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# Impact of ether coordination on the solid-state luminescence of a lithium $\beta$ -diketiminate complex†

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A solid-state luminescent lithium  $\beta$ -diketiminate complex was synthesized by the reaction of the corresponding proligand and *n*-butyllithium in hexane. The complex showed a low-coordinated structure in the solution and crystalline states. On the other hand, the lithium complex reacted with diethyl ether, tetrahydrofuran, and 1,4-dioxane to form three-coordinated complexes, which showed lower emission efficiency. Remarkably, the photoluminescence of the crystal of the low-coordinate species was quenched by the treatment with the diethyl ether vapor. NMR spectroscopy and X-ray diffraction analyses revealed that diethyl ether reacts with the complex to afford the three-coordinated ether adduct even in the crystalline state. To the best of our knowledge, this is the first report on the luminescence switching in crystalline materials based on the modification of the coordination number on a lithium atom.

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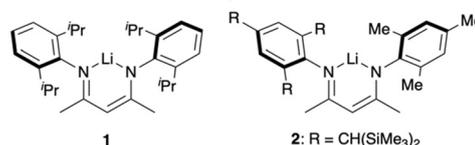
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## Introduction

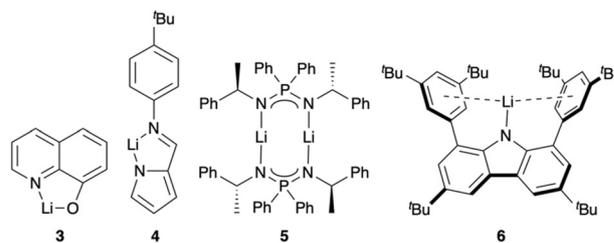
Inorganic and organometallic alkali metal compounds are intermediates of a tremendous variety of organic and organometallic compounds because of their high reactivity. The coordination environment around metals is well known to be a key factor in controlling the reactivity of compounds and the selectivity of reactions.<sup>1–13</sup> The most common coordination number of alkali metals is four. Nevertheless, over the past two decades, the isolation of two-coordinated or donor-free alkali metal complexes has been accomplished because of the development of sterically encumbered ligands.<sup>5,14–22</sup> This class of complexes exhibits strong Lewis acidity originating from their coordinative unsaturation.  $\beta$ -Diketiminato ligands are one of the important scaffolds for isolating base-free alkali metal complexes, such as **1**<sup>15</sup> and **2** (Chart 1, top).<sup>16</sup> The sterically hindered substituents on the nitrogen atoms are essential for preventing the strong interactions with Lewis bases. In addition to studies on their structures, recent studies have also focused on their catalytic activities<sup>23</sup> and luminescent properties.<sup>24–28</sup> In particular, lithium quinolinolate **3** has been studied as an emitting and electron-transporting layer in organic light-emitting diodes.<sup>24</sup> More recently, the lithium

complexes of pyrrolide **4**,<sup>25</sup> iminophosphonamide **5**,<sup>26</sup> and carbazolide **6**<sup>27</sup> have been developed, exhibiting fruitful properties (Chart 1, middle), for instance, efficient luminescence, thermally activated delayed fluorescence, and solid-state luminescence. Importantly, it was suggested that the base-free

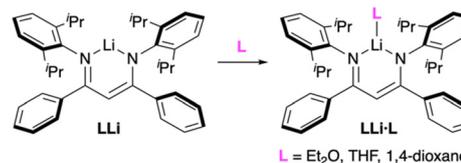
Solvent-free lithium  $\beta$ -diketiminate complexes



Luminescent lithium complexes



This work



**Chart 1** Chemical structures of selected lithium complexes and molecules investigated in this work.

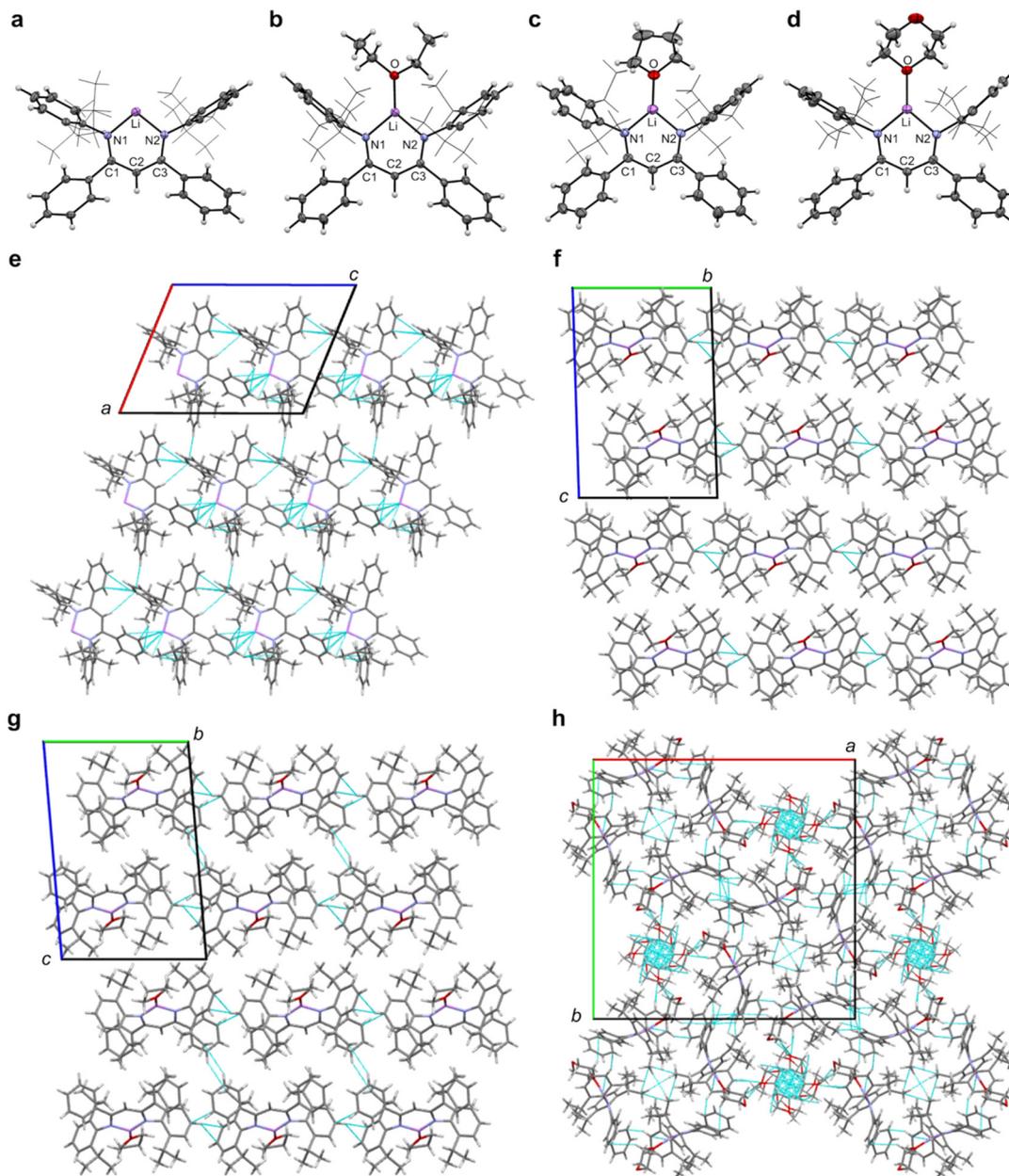
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**Fig. 1** Single-crystal structures of (a) **LLi**, (b) **LLi·OEt<sub>2</sub>**, (c) **LLi·THF**, and (d) **LLi·DOX** (50% displacement ellipsoids). Only the crystal of **LLi·DOX** was obtained as a 1 : 1 solvated crystal with 1,4-dioxane. Isopropyl groups, and the free 1,4-dioxane molecule in a solvent channel are shown in wire-frame style and omitted, respectively, for clarity. Packing structures of (e) **LLi**, (f) **LLi·OEt<sub>2</sub>**, (g) **LLi·THF**, and (h) **LLi·DOX**. Dashed cyan lines show short contacts (<sum of van der Waals radii). Atom legends: gray, carbon; white, hydrogen; blue, nitrogen; purple, lithium; red, oxygen.

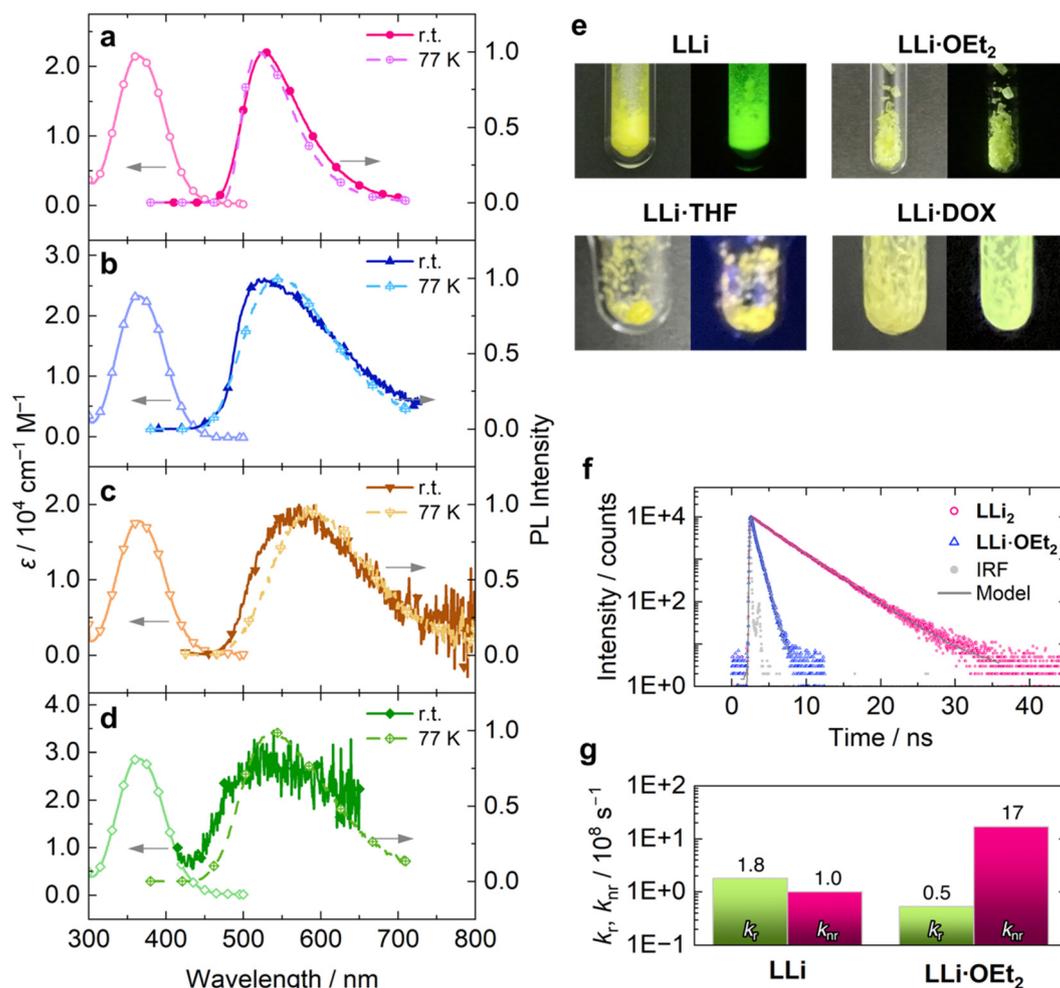
measurements (Fig. 2a–d and Table 2). All measurements were carried out under a N<sub>2</sub> atmosphere because of the instability of the complexes under aerobic conditions. All complexes showed similar absorption spectra peaked at around 365 nm. Additionally, emission from their dilute solution (2-methylpentane (2MP)/toluene = 99/1, 1 × 10<sup>−5</sup> M) was not detectable at room temperature and even in the frozen solution at 77 K. The absolute luminescence quantum yields were not determined due to small values below the detection limit (<0.01). On the other hand, intense green PL was observed from crys-

talline **LLi** even at room temperature ( $\Phi_{\text{PL}} = 0.65$ , Fig. 2e). This result clearly indicates that **LLi** possesses the CIE property, similarly to the related  $\beta$ -diketiminato complexes containing the group 13 elements.<sup>29–39</sup> The molecular motions in the excited states, like intramolecular vibrations<sup>34</sup> and structural deformations,<sup>37</sup> could open the channel for the nonradiative decay of the excited molecules in solution states, while the rigid crystalline packing could annihilate such nonradiative decay processes. Remarkably, in contrast to the base-free complex, the ether adducts exhibited only slight enhancement



**Table 1** Selected structural parameters of LLi, LLi-OEt<sub>2</sub>, LLi-THF, and LLi-DOX

LLi	LLi-OEt <sub>2</sub>		LLi-THF		LLi-DOX		
Bond	<i>d</i> /Å	<i>d</i> /Å	<i>d</i> /Å	<i>d</i> /Å	<i>d</i> /Å	<i>d</i> /Å	
Li-N1	1.901(3)	Li-N1	1.919(4)	Li-N1	1.903(3)	Li-N1	1.902(4)
Li-N2	1.881(3)	Li-N2	1.929(4)	Li-N2	1.910(3)	Li-N2	1.918(4)
N1-C1	1.329(3)	N1-C1	1.329(3)	N1-C1	1.326(2)	N1-C1	1.324(2)
N2-C3	1.321(3)	N2-C3	1.330(3)	N2-C3	1.326(2)	N2-C3	1.324(2)
C1-C2	1.411(2)	C1-C2	1.413(3)	C1-C2	1.409(2)	C1-C2	1.411(3)
C2-C3	1.413(2)	C2-C3	1.419(3)	C2-C3	1.415(2)	C2-C3	1.415(3)
		O-Li	1.929(5)	O-Li	1.899(4)	O-Li	1.918(4)
Angle	$\theta$ /°	$\theta$ /°	$\theta$ /°	$\theta$ /°	$\theta$ /°	$\theta$ /°	
N1-Li-N2	99.4(1)	N1-Li-N2	98.4(2)	N1-Li-N2	98.8(1)	N1-Li-N2	98.5(2)
		O-Li-N1	129.7(2)	O-Li-N1	132.1(2)	O-Li-N1	128.8(2)
		O-Li-N2	129.8(2)	O-Li-N2	127.1(2)	O-Li-N2	130.7(2)



**Fig. 2** UV-vis absorption spectra in dilute 2MP/toluene (99/1) solutions ( $1 \times 10^{-5}$  M) and PL spectra of crystals of (a) LLi, (b) LLi-OEt<sub>2</sub>, (c) LLi-THF, and (d) LLi-DOX. (e) Pictures of crystals of the complexes under room light (left panels) and UV irradiation (right panels). (f) PL decay profiles of the crystals of LLi (magenta open circles) and LLi-OEt<sub>2</sub> (blue open squares) upon excitation at 375 nm with an LED laser. Gray solid lines are the fitted curves. Instrument response function (IRF) is also shown (gray closed circles). (g) Calculated radiative ( $k_r$ ) and non-radiative ( $k_{nr}$ ) decay rate constants of the complexes.



Table 2 Photophysical properties of the complexes<sup>a</sup>

	Temperature	Solution <sup>b</sup>		$\Phi_{\text{PL}}^d$
		$\lambda_{\text{abs}}/\text{nm}$	$\lambda_{\text{PL}}/\text{nm}$	
<b>LLi</b>	r.t.	364	527	0.65
	77 K	— <sup>c</sup>	521	0.90
<b>LLi-OEt<sub>2</sub></b>	r.t.	363	527	0.03
	77 K	— <sup>c</sup>	543	0.14
<b>LLi-THF</b>	r.t.	366	572	0.03
	77K	— <sup>c</sup>	594	0.06
<b>LLi-DOX</b>	r.t.	364	539	0.08
	77K	— <sup>c</sup>	535	0.32

<sup>a</sup> PL spectra and absolute quantum yields were recorded with the photoexcitation at 360 nm. <sup>b</sup> 2MP/toluene (99/1, v/v) solution ( $1 \times 10^{-5}$  M). <sup>c</sup> Not determined. <sup>d</sup> Absolute PL quantum yield determined with the integration sphere method.

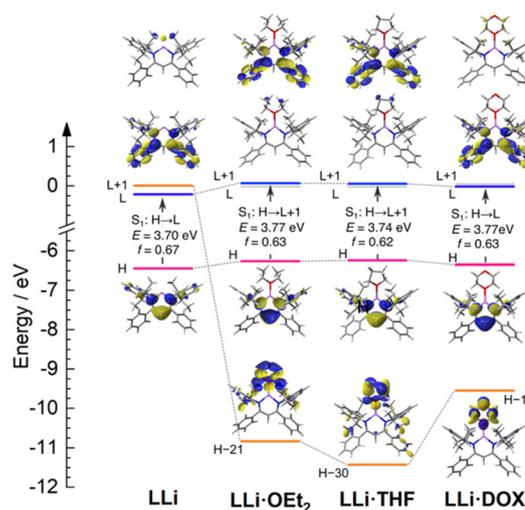


Fig. 3 Calculated Kohn–Sham (KS) orbital energy diagram, transition energy ( $E$ ), and oscillator strength ( $f$ ). KS frontier orbital distributions are shown (isovalued = 0.03). H and L denote the HOMO and LUMO, respectively.

of the emission by crystallization ( $\Phi_{\text{PL}} = 0.03$  for **LLi-OEt<sub>2</sub>**, 0.03 for **LLi-THF**, and 0.08 for **LLi-DOX**, Fig. 2e).

In order to gain further insights into the photophysical processes of the complexes, luminescence lifetimes of the crystals of **LLi** and **LLi-OEt<sub>2</sub>** were measured by the time-correlated single photon counting (TCSPC) measurement (Fig. 2f and Table 3). The nanosecond-order lifetimes strongly suggested that the luminescence from both crystals should be assigned to fluorescence. As shown in Table 3, the emission decay of **LLi** includes the relatively long components (1.71 and 3.81 ns), whereas the luminescence from **LLi-OEt<sub>2</sub>** decays with the subnanosecond lifetimes (0.26 and 0.65 ns). The nonradiative decay rate constant ( $k_{\text{nr}}$ ) of **LLi-OEt<sub>2</sub>** was 17 times larger than that of **LLi**, while the radiative decay rate constant ( $k_{\text{r}}$ ) of the ether adduct was less than one-third of that of the base-free complex (Fig. 2g and Table 3). These results indicate that significantly fast nonradiative decay processes hamper the efficient luminescence of **LLi-OEt<sub>2</sub>**, and such processes are absent in the crystals of **LLi**. The molecular motions of the coordinated ether molecules could be responsible for such quenching paths.

To investigate the electronic nature of the complexes, density functional theory (DFT) calculations were carried out. Geometries of the compounds were optimized at the CAM-B3LYP/6-31G(d,p) level of theory. The optimized structures were confirmed as local minima by performing frequency calculations at the same level. The calculated energy diagrams and the Kohn–Sham (KS) frontier orbital distributions of the complexes are shown in Fig. 3. The energies of the KS highest occupied molecular orbitals (HOMOs) were hardly affected by

the ether. The KS lowest unoccupied molecular orbital (LUMO) of **LLi**, the next LUMO (LUMO+1) of **LLi-OEt<sub>2</sub>** and **LLi-THF**, and the LUMO of **LLi-DOX** assigned to the LUMO of the  $\text{N}_2\text{C}_3$  moiety were located in almost the same energy region. On the other hand, the LUMO+1 of **LLi**, which was composed mainly of the vacant 2p orbital of the lithium atom, was significantly stabilized by the complexation owing to the bonding interaction between the vacant orbital and the lone pair of ether. Additionally, the LUMO of **LLi-OEt<sub>2</sub>** and **LLi-THF** and LUMO+1 of **LLi-DOX**, which were assigned to the antibonding orbital in the ether moiety, seemed to be degenerated with the LUMO+1 because there is almost no interaction between the  $\pi^*$  orbital of the ligand and the  $\sigma^*$  orbital of the ether.

The  $S_0$ – $S_1$  electronic transitions were calculated with time-dependent DFT (TD-DFT) at the CAM-B3LYP/6-311+G(d,p) level of theory and the results are shown in Fig. 3 and Table 4. The  $S_1$  states of all complexes possessed the same character of the  $\pi$ – $\pi^*$  transition mainly located at the  $\text{N}_2\text{C}_3$  moiety. Consequently, the transition energy was not disturbed independently of whether the lithium atom was coordinated by the ethers or not. These results are consistent with the experimental observation of the UV–vis absorption spectra in which the complexes exhibit almost identical absorption bands. It is assumed that an increase in the degree of molecular motions

Table 3 Luminescence lifetimes and decay rate constants of the complexes at crystalline states<sup>a</sup>

	$\tau_1/\text{ns}$ ( $f_1^b$ )	$\tau_2/\text{ns}$ ( $f_2^b$ )	$\chi^2$	$\langle\tau\rangle^c/\text{ns}$	$k_{\text{r}}^d/10^8 \text{ s}^{-1}$	$k_{\text{nr}}^d/10^8 \text{ s}^{-1}$
<b>LLi</b>	1.71 (8.26%)	3.81 (91.74%)	1.17	3.63	1.8	1.0
<b>LLi-OEt<sub>2</sub></b>	0.26 (20.70%)	0.65 (79.30%)	1.22	0.57	0.5	17

<sup>a</sup> Excited at 375 nm with an LED laser. Decay curves were fitted with the multi-component exponential function:  $I(t) = \sum_i [a_i \exp(-\tau_i/t)]$ .

<sup>b</sup> Fraction of component  $i$ :  $f_i = a_i\tau_i / \sum_i a_i\tau_i$ . <sup>c</sup> Average lifetime:  $\langle\tau\rangle = \left[ \sum_i a_i\tau_i^2 \right] / \left[ \sum_i a_i\tau_i \right] = \sum_i f_i\tau_i$ . <sup>d</sup>  $k_{\text{r}} = \Phi_{\text{PL}}/\langle\tau\rangle$ ,  $k_{\text{nr}} = (1 - \Phi_{\text{PL}})/\langle\tau\rangle$ .



**Table 4** Results of TD-DFT calculations for the  $S_1$  state of the complexes<sup>a</sup>

	Energy <sup>b</sup> /eV	$\lambda$ /nm	$f^c$	Composition	Coefficient <sup>d</sup>
<b>LLi</b>	3.70	336	0.6707	HOMO $\rightarrow$ LUMO	0.68589
<b>LLi·OEt<sub>2</sub></b>	3.77	329	0.6281	HOMO $\rightarrow$ LUMO+1	0.67493
<b>LLi·THF</b>	3.74	332	0.6171	HOMO $\rightarrow$ LUMO+1	0.67896
<b>LLi·DOX</b>	3.77	329	0.6266	HOMO $\rightarrow$ LUMO	0.68099

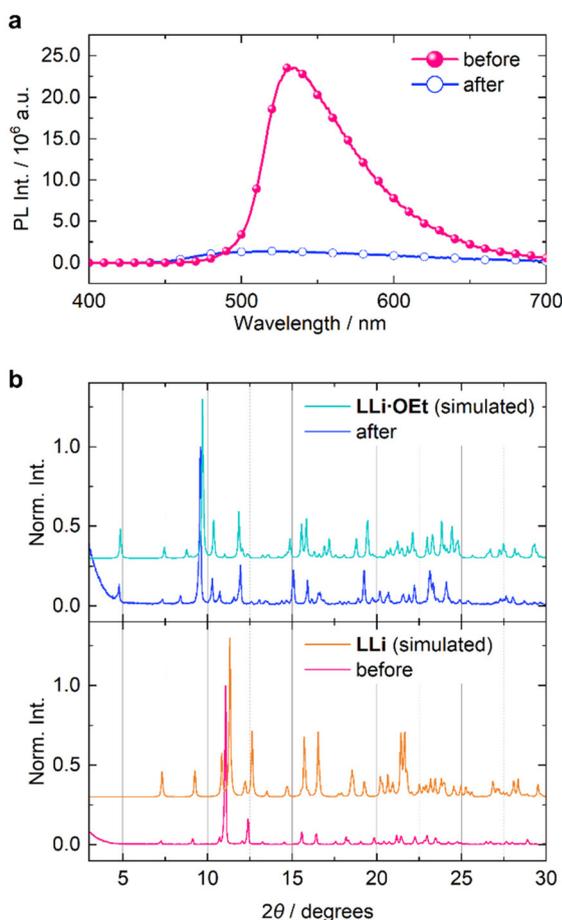
<sup>a</sup> Calculated at the TD-CAM-B3LYP/6-311++G(d,p) level of theory. <sup>b</sup> Excitation energies between  $S_0$  and  $S_1$  states. <sup>c</sup> Oscillator strength.

<sup>d</sup> Configuration interaction coefficient of the component for the  $S_0$ - $S_1$  transition.

might cause the luminescence quenching in the ether adducts. Indeed, the crystal structures of the complexes imply that the Li–N coordination could be weakened by the ether coordination. On the other hand, TD-DFT calculations for the monomeric and dimeric structures in the **LLi** crystal suggested that the relatively weak Li– $\pi$  interactions in the crystal should not significantly affect the electronic transition properties (Table S12 and Fig. S1†).

To test whether the luminescence of the crystals of **LLi** was switched to that of **LLi·OEt<sub>2</sub>** even in the solid state, the crystal-

line powder of **LLi** was exposed to the vapor of diethyl ether in a glovebox. In a 50 mL sealed vial containing 3 mL of diethyl ether, a 5 mL open vial equipped with the crystalline powder of **LLi** was placed and subsequently stored at room temperature for 12 h. After the treatment, we confirmed that the emission from the crystals was critically weakened. The PL spectrum and the absolute PL quantum yield of the treated sample (0.02) were almost identical to those of the crystals of **LLi·OEt<sub>2</sub>** (Fig. 4a). Powder X-ray diffraction (PXRD) patterns for the samples before and after treatment were identical with the simulated patterns of **LLi** and **LLi·OEt<sub>2</sub>**, respectively, indicating that a crystal–crystal transition occurred by the ether vapor treatment (Fig. 4b). The <sup>1</sup>H NMR spectrum (Chart S5†) of the treated crystal after drying under vacuum showed the same peaks as those of **LLi·OEt<sub>2</sub>**, and there was no residual signal assigned to **LLi**, indicating that the complexation between **LLi** and Et<sub>2</sub>O is not reversed on simple vacuum drying. It can be said that the base-free lithium complex could be able to be utilized not only for the detection of Lewis bases but also for constructing other types of stimuli-responsive materials.



**Fig. 4** (a) PL spectra before (magenta) and after (blue) the exposure of the diethyl ether vapor. (b) PXRD profiles before (magenta) and after (blue) Et<sub>2</sub>O vapor treatment. Diffraction profiles simulated from SCXRD measurements are also shown.

## Conclusion

The essentially two-coordinated lithium  $\beta$ -diketiminato complex **LLi** was synthesized by the reaction of *n*-butyllithium and the corresponding ligand in hexane. The complex exhibited strong green emission in the crystalline state, whereas its solution showed no PL. This is the first example of a CIE-active lithium complex. Single-crystal X-ray diffraction revealed that the lithium atom of **LLi** moderately interacts with the C-bounded phenyl rings of the  $\beta$ -diketiminato ligand of a neighboring molecule. On the other hand, the ether adducts **LLi·OEt<sub>2</sub>**, **LLi·THF**, and **LLi·DOX** exhibited only weak emission even in the crystalline state. Furthermore, it was demonstrated that the luminescence of the crystal of **LLi** was annihilated by fuming diethyl ether vapor through the formation of **LLi·OEt<sub>2</sub>**. These results shed new light on the development of stimuli-responsive materials based on the high Lewis acidity of base-free alkali metal complexes.

## Data availability

The data supporting this article have been included as part of the ESI.†



## Conflicts of interest

The authors declare no conflict of interest.

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