Thermal property and structural molecular dynamics of organic–inorganic hybrid perovskite 1,4-butanediammonium tetrachlorocuprate †

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We investigate the thermal behaviour and physical properties of the crystals of the organic inorganic hybrid perovskite [(NH3)(CH2)4(NH3)]CuCl4. The compound’s thermal stability curve as per thermogravimetric analysis exhibits a stable state up to ~495 K, while the weight loss observed near 538 K corresponds to partial thermal decomposition. The 1H nuclear magnetic resonance (NMR) chemical shifts for NH3 change more significantly with temperature than those for CH2 because the organic cation motion is enhanced at both ends of the organic chain. The 13C NMR chemical shifts for the ‘CH2-1’ units of the chain show an anomalous change, and those for ‘CH2-2’ (units closer to NH3) are shifted sharply. Additionally, the 14N NMR spectra reflect the changes of local symmetry near Tc (=323 K). Moreover, the 13C T1p values for CH2-2 are smaller than those for CH2-1, and the 13C T1p data curve for CH2-1 exhibits an anomalous behaviour between 260 and 310 K. These smaller T1p values at lower temperatures indicate that 1H and 13C in the organic chains are more flexible at these temperatures. The NH3 group is attached to both ends of the organic chain, and NH3 forms a N–H···Cl hydrogen bond with the Cl ion of inorganic CuCl4. When H and C are located close to the paramagnetic Cu2+ ion, the T1p value is smaller than when these are located far from the paramagnetic ion.

I. Introduction

The search for new and improved functional materials in recent years has resulted in considerable progress in the synthesis of many families of organic–inorganic compounds. The properties and structural phase transition of these compounds are related to their structures and the interaction of the cationic units with complex anionic sublattices. One such group of hybrid compounds, whose structure can be expressed by the general formula [NH3(CH2)4NH3]MX4 (M = divalent metal ion and X = Cl, Br) is known to crystallise in a 2D perovskite-like structure, and these compounds are usually referred to as organic–inorganic hybrid perovskites or organic–metal-halide composites. These perovskites combine the advantages of both organic and inorganic materials in a single molecular scale. In particular, in the diazammonium hybrid perovskite with its formula of [NH3(CH2)4NH3]MX4, the NH3 group is attached to both ends of the organic chain. At the end of the organic part of the chain, the ammonium ion forms a N–H···X hydrogen bond with the halide ion of the metallic inorganic layer. These perovskites tend to exhibit a number of phase transitions such as order-disorder transitions. Here, we note that the properties of organic–inorganic hybrid perovskites depend on the organic cation, divalent metal, and halogen ion, and thus, it is necessary to investigate the ‘structure-directing’ properties of these new materials. In general, 2D hybrid perovskites can find use in the fields of energy, optoelectronics, photonics, and catalysis in green chemistry applications.

The compound [(NH3)(CH2)4(NH3)]CuCl4, or 1,4-butanediammonium tetrachlorocuprate, with M = Cu and X = Cl, undergoes a reversible phase transition at 325 K (=Tc) between the two monoclinic phases II and I. The transition can be explained by order-disorder mechanisms involving a model of twisted conformation chains, which was introduced to explain the decrease in interlayer distance with increasing temperature from X-ray diffraction experiment. From structural considerations, these results can be explained by the conformational change of organic chains from the left-handed conformation in phase II to an all-trans conformation in phase I. The structural geometry of [(NH3)(CH2)4(NH3)]CuCl4 in the room-temperature phase II and high-temperature phase I are represented in Fig. 1(a) and (b), respectively. The crystal structure at room temperature is monoclinic, corresponding to space group P21/c. The unit cell dimensions are a = 9.270 Å, b = 7.600 Å, c = 7.592 Å, β = 103.14°, and Z = 2. Structural

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cohesion is achieved via N–H···Cl hydrogen bonds. \([\text{NH}_3](\text{CH}_2)_4(\text{NH}_3)]\text{CuCl}_4\) is composed of alternating inorganic \(\text{CuCl}_2\) layers and organic \([\text{NH}_3](\text{CH}_2)_2\text{CuCl}_2\) sheets. The \(\text{CuCl}_2\) layers are sandwiched by \([\text{NH}_3](\text{CH}_2)_2\text{NH}_3\) cations, which possess centrosymmetrical chains with left-handed conformations at both ends, thereby forming the organic sheets. Above 325 K, the symmetry changes to that of a monoclinic structure with space group \(P2_1/c\), and the corresponding lattice constants are \(a = 10.420\ \text{Å}, b = 7.442\ \text{Å}, c = 7.225\ \text{Å}, \beta = 93.46^\circ\), and \(Z = 2\).15

In the context of the property measurements of such compounds, Snively et al. conducted magnetic susceptibility measurements of \([\text{NH}_3](\text{CH}_2)_4(\text{NH}_3)]\text{CuCl}_4\) powdered and single-crystals in the temperature range of 4 to 200 K.16,17 They reported that the interplanar superexchange interaction along the linear Cu–Cl–Cl–Cu path exhibits a significantly stronger Cu–Cu-distance dependence than that along the Cu–Cl–Cu path. This crystal structure has been reported in phases I and II by Garland et al.18 Subsequently, the phase transitions occurring in the perovskite-type 2D molecular composite \([\text{NH}_3](\text{CH}_2)_4(\text{NH}_3)]\text{CuCl}_4\) have been studied by means of differential scanning calorimetry (DSC), X-ray diffraction (XRD),19 and electron paramagnetic resonance (EPR).14,19,20

Understanding the structural dynamics of organic–inorganic hybrid perovskite \([\text{NH}_3](\text{CH}_2)_4(\text{NH}_3)]\text{CuCl}_4\) is essential for their advanced use as new materials. Here, we study the structural dynamics of the organic–inorganic hybrid perovskite \([\text{NH}_3](\text{CH}_2)_4(\text{NH}_3)]\text{CuCl}_4\) via magic angle spinning (MAS) nuclear magnetic resonance (NMR) and static NMR experiments. The chemical shifts and spin–lattice relaxation times in the rotating frame \(T_{1p}\) in the low- and high-temperature phases are measured by means of MAS \(^1\text{H}\) NMR and cross-polarisation (CP)/MAS \(^{13}\text{C}\) NMR to understand the role of the organic cation in this crystal. The \(^{14}\text{N}\) NMR spectra of the compound in the laboratory frame are also obtained as a function of temperature. We use these results to discuss the structural dynamics of the \(\text{NH}_3\text{CH}_2\text{CH}_2\text{CH}_2\text{NH}_3\) chain below and above the phase transition temperature \(T_C\). In particular, an examination of the hydrogen bonding of N–H···Cl between the Cu–Cl layer and the alkylammonium chain within \([\text{NH}_3](\text{CH}_2)_4(\text{NH}_3)]\text{CuCl}_4\) can provide important insights into the operational mechanism as regards potential applications.

II. Experimental method

Crystals of \([\text{NH}_3](\text{CH}_2)_4(\text{NH}_3)]\text{CuCl}_4\) were prepared by mixing equimolar amounts of \(\text{NH}_2\text{CH}_2\text{CH}_2\text{HCl}\) and \(\text{CuCl}_2\) in aqueous solution and allowing the resulting mixture to slowly evaporate. The light-green-coloured crystals grew as rectangular parallelepipeds with dimensions of 5 mm \(\times\) 5 mm \(\times\) 1 mm.

The crystal structure of \([\text{NH}_3](\text{CH}_2)_4(\text{NH}_3)]\text{CuCl}_4\) was determined with a X-ray diffraction system, using a Cu-Kα radiation source at the KBSI, Seoul Western Center. DSC (TA, DSC 25) experiments were carried out at a heating rate of \(10^\circ\text{C}\ \text{min}^{-1}\) in the temperature range of 190 to 600 K in a nitrogen-gas atmosphere. Thermogravimetry analysis (TGA) experiments were conducted using a thermogravimetric analyser (TA Instruments) under conditions identical to those of DSC over a temperature range of 300 to 680 K. The DSC and TGA experiments were performed by using crystal sample quantities of 6.23 and 7.53 mg, respectively.

Solid-state MAS NMR investigations of the \([\text{NH}_3](\text{CH}_2)_4(\text{NH}_3)]\text{CuCl}_4\) crystals were conducted by using a 400 MHz Avance II+ Bruker NMR spectrometer at the same facility. The MAS \(^1\text{H}\) NMR and CP/MAS \(^{13}\text{C}\) NMR experiments were performed at the Larmor frequencies of \(\omega_0/2\pi = 400.13\) and 100.61 MHz, respectively. Solid samples were packed into 4 mm-diameter zirconia rotors and closed off using Vespel caps. The samples were spun at 10 kHz MAS by using dry nitrogen gas. The \(^1\text{H}\) and \(^{13}\text{C}\) NMR chemical shifts were obtained with the use of tetramethylsilane (TMS) as a standard. The \(T_{1p}\) data for \(^1\text{H}\) and \(^{13}\text{C}\) were obtained by applying a \(\pi/2\) pulse, immediately followed by a long spin-locking pulse phase-shifted by \(\pi/2\) with respect to the \(\pi/2\) pulse. The width of the \(\pi/2\) pulse used for \(T_{1p}\) measurements was 3.3 \(\mu\text{s}\), which yields the frequency of the rotating frame as \(\omega_1 = 75.75\ KHz\). The \(T_{1p}\) data were obtained by varying the length of the spin-locking pulse. In addition, the \(^{14}\text{N}\) NMR spectra of a \([\text{NH}_3](\text{CH}_2)_4(\text{NH}_3)]\text{CuCl}_4\) single-crystal were obtained at the Larmor frequency of \(\omega_0/2\pi = 28.90\ \text{MHz}\) in the laboratory frame. The \(^{14}\text{N}\) resonance frequency was referenced with \(\text{NH}_4\text{NO}_3\) as the standard sample. The \(^{14}\text{N}\) NMR spectrum was obtained by the application of the following solid-state echo sequence: 8 \(\mu\text{s}\)-tau (16 \(\mu\text{s}\))–8 \(\mu\text{s}\)-tau (16 \(\mu\text{s}\)). The temperature change was maintained within the error range of \(\pm 0.5\ \text{K}\) by adjusting the nitrogen gas flow and heater current.

III. Results and discussion

The powder X-ray diffraction pattern of \([\text{NH}_3](\text{CH}_2)_4(\text{NH}_3)]\text{CuCl}_4\) at 300 K is described in the ESI, and this data is consistent with previously reported results.14 Fig. 2 shows the DSC curve of the \([\text{NH}_3](\text{CH}_2)_4(\text{NH}_3)]\text{CuCl}_4\) crystals obtained with the heating rate of \(10^\circ\text{C}\ \text{min}^{-1}\). An endothermic peak corresponding to the previously reported II–I phase transition is detected at 323 K. In addition, a very large exothermic peak is
observed at 538 K. To understand the origins of this peak, we performed TGA experiments; these results are also shown in Fig. 2. In the TGA curve, a stable state is observed up to 495 K, whereas a weight loss is observed at higher temperatures, which represents partial thermal decomposition. Here, we note that \(\text{[(NH}_3\text{)(CH}_2\text{)}_4\text{(NH}_3\text{)]CuCl}_4\) crystals show the weight loss with temperature increase. From the TGA experimental results and possible chemical reactions, we compared the weight loss. The weight loss of 12% around 538 K obtained from the DSC experiment is consistent with the calculated decomposition of HCl moieties. From the figure, we note that the weight sharply decreases between 500 and 650 K, with a corresponding weight loss of 67% near 650 K.

Next, we acquired the MAS 1H NMR spectrum of the \(\text{[(NH}_3\text{)(CH}_2\text{)}_4\text{(NH}_3\text{)]CuCl}_4\) crystals at various temperatures (Fig. 3). In the figure, we can observe two resonance signals for 1H. The spinning sidebands corresponding to CH2 are indicated by open circles, and those of NH3 are indicated by crosses. At 300 K, the 1H NMR chemical shifts for CH2 and NH3 are observed at \(\delta = 2.73\) ppm and \(\delta = 11.86\) ppm, respectively. Below 300 K, the 1H resonance signal bonded to CH2 mostly merges with the 1H resonance signal bonded to NH3, which makes it difficult to distinguish the two signals. In addition, the 1H resonance signal for CH2 is related to the number of bonded protons, which means that the signal exhibits a stronger intensity and wider linewidth than the corresponding ones for NH3. The 1H NMR chemical shifts according to the temperature exhibit a greater change for NH3 than CH2. These results indicate that NH3 is temperature-sensitive.

Next, we measured the MAS 1H NMR spectrum at various temperatures, and the intensity change for the delay time was observed to obtain the spin–lattice relaxation time in the rotating frame \(T_{1p}\) for 1H at each temperature. Normally, the \(T_{1p}\) data can be obtained as the slope of the intensity or the ratio of the area of the resonance signal to the delay time. The change in the proton magnetisation intensity in terms of \(T_{1p}\) is expressed as below:\textsuperscript{21–23}

\[
P(\tau) = P(0) \exp(-\tau/T_{1p}),
\]

where \(P(\tau)\) and \(P(0)\) denote the signal intensities at time \(\tau\) and \(\tau = 0\), respectively. Next, at 300 K, the MAS 1H NMR signals of CH2 and NH3 were plotted for various delay times in the range from 0.2 to 80 ms (Fig. 4); we note that the intensities of the 1H NMR signal as a function of the delay times exhibit considerable variation. From the slope of the intensity vs. delay time curve, the \(1^H\) \(T_{1p}\) data of \(\text{[(NH}_3\text{)(CH}_2\text{)}_4\text{(NH}_3\text{)]CuCl}_4\) were obtained for CH2 and NH3 in the low-temperature phase II and high-temperature phase I. From Fig. 4, we note that no changes are observed in the \(T_{1p}\) value near \(T_C\), and \(T_{1p}\) slightly increases according to the temperature change. The \(1^H\) \(T_{1p}\) values for CH2 and NH3 at 300 K are 14.37 and 12.88 ms, respectively. Here, the \(1^H\) \(T_{1p}\) value for NH3 is smaller than that for CH2. This is
possibly because NH$_3$ is closer to the inorganic CuCl$_4$ layer; the $T_1^r$ value becomes smaller as the distance between H and the paramagnetic Cu$^{2+}$ ion reduces. This is because $T_1^r$ is inversely proportional to the square of the magnetic moment of the paramagnetic ion.$^{21}$

The CP/MAS $^{13}$C NMR chemical shifts measured at various temperatures are shown in Fig. 5. In the study, the MAS $^{13}$C NMR spectrum for TMS was recorded at 38.3 ppm at 300 K, and this value was calibrated to determine the chemical shift in $^{13}$C. Here, the two inner CH$_2$ groups of the four CH$_2$ ones are together designated as CH$_2$-1, and the two CH$_2$ units close to the NH$_3$ ones are designated as CH$_2$-2. We note from the figure that the $^{13}$C chemical shifts for CH$_2$-1 (far from NH$_3$) are different from those for CH$_2$-2, which is closer to NH$_3$. In the $^{13}$C NMR spectra obtained for CH$_2$-1 and CH$_2$-2, two unusual resonance lines are observed between 260 and 310 K. At 300 K, the two resonance signals for CH$_2$-1 are recorded at chemical shifts of $\delta = 38.44$ and 59.56 ppm. Furthermore, the signal of $\delta = 98.23$ ppm corresponds to CH$_2$-2. The $^{13}$C chemical shifts for CH$_2$-1 exhibit an anomalous change with increase in temperature, whereas those for CH$_2$-2 shift abruptly with increasing temperature, as shown in Fig. 6. The two resonance lines between 260 and 310 K correspond to CH$_2$-1, and hitherto unreported anomalous phenomena are observed in this temperature range.

We next remark that line broadening in the MAS $^{13}$C NMR spectra is influenced by relaxation processes such as the motional modulations of the chemical shift anisotropy and dipolar carbon-proton coupling. Fig. 7 shows the $^{13}$C full-width at half-maximum (FWHM) linewidth of [(NH$_3$)(CH$_2$)$_4$(NH$_3$)] CuCl$_4$. The $^{13}$C NMR line shapes vary from the Gaussian type at lower temperatures to the Lorentzian shape at higher temperatures. The appearance of these two-component spectra is caused by difference in different molecular motions. The line-width near the phase transition temperature $T_C$ shows

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**Fig. 5** In situ MAS $^{13}$C NMR spectra for CH$_2$-1 and CH$_2$-2 of [(NH$_3$)(CH$_2$)$_4$(NH$_3$)] CuCl$_4$ as a function of temperature.

**Fig. 6** MAS $^{13}$C chemical shifts for CH$_2$-1 and CH$_2$-2 of [(NH$_3$)(CH$_2$)$_4$(NH$_3$)] CuCl$_4$ at low temperature phase II and high temperature phase I.

**Fig. 7** MAS $^{13}$C NMR spin-lattice relaxation times $T_1^r$ for CH$_2$-1 and CH$_2$-2 of [(NH$_3$)(CH$_2$)$_4$(NH$_3$)] CuCl$_4$ as a function of temperature (inset: line widths for CH$_2$-1 and CH$_2$-2 according to the temperature).

**Fig. 8** $^{14}$N resonance frequency of [(NH$_3$)(CH$_2$)$_4$(NH$_3$)] CuCl$_4$ single crystal as a function of temperature (inset: in situ $^{14}$N resonance frequency at several temperatures).
Table 1  Phase transition temperature $T_C$, decomposition temperature $T_d$, structure, space group, lattice constant, spin-lattice relaxation time $T_{1p}$ for [C$_2$H$_5$NH$_3$]$_2$CuCl$_4$ and [NH$_3$(CH$_2$)$_4$NH$_3$]CuCl$_4$ crystals

| Structure       | $T_C$ (K) | $T_d$ (K) | [C$_2$H$_5$NH$_3$]$_2$CuCl$_4$ | [NH$_3$(CH$_2$)$_4$NH$_3$]CuCl$_4$ |
|-----------------|-----------|-----------|-------------------------------|---------------------------------|
| Orthorhombic    | 326, 330, 357, 371 (ref. 26–31) | 430 | 325 (ref. 14) | Monoclinic$^{14,15}$ |
| Pbca            |           |           |                               | $P2_1/c$                        |
| Lattice constant| $a = 7.47$ | $b = 7.35$ | $a = 9.270$                   | $b = 7.600$                     |
| $c = 21.18$     |           |           | $c = 7.592$                   | $\beta = 103.14^\circ$         |

IV. Conclusions

In this study, we investigated the thermal behaviour and physical properties of organic–inorganic hybrid perovskite [[NH$_3$]$_2$(CH$_2$)$_4$(NH$_3$)]CuCl$_4$ crystals. The structural dynamics of [[NH$_3$]$_2$(CH$_2$)$_4$(NH$_3$)]CuCl$_4$ with emphasis on the role of the [[NH$_3$]$_2$(CH$_2$)$_4$(NH$_3$)] cation, were discussed by MAS $^1$H NMR, MAS $^{13}$C NMR, and static $^{14}$N NMR as a function of temperature. Firstly, we found that the TGA curve exhibited stability until 495 K, and the observed weight loss of 12% near 538 K was due to the partial thermal decomposition of HCl moieties.

Secondly, the $^1$H NMR chemical shift of NH$_3$ for crystallographic environments changed more significantly with temperature than that for CH$_2$ because the [[NH$_3$]$_2$(CH$_2$)$_4$(NH$_3$)] cation motion is enhanced at both ends of the cation of the NH$_3$ group. The $^{13}$C NMR chemical shifts for CH$_2$-1 showed an anomalous change, and those for CH$_2$-2 shifted sharply to lower values when compared with that of CH$_2$-1. The $^{13}$C chemical shifts of the CH$_2$-2 unit (closer to the N–H…Cl bond) sharply changed relative to those of CH$_2$-1. In addition, the $^{14}$N NMR spectra reflected the changes in the local symmetry of the crystal near $T_C$.

The $^1$H $T_{1p}$ values for CH$_2$ and NH$_3$ slightly increased with temperature increase. Moreover, the $^{13}$C $T_{1p}$ value for CH$_2$-2 was smaller than that of CH$_2$-1, and the $^{13}$C $T_{1p}$ value for CH$_2$-1 exhibited an anomalous trend between 260 and 310 K. At low temperatures, the $^1$H and $^{13}$C $T_{1p}$ values were smaller than at high temperatures. Smaller $T_{1p}$ values at lower temperatures indicate that $^1$H and $^{13}$C in the organic chains are more flexible at these temperatures. Moreover, the $^{13}$C of CH$_2$-2 close to NH$_3$ of the organic chain is more flexible than the $^{13}$C of CH$_2$-1 and 310 K, an abnormal phenomenon is observed in the 14 N resonance line in this region. At 320 K near $T_C$, there is only one $^{14}$N resonance line, and at temperatures >320 K, no resonance lines are observed. At the transition point of 323 K, the $^{14}$N NMR lines merge into one line. This single $^{14}$N resonance line indicates that there is no electric field gradient (EFG) tensor at the N site in phase I because of site symmetry. The EFG tensor changes around the N site, which indicates a change in the structural configuration around the N site near $T_C$.
between the four CH2 sites. The NH3 group is attached to both ends of the organic chain, and it forms a N–H···Cl hydrogen bond with the Cl ion of the inorganic CuCl4. The T1ρ value is smaller when H and Cl are located close to the paramagnetic Cu2+ ion than when far away. Additionally, the NH3 groups are coordinated by CuCl4, and thus, atomic displacements in the environment of the 14N nuclei with temperature are correlated with CuCl4. We also note here that detailed studies are required to examine the anomalies observed in the range of 260 to 310 K.

Here, we compared the phase transition temperatures, decomposition temperatures, crystal structures, space groups, lattice constants, and spin-lattice relaxation times of the previously reported [C2H5NH3]2CuCl4 (ref. 26–35) and those of [NH3(CH2)4NH3]CuCl4 examined in this study; this is summarised in Table 1. The difference between the two crystals is only the presence of organic cation. The two compounds have four anions, the molecular motions according to the 13C bond

Conflicts of interest

There are no conflicts to declare.

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