Scintillation studies of $CaWO_4$ in the millikelvin temperature range

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We extended the multiphoton counting technique to investigate the scintillation characteristics of crystals at millikelvin temperatures using $^3He/^4He$ dilution refrigerator. The temperature dependence of the scintillation time constant and the light response of $CaWO_4$ were measured down to 20 mK. The scintillation light response is found to be independent of temperature below 10 K—so far taken as an assumption in many cryogenic scintillator experiments, but yet previously unproved. The decay kinetics of $CaWO_4$ and its temperature dependence are analyzed in terms of a simple three-level model of the emission center. The model agrees well with the data over the temperature range (0.02–350 K) investigated, and is used to determine the parameters of the relaxed excited state.

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I. INTRODUCTION

Since its first use as a detector of ionizing radiation following Röntgen's discovery of x-rays,[1] calcium tungstate has found extensive application as a phosphor,[2] scintillator,[3] and laser host.[4] Resulting from this was a whole range of studies of the optical characteristics of the compound, some of which are presented in Refs. 5–13. In recent years, interest in $CaWO_4$ has been revived, mainly driven by materialist use in a new class of hybrid cryogenic phonon-scintillation detectors of radiation operating at millikelvin temperatures.[14] These detectors combine a very low energy threshold and excellent resolution with the ability to distinguish different types of particle interactions (neutrons, α particles, β and/or γ quanta). This combination is ideally suited to experimental searches for extremely rare events, such as the interaction of weakly interacting massive particle dark matter with nuclei[15] or neutrinoless double-beta decay.[16]

With the advent of cryogenic phonon-scintillation detectors, numerous studies of the luminescence and scintillation properties of suitable target materials have been made. While a significant amount of research has been carried out on the temperature dependence of the decay time constant and the light response of scintillators down to ~10 K (see recent review in Ref. 17, and references therein), investigations at lower temperatures are rare. Although it was assumed that the results obtained at ~10 K can provide instructive information on the major scintillation characteristics also at millikelvin temperatures, direct experimental proof of this presumption was missing so far. Providing this missing experimental evidence, as well as our desire to get an insight into scintillation processes at such low temperatures, motivated this study.

II. EXPERIMENT

A. Problems associated with low-temperature scintillation studies

Investigating scintillation properties at extremely low temperatures is a significant experimental challenge. In addition to the familiar considerations associated with experiments at millikelvin temperatures (thermal insulation, heat sinks, thermometry, etc.), there are two problems specific to this kind of measurement, related to the temperature dependence of photodetector efficiency and multiple excitation of the sample.

Experiments frequently measure the scintillator response using a high-gain photodetector, a photomultiplier, or an avalanche photodiode, the sensitivity of which is temperature dependent. Therefore, in order to obtain reliable measurements of scintillation characteristics as a function of temperature, it is imperative to keep the detector at a constant temperature while cooling or heating the sample. For all practical purposes, this means separating detector and sample, leading inevitably to poor light collection efficiency.

Measurements of the temperature dependence of the decay time constant must allow for its variation over a large range (from tens of microseconds to tens of milliseconds). This requires a correspondingly long recording time. As it is not practically possible to fully control the rate at which ionizing radiation interacts with the scintillator, there are always events recorded in which additional scintillation events have occurred during the measurement period of the first, so-called multiple excitation or pileup. These events contribute to the total signal, resulting in a roughly even time difference distribution throughout the decay curve, hence creating a noticeable tailing effect. Thus, when analyzing such data, one might easily misinterpret this background as an additional decay component with a slow time constant (see Ref. 18). This effect becomes especially pronounced in the case of measurements at cryogenic temperatures, where the scintillation decay rate becomes comparable with that of the excitation events. Together, these two issues impose a practical limit on the scope of any such experiment to make measurements of the temperature dependence of scintillation characteristics, and as a consequence most studies have hitherto been performed at temperatures no lower than that of liquid nitrogen.[19–23]

In order to overcome these difficulties, we recently developed and successfully tested the multiphoton counting (MPC) technique in which the sequence of individual photon pulses resulting from a scintillation event are recorded and analyzed. The distribution of arrival times of these pulses provides information on the decay characteristics of the scintillation process, while the number of pulses recorded per

event is proportional to the light response. Off-line analysis is used to identify and discard events associated with multiple excitation. A detailed description of the original experimental setup and data analysis procedure can be found elsewhere.[18] The technique is especially advantageous for the analysis of slow scintillation processes and the investigation of temperature-dependent scintillator properties. Encouraged by this success, we implemented the MPC technique for the study of scintillation properties in the millikelvin temperature range.

B. Description of experimental setup and data acquisition hardware and software

This study focuses on a $CaWO_4$ crystal sample that we have investigated extensively earlier.[18,24] The $5\times5\times2$ mm^3 sample is held between two copper blocks fixed by screws. This holder also encapsulates a Cerinox resistor temperature sensor (CX-1010-SD model, Lake Shore) placed in direct contact with the sample, and it contains a radioactive α or γ source (^{241}Am or ^{60}Co, respectively). The holder is firmly attached to a copper plate, which in turn is connected to the mixing chamber of a ^{3}He/^{4}He dilution refrigerator cryostat (S-400, Oxford Instruments). The copper plate is equipped with another temperature sensor, a ruthenium oxide resistor (T1-202 model, Oxford Instruments) that, together with a 100 Ω resistance heater, is used for temperature control. The temperature of the copper plate can be stabilized at any value below 40 K using a TS-530 temperature controller connected to an AVS-47 bridge. For each point, ~10 min was allowed for reaching thermal equilibration, and then the variation of the resistance of the temperature sensor was held within ±0.2% of the chosen value.

One end of an optical fiber was put in contact with the scintillator sample, the other with a photomultiplier tube (PMT) mounted outside the cryostat. The optical fiber has a 1 mm core diameter and is made from quartz that has low transmittance in the far-IR range; therefore, when thermally anchored to the main He bath at 4.2 K, it provides negligible heat load onto the sample. Scintillation light is guided along the optical fiber to the PMT, which detects the individual scintillation photon pulses in the photon counting regime. The charge signal produced by the PMT is converted by a preamplifier into a voltage pulse and fed into a transient recorder and also into an integrating amplifier. Further signal processing occurs as described in Ref. 18 except that we employ the latest development of the MPC technique,[25] operating with one PMT (9125BQ Electron Tube) with self-trigger option and using a modified off-line analysis routine to eliminate spurious events.

The transient recorder offers sampling intervals ranging from 10 to 80 ns. The electronics used enables us to record up to 131 072 samples per record, giving a 2.62 ms record length with 20 ns sampling interval. This allows us to detect and analyze the scintillation decay in the millisecond time domain, which is of vital importance for low-temperature experiments. All scintillation events recorded are stored in a binary data file and are subsequently subjected to data analysis. The algorithm used for searching for and eliminating multiple events is based on the idea that the decay time constant τ obtained for each event should be noticeably dissimilar in single events when compared to multiple events. The statistical analysis consists of a combination of a cut on the number of photons, a Shapiro-Wilk likelihood cut and a Poisson statistic cut for the distribution of arrival times of the photons.[26]

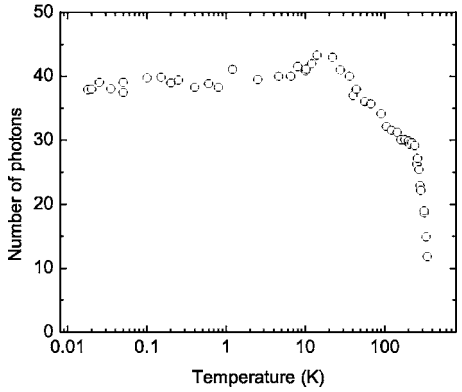

FIG. 1. Temperature dependence of scintillation light response of $CaWO_4$.

III. RESULTS

A. Scintillation light response as function of temperature

For the excitation of $CaWO_4$, we used α particles from ^{241}Am (5.44 and 5.48 MeV) and γ quanta from ^{60}Co (1173 and 1333 keV). Due to the small size of the crystal and the low light collection efficiency of the experimental setup, deposition of the full γ energy cannot be detected. Most excitations result from Compton scattered photons yielding a continuum of energies. In contrast, α particles are absorbed within ~10 μm of the scintillator surface and deposit their full energy in the crystal. The number of scintillation photons produced, proportional to the deposited energy, has a Gaussian distribution. This can easily be identified by MPC as a few tens of photons detected in a single scintillation event.[18] The amplitude characteristic of α scintillations as a function of sample temperature is, therefore, a direct measure of the temperature dependence of the light response.

Figure 1 shows the variation of scintillation light response of $CaWO_4$ over the 0.020–350 K temperature range. At high temperatures (>200 K), the light yield is dominated by thermal quenching, and as the probability of nonradiative decays increases strongly with temperature, the emission intensity decreases. From 20 to 200 K, one observes only a small decrease in the light yield, and in the region between 0.02 and 10 K, it remains constant within the error limits. The latter is the most important practical finding of this experiment, as it provides an explicit demonstration of the invariance of the light response in the operating temperature range of cryogenic phonon-scintillation detectors.

We make three remarks on the measurement of the temperature-dependent light yield. First, detailed measurements for the high-temperature part of Fig. 1 (10–350 K) were made during a previous experiment using a conven-

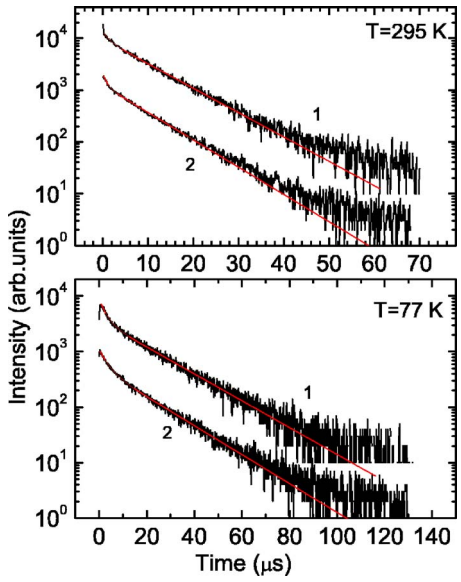

FIG. 2. (Color online) Scintillation decay curves of $CaWO_4$ measured at $T=295$ and 77 K using γ (1) and α (2) excitations. The solid line shows the best fit to the experimental data using two exponentials. Parameters of the fit are listed in Table I.

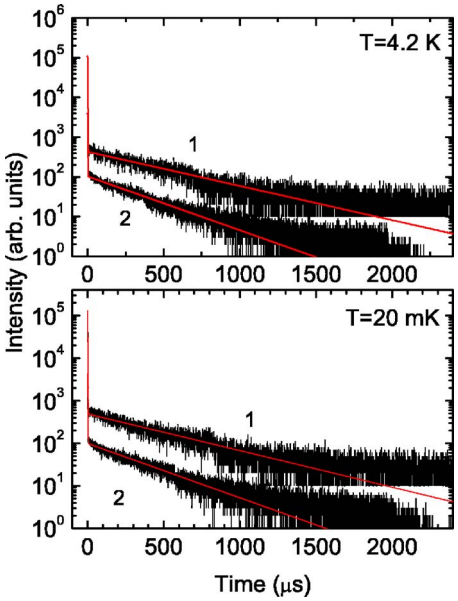

FIG. 3. (Color online) Scintillation decay curves of $CaWO_4$ measured at $T=4.2$ and 0.02 K using γ (1) and α (2) excitations. The solid line shows the best fit to the experimental data using two exponentials. Parameters of the fit are listed in Table I.

tional He-flow cryostat,[18] with only a few reference points above 40 K recorded with the current setup to ensure a proper match of the two curves. Second, use of a transient recorder with extended memory allowed for a fivefold increase in record length compared with the previous study.[18] Therefore, photons from the tail end of a scintillation event that used to escape detection can now be recorded. This reduces the systematic error of the measurement at low temperatures, which led previously to underestimation of the number of photons emitted. Third, at $T\sim 10$ K we detected $\sim$40 photons per α scintillation, a factor of 2 lower than detected using the He-flow cryostat setup, due to lower light collection efficiency. We collect light only from the 3% of the crystal area which is in contact with the optical fiber, something we plan to improve in the future.

B. Decay kinetics as a function of temperature

As mentioned above, in this study we used both α and γ sources for high-energy excitation. There is a noticeable dissimilarity in the decay time characteristics of inorganic scintillators following excitation by α particles or γ quanta. Given that this difference underpins the pulse-shape analysis technique that allows the identification of the type of interaction events,[27] the study of scintillation decay kinetics for both α and γ excitations is of importance.

Figures 2 and 3 present the scintillation decay curves of $CaWO_4$ measured at 295, 77, 4.2, and 0.02 K under excitation with α particles or γ quanta. A fit to the experimental data was done using a sum of two exponentials. The amplitudes and decay time constants derived from the fit are listed in Table I. The room-temperature values are in good agreement with recent measurements.[18,24,27] It should be noted that previously we quoted the value of the slow decay time constant measured under α excitation at $T=9$ K as 390 ± 20 μs.[18] Since then, as mentioned above, we have extended the capability of the electronics and significantly improved the data treatment procedure.[26] This allowed us to make a more rigorous identification of multiple excitation events responsible for the previous overestimation of the decay constant. Therefore, we revise this value to $\tau_2=340\pm 40$ μs, which is consistent with literature data.[10,28,29]

Inspection of the table shows that the value of the slow decay time constant (τ_2) for γ excitation is consistently greater than that for α excitation, and the difference increases as temperature decreases. It is also noteworthy that as the temperature decreases, the amplitude of the slow component (A_2) decreases more rapidly for γ excitation. This means that the difference in the shape of the decay curves for excitation with α particles or γ quanta remains noticeable at low temperature and hence the use of the pulse-shape analysis technique can be extended down to cryogenic temperatures.

Another feature that can be seen from the analysis is a significant change in the shape of scintillation decay curves at low temperatures. The scintillation decay curves show a gradual decay with time at 295 and 77 K (Fig. 2). This is because the difference between fast and slow decay time constants is low (less than 1 order of magnitude) and their amplitude contributions are comparable (see Table I). However, for the scintillation decay curves obtained at 4.2 and 0.02 K, the fast ($\tau_1\sim 1$ μs) and very intense component is followed by a weak ($A_2\leqslant 1\%$) slow component (see Fig. 3). The decay time constant of the fast component (τ_1) changes little throughout the measured temperature range. The τ_2 component, however, undergoes very significant changes, as depicted in Fig. 4, which shows the temperature dependence of the slow decay time constant measured under α-particle excitation. Such behavior of the slow decay constant is char-

TABLE I. Parameters of scintillation kinetics of $CaWO_4$ at different temperatures obtained from a fit to two exponentials.

Temperature (K)	α particles (^{241}Am)				γ quanta (^{60}Co)			
	A_1 (%)	τ_1 (μs)	A_2 (%)	τ_2 (μs)	A_1 (%)	τ_1 (μs)	A_2 (%)	τ_2 (μs)
295	40	1.0(2)	60	8.6(3)	30	1.4(2)	70	9.2(3)
77	50	3.2(3)	50	16.6(4)	60	2.1(3)	40	17.6(3)
4.2	98	1.0(2)	2	330(40)	>99	0.9(2)	0.5	480(60)
0.02	98.4	1.2(2)	1.6	340(40)	>99	1.0(2)	0.4	500(60)

acteristic of the decay kinetics of $CaWO_4$ and other tungstates. It is attributed to the existence of a metastable level a few meV below the emitting level. We will now discuss this feature in more detail.

IV. DISCUSSION

It is generally accepted that the emission of $CaWO_4$, as well as of other tungstates, is excitonic in nature: it is attributed to the radiative recombination of self-trapped excitons (STEs) localized at WO_4^{2-} molecular ions.[7–13] The point symmetry of the WO_4^{2-} oxyanion complex is lower than T_d. Due to symmetry lowering, the spin-orbit and Jahn-Teller interactions split the lowest excited $^3T_{1,2}$ triplet states of the STE into several sublevels, making radiative transitions to the ground state $^1A_1$ partially allowed. Such an energy-level scheme is usually sufficient to explain the major features of excited-state dynamics in tungstates and molybdates,[30–32] as the temperature-induced phonon-assisted processes provide an efficient mechanism for the depopulation of nonemitting levels. However, at temperatures of ~1 K this process is not effective, and to explain the dramatic increase of the decay time constant one should take into account that emission originates from the pair of closely lying energy levels originating from the lowest triplet state.

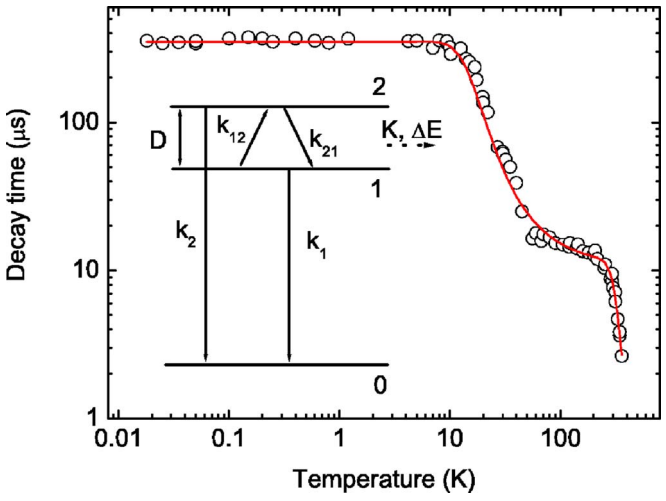

FIG. 4. (Color online) Temperature dependence of the slow scintillation decay time constant of $CaWO_4$ (excitation with ^{241}Am α particles). The solid curve displays the result of the best fit to the experimental data using the three-level model shown in the inset. Parameters of the fit are $k_1=3.0\times10^3$ s^{-1}, $k_2=1.1\times10^5$ s^{-1}, $D=4.4$ meV, $K=8.6\times10^9$ s^{-1}, and $\Delta E=320$ meV.

In order to explain the observed results on the temperature dependence of the decay time constants of $CaWO_4$, we consider a simplified configuration coordinate model that includes the ground and two excited levels 1 and 2 of which the lower one is metastable (see inset in Fig. 4). The key features of this model have been suggested by Beard *et al.*[6] to provide a qualitative interpretation of the features of the radiative decay of $CaWO_4$ and $CdWO_4$. Later, this model has been developed further and is extensively used to give a theoretical description of the decay process of mercurylike ions[33–35] and exciton emission.[11,36–38] It should be noted that this model does not include consideration of the fast component in $CaWO_4$ that is assumed to be associated with the radiative decay of upper-lying singlet states.[31]

Excitation of the emission center to the upper-lying band is followed by relaxation to emission levels 2 and 1. Let the initial populations of the levels be n_2 and n_1, with the total population of the emission center being

$$n = n_1 + n_2. \tag{1}$$

The probabilities of radiative decay from levels 1 and 2 are given by k_1 and k_2, respectively ($k_2 >> k_1$). The levels are separated by energy D and the condition of thermal equilibrium is

$$n_2 = g n_1 \exp(-D/kT), \tag{2}$$

where g is the ratio of the degeneracies of levels 2 and 1, k is the Boltzmann constant, and T is the temperature. In our case the emission levels are both triplets and hence $g=1$. The probability of nonradiative quenching to the ground state is given by the classical equation

$$k_x = K \exp(-\Delta E/kT), \tag{3}$$

where K is a decay rate and ΔE is the energy barrier. Provided that the energy difference between the two emitting levels is much lower than this barrier ($D \ll \Delta E$), it is reasonable to infer that for temperatures at which this escape channel is active, the metastable level can be depleted efficiently through the thermal activation process. Therefore k_x as a characteristic of the thermal quenching process of the emission center is the same for both emitting levels.

After excitation, the variation of the total population with time of the emission center is given by the following differential equation:

$$\frac{dn}{dt} = -k_1 n_1 - k_2 n_2 - k_x(n_1 + n_2). \tag{4}$$

Combining Eqs. (1) and (2), Eq. (4) can be transformed into

$$\frac{dn}{dt} = -n\left(\frac{k_1}{1+\exp(-D/kT)} + \frac{k_2 \exp(-D/kT)}{1+\exp(-D/kT)} + K\exp\left(-\frac{\Delta E}{kT}\right)\right), \tag{5}$$

the solution of which gives the following expression for the radiative decay time constant τ_r:

$$\frac{1}{\tau_r} = \frac{k_1 + k_2 \exp(-D/kT)}{1+\exp(-D/kT)} + K\exp\left(-\frac{\Delta E}{kT}\right). \tag{6}$$

We will now analyze a few special cases for this equation. At very low temperature ($kT \ll D \ll \Delta E$) the decay time constant tends to $1/k_1$. As the temperature increases to the value where $kT \approx D \ll \Delta E$, the second term of Eq. (6) can be ignored and one recovers the formula for the decay time constant used to describe the temperature variation of the decay kinetics of a three-level system in the absence of the nonradiative depopulation of excited states:[10,34–36]

$$\frac{1}{\tau_r} \approx \frac{k_1 + k_2 \exp(-D/kT)}{1+\exp(-D/kT)}. \tag{7}$$

Further increase of temperature $D \ll kT \approx \Delta E$ causes the contribution of the first term to tend to a constant and Eq. (6) translates into the following:

$$\frac{1}{\tau_r} \approx \frac{k_1 + k_2}{2} + K\exp\left(-\frac{\Delta E}{kT}\right). \tag{8}$$

Writing $(k_1+k_2)/2 = 1/\tau_{12}$, one can obtain the canonical equation for the temperature dependence of the radiative decay time constant derived for the simple configuration coordination model:[39]

$$\frac{1}{\tau_r} \approx \frac{1}{\tau_{12}} + K\exp\left(-\frac{\Delta E}{kT}\right). \tag{9}$$

It is worth noting that in the case that the conditions $D \ll kT$ and $kT \ll \Delta E$ can be satisfied simultaneously over a certain temperature range, one should observe a plateau in the temperature dependence of the decay time constant. Taking into account the aforementioned proviso, $k_2 \gg k_1$ for the emission center with metastable level 1, the value of the decay time constant at the plateau region is determined by the probability of radiative decay from level 2 as

$$\tau_r \approx 2/k_2. \tag{10}$$

Formula (6) has been used to fit the measured temperature dependence of the slow decay time constant τ_3. The result of the fit, displayed in Fig. 4, shows fairly good agreement between the theoretical model and the experimental data. The parameters of the emission center of $CaWO_4$ obtained from the fit of the experimental results to Eq. (6) are as follows:

$$k_1 = 3.0 \times 10^3\ \mathrm{s}^{-1}, \quad k_2 = 1.1 \times 10^5\ \mathrm{s}^{-1}, \quad D = 4.4\ \mathrm{meV},$$

$$K = 8.6 \times 10^9\ \mathrm{s}^{-1}, \quad \Delta E = 320\ \mathrm{meV}.$$

Comparison of these parameters with the results of previous studies on $CaWO_4$ and other tungstates shows that the value of the energy gap between two emitting levels D obtained in this experiment exceeds that quoted by Beard *et al.*[6] (2.3 meV) by almost a factor of 2. The recent study of decay kinetics of other tungstates and molybdates by Babin *et al.*[38] gave an even lower value of D, between 0.3 and 1.2 meV. To explain the reason for this difference, we recall that the value of D strongly depends on the precision of the decay time constant determination in the temperature range of its decrease (10–50 K). In this temperature region, the decay curves exhibit a contribution from the recombination process [see Fig. 6(a) in Ref. 18] that is associated with the additional decay channel[11] which is not taken into account by the model. This has an adverse effect on the accuracy of the determination of decay time constants and eventually affects the parameter of fit. An additional factor that contributes to this discrepancy is the difference in the excitation condition, i.e., α particles in our study versus ultraviolet light in Ref. 38, which leads to noticeably different decay time characteristics due to the excitation density effect.[13] The measured activation energy of thermal quenching, ΔE, is in good agreement with the value reported for $CaWO_4$ (340 meV),[6] and for other tungstates.[11,38] This indicates that these differences are less severe in the high-temperature region and hence a better fit to the experimental data is obtained here.

V. CONCLUSION

The development of the cryogenic scintillation technique for particle detection means there is an urgent need for the investigation of the scintillation properties of materials at millikelvin temperatures. To address this need, we developed an experimental technique to characterize inorganic scintillators in the temperature range of interest. Using this technique, we measured the scintillation decay time and light response of $CaWO_4$ as a function of temperature down to 0.02 K, and we received direct and unambiguous proof that the light response of $CaWO_4$ is constant below 10 K. This finding provides long-awaited support for the assumption that data from studies above 10 K can be used to assess the suitability of scintillation materials for cryogenic applications.[17]

We investigated the scintillation decay curves of $CaWO_4$ under different excitations (α particles and γ quanta) and found that the difference in the decay time constants remains substantial throughout the examined temperature range of 295–0.02 K. This finding paves the way for the use of pulse-shape discrimination techniques at low temperatures.

The results on the measurements of the temperature dependence of the decay time were analyzed within the frame-

work of a simple three-level model of the emission center with one metastable level. The model successfully described the features of the decay kinetics, giving the parameters of the relaxed excited state of the emission center.

The results of this study demonstrate the feasibility of the experimental technique for the characterization of scintillators down to millikelvin temperatures. We envisage the technique will be a vital tool for future research on and development of materials for cryogenic phonon-scintillation detectors.

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