent in figure 5. This effect is extremely evident in the α -peaks induced by ^{226}Ra , ^{222}Rn and ^{218}Po . This results in a large smearing of the energy resolution of these peaks. The “doublet” due to ^{210}Po , instead, shows this unexpected feature only -very marginally- on the full energy peak, at 5407 keV line.

This strange behavior is likely when the contaminations are close to the surfaces, both of the crystal and of the surrounding materials. The presence of large quantity of surface contaminants, in fact, is corroborated by the large α -continuum background, visible in the same figure. This issue should be studied in more details. This is however beyond the scope of this work. Moreover this effect — even if not well understood — increases the separation between α and γ/β interactions

Table 3. Radioactive α activity of ZnMoO_4 crystal. The activity in ^{232}Th corresponds to a limit in the contamination of $7 \cdot 10^{-11}$ g/g. The contamination in ^{238}U has a limit of $2 \cdot 10^{-11}$ g/g. The observed ^{226}Ra contamination, instead, would correspond — assuming secular equilibrium — to a contamination of $^{238}\text{U} = 6 \cdot 10^{-10}$ g/g.

Chain	Nuclide	Activity (mBq/kg)
^{232}Th	^{232}Th	≤ 0.3
	^{228}Th	≤ 0.3
^{238}U	^{238}U	≤ 0.2
	^{234}U	≤ 0.8
	^{230}Th	≤ 0.3
	^{226}Ra	8.1 ± 0.3
	^{210}Po	28 ± 2
Total α activity		73 ± 2

in the scatter plot. Nonetheless, by assuming, in a very conservative way, that all the observed α lines are induced by internal contamination, we obtained the values in table 3.

It can be seen that, as it very often happens, the ^{238}U chain is broken at ^{226}Ra . With respect to the ^{232}Th decay chain, the most dangerous [19] internal contaminant for this kind of detectors, we only observe a limit.

A very interesting feature of this compound comes from the observation that α and γ/β events give rise to slightly different thermal pulses. In fact this promising feature was very recently exploited in CaMoO_4 [42] as well as, even if less pronounced, in ZnSe [43].

In figure 6 we show the Pulse Shape Analysis performed only on the heat signal read in the ZnMoO_4 crystal. The separation between α and γ/β events is rather impressive, especially in the left panel of figure 6. A comparison of the two distributions in the 2400–2620 keV energy region shows a separation of 7.2σ . This result needs further investigation with a high statistics comparison in the 3 MeV energy region (the $Q_{2\beta}$ value of ^{100}Mo). It expresses however in a quantitative way the excellent rejection power of this technique.

5 Conclusions

Zinc molybdate (ZnMoO_4) single crystals were grown for the first time by the Czochralski method. Luminescence was measured under X-ray excitation in the temperature range 85–400 K. Emission maximum of ZnMoO_4 occurs at 544 nm at 295 K, and then shifts to 588 nm at 85 K.

Applicability of ZnMoO_4 crystals as cryogenic phonon-scintillation detectors was demonstrated for the first time. Despite the “non perfect” quality of our crystal sample, we obtained very encouraging results. The light output of ZnMoO_4 was estimated $< 6\%$ comparatively to CdWO_4 . Even if rather small, the scintillation signal permits a very efficient discrimination between α and γ/β particles.

Especially this compound shows a different heat pulse shape between α and γ/β events. This permits the discrimination of the α induced background -without using the scintillation signal-

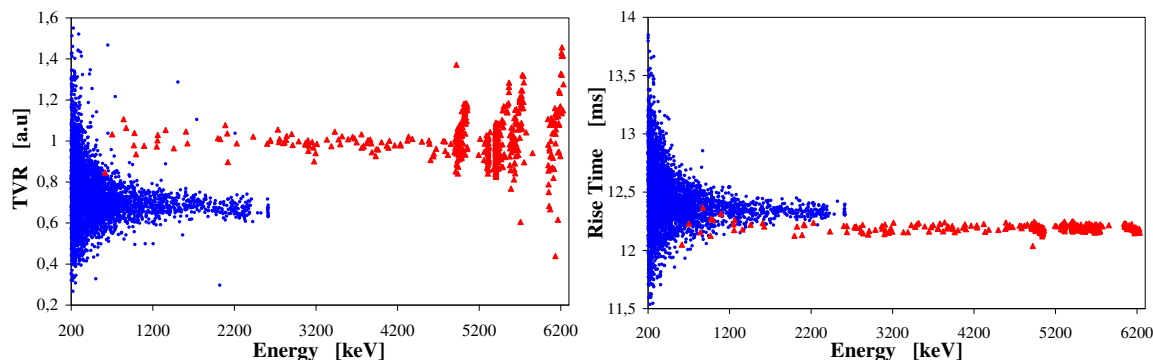


Figure 6. Pulse Shape Analysis of the events of figure 5. Left panel: TVR of the heat signal in ZnMoO_4 vs. energy (heat). The α events are effectively discriminated with respect to γ/β events down to low energies. Right panel: τ_{rise} of heat pulses vs. energy (heat) for the same events. It has to be pointed out, however, that the two parameters (τ_{rise} and TVR) are largely uncorrelated since one is calculated on the left part of the pulse maximum, while the latter on the right. Thus their combination will further increase the discrimination factor.

at a level of 7.2σ . This feature could play a crucial role for a possible large scale double beta decay experiment.

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