Near-infrared luminescent materials exhibit unique photophysical properties that make them crucial components in photonic, optoelectronic and biological applications. As broadband near infrared phosphors activated by transition metal elements are already widely reported, there is a challenge for next-generation materials discovery by introducing rare earth activators with 4f-5d transition. Here, we report an unprecedented phosphor $K_3LuSi_2O_7:Eu^{2+}$ that gives an emission band centered at 740 nm with a full-width at half maximum of 160 nm upon 460 nm blue light excitation. Combined structural and spectral characterizations reveal a selective site occupation of divalent europium in $LuO_6$ and $K_2O_6$ polyhedrons with small coordination numbers, leading to the unexpected near infrared emission. The fabricated phosphor-converted light-emitting diodes have great potential as a non-visible light source. Our work provides the design principle of near infrared emission in divalent europium-doped inorganic solid-state materials and could inspire future studies to further explore near-infrared light-emitting diodes.
ear-infrared (NIR) light source has attracted attention for applications in medical fields, bio-sensing, food processing industry, and night-vision technologies. Traditional NIR light sources, such as halogen lamps and tungsten-halogen lamps, suffer from large sizes, low efficiency, short lifetime, and heat dissipation. As an emerging field, researchers have made considerable efforts to explore new NIR emitters, such as InAs-ZnSe polymer nanocrystal, CH3NH3PbI3, organometal halide perovskite, PbSe/MAPbX3 (X = I, Br) quantum dots, and Pr(II) complex based organic light-emitting diodes (LEDs). However, the low efficiency, poor stability or complex fabrication technique restrict their development. As a contrast, the high luminous efficiency, long operating life, and compact sizes make NIR LEDs useful, however, a narrow full-width at half maximum (FWHM) below 50 nm seriously limit their applications in 3D sensing, food analyzing, and other specific fields. Therefore, discovery of broadband-emitting NIR light sources is a necessary and urgent requirement. In this regard, phosphor-converted light-emitting diodes (pc-LEDs) have attracted increasing interests considering more opportunities for tunable emission during the phosphors’ screening, and availability of simpler strategies for both materials design and fabrication of NIR pc-LEDs. Furthermore, the low cost of NIR phosphors is beneficial. However, one of the most significant challenges is the discovery of blue light pumped and efficient broadband NIR-emitting phosphors.

Numerous studies on exploring NIR phosphors have been realized by doping transition metal ions (Cr3+, Ni2+, and Mn4+), Bi3+ or rare earth ions (Pr3+, Nd3+, Tb3+, and Yb3+) into inorganic host materials. However, the sharp line emission of rare earth ions with 4f-4f transition is not broad enough for the desired applications, and the absorption region is also very narrow and weak. The Ni2+ doped materials show broad emission band around NIR region, but the low efficiency and IR laser excitation suspends the application. Recently, Cr3+ ion has been considered as an ideal NIR luminescent center, which can exhibit broad-band emission ~650–1200 nm with 4T1 → 4A2 transition. However, mixed valence of Cr6+ ions simultaneously exist in most Cr3+ doped phosphors, which seriously quench NIR luminescent efficiency. As one of important rare earth activators with 4f-5d transition, Eu2+ -activated phosphors normally possess high efficiency and have been applied in the field of commercial white LEDs. Moreover, the tunable emission in the visible range is very popular to discover useful phosphors, and the typical examples include Sr[LiAl3N4]:Eu2+ (red), BaSiON:Eu2+ (green) and BaMgAl10O17:Eu2+ (blue). If Eu2+-doped phosphors can realize NIR emissions, it would launch a new era in NIR light source. However, in addition to the recent report on Ca3Sc2Si3O12:Eu2+, no other Eu2+-doped phosphors can achieve NIR emissions with the peak beyond 700 nm. Unfortunately, though Ca3Sc2Si3O12:Eu2+ gives a broad-band emission ranging from 720 to 1100 nm, it cannot be pumped by blue LEDs due to the lack of blue light absorption of the sample. Therefore, discovery of Eu2+ -activated NIR phosphors for blue light-pumped LEDs is an essential and difficult task for the emerging photonic, optoelectronic and biological applications.

The emission energy distribution of Eu2+ is generally related with the average bond lengths, coordination numbers and the symmetry of the cations and corresponding polyhedron occupied by Eu2+. Many strategies, including neighboring-cation substitution, chemical unit cosubstitution and cation-size-mismatch have been used to tune visible photoluminescence of Eu2+ ions. However, there is still a challenge to realize the NIR emission for the difficulties of obtaining strong crystal field splitting (CFS) and large centroid shift originating from Eu2+. Recently, our group has reported a new approach showing that Eu2+ selectively occupies a site with small coordination number, which tends to large redshift as found in Rb2YSi2O7:Eu2+ with red emission. If the cations’ sites occupied by Eu2+ in Rb2YSi2O7 are substituted by smaller ions, this effect will produce a larger CFS and even realize NIR emission. Accordingly, we have designed a Eu2+-doped K2LiSi2O7 NIR phosphor from isostructural Rb2YSi2O7:Eu2+ by substituting the large Rb+ and Y3+ ions with small K+ and Lu3+ ions to further increase the CFS effect.

Here in this work, we show that K2LiSi2O7:Eu2+ is the first Eu2+-doped NIR phosphor under 460 nm blue light excitation and gives a broad-band emission peak at 740 nm in the NIR region (600–900 nm). Structural and spectral analysis results indicate that the selective Eu2+ site occupation in LuO2 and K2O polyhedrons contributes to the NIR light emission in K2LiSi2O7:Eu2+. The as-fabricated phosphor-converted LEDs have the potential to be used for non-visible light source, and the result could initiate the exploration of Eu2+-doped NIR phosphors and other NIR spectroscopy applications.

Results

Crystal structure of K2LiSi2O7:Eu2+. Figure 1a shows the simulated X-ray diffraction (XRD) profile of K2LiSi2O7 and the measured XRD patterns for the K2LiSi2O7:Eu2+ (x = 0–0.05) with different Eu2+-doping concentrations. All diffraction peaks can be well indexed by hexagonal cell (P63/jmmc), and the parameters are similar to those of K2LiSi2O7 phase, confirming the successful synthesis of K2LiSi2O7:Eu2+ phosphors. To further understand the phase structure and sites occupation of Eu2+ ions, Rietveld refinement were performed for K2LiSi2O7:Eu2+ (x = 0, 0.01, 0.02, and 0.04), as shown in Fig. 1b and Supplementary Fig. 1. All the refinements demonstrated low R-factors (Supplementary Table 1). Moreover, Supplementary Tables 2 and 3 listed the parameters of atoms’ coordinates and bond lengths of the studied phases for the comparison.

K2LiSi2O7 host (Fig. 1c) offers one Lu sites with 6-fold coordination, and two different K sites: K1 (9-fold) and K2 (6-fold), for the possible occupation by Eu2+ ions. The cell volume increases with x (Fig. 1d), indicating that some Eu2+ ions occupy Lu sites since the radius of Lu3+ is smaller than that of Eu2+ (Supplementary Table 4). However, the unit cell volume, V(x) shows a nonlinear increasing trend with increasing Eu content, thus another occupation mechanism should exist simultaneously, which competes with the main trend and reduces cell volume. Such mechanism can be ascribed to K → Eu replacement because K ion is bigger than Eu ion (Supplementary Table 4). To further explore the Eu2+ selective site occupation, Fig. 1e illustrates the average bond lengths of LuO6, K1O6 and K2O polyhedron in different concentration of Eu2+ doped K2LiSi2O7 by extracting the data from Rietveld refinement. With increasing Eu2+ doping concentration, the average bond lengths of K1-O remain nearly unchanged, while the bond lengths of K2-O and Lu-O change significantly. In addition, the d(K2-O) and d(Lu-O) exhibit the same trend. It is owing to the fact that the occupation of Eu in Lu sites will lead to longer d(Lu-O) bond length, simultaneously, the amount of Eu in K2 sites is reduced and thus d(K2-O) bond lengths also become longer. Therefore, one can conclude that the main doping mechanism is ascribed to the synergetic effect of Lu → Eu and K → Eu replacements in K2LiSi2O7, which is similar to the site occupation of Eu2+ ions in isomorphic Rb2YSi2O7. The scanning electron microscopy (SEM) images and elemental mapping images in Supplementary Fig. 2 reveal that K2LiSi2O7:Eu2+ microcrystals are in well crystallized and K, Lu, Si, and O elements are homogeneously distributed.

Photoluminescence properties of K2LiSi2O7:Eu2+. Figure 2a gives the room temperature photoluminescence emission (PL)
and excitation (PLE) spectra of K₃LuSi₂O₇:0.01Eu²⁺. When monitored at 740 nm, K₃LuSi₂O₇:0.01Eu²⁺ gives an ultra-broad excitation band and a maximum excitation peak is found ~460 nm in the range of 250 and 600 nm. Such a broad excitation band should be ascribed to the 4f → 5d transition of two different Eu²⁺ sites in K₃LuSi₂O₇, as verified by the structural analysis discussed previously, and some sharp lines are related with the spectrophotometer itself. Supplementary Figure 3a illustrates the diffuse reflection spectra of Eu²⁺ doped and undoped K₃LuSi₂O₇ samples. K₃LuSi₂O₇ host shows nearly no absorption from 300 to 800 nm, however, K₃LuSi₂O₇:Eu²⁺ exhibits an ultra-broad absorption band from 250 to 600 nm with the introduction of Eu²⁺ in the host. K₃LuSi₂O₇ exhibits a white body color under natural light suggesting a band gap of about 5 eV (Supplementary Fig. 3b). The diffuse reflection spectra of K₃LuSi₂O₇:Eu²⁺ are in accordance with the excitation spectrum discussed above. Such a broad absorption band from ultraviolet to visible light region leads to deep red body color under natural light (the inset of Fig. 2a) and favors its application for blue light-pumped pc-LEDs.

Thus, the NIR emission spectrum under 460 nm excitation upon the excitation of 460 nm is exactly deconvoluted into two Gaussian spectral profiles with two peaks at 13900 cm⁻¹ (719 nm) and 12750 cm⁻¹ (784 nm), suggesting that Eu³⁺ substitute two different cations in K₃LuSi₂O₇ (Fig. 2c). Figure 2d shows the PL decay curves of K₃LuSi₂O₇:0.01Eu²⁺ measured at low temperature (80 K) and at room temperature (300 K) under 450 nm pulse laser diode excitation. The decay curves were comparatively fitted by mono- (n = 1), bi- (n = 2) and triple- (n = 3) exponential functions:

\[ I = \sum_{i=1}^{n} A_i \exp \left( -\frac{t}{\tau_i} \right), \quad (n = 1, 2, 3) \]  

where \( I \) denotes luminescence intensity, \( t \) represents time, \( \tau_i \) is lifetime for different components; and \( A_i \) is the corresponding fitting constants. As compared in Fig. 2d, the decay curve measured at 80 K is well fitted by both the bi- and triple-exponential function, and two fitting curves nearly overlap. However, the decay curves at 300 K can be only fitted by the triple-exponential function, which is ascribed to an additional room temperature nonradiative transition owing to the lattice thermal vibration. Accordingly, the average lifetime values are 1.58 μs (at 300 K) and 2.28 μs (at 80 K), respectively. The detailed fitting results are presented in Supplementary Table 5. Moreover, the decay times are shortened with increasing Eu concentration (Supplementary Fig. 5a) due to the energy transfer between different Eu²⁺ luminescent centers. Upon the excitation of 340 nm pulse laser diode, the average lifetime (1.69 μs) is longer than that under 450 nm excitation (1.58 μs) (Supplementary Fig. 5b and Supplementary Table 5), which is attributed to the more contribution of long-lived emitters to average decay time under 340 nm excitation.
To evaluate the robustness of phosphors to heat and moisture, two sets of K$_3$LuSi$_2$O$_7$:Eu$^{2+}$ samples were exposed to the extremely environmental condition at 80% relative humidity and 80 °C. The results suggest that the chemical stability of K$_3$LuSi$_2$O$_7$:Eu$^{2+}$ is relatively good (see details in Supplementary Fig. 6). The Eu$^{2+}$ content rarely changed to Eu$^{3+}$ depending on the prolonging duration time suggesting the good stability. One can also note that a small amount of Eu$^{3+}$ can be detected for the freshly prepared materials (Supplementary Fig. 7). Samples with different Eu$^{2+}$-doping concentrations (x = 0.005–0.05) have been prepared to optimize the PL intensity of K$_3$LuSi$_2$O$_7$:xEu$^{2+}$. As displayed in Supplementary Fig. 8a, the emission intensities increase slightly till up to x = 0.01 and then decrease with further increase in x. Based on the Dexter’s theory and the Eu-Eu distances in K$_3$LuSi$_2$O$_7$:Eu$^{2+}$, energy transfer among the nearest neighbor ions accounts for the observed concentration quenching (see details in Supplementary Fig. 8a). The internal quantum efficiency of selected K$_3$LuSi$_2$O$_7$:0.01Eu$^{2+}$ is measured to be about 15% (λ$_{em}$ = 460 nm). Furthermore, one can find that the normalized emission peaks of K$_3$LuSi$_2$O$_7$:xEu$^{2+}$ show no shift as Eu$^{2+}$ doping content increase (Supplementary Fig. 8b).

**Mechanism of NIR emission in K$_3$LuSi$_2$O$_7$:Eu$^{2+}$.** First of all, we need to correlate the Eu$^{2+}$ site contribution and different emission center by considering local environments of activators. The Eu$^{2+}$ emission is dependent on the CFS. In general, when the average bond length ($d_{av}$) is shorter and the distortion indices ($D$) is larger, then there will be a larger CFS$^{35}$. The average bond lengths, $d$(Lu-O) and $d$(K2-O) in K$_3$LuSi$_2$O$_7$ are 2.22 Å and 2.77 Å, respectively. The distortion indices are 0 for both LuO$_6$ and K2O$_6$ polyhedron. Thus, we believe that the higher energy emission (719 nm) peak is associated with Eu$^{2+}$ occupying K2 sites, while the lower energy emission (784 nm) peak is attributed to Eu$^{2+}$ occupying Lu sites. The conclusions of the site occupation for Eu$^{2+}$ in K$_3$LuSi$_2$O$_7$ are consistent with the results in Rb$_3$YSi$_2$O$_7$:Eu$^{2+}$ phosphor reported by our group$^{31}$. It is well-known that the free Eu$^{2+}$ ions possess a large energy gap (4.2 eV) between the 5d excited state and the 4f ground state$^{36}$. Once Eu$^{2+}$ are introduced into a given compound, the resulting emission wavelengths are affected by centroid shift, crystal splitting, and Stokes shift effects$^{37}$. For K$_3$LuSi$_2$O$_7$:Eu$^{2+}$ phosphor, the centroid shift and Stokes shift have weak effect on the redshift because of the low covalence between Eu$^{2+}$ and O$^{2-}$ ions and the highly symmetric LuO$_6$ and K2O$_6$ polyhedrons, respectively$^{38,39}$. The CFS depends especially on the coordination numbers, distortion index and size of the polyhedrons$^{38}$. The substitution of Eu$^{2+}$ ions in LuO$_6$ and K2O$_6$ polyhedrons, and thus a design principle of selective occupation of Eu$^{2+}$ at small coordination numbers takes effect. It results in large CFS, which in turn leads to a large redshift. A comparison of the local environment of Eu$^{2+}$ ions in K$_3$LuSi$_2$O$_7$ and Rb$_3$YSi$_2$O$_7$ benefits further understanding of the abnormal NIR emission in K$_3$LuSi$_2$O$_7$. Firstly, they are isomorphic, and Eu$^{2+}$ ions occupy the same crystallographic lattices in both materials, and the distortion index of YO$_6$/LuO$_6$ and K2O$_6$ polyhedron are all 0$^{31}$. Thus, the effect of polyhedral distortion on the NIR emission can be neglected. Secondly, the $d_{av}$ values of Lu-O and K2-O in K$_3$LuSi$_2$O$_7$ are 2.22 Å and 2.77 Å, respectively, which are much smaller than that of Y-O and K2-O (2.29 Å and 2.86 Å) in Rb$_3$YSi$_2$O$_7$, resulting in a larger CFS in 5d energy levels.
and a larger redshift\cite{40}. Therefore, the NIR light emission (740 nm) in K₃LuSi₂O₇:Eu²⁺ is reasonable compared with the red emission (622 nm) in Rb₂YSi₂O₇:Eu²⁺. In summary, the selective occupation of Eu²⁺ ions in polyhedrons with small coordination numbers and small average bond length contribute to the NIRM emission and the broad excitation band in K₃LuSi₂O₇:Eu²⁺.

**Thermal quenching of PL.** Normally, the thermal effect of the NIR phosphor is crucial for the LED application, and thus the thermal quenching behavior of K₃LuSi₂O₇:Eu²⁺ was investigated at different temperature from 80 to 500 K. Figure 3a demonstrates that the emission intensity decreases slowly with the increase of temperature owing to the enhanced nonradiative transition probabilities. As given in Fig. 3b, the integrated intensity at 150 °C retains ~59% of that at room-temperature. According to Dorenbos’s model, the activation energy $\Delta E$ was calculated by using the following empirical formula:\cite{40,41}

$$\Delta E = \frac{T_{0.5} \text{ eV}}{680}$$

where $T_{0.5}$ denotes the quenching temperature, and it is defined as the temperature when the emission declines to half of its initial intensity. Here, $\Delta E$ is calculated to be ~0.65 eV.

One can find from Fig. 3a that the emission peaks shift to shorter wavelength (blueshift) at evaluated temperature from 300 to 500 K. Furthermore, the temperature-dependent PL spectra under excitation of 460 nm of K₃LuSi₂O₇:0.01Eu²⁺ were measured from 80 to 500 K, and the normalized spectra are presented in Fig. 3c. The emission peaks demonstrate a remarkable blueshift from 790 nm (80 K) to 710 nm (500 K) with increasing temperature, and the FWHM of emission bands gradually broadens with the increase of temperature. These observations are associated with the different response of Eu²⁺ ions located at the Lu and K2 sites to the temperature. In addition, the emission peaks at high temperature (790 nm) and low temperature (710 nm) are close to the two Gaussian fitted peaks at room temperature (784 nm, 719 nm) in Fig. 2c. This further validates the feasibility of the previous analysis about the site occupation of Eu²⁺ ions.

Thus, a simple schematic Eu²⁺ energy level diagram in K₃LuSi₂O₇ host is depicted in Fig. 3d. Based on the thermal ionization model proposed by Dorenbos, the smaller the gap (photoionization barrier $\Delta E_A$) between the bottom of conduction band and the upper edge of 5d energy level, the greater is the thermal quenching\cite{40}. In K₃LuSi₂O₇:Eu²⁺, the larger CFS of Eu²⁺ occupied in Lu site produces a smaller $\Delta E_A$, and thus a poor thermal stability compared with Eu²⁺ in the K2 site (Fig. 3d). Similarly, the larger CFS of Eu²⁺ in K₃LuSi₂O₇ results in a smaller $\Delta E_A$, and thus it enables a poor thermal stability compared with that of Rb₂YSi₂O₇:Eu²⁺\cite{31}. Therefore, the blueshift occurred when heated. At low temperature of 80 K, the thermal ionization of 5d electrons becomes weak, and the dominant emission peak centers at longer wavelength (790 nm) associated with the more contribution from Eu²⁺ ions occupying Lu sites. In contrast, thermal quenching behavior at high temperatures has a much weaker effect on the Eu²⁺ ions in K2 sites than on the Eu²⁺ ions in Lu sites, resulting in a relatively stronger emission at short wavelength. Therefore, with temperature increasing, the blueshift and the broadening of emission spectra occur simultaneously.

**NIR pc-LED device and applications.** A pc-LED with NIR emission was fabricated by combining K₃LuSi₂O₇:Eu²⁺ phosphor and the commercial blue light-emitting InGaN chip (460 nm). The emission spectrum of the lighted lamp was monitored by a
visible-NIR continuous fiber spectrophotometer. The broad-band PL spectrum and the as-obtained and lighted NIR pc-LED lamps are given in Fig. 4a. The slight spectral deviation from the lamp and the powder is related with the calibration difference of the used spectrometers. Figure 4b demonstrates the PL spectra of LED device upon different forward bias currents (20–120 mA). The NIR emission output power increase with the increase of forward currents and reaches 21.5 mW when the forward current is 100 mA (Supplementary Table 6). Figure 4c shows the photographs obtained by different cameras under natural light and NIR pc-LED light, respectively. Nothing can be detected by NIR camera once the NIR pc-LED is off. In contrast, the NIR camera can capture black-and-white images while the NIR pc-LED lamp is lighted. These results indicate the application of the K₃LuSi₂O₇:Eu²⁺ phosphor in night-vision technologies, and other potential fields including medical and food industries can be also expected for such NIR phosphors.

**Discussion**

To conclude, we have designed and successfully synthesized the Eu²⁺-activated broad-band-emitting NIR phosphor with super-broad excitation band from 250 to 600 nm. Under 460 nm blue light excitation, K₃LuSi₂O₇:Eu²⁺ shows an unprecedented broad NIR emission band peaking at 740 nm and also demonstrates a high FHWM of 160 nm. Eu²⁺ selectively occupancy at Lu and K2 sites, and the small coordination numbers and small average bond lengths lead to the as-observed NIR emission with characteristic 4f-5d transition of Eu²⁺. The as-fabricated NIR pc-LEDs based on K₃LuSi₂O₇:Eu²⁺ phosphors have been applied in night-vision devices and demonstrated potential applications. This finding gives the feasibility of the feasible strategy to discover Eu²⁺ doped NIR phosphor with large redshift by occupying polyhedrons with small coordination numbers. More importantly, it initiates a new way to explore NIR phosphors, i.e. achieve NIR emission by Eu²⁺ doping in the solid-state materials.
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Author contributions
Z.X. initiated and guided the research. J.Q. synthesized the samples and wrote the manuscript, and Z.X. and Q.Z. revised it. G.Z. and Y.Z. discussed the luminescence properties and made the NIR pc-LED device. All authors discussed the results and commented on the manuscript.

Competing interests
The authors declare no competing interests.

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Correspondence and requests for materials should be addressed to Z.X.

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