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Control of assembly and magnetism of metal–dmit complexes by supramolecular cations

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Abstract

The assembly of monovalent species of [Ni(dmit)$_2$] (dmit^{2-} = 2-thioxo-1,3-dithiole-4,5-dithiolate), which bear $S = 1/2$ spin on each molecule, was controlled by using crown-ether based supramolecular cations. We could regulate the shape and valence of the supramolecular cations by changing the ring size of the crown ethers as well as the metal cations included. Since the structure of the ionic crystals is mainly determined by the Madelung energy and the Lennard-Jones potential (shape of molecules), a large diversity in crystal structure was observed. A relationship between the overlap of [Ni(dmit)$_2$] frontier orbitals and the magnetism is discussed. © 2002 Elsevier Science B.V. All rights reserved.

Keywords: Supramolecular cations; Ni(dmit)$_2$; Crown ethers; Molecular magnets

1. Introduction

Transition metal complexes are useful building blocks for molecular conductors and magnets. $K_2Pt(CN)_4Br_{0.3}(3.2H_2O)$ (KCP) is the first example of a highly conducting molecular solid of a transition metal complex, in which electronic conduction is achieved through a Pt–Pt one-dimensional chain [1]. Ferromagnetic interactions have been observed in a variety of the transition metal complexes having d-spins [2]. On the other hand, there is a class of transition metal complexes whose electronic and magnetic properties in the solid state arise from the extended π-ligands (π-electrons and π-spins). A ferromagnetic intermolecular interaction was observed in a nickel bis(maleonitrile) complex NH_4[Ni(mnt)$_2$](H_2O), and ascribed to the metal–sulfur interaction between the central metal cation and π-conjugated ligands [3].

Among the transition metal complexes with extended π-ligands, metal dithiolate complexes have been extensively studied. The complex NH_4[Ni(mnt)$_2$](H_2O) is one example, and recently, even a single component molec-

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ular metal has been reported using a tetrathiafulvalene (TTF)-type dithiolate ligand, although the molecule has a completely filled highest occupied molecular orbital (HOMO) [4]. One of the most popular building block is [Ni(dmit)$_2$] (dmit^{2-} = 2-thioxo-1,3-dithiole-4,5-dithiolate), which has been used for constructing more than half a dozen molecular superconductors [5] as well as interesting molecular materials including spin-ladders [6].

The [Ni(dmit)$_2$] molecule is a good acceptor giving molecular conductors in partially charged state. The anion radical species with unit charge usually do not give highly conducting molecular solids due to their relatively large on-site Coulomb repulsive energy (U), and are utilized to construct molecular magnetic materials. We have been introducing supramolecular cation structures based on crown ether macrocycles as a counter cation for [Ni(dmit)$_2$] anionic species [7]. By utilizing supramolecular cation structures, we can regulate to some extent the charge and molecular arrangement of the [Ni(dmit)$_2$] species in the crystal, through which we can prepare a variety of molecular conductors and magnetic materials.

As for electrically conducting solids, we succeeded in introducing ionic channel structures in [Ni(dmit)$_2$] based molecular conductors [8]. In the crystals, [Ni(dmit)$_2$] forms highly conducting one-dimensional columns (both structurally and electronically) and crown ethers, also stacked one-dimensionally, form ionic channel structures. The number of conduction electrons corresponded to the number of ions in the ionic channel, and the distribution and motion of ions strongly affected the conducting properties of the crystal. We could observe novel functions arising from the synergistic and cooperative effects between the electronic system of [Ni(dmit)$_2$] and the supramolecular cation structure in these solids. In this paper, we will review recent results on [Ni(dmit)$_2$]$^-$ based magnetic materials obtained through the supramolecular cation approach, mainly focusing on the relationship between the [Ni(dmit)$_2$]$^-$ arrangements determined by supramolecular cations and magnetic properties of the crystals.

2. Design of [Ni(dmit)$_2$] based molecular magnets

The main parameters determining the conducting and magnetic properties of molecular solids are, among others, the average charge on a molecule (δ), U_{eff} (suffix 'eff' means the effective U in the solid state) and transfer integral (t) between the adjacent molecules (within the extended Hückel molecular orbital calculation regime). The parameter t is related to the band structure, which is described well by the tight-binding approximation in these molecular solids as

$$\varepsilon_k = \varepsilon_0 - 2t \cos ka \tag{1}$$

The crystal is a conductor when the conduction band is partially filled to an extent determined by δ. The bandwidth $4t$ is usually smaller than U_{eff} in a molecular solid. Electrons (or holes) tend to localize on each molecule when a molecule is in a fully charge transferred state [9]. In most cases, such molecules form dimers or higher aggregates and the systems become band insulators. The magnetic exchange interaction energy (J) between the localized spins is given by

$$|J| = 2t^2/U_{\mathrm{eff}} \tag{2}$$

Therefore, the design and regulation of t between molecules in the crystal is most important in obtaining the desired molecular conductors and magnets. The U_{eff} is controlled by the extension of the π-electron system and is important in designing molecular conductors, since the electron localization is suppressed in small U_{eff} systems [9]. The design of U_{eff} in molecular magnets is also important, although the subject is outside the scope of this review.

The parameter t is determined by the overlap between the frontier orbitals, and thus, the regulation of molecular arrangements in the crystal is essential for controlling t. The main factors determining the structure of ionic crystals are the Madelung energy and the Lennard-Jones potential (shape of molecules). We adopted the supramolecular approach to modify these two factors. We used crown ether based supramolecular cations as a counter cation for [Ni(dmit)$_2$]$^-$. Through this approach, we can systematically change the shape of the cation without changing the valence of the supramolecular cation by using a variety of crown ether derivatives with different ring sizes. We can also change the valence without significant change in the supramolecular cation by using inorganic cations with different valences. In addition, we can introduce transition metal cations with magnetic moments in the crown ether cavity, which will coexist with the [Ni(dmit)$_2$]$^-$ magnetic moments in the crystal.

3. Synthesis, structure and properties of [Ni(dmit)$_2$]$^-$ salts with supramolecular cations

3.1. Preparation of the salts

The single crystals were grown using a slow diffusion method, or using electrocrystallization, between (tetrabutylammonium)[Ni(dmit)$_2$] and inorganic salts/crown ether in CH_3CN. $M^+(ClO_4^-)$ or $M^{2+}(ClO_4^-)_2$ was used as a cation source. The composition and the method of preparation of the crystals are summarized in Table 1. In the case of electrocrystallization, cation exchange occurred before the oxidation of [Ni(dmit)$_2$]$^-$, and [Ni(dmit)$_2$] monovalent salts were obtained.

Table 1
Crystal composition and crystal preparation condition

	Composition	Method [a]	Solvent [b]	Period (day)
1	K^+(1,10-diaza-18-crown-6)[Ni(dmit)$_2$]	Electrocrystallization	CH_3CN	7
2	Rb^+(1,10-diaza-18-crown-6)[Ni(dmit)$_2$]	Electrocrystallization	CH_3CN	7
3	(NH_4^+)(15-crown-5)$_2$[Ni(dmit)$_2$]	Diffusion	CH_3CN	7
4	Ca^{2+}(12-crown-4)$_2$[Ni(dmit)$_2$]$_2$	Diffusion	CH_3CN	12
5	Ca^{2+}(15-crown-5)$_2$[Ni(dmit)$_2$]$_2$(CH_3CN)$_{0.7}$	Diffusion	CH_3CN	12
6	Ca^{2+}(1,10-diaza-18-crown-6)[Ni(dmit)$_2$]$_2$(CH_3CN)$_2$	Diffusion	CH_3CN	12

[a] Between the (TBA)[Ni(dmit)$_2$] and crown ethers/M(ClO_4) or M(ClO_4)$_2$.
[b] CH_3CN was distilled over CaH_2 prior to use.

3.2. Monovalent supramolecular cation salts

The potassium and rubidium cations fit well in the cavity of 18-crown-6 derivatives and these combinations will give disk-shaped supramolecular cations. We utilized this type of supramolecular cation to regulate the [Ni(dmit)$_2$] arrangements [10]. Fig. 1 shows the crystal structure of K^+(1,10-diaza-18-crown-6)[Ni(dmit)$_2$] (**1**). The rubidium salt, Rb^+(1,10-diaza-18-crown-6)[Ni(dmit)$_2$] (**2**), also gave the same crystal structure. 1,10-Diaza-18-crown-6 forms a disk-shaped supramolecular cation structure in the crystal as expected, and is stacked alternately with [Ni(dmit)$_2$]$^-$ along the *a*-axis. Since the monovalent [Ni(dmit)$_2$]$^-$ molecule has a singly occupied molecular orbital, the molecules tend to form dimers in the crystal to gain exchange energy. In the crystals of **1** and **2**, the disk-shaped supramolecular cation is inserted between two [Ni(dmit)$_2$]$^-$ anions to reduce the Coulombic energy (Fig. 1). As a result, strong face-to-face π–π interaction between [Ni(dmit)$_2$]$^-$ is prevented and the magnetic interaction is mainly achieved through the S–S interaction at the terminal sulfur atoms of [Ni(dmit)$_2$]$^-$ molecules along the *b*-axis. The transfer integrals between the [Ni(dmit)$_2$]$^-$ molecules obtained by the extended Hückel molecular orbital calculation (t_1 in Fig. 1) are -1.03 and 2.77 meV for **1** and **2** salts, respectively, and [Ni(dmit)$_2$]$^-$ molecules form one-dimensional chains along the *b*-axis through this interaction.

Fig. 2a and b shows the temperature dependence of the magnetic susceptibility of **1** and **2** salts, respectively. Their behavior was well reproduced by the one-dimensional Heisenberg antiferromagnetic (1D AF) chain model with a magnetic exchange energy J/k_B of -24.7 and -22.7 K for **1** and **2**, respectively. The magnetic behavior, therefore, is in good agreement with the one-dimensional [Ni(dmit)$_2$]$^-$ chain structure in the crystal formed through relatively weak intermolecular interaction.

The size of the NH_4^+ cation is almost the same as those of K^+ and Rb^+. However, NH_4^+ has not given the same crystal structure as **1** with 1,10-diaza-18-crown-6 so far. Instead, we have prepared (NH_4^+)(15-crown-5)$_2$[Ni(dmit)$_2$] (**3**), which will be discussed later [11].

3.3. Divalent supramolecular cation salts

Table 2 summarizes the crystal data for [Ni(dmit)$_2$]$^-$ salts containing a supramolecular cation structure: Ca^{2+}(12-crown-4)$_2$[Ni(dmit)$_2$]$_2$ (**4**), Ca^{2+}(15-crown-5)$_2$[Ni(dmit)$_2$]$_2$(CH_3CN)$_{0.7}$ (**5**) and Ca^{2+}(1,10-diaza-18-crown-6)[Ni(dmit)$_2$]$_2$(CH_3CN)$_2$ (**6**). It was possible to change the size and shape of the supramolecular cation by using 12-crown-4, 15-crown-5 and 18-crown-6 derivatives for the building blocks. Although we have not succeeded in the regulation of the [Ni(dmit)$_2$]$^-$ arrangement in the crystal according to the advanced design, the crystal structures show a large diversity compared to the monovalent salts and a variety of intermolecular overlap modes were observed through the systematic change in the ring size of the crown ether [12].

Fig. 3 summarizes the intermolecular interaction modes of [Ni(dmit)$_2$]$^-$ anions observed in the divalent Ca^{2+}(crown ethers)[Ni(dmit)$_2$]$_2$ (lower six) in addition to those observed in the monovalent M^+(crown ethers)[Ni(dmit)$_2$]$^-$ (upper two). Table 3 summarizes the transfer integrals *t* of [Ni(dmit)$_2$]$^-$ arrangements of salts **1**–**6**. Salt **3** forms a π–π dimer with an interdimer interaction of $t \sim 5$, while salts **1** and **2** have a regular

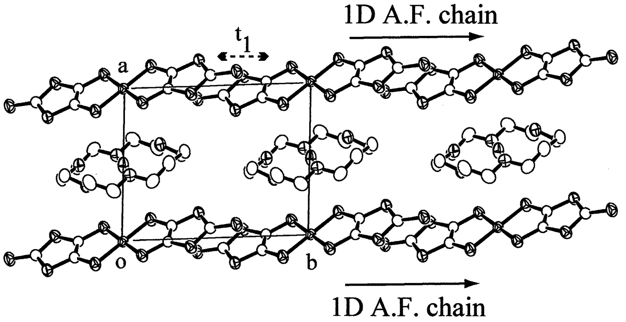

Fig. 1. Crystal structure of K^+(1,10-diaza-18-crown-6)[Ni(dmit)$_2$] (**1**). Unit cell of salt **1** viewed along the *c*-axis. The [Ni(dmit)$_2$]$^-$ molecules form a one-dimensional antiferromagnetic chain along the *b*-axis through the t_1 interaction.

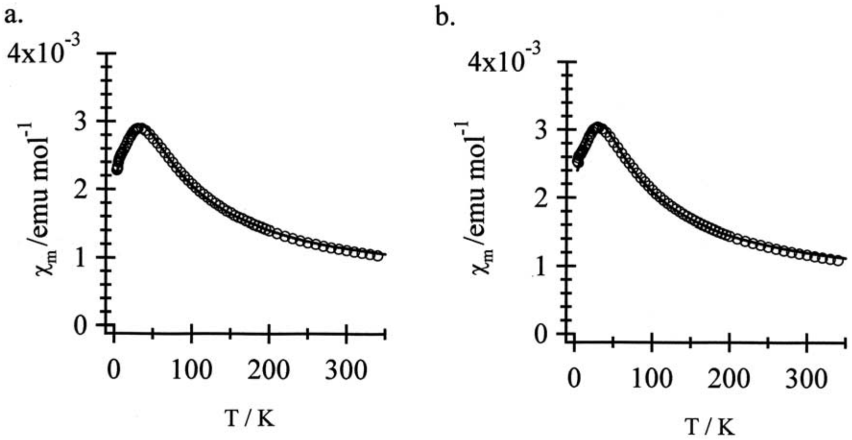

Fig. 2. Temperature dependence of the magnetic susceptibility of the salts (a) **1** and (b) **2**. The solid lines are the fitting curves using the one-dimensional Heisenberg antiferromagnetic linear chain model ($2 < T < 350$ K).

Table 2
Crystal data of divalent Ca^{2+}(crown ethers)[Ni(dmit)$_2$]$_2$ (**4**–**6**)

	4	**4**	**5**	**6**
Empirical formula	$C_{28}H_{32}O_8S_{20}CaNi_2$	$C_{28}H_{32}O_8S_{20}CaNi_2$	$C_{100}H_{126}N_2O_{30}S_{60}Ca_3Ni_6$	$C_{28}H_{31}S_{20}N_3O_5CaNi_2$
Formula weight	1295.24	1295.24	4232.13	1288.25
Temperature (K)	297	150	297	297
Space group	$P2_1/n$ (# 14)	$P2_1/n$ (# 14)	$P\bar{1}$ (# 2)	$P4_2/mnm$ (# 136)
Unit cell dimensions				
a (Å)	19.872(1)	19.8366(4)	12.4442(2)	15.598(3)
b (Å)	19.7547(8)	19.5053(3)	12.5299(2)	
c (Å)	12.6242(6)	12.5358(2)	26.872(1)	10.222(4)
α (°)			97.0670(7)	
β (°)	95.442(1)	94.7610(7)	91.7234(8)	
γ (°)			97.214(1)	
V (Å^3)	4933.55(4)	4833.6(1)	4120.8(2)	2486.9(6)
Z	4	4	1	2
R [a]	0.041	0.023	0.049	0.050
$R_w(F^2)$ [b]	0.050	0.037	0.126	0.081

[a] $R = \Sigma\|F_o| - |F_c\|/\Sigma|F_o|$.

[b] $R_w = (\Sigma w|F_o| - |F_c|)^2/\Sigma w F_o^2)^{1/2}$.

[Ni(dmit)$_2$] chain with the intermolecular interaction of $t \sim 2$ through the terminal S–S interactions as described above. The temperature dependent magnetic behavior of salt **3** was well reproduced by the Curie–Weiss equation. The magnitude of π–π dimerization in NH_4^+(15-crown-5)$_2$[Ni(dmit)$_2$] is not strong enough to stabilize the singlet pair as in the case of salts **1** and **2**.

The divalent Ca^{2+}(crown ethers)[Ni(dmit)$_2$]$_2$ salts showed more diversity in their structures and in the magnitude of the π–π interaction than those in the monovalent system. The magnitude of the transfer integral in those crystals ranges from $t \sim 3$ to ~ 9 at room temperature for the Ca^{2+}(crown ethers)[Ni(dmit)$_2$]$_2$ system, resulting in a variety of magnetism properties. Fig. 4 shows the temperature dependent magnetic susceptibility (χ_m) of salts **4**–**6**.

There are two different origins for the magnetism in **4**, that arising from the dimers and that from the monomers of [Ni(dmit)$_2$]. The χ_m value of **4** increases monotonically by lowering the temperature from 350 to 190 K, then a sudden decrease of the χ_m value is observed at 190 K (Fig. 4a). The crystal of **4** at 150 K has the same crystal symmetry as at 297 K. However, the magnitude of the π–π dimer interaction in the **A**–**A** unit is significantly different. The intradimer t_1 interaction at 150 K (11.34) is ca. 30% larger than that at 297 K.

The magnetic exchange energy J within the dimer at 150 K is 1.5 times larger than that at 297 K from proportional relationship $J \propto t^2$, thus the magnetic transition at 190 K is due to the spin singlet formation of the **A**–**A** dimer. The spin from monomer **B** contributes to the magnetism below 190 K, which has a novel π–π overlap mode with 120° bending of the long axis of the [Ni(dmit)$_2$]$^-$ anions.

The temperature dependent χ_m behavior observed in salt **5** obeys the Curie–Weiss law in the temperature range from 40 to 350 K (Fig. 4b). The almost constant $\chi_m T$ value (0.73 emu K mol^{-1}) suggests the absence of any effective magnetic interaction between the [Ni(dmit)$_2$]$^-$ anions above 40 K. Below 40 K, a weak antiferromagnetic interaction is observed ($\theta = -8.5$ K). Since the magnitude of the intradimer π–π interaction in salt **5** is much smaller than that observed in salt **4**, the formation of a singlet pair on **A**–**A** or **B**–**B** dimers is not observed.

The temperature dependent χ_m of salt **6** has a broad χ_m maximum at around 15 K, and the behavior can be fitted by the one-dimensional Heisenberg antiferromagnetic linear chain model (solid line in Fig. 4c). The uniform zig-zag chain of [Ni(dmit)$_2$]$^-$ anions as confirmed by the X-ray crystal structural analysis is consistent with the temperature dependent magnetic behavior. The intrachain magnetic exchange energy estimated from the fitting result in salt **6** was $J/k_B = -11.7$ K, which originated from the regular π–π interaction ($t \sim 4$) of [Ni(dmit)$_2$]$^-$ anions in the crystal.

The orthogonal S–S interactions between [Ni(dmit)$_2$]$^-$ are characteristic of the divalent supramolecular cation salts. The magnitude of the orthogonal S–S interactions found in **3**–**6** is observed in the t range from 0.5 to 2, whose absolute value is not as large compared to those of the intradimer interactions. However, these anisotropic S–S interactions will be important for connecting the magnetic interactions over the crystal.

One of the advantages in using a supramolecular cation structure is that we can replace metal cations with the other cations without significant lattice distortion. Since the divalent Ca^{2+} cations were successfully incorporated into the [Ni(dmit)$_2$]$^-$ salts, we attempted to introduce divalent transition metal cations having magnetic moments. However, it was necessary to find appropriate conditions for crystal preparation due to the high chemical reactivity of transition metal cations with the [Ni(dmit)$_2$]$^-$ species.

Fig. 5 shows the crystal structure of Co^{2+}(15-crown-5)[Ni(dmit)$_2$]$_2$(CH$_3$CN)$_2$ (**7**) which has the same crystal structure as **6** [13]. The crystal exhibited a larger mag-

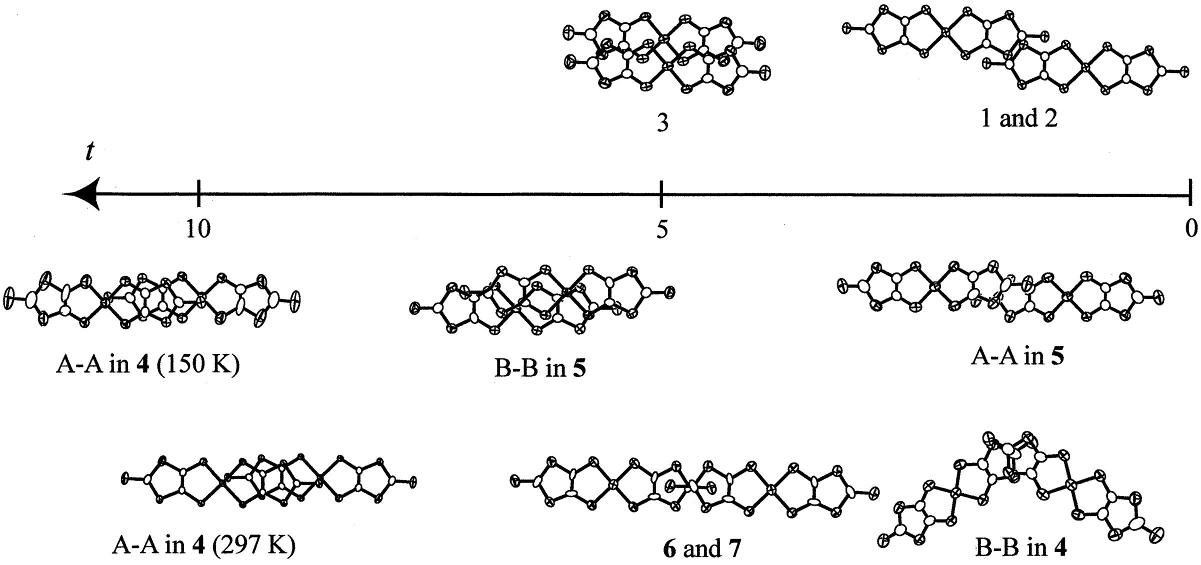

Fig. 3. Intermolecular interaction modes of [Ni(dmit)$_2$]$^-$ anions observed in the monovalent M$^+$(crown ethers)[Ni(dmit)$_2$]$^-$ (upper two) and divalent Ca^{2+}(crown ethers)[Ni(dmit)$_2$]$_2$ (lower six).

Table 3
π–π Interaction mode and transfer integral ($t \times 10^{-2}$ eV) [a] in salts **1**–**6**

	1	**2**	**3**	**4**	**4′** [b]	**5**	**6**
Packing mode [c]	Regular	Regular	D	D–M	D–M	D–M	Regular
t_1 (**A**–**A**)	1.03	2.77	5.82	8.96	11.34	2.54	4.26
t_2 (**B**–**B**)	–	–	–	−3.47	−2.21	5.67	–
Magnetic property	1D AF	1D AF	Curie–Weiss	Curie–Weiss	Curie–Weiss	Curie–Weiss	1D AF

[a] The transfer integrals (t) were obtained from the LUMO of [Ni(dmit)$_2$]$^-$ based on the extended Hückel calculation ($t = -10S$ eV, S is overlap integral).

[b] Based on the crystal structure at 150 K.

[c] Dimers (D) and monomers (M) coexist in the crystal.

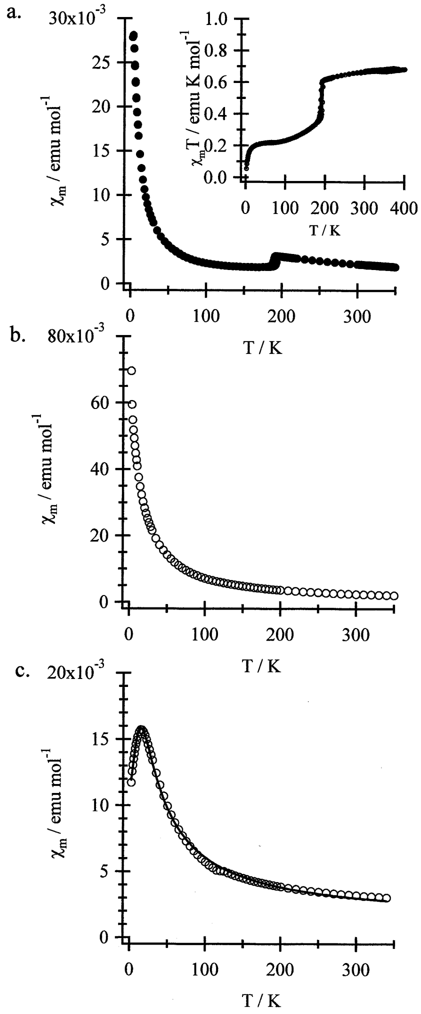

Fig. 4. Temperature dependent magnetic susceptibility (χ_m) of the salts (a) **4**, (b) **5** and (c) **6**.

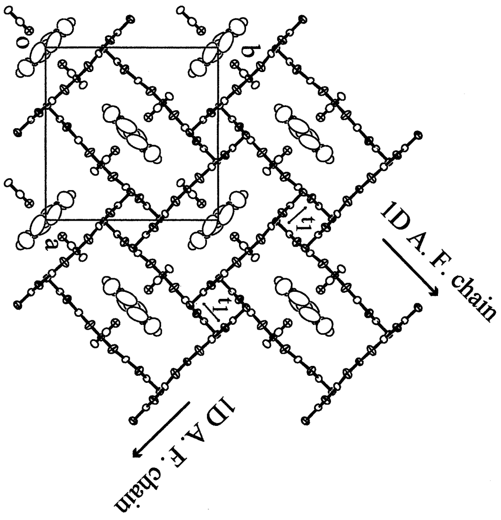

Fig. 5. Crystal structure of Co^{2+}(15-crown-5)[Ni(dmit)$_2$]$_2$(CH_3CN)$_2$ (**7**). Unit cell viewed along the *c*-axis. The [Ni(dmit)$_2$]$^-$ molecules form a one-dimensional antiferromagnetic chain along the $a+b$ and $a–b$ axes through the t_1 interaction.

netic susceptibility, which was the sum of that of [Ni(dmit)$_2$]$^-$ and the Co^{2+} Curie spin. We did not observe a significant magnetic interaction between [Ni(dmit)$_2$]$^-$ and Co^{2+}, because the interaction is prevented by the crown ether moiety surrounding Co^{2+}.

A magnetic interaction between the supramolecular cations and [Ni(dmit)$_2$]$^-$ was, however, made possible by introducing a π-conjugated system in the supramolecular cation moiety. We recently reported that Cu^{2+} spins in aza-annulene moiety interact with [Ni(dmit)$_2$]$^-$ in the crystal [14].

3.4. Trivalent supramolecular cation salts

Trivalent or higher-valence cations may be introduced into the supramolecular structure. Thus, we can also utilize the f-spins of lanthanoid cations in addition to d-spins of transition metal cations to coexist with [Ni(dmit)$_2$]$^-$ magnetic moments. Fig. 6 shows the crystal structure of Sc^{3+}(12-crown-4)$_2$[Ni(dmit)$_2$]$_3$ (**8**) as an example of a trivalent supramolecular cation salt of [Ni(dmit)$_2$]$^-$. [Ni(dmit)$_2$]$^-$ formed a strong trimer structure and one $S=1/2$ spin on each trimer showed Curie-type paramagnetism [15]. The introduction of a trivalent cation with f-spins is also difficult due to the chemical reactivity as in the case of transition metal cations with d-spins. However, it is possible to synthesize [Ni(dmit)$_2$]$^-$ salts of lanthanoid containing supramolecular cations. Analysis of their crystal structures is now underway [16].

4. Conclusions

Supramolecular cation structures were assembled with the [Ni(dmit)$_2$]$^-$ anions bearing $S=1/2$ spin. By

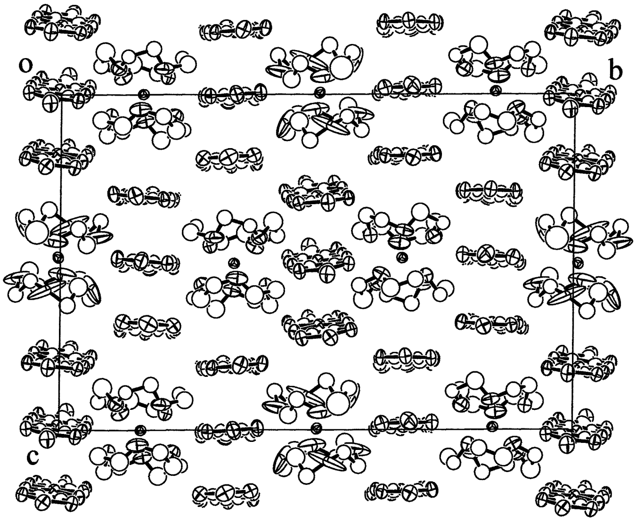

Fig. 6. Crystal structure of Sc^{3+}(12-crown-4)$_2$[Ni(dmit)$_2$]$_3$(CH_3CH)$_4$ (**8**). Unit cell viewed along the *c*-axis. The [Ni(dmit)$_2$]$^-$ molecules form the π–π trimer unit.

using supramolecular cation structures with the [Ni(dmit)$_2$] assembly, a diversity in structures as well as magnetic behavior was observed. Further design for obtaining novel magnetic properties of a [Ni(dmit)$_2$] based magnetic system will be possible using a variety of cations and crown ethers. We can use cyclohexyl crown ethers [17] or benzo derivatives as well as linear chain polyethers [18] and cryptands. The cations are not necessarily limited to inorganic species. It is well known that organic ammonium moieties can be contained inside the crown ether cavity. Novel [Ni(dmit)$_2$]$^-$ assemblies and magnetism should be observed among these supramolecular cation based [Ni(dmit)$_2$]$^-$ crystals.

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