

Narrow-Band Phosphor $\text{K}_2\text{ZnP}_2\text{O}_7\text{:Eu}^{2+}$ Discovered Using Local Structure Similarity.

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Abstract

A new narrow-band emitting phosphor $\text{K}_2\text{ZnP}_2\text{O}_7\text{:Eu}^{2+}$ was discovered by extracting promising crystal structures from a database. Regarding the local structures consisting of the central cation and surrounded ligands extracted from the crystal structure, we obtained the quantitative dissimilarity between the local structures and visualized the data as a 2D scatter plot using the t-SNE method. As a result, one of the K-sites of $\text{K}_2\text{ZnP}_2\text{O}_7$ was located near the reference local structure Ba-site of the known narrow-band phosphor $\text{BaSi}_2\text{O}_2\text{N}_2\text{:Eu}^{2+}$ in the scatter plot. We synthesized $\text{K}_2\text{ZnP}_2\text{O}_7\text{:Eu}^{2+}$ and measured the luminescence properties. $\text{K}_2\text{ZnP}_2\text{O}_7\text{:Eu}^{2+}$ showed a narrow-band emission at 440 nm with a full width at half maximum of 30 nm.

Keywords: Narrow-band Phosphor; Eu^{2+} ; Local structure similarity, t-SNE visualization; Materials informatics.

Phosphor-converted light-emitting diodes (pc-LEDs) replace conventional cold cathode fluorescent lamps in lighting and display backlights. Pc-LEDs combine phosphors and blue LEDs, realizing a high efficiency, energy savings, and long life [1-3]. The luminescent centers of transition metal ions and rare-earth ions doped in the host crystal are responsible for the excitation and emission of phosphors. The emission color of the phosphor depends on the luminescence spectrum. The emission is characterized by the peak wavelength and full width at half maximum (FWHM). The recent development of phosphors focuses on narrowing the peak of the emission spectrum to improve the luminescent efficiency for solid-state lighting and expand the color gamut of displays. Backlighting for liquid-crystal displays (LCDs) requires red-green-blue (RGB) lights with a high color purity. Pc-LED backlighting is commonly used in televisions because it has higher stability, lower cost, and lower toxicity than other materials such as RGB LEDs [4,5] and quantum dots [6,7]. Commercial narrow-band phosphors for pc-LED backlighting include β -SiAlON:Eu²⁺ (Si_{6-x}Al_zO_zN_{8-z}:Eu²⁺) as a green phosphor [8-10] and K₂SiF₆:Mn⁴⁺ as a red phosphor [11]. Although backlighting composed of a blue LED and red and green phosphors satisfies the National Television System Committee (NTSC) standard, [12,13] an even wider color gamut is necessary to satisfy the BT.2020 standard for 8K televisions. Consequently, the next-generation phosphors with a narrow-band emission are required.

Since Eu^{2+} has a $4f^6 5d^1 \rightarrow 4f^7$ ($^8S_{7/2}$) emission and the ground state $^8S_{7/2}$ is degenerated with almost no splitting, the emission shows a single peak. Therefore, Eu^{2+} is suitable as an emitting ion for a narrow-band phosphor. Recently, a very narrow-band red emission was observed in the nitride phosphor $\text{SrLiAl}_3\text{N}_4:\text{Eu}^{2+}$ [14]. This phosphor has a UCr_4C_4 -type crystal structure [15], and the local structures of the Sr-sites, which are the substitution sites for Eu, are similar to a cubic structure. As a result, a crystal structure with a cubic-like local structure has attracted much attention. To date, nitride narrow-band phosphors of the same UCr_4C_4 -type, including $\text{SrMg}_3\text{SiN}_4:\text{Eu}^{2+}$ [16], $\text{CaLiAl}_3\text{N}_4:\text{Eu}^{2+}$ [17], and oxide phosphors $\text{RbLi}(\text{Li}_3\text{SiO}_4)_2:\text{Eu}^{2+}$ [18], $\text{RbNa}(\text{Li}_3\text{SiO}_4)_2:\text{Eu}^{2+}$ [19], and $\text{CsKNaLi}(\text{Li}_3\text{SiO}_4)_4:\text{Eu}^{2+}$ [20], have been discovered. However, since the narrow-band emission is also observed such as the characteristic nine-coordinate structure in $\beta\text{-SiAlON}$ ($\lambda_{\text{em}} = 535 \text{ nm}$, $\text{FWHM} \approx 1,920 \text{ cm}^{-1}$) [8], the Ba-site of the flat rectangular structure in $\text{BaSi}_2\text{O}_2\text{N}_2$ ($\lambda_{\text{em}} = 499 \text{ nm}$, $\text{FWHM} \approx 1,430 \text{ cm}^{-1}$) [21], and the Ba-site called truncated square pyramid in $\text{BaLi}_2\text{Al}_2\text{Si}_2\text{N}_6$ ($\lambda_{\text{em}} = 515 \text{ nm}$, $\text{FWHM} \approx 2,016 \text{ cm}^{-1}$) [22], a narrow-band emission may occur not only for cubic-like local structure. It is empirically known that the FWHM of the emission spectrum depends on the local structure around Eu^{2+} in the phosphor. However, the FWHM cannot be explained by ligand-field theory. The ideal local structure with the narrowest FWHM remains theoretically unknown.

Recently, attempts have been made to explore new phosphor materials by data-driven

approaches. Zhuo et al. found new thermally robust phosphors by extracting promising from crystal structure databases [23]. In the search for new narrow-band phosphors, crystal structures with a local structure similar to those in known narrow-band phosphors have been extracted. Kim et al. extracted crystal structures with cubic-like local structures by qualitatively screening the crystal structures registered in Inorganic Crystal Structure Database (ICSD) [24] under various conditions using cubic structure as a reference structure [25].

To expand the search area, it is necessary to prepare distorted local structures with low symmetry as a reference structure. Therefore, a search for host crystals containing the target local structure based on the quantitative similarity of local structures was required. Previously, we proposed a quantitative dissimilarity measure of local structures in inorganic crystals using the Wasserstein distance, which is often used in data science [26]. In this method, the local structure is defined as a geometric polyhedron consisting of the central ion and its surrounding ligands. The polyhedron is represented by a distribution of interatomic distances consisting of center-ligand and ligand-ligand distances. The obtained Wasserstein distance indicates the minimum deformation to change the local polyhedral structure. The smaller the value of the Wasserstein distance, the more similar the structures. Therefore, more candidates for narrow-band emissions can be extracted from crystal structure databases using this dissimilarity.

In this study, we searched for host crystals which show a narrow-band emission from ICSD

based on the dissimilarity of local structures, using the local structures in known narrow-band phosphor hosts as reference structures. As a result, we discovered $\text{K}_2\text{ZnP}_2\text{O}_7$ as a host crystal and developed a new narrow-band phosphor $\text{K}_2\text{ZnP}_2\text{O}_7:\text{Eu}^{2+}$.

First, we screened 200 thousand crystal structures registered in ICSD. Metallic compounds and hydrogen-containing crystal structures were excluded because they are not suitable as host crystals for phosphors. Additionally, solid solutions, partially occupied crystal structures, and crystal structures containing atoms with non-integer charges were excluded because determining the coordination number of the local structure is difficult. From the remaining ionic crystals, 6–14 coordinated local structures consisting of the central alkali metal or alkaline-earth metal ion and ligands were extracted. In many reported Eu^{2+} phosphors, Eu^{2+} substitutes for alkali or alkaline earth ions sites since its ionic radius is close to those of alkali or alkaline earth ions. The coordination numbers were determined using the CrystalNN method by Zimmermann et al. [27] Table 1 shows the number of extracted local structures by metal ion.

We prepared six references of local structures: ideal cubic local structure, the two Sr-sites in $\text{SrLiAl}_3\text{N}_4$, the nine-coordinated structure of $\beta\text{-SiAlON}$, the Ba-site in $\text{BaSi}_2\text{O}_2\text{N}_2$, and the Ba-site in $\text{BaLi}_2\text{Al}_2\text{Si}_2\text{N}_6$. The first-order Wasserstein distance was calculated for all pairs of extracted and reference local structures using the SciPy package [28]. To search for similar local structures to the reference structures, we visualized the distributions of all the local

structures as a scatterplot on a 2-dimensional (2D) plane using the t-distributed stochastic neighbor embedding (t-SNE) method [29]. The t-SNE method transforms a data distribution into lower dimensions, usually 2D or 3D for visualization, keeping the distance distribution of neighbors. Therefore, data points with similar local structures are placed closer and tend to form a cluster. A scatterplot was created for each center ion. Figure 1 shows the scatterplot of the K-centered local structures. The 2D scatter plots for Na-, Rb-, Cs-, Ca-, Sr-, and Ba-centered local structures are summarized Figure S1-S6 in the Supporting Information. Each data point indicates one local structure, while the color denotes the coordination number. As can be seen, the data points are generally grouped by coordination number.

In Figure 1, the K sites of simple halides such as KCl and KI with the CsCl-type structure, which are high-pressure phases, were located near the reference ideal cubic structure. Most of the host crystals with the local structures located near the Sr sites of $\text{SrLiAl}_3\text{N}_4$ contain transition metal, radioactive element, or toxic element, and thus they are unsuitable for Eu^{2+} phosphors. Other structures near the Sr sites of $\text{SrLiAl}_3\text{N}_4$ were known phosphors including the narrow-band phosphor $\text{CsKNaLi}(\text{Li}_3\text{SiO}_4)_4\text{:Eu}^{2+}$, or the crystal structures with cubic-like local structures listed by Kim et al. [24] There is no structure near the nine-coordinated structure of $\beta\text{-SiAlON}$ and the Ba-site in $\text{BaLi}_2\text{Al}_2\text{Si}_2\text{N}_6$. These two local structures are unique. The K1-site of $\text{K}_2\text{ZnP}_2\text{O}_7$ is located near the Ba-site of the narrow-band phosphor $\text{BaSi}_2\text{O}_2\text{N}_2$ in the enlarged

plot of Figure 1 which indicates only the eight-coordinated local structures. $\text{K}_2\text{ZnP}_2\text{O}_7$ has two K-sites, and the K2-site is also near the Ba-site in $\text{BaSi}_2\text{O}_2\text{N}_2$. The band gap of $\text{K}_2\text{ZnP}_2\text{O}_7$ is 4.134 eV [30]. It is suitable for phosphors which emitting visible-light, therefore, we performed a synthesis of the $\text{K}_2\text{ZnP}_2\text{O}_7:\text{Eu}^{2+}$ phosphor and measured its luminescence properties.

$\text{K}_2\text{ZnP}_2\text{O}_7:\text{Eu}^{2+}$ was synthesized by a solid-state method. Starting materials of KH_2PO_4 (FUJIFILM Wako Pure Chemical, 2N5), ZnO (Kojundo Chemical, 4N), and Eu_2O_3 (Shin-etsu Chemical, 3N) were mixed at a K:Eu:Zn ratio of 1.96:0.02:1 and fired in a tube furnace at 650 °C for 4 hours under a reducing atmosphere (5% H_2/N_2). The product was characterized by Powder x-ray diffraction (XRD) (Rigaku, Smartlab, $\text{Cu K}\alpha_1$ radiation). The XRD analysis indicated that the product was a mixture of $\text{K}_2\text{ZnP}_2\text{O}_7$, KPO_3 and KZnPO_4 as illustrated in Figure S7 in the Supporting Information. Therefore, a single particle of $\text{K}_2\text{ZnP}_2\text{O}_7:\text{Eu}^{2+}$ was picked up from the product and analyzed [31,32]. Figure 2 shows the image of the selected $\text{K}_2\text{ZnP}_2\text{O}_7:\text{Eu}^{2+}$ particle. It shows blue luminescence under ultraviolet (UV) irradiation.

The single crystal XRD data of the particle were collected using a diffractometer (Rigaku, XtaLab Synergy-R-custom) with $\text{Mo K}\alpha$ radiation ($\lambda = 0.71073 \text{ \AA}$). The data were integrated and corrected for absorption using CrysAlisPro. The crystal structure was solved and refined with SHELX[33,34]. The crystal structure of this new phosphor has a tetragonal unit cell with $a = b = 7.84328(13) \text{ \AA}$, $c = 11.3195(3) \text{ \AA}$, and $P4_2/mnm$ space group (No.136). The refined

structural parameters are shown in Tables S1 and S2 in the Supporting Information, and they are in good agreement with the literature [35]. Figure 3 shows the crystal structure of $\text{K}_2\text{ZnP}_2\text{O}_7$ and the local structures of the K1- and K2-sites. The local structure of the Ba-site of $\text{BaSi}_2\text{O}_2\text{N}_2$ is also shown in Figure 3 for comparison. The eight-coordinated K1- and K2-sites have a C_{2v} symmetry with average bond lengths of 2.799 Å and 2.819 Å, respectively. The K1-site looks very similar to the Ba-site of $\text{BaSi}_2\text{O}_2\text{N}_2$.

Figure 4 shows the emission and excitation spectra from the particle of the $\text{K}_2\text{ZnP}_2\text{O}_7\text{:Eu}^{2+}$ phosphor. The excitation and emission spectra of the particle were obtained using a spectrometer (Otsuka electronics, MCPD7700) through a microscope (Olympus, BX51M) under excitation of 150-W Xenon light (Bunkoukeiki, SM-10ND). The emission spectrum had a peak at approximately 440 nm with a FWHM of 30 nm ($1,549\text{ cm}^{-1}$). This peak is attributed to the electric dipole transition from $4f^65d^1$ to $4f^7$ of Eu^{2+} . The observed FWHM is comparable to other reported narrow-band phosphors ($\text{BaSi}_2\text{O}_2\text{N}_2\text{:Eu}^{2+}$, $1,430\text{ cm}^{-1}$; $\text{SrLiAl}_3\text{N}_4\text{:Eu}^{2+}$, $1,137\text{ cm}^{-1}$; $\text{BaLi}_2\text{Al}_2\text{Si}_2\text{N}_6\text{:Eu}^{2+}$, $2,016\text{ cm}^{-1}$). The excitation spectrum spanned from 300–400 nm. Two exponential curves fit the emission decay curve shown in Figure 5. The luminescence decay was measured by a streak camera (Hamamatsu Photonics, C14831-110) under excitation of a 365-nm pulsed laser through a microscope (Olympus, BX51M). The decay times were 0.19 μs (10%) and 0.70 μs (90%). Since the $\text{K}_2\text{ZnP}_2\text{O}_7$ crystal has two K-sites, this result is

reasonable and suggests that Eu^{2+} is preferentially substituted for one of the two K-sites.

Generally, a single substitution site for Eu^{2+} is preferred for narrow-band emission. Although $\text{K}_2\text{ZnP}_2\text{O}_7$ has two substitution sites, doped Eu^{2+} showed narrow-band emission. There are two possible origins for this narrow-band emission. One is that Eu preferentially substitutes for one K-site as observed in the decay curve. Another possible origin is the similar local structures of the K1- and K2-sites, leading to the restraint of inhomogeneous broadening. $\text{SrLiAl}_3\text{N}_4:\text{Eu}^{2+}$ is a well-known example: it has a very small FWHM, even though there are two types of Sr-sites. The dissimilarity between the K1- and K2-sites of $\text{K}_2\text{ZnP}_2\text{O}_7:\text{Eu}^{2+}$ was 0.042. We have set a threshold at 0.047 for similar local structures in our previous work based on the dissimilarity between the cubic and square antiprism structures.²⁶ Based on this threshold, the K1- and K2-sites are considered to have similar local structures to each other. Further research is necessary to clarify which origin is dominant for the narrow-band emission of $\text{K}_2\text{ZnP}_2\text{O}_7:\text{Eu}^{2+}$, but it is beyond the scope of this study.

In conclusion, we extracted the $\text{K}_2\text{ZnP}_2\text{O}_7$ crystal structure from ICSD as a host crystal of the Eu^{2+} narrow-band phosphor based on quantitative evaluation of dissimilarity between local structures using Wasserstein distance and visualization by t-SNE dimensionality reduction. Furthermore, we developed a new narrow-band phosphor $\text{K}_2\text{ZnP}_2\text{O}_7:\text{Eu}^{2+}$, which shows a blue luminescence peak at approximately 440 nm with the FWHM of 30 nm ($1,549\text{ cm}^{-1}$). The

narrowness of the FWHM is comparable to other reported UCr_4C_4 -type narrow-band phosphors. Because this approach can search for host crystals efficiently and quantitatively, it should lead to discovering new phosphors with specific luminescence properties.

Supporting Information

Each 2D t-SNE plot of the Na, Rb, Cs, Ca, Sr and Ba-centered local structures. The powder XRD pattern and the refined single crystal structural parameters.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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Table 1. Number of local structures of each central ion used in this work.

Center ion	Number of local structures (CN = 6-14)
Na^+	4,609
K^+	5,290
Rb^+	2,512
Cs^+	3,301
Ca^{2+}	3,711
Sr^{2+}	3,198
Ba^{2+}	5,364

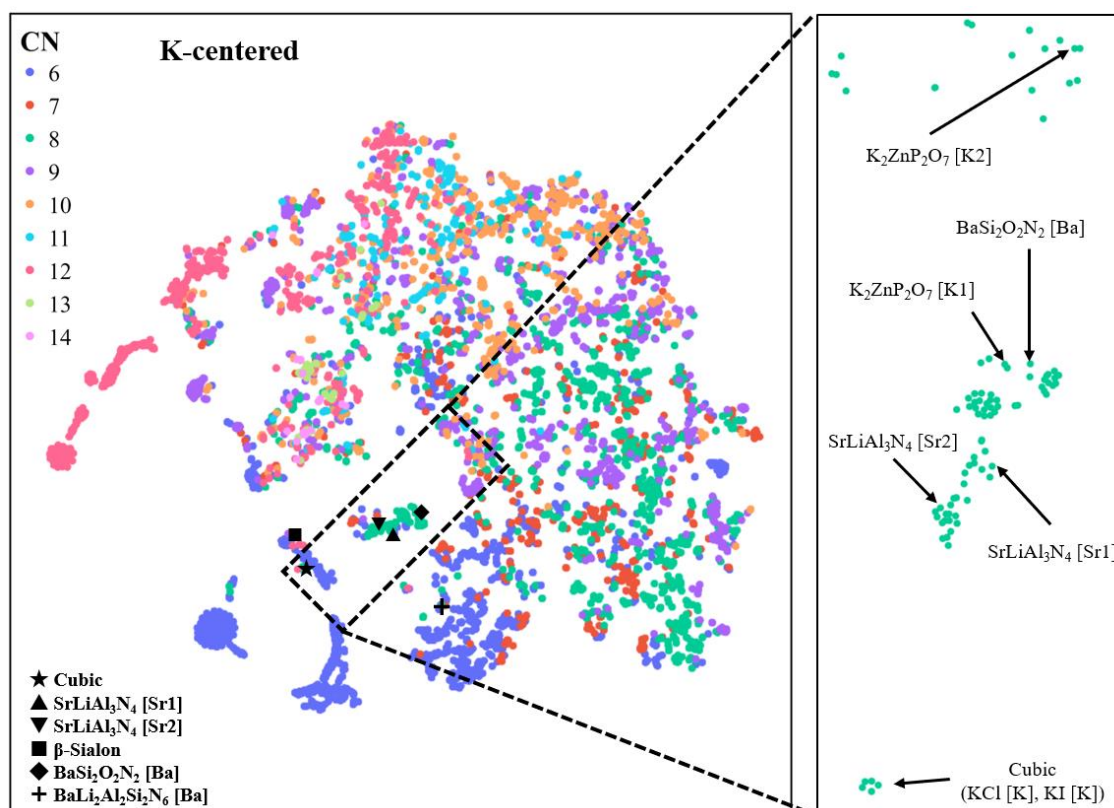


Figure 1. 2D t-SNE plot of the K-centered local structures. Enlarged view indicates only eight-coordinated local structures in the region enclosed by the black dotted line.

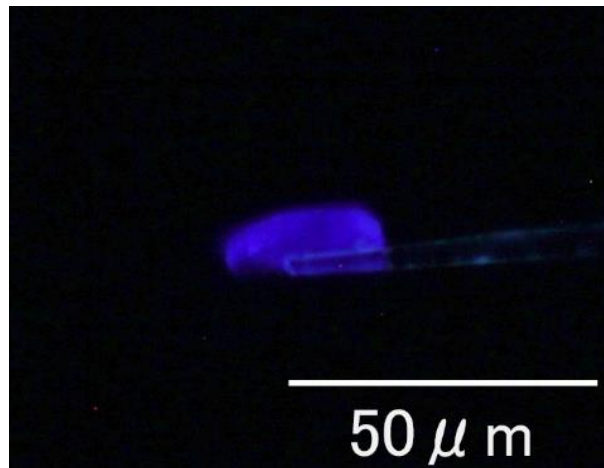


Figure 2. Single particle of the $\text{K}_2\text{ZnP}_2\text{O}_7:\text{Eu}^{2+}$ phosphor emitting blue color under UV irradiation.

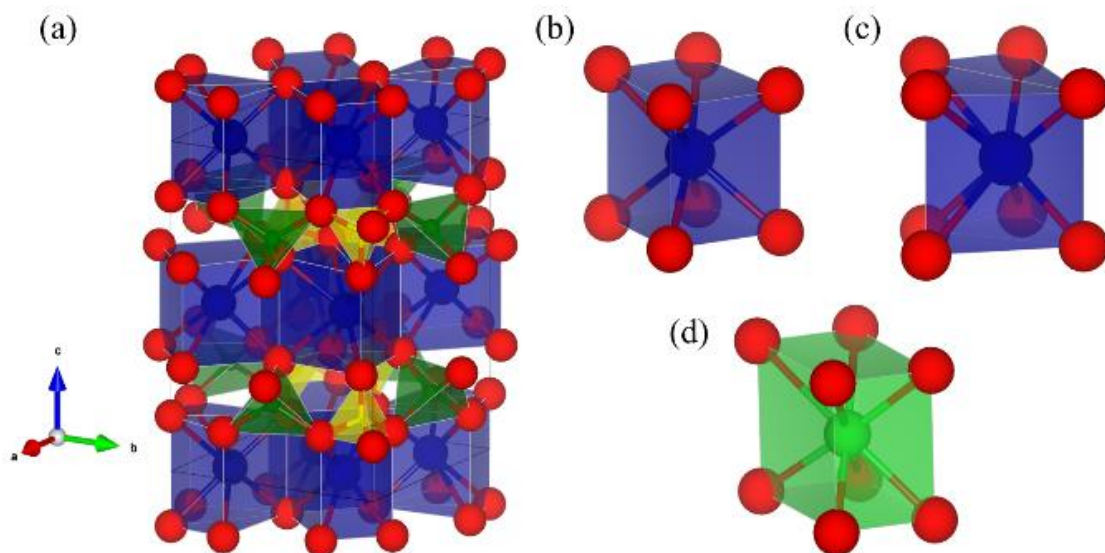


Figure 3. (a) Crystal structure of $\text{K}_2\text{ZnP}_2\text{O}_7$. Blue, green, and yellow polyhedra are KO_8 , ZnO_4 , and PO_4 , respectively. (b, c) Local structures of the K1- and K2-sites, respectively. (d) Local structure of the Ba-site of $\text{BaSi}_2\text{O}_2\text{N}_2$.

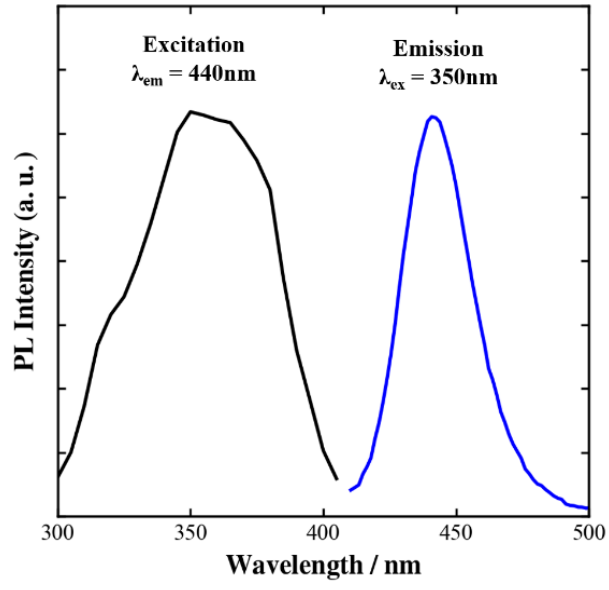


Figure 4. Emission (blue line, $\lambda_{\text{ex}} = 350\text{ nm}$) and excitation (black line, $\lambda_{\text{em}} = 440\text{ nm}$) spectra from a single particle of $\text{K}_2\text{ZnP}_2\text{O}_7:\text{Eu}^{2+}$.

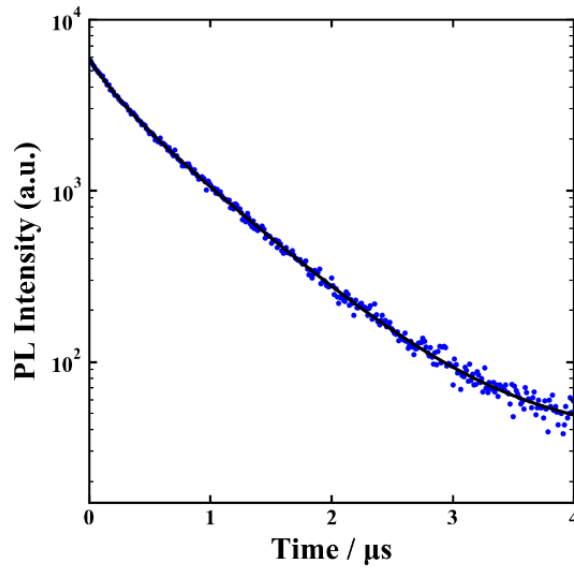


Figure 5. Observed (blue dot) and fitted (black line) decay curves of one $\text{K}_2\text{ZnP}_2\text{O}_7:\text{Eu}^{2+}$ particle upon exciting with 350-nm pulsed light.