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Synthesis, Crystal Structure, and Luminescence Properties of Coordination Polymers Based on Cadmium Isonicotinates

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Abstract—Heating a solution of cadmium nitrate and isonicotinic acid in N,N'-dimethylformamide or dimethyl sulfoxide gave three new coordination polymers, $[Cd(Inic)_2] \cdot 0.5DMF$ (**I**), $[Cd_3(DMSO)_6(Inic)_2(SO_4)_2]$ (**II**), and $[\{Cd(DMSO)(Inic)\}_2(SO_4)] \cdot 2H_2O$ (**III**), which were studied by X-ray crystallography. Compounds **I** and **III** were characterized by IR spectroscopy and elemental and thermogravimetric analyses and luminescence spectroscopy.

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Metal-organic coordination polymers (MOCPs) are compounds in which metal cations are linked by polytopic organic ligands to give one-, two-, and three-dimensional structures. This class of compounds is vigorously studied due to the extensive possibilities of the chemical design of coordination polymers and diversity of properties they exhibit [1–3]. Since coordination polymers can be prepared using a wide range of metal cations and organic ligands with various functional groups, they can exhibit a variety of functional properties, for example, catalytic [4], luminescence [5], and magnetic [6, 7] properties. Porous MOCPs can sorb gases and small organic or inorganic molecules [8]. The formation of MOCPs in the synthesis is fairly sensitive to experimental conditions, the reaction outcome being dependent on the solvent, temperature, reaction time, and reactant ratio and concentrations [9, 10]. By varying reaction conditions, it is possible to obtain different products from the same reactants. This communication reports the synthesis and crystal structure of three new MOCPs, namely, $[Cd(Inic)_2] \cdot 0.5DMF$ (**I**), $[Cd_3(DMSO)_6(Inic)_2(SO_4)_2]$ (**II**), and $[\{Cd(DMSO)(Inic)\}_2(SO_4)] \cdot 2H_2O$ (**III**) (HInic is isonicotinic acid, DMF is N,N'-dimethylformamide, DMSO is dimethyl sulfoxide) prepared by heating cadmium nitrate and isonicotinic acid in different solvents, DMF or DMSO.

EXPERIMENTAL

The starting compounds, HInic, cadmium nitrate(II) tetrahydrate, DMF, and DMSO were at least reagent grade chemicals. IR spectra in the 4000–400 cm^{-1} range were measured on a Scimitar FTS 2000 Fourier transform spectrometer (KBr pellets). Elemental analysis was performed on a EURO EA 3000 EuroVector instrument. Thermogravimetric analysis (TGA) was done using a TG 209 F1 Netzsch thermobalance; the samples were decomposed in a helium atmosphere, and the heating rate was 10 K/min. The fluorescence spectra were measured on a Varian Cary Eclipse instrument.

Synthesis of I. Weighed portions of $Cd(NO_3)_2 \cdot 4H_2O$ (0.308 g, 1 mmol) and HInic (0.123 g, 1 mmol) were dissolved in DMF (10 mL). The resulting solution was heated in a sealed tube at 100°C. After 3 days, colorless needle crystals formed. The precipitate was washed with dimethylformamide and methanol and dried in air. Yield 0.148 g (39% in relation to Cd).

For $C_{13.5}H_{11.5}N_{2.5}O_{4.5}Cd$			
calculated (%):	C, 41.2;	H, 2.9;	N, 8.9.
Found (%):	C, 40.7;	H, 2.9;	N, 8.8.

IR (ν, cm^{-1}): 3466, 3056, 2937, 2871, 1966, 1675, 1604, 1556, 1546, 1416, 1393, 1322, 1221, 1088, 1063, 1021, 861, 839, 777, 727, 707, 682, 582, 549, 449, 416.

TGA. Found: $\Delta m = 10\%$, calculated for the loss of 0.5 DMF in **I**: $\Delta m = 10\%$.

Synthesis of II. Compound **II** was prepared under synthesis conditions similar to those of the preparation of **I** but using 10 mL of DMSO as the solvent. The pale yellow plate-shaped crystals were formed at 120°C in 3 weeks. The precipitate was washed with dimethyl sulfoxide and methanol and dried in air. Yield 0.081 g (20% in relation to Cd).

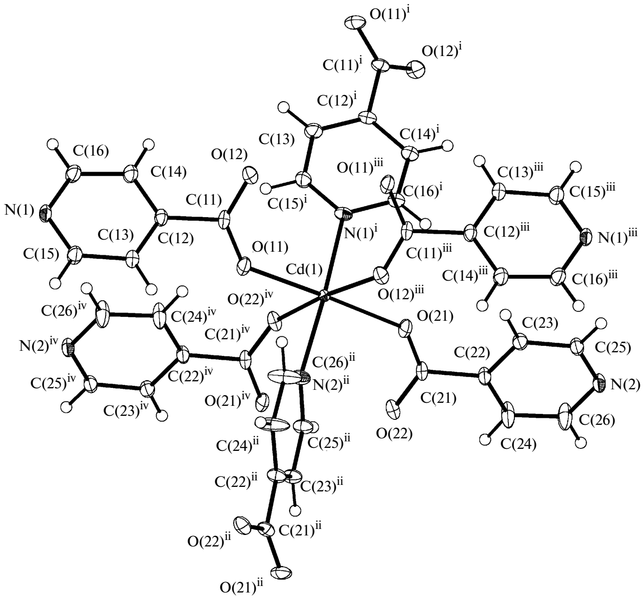

Fig. 1. Coordination environment of the cadmium cation in **I**. 50% probability ellipsoids. The coordinates of dependent atoms were obtained by the following symmetry operations: ^{i} $1 - x$, $1/2 + y$, $3/2 - z$; ^{ii} $-x$, $-1/2 + y$, $1/2 - z$; ^{iii} $1 - x$, $-y$, $1 - z$; ^{iv} $-x$, $-y$, $1 - z$.

Synthesis of III. Compound **III** was prepared under the same conditions as compound **II** but the reaction mixture was heated at 100°C for 4 weeks. Yield 0.110 g (30% in relation to Cd). The single crystals for X-ray diffraction were picked from the reaction solution. According to X-ray diffraction data, the composition of the crystals corresponded to the formula $[\{Cd(DMSO)(Inic)\}_2(SO_4)] \cdot 2H_2O$. After separation, the crystals were washed with methanol and dried at room temperature. According to chemical analysis, TGA, and IR spectroscopy, the composition of the crystalline compound was described as $[\{Cd(DMSO)(Inic)\}_2(SO_4)]$.

For $C_{16}H_{20}N_2O_{10}S_3Cd_2$

calculated (%):	C, 26.6;	H, 2.8;	N, 3.9.
Found (%):	C, 26.8;	H, 2.8;	N, 3.7.

IR (ν, cm^{-1}): 3097, 2908, 1617, 1587, 1546, 1427, 1408, 1319, 1229, 1128, 1105, 1024, 1001, 950, 874, 860, 778, 712, 703, 625, 606, 438, 411.

TGA. Found: $\Delta m = 22\%$, calculated for the loss of 2 DMSO in **III**: $\Delta m = 22\%$.

X-ray diffraction. X-Ray diffraction data for the single crystals of **I** and **III** were collected on Bruker X8Apex CCD and Bruker Apex Duo four-circle automated diffractometers, respectively ($\lambda(MoK_\alpha) = 0.71073$ Å, graphite monochromator, two-coordinate detector, φ-scan mode). The primary data including the unit cell parameters, the Miller indices, and the intensities of measured reflections were processed by means of software supplied with the diffractometer. The absorption corrections were based on the divergence of equivalent reflections using the SADABS program [11]. The X-Ray diffraction data for the single crystal of **II** were collected at the Macromolecular Crystallography Wiggler Beamline 4A (Pohang Accelerator Laboratory) equipped with a one-circle goniometer and a two-coordinate Quantum ADSC 210 detector ($\lambda = 0.80000$ Å, silicon monochromator, φ scan mode). The data collection, frame integration, processing of the set of reflections, and absorption correction were performed using the HKL2000 program package [12]. The structures **I–III** were solved by the direct method and refined by full-matrix least-squares method in the anisotropic approximation (except for hydrogen atoms) using the SHELX-97 program package [13]. The positions of organic ligand hydrogen atoms were calculated geometrically and refined using the riding model. The water hydrogen atoms were not located. The crystallographic data and X-ray experiment details are summarized in Table 1. Selected interatomic distances and bond angles for **I–III** are presented in Table 2. The full tables of interatomic distances and bond angles, atom coordinates and displacement parameters are deposited with the Cambridge Crystallographic Data Centre (nos. 876504–876506; deposit@ccdc.cam.ac.uk or http://www.ccdc.cam.ac.uk/data_request/cif) and are available from the authors.

RESULTS AND DISCUSSION

Cadmium coordination polymers **I–III** were obtained using the same starting reactants, cadmium nitrate and isonicotinic acid, taken in the same ratios but using different solvents and different heating schedules. The reaction carried out in DMF affords coordination polymer **I**. During the synthesis of compounds **II** and **III**, DMSO is partially oxidized due to long-term heating (3 to 4 weeks). The sulfate anion thus formed participates in the formation of coordination polymers.

In compound **I**, the cadmium cation is located in the octahedral environment composed of two nitrogen atoms of the isonicotinate ligands, which are located in *trans*-positions relative to each other, and four oxygen atoms of the carboxyl groups of four isonicotinate anions (Fig. 1). The Cd–O and Cd–N bond lengths (2.270(3)–2.337(4) Å) fit into the normal bond length distribution in Cd(II) octahedral complexes. Two types of isonicotinate anions are present. Each anion is coordinated to three cadmium cations either through nitrogen (one cation) or through carboxyl oxygens (two cations). This gives a metal-organic framework with channels of square cross-section (4 × 4 Å) running along the *x* axis (Fig. 2). According to X-ray diffraction, elemental analysis, and TGA data, the channels accommodate DMF guest molecules disordered over two sites around the inversion center (0.5 mole-

Table 1. Crystal data and structure refinement parameters for **I–III**

Parameter	Value		
	I	**II**	**III**
Molecular formula	$C_{13.5}H_{11.5}N_{2.5}O_{4.5}Cd$	$C_{24}H_{44}N_2O_{18}S_8Cd_3$	$C_{16}H_{24}N_2O_{12}S_3Cd_2$
M	393.15	1242.29	757.35
Temperature, K	150(2)	100(2)	100(2)
System	Monoclinic	Triclinic	Tetragonal
Space group	$P2_1/c$	$P\bar{1}$	$I4/m$
a, Å	9.8383(6)	9.522(1)	15.7960(4)
b, Å	13.1984(9)	10.354(1)	15.7960(4)
c, Å	11.3829(8)	12.014(1)	10.3855(2)
α, deg	90	108.742(1)	90
β, deg	101.437(2)	109.761(1)	90
γ, deg	90	96.696(1)	90
V, Å^3	1448.72(17)	1021.34(17)	2591.32(11)
Z	4	1	4
ρ(calcd.), g/cm^3	1.803	2.020	1.941
μ, mm^{-1}	1.529	2.767	1.943
$F(000)$	776	618	1496
Crystal size, mm	0.20 × 0.04 × 0.04	0.10 × 0.10 × 0.04	0.10 × 0.08 × 0.08
Scanning range over θ, deg	2.11–33.13	2.20–30.39	1.82–30.60
Range of indices h, k, l	$-9 < h < 16$, $-19 < k < 21$, $-18 < l < 15$	$-13 < h < 13$, $-14 < k < 14$, $-16 < l < 16$	$-23 < h < 23$, $-23 < k < 22$, $-8 < l < 15$
The number of measured/independent reflections	14660/5499	6821/3664	11086/2094
R_{int}	0.0331	0.0293	0.0186
The number of reflections with $F > 4\sigma(F)$	4167	3479	1953
T_{max}/T_{min}	0.9414/0.7496	0.8974/0.7694	0.8601/0.8295
GOOF on F^2	1.218	1.146	1.062
Final R-indices ($F > 4\sigma(F)$)	$R_1 = 0.0478$, $wR_2 = 0.1102$	$R_1 = 0.0304$, $wR_2 = 0.0881$	$R_1 = 0.0178$, $wR_2 = 0.0466$
R-indices (for all data)	$R_1 = 0.0698$, $wR_2 = 0.1142$	$R_1 = 0.0328$, $wR_2 = 0.0996$	$R_1 = 0.0199$, $wR_2 = 0.0476$
$\Delta\rho_{max}/\Delta\rho_{min}$, e/Å^3	1.967/–0.931	0.776/–1.764	0.888/–1.999

cules per formula unit), which can be removed only by heating to 330°C (Fig. 3). According to TGA data, framework destruction in a helium atmosphere occurs above 360°C.

The vibrational spectrum of **I** exhibits absorption bands for the coordinated carboxyl group of the isonicotinate residue at 1392 cm^{-1} (symmetric) and 1605 cm^{-1} (asymmetric). The presence of guest DMF molecules is confirmed by the signals at 2800–3000 cm^{-1} (methyl groups), 1675 cm^{-1} (carbonyl group) [14].

The structure of **II** contains two types of cadmium cations, the number of Cd(2) being twice that of Cd(1). The latter (at the inversion center) is located in the octahedral environment composed of two dimethyl sulfoxide oxygen atoms, two oxygen atoms of bridging sulfate anions, and two nitrogen atoms of two isonicotinate ligands (Fig 4). The Cd(1)–O and Cd(1)–N bond lengths are in the range of 2.292(2)–2.347(3) Å.

The Cd(2) atom has a trigonal bipyramidal geometry. The axial positions are occupied by DMSO oxygen atoms. Two oxygen atoms of the bridging sulfate anions lie in the equatorial plane. It is likely that the remaining coordination site is occupied by two oxygen atoms of the bidentately coordinated carboxyl group of the isonicotinate anion (Fig. 4). The Cd(2)–O bond lengths are in the range of 2.211(2)–2.396(2) Å.

Table 2. Selected bond lengths and bond angles in **I–III**

I

Bond	*d*, Å	Bond	*d*, Å
Cd(1)–O(11)	2.281(3)	Cd(1)–O(21)	2.270(3)
Cd(1)–O(12)$^{\mathrm{i}}$	2.329(3)	Cd(1)–O(22)$^{\mathrm{iii}}$	2.323(3)
Cd(1)–N(1)$^{\mathrm{ii}}$	2.337(4)	Cd(1)–N(2)$^{\mathrm{iv}}$	2.290(4)
Angle	ω, deg	Angle	ω, deg
O(11)Cd(1)O(12)$^{\mathrm{i}}$	95.59(12)	O(21)Cd(1)O(22)$^{\mathrm{iii}}$	92.46(12)
O(11)Cd(1)N(1)$^{\mathrm{ii}}$	96.93(13)	O(21)Cd(1)N(2)$^{\mathrm{iv}}$	94.89(14)
O(11)Cd(1)O(22)$^{\mathrm{iii}}$	89.70(12)	O(22)$^{\mathrm{iii}}$Cd(1)O(12)$^{\mathrm{i}}$	174.49(12)
O(11)Cd(1)N(2)$^{\mathrm{iv}}$	82.19(14)	O(22)$^{\mathrm{iii}}$Cd(1)N(1)$^{\mathrm{ii}}$	85.42(13)
O(12)$^{\mathrm{i}}$Cd(1)N(1)$^{\mathrm{ii}}$	95.44(13)	N(2)$^{\mathrm{iv}}$Cd(1)O(12)$^{\mathrm{i}}$	89.02(13)
O(21)Cd(1)O(11)	176.37(13)	N(2)$^{\mathrm{iv}}$Cd(1)N(1)$^{\mathrm{ii}}$	175.52(14)
O(21)Cd(1)O(12)$^{\mathrm{i}}$	82.18(12)	N(2)$^{\mathrm{iv}}$Cd(1)O(22)$^{\mathrm{iii}}$	90.17(13)
O(21)Cd(1)N(1)$^{\mathrm{ii}}$	86.15(14)		

Symmetry codes for **I**: $^{\mathrm{i}}$ $-x + 1, -y, -z + 1$; $^{\mathrm{ii}}$ $-x + 1, y + 1, -z + 3/2$; $^{\mathrm{iii}}$ $-x, -y, -z + 1$; $^{\mathrm{iv}}$ $x, y - 1/2, -z + 1/2$.

II

Bond	*d*, Å	Bond	*d*, Å
Cd(1)–N(1)	2.347(3)	Cd(2)–O(12)$^{\mathrm{i}}$	2.211(2)
Cd(1)–O(11)	2.292(2)	Cd(2)–O(13)$^{\mathrm{ii}}$	2.250(2)
Cd(1)–O(21)	2.306(2)	Cd(2)–O(31)	2.296(2)
Cd(2)–O(1)	2.382(2)	Cd(2)–O(41)	2.350(2)
Cd(2)–O(2)	2.390(3)		
Angle	ω, deg	Angle	ω, deg
O(11)Cd(1)N(1)	90.58(9)	O(13)$^{\mathrm{ii}}$Cd(2)O(1)	94.95(8)
O(11)Cd(1)O(21)	84.30(9)	O(13)$^{\mathrm{ii}}$Cd(2)O(2)	148.45(8)
O(21)Cd(1)N(1)	86.75(10)	O(13)$^{\mathrm{ii}}$Cd(2)O(31)	96.67(9)
O(1)Cd(2)O(2)	55.43(8)	O(13)$^{\mathrm{ii}}$Cd(2)O(41)	83.13(9)
O(12)$^{\mathrm{i}}$Cd(2)O(1)	154.86(9)	O(31)Cd(2)O(1)	87.11(9)
O(12)$^{\mathrm{i}}$Cd(2)O(2)	99.62(8)	O(31)Cd(2)O(2)	92.71(9)
O(12)$^{\mathrm{i}}$Cd(2)O(13)$^{\mathrm{ii}}$	108.71(9)	O(31)Cd(2)O(41)	174.48(8)
O(12)$^{\mathrm{i}}$Cd(2)O(31)	98.15(9)	O(41)Cd(2)O(1)	87.42(9)
O(12)$^{\mathrm{i}}$Cd(2)O(41)	87.11(8)	O(41)Cd(2)O(2)	84.74(9)

Symmetry codes for **II**: $^{\mathrm{i}}$ $-x + 1, -y + 1, -z + 1$; $^{\mathrm{ii}}$ $x + 1, y + 1, z$; $^{\mathrm{iii}}$ $-x, -y, -z$.

III

Bond	*d*, Å	Bond	*d*, Å
Cd(1)–N(1)	2.3309(18)	Cd(1)–O(11)	2.2566(11)
Cd(1)–O(1)$^{\mathrm{i}}$	2.3549(11)	Cd(1)–O(21)	2.3129(16)
Angle	ω, deg	Angle	ω, deg
N(1)Cd(1)O(1)$^{\mathrm{i}}$	86.98(5)	O(11)$^{\mathrm{iii}}$Cd(1)O(11)	100.74(6)
O(1)$^{\mathrm{ii}}$Cd(1)O(1)$^{\mathrm{i}}$	56.41(6)	O(11)Cd(1)O(21)	84.74(4)
O(11)Cd(1)N(1)	100.40(4)	O(21)Cd(1)N(1)	171.83(6)
O(11)Cd(1)O(1)$^{\mathrm{ii}}$	100.58(4)	O(21)Cd(1)O(1)$^{\mathrm{i}}$	85.82(5)
O(11)Cd(1)O(1)$^{\mathrm{i}}$	155.73(4)		

Symmetry codes for **III**: $^{\mathrm{i}}$ $-y + 1, x, z$; $^{\mathrm{ii}}$ $-y + 1, x, -z$; $^{\mathrm{iii}}$ $x, y, -z$.

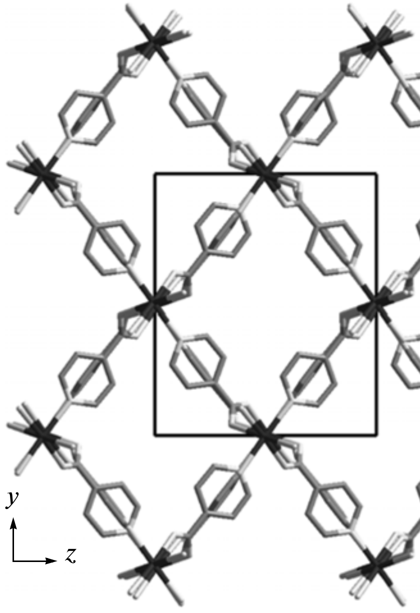

Fig. 2. Structure of the framework of **I**.

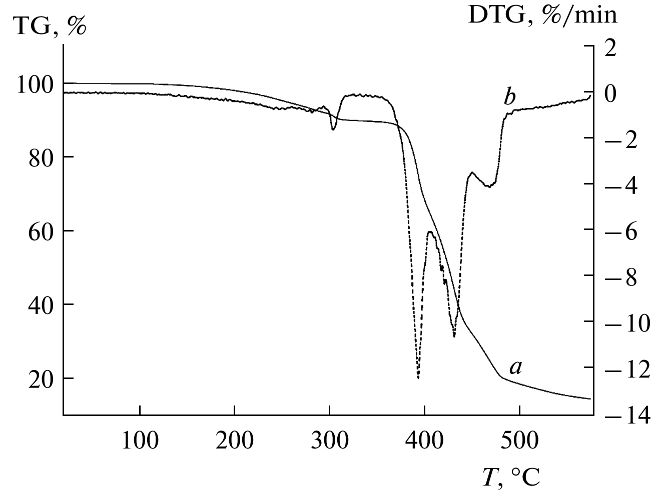

Fig. 3. (*a*) TG and (*b*) DTG curves for compound **I**.

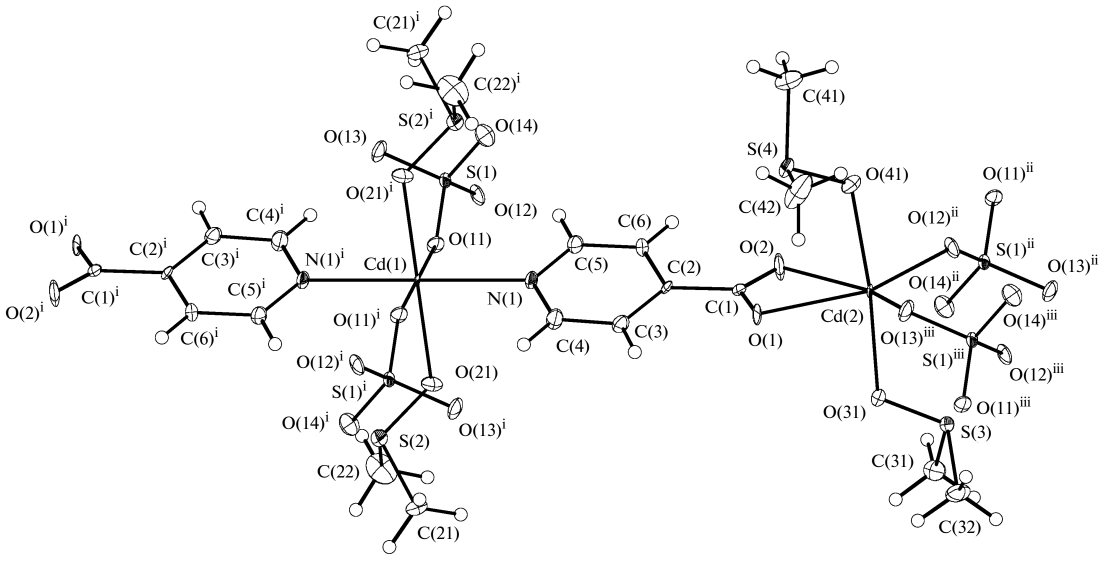

Fig. 4. Coordination environment of cadmium cations in **II**. 70% probability ellipsoids. The coordinates of dependent atoms were obtained by the following symmetry operations: $^{\mathrm{i}}$ 1 − *x*, 1/2 + *y*, 3/2 − *z*; $^{\mathrm{ii}}$ −*x*, −1/2 + *y*, 1/2 − *z*; $^{\mathrm{iii}}$ 1 − *x*, −*y*, 1 − *z*; $^{\mathrm{iv}}$ −*x*, −*y*, 1 −*z*.

Each isonicotinate ligand is coordinated to the Cd(1) and Cd(2) cations. The bridging sulfate anions connect one Cd(1) and two Cd(2) cations to form layers (Fig. 5a) in the (−1 1 0) planes (Fig 5b). The coordinated solvent molecules directed toward the space between the layers.

In compound **III**, the cadmium cation occurs in a trigonal bipyramidal environment (Fig. 6). The axial positions are occupied by dimethyl sulfoxide oxygen atoms and isonicotinate nitrogen atoms. In the equatorial plane, there are two oxygen atoms of the bridging sulfate anions, and the remaining site is occupied by two oxygen atoms of the bidentate carboxyl group of the isonicotinate anion.

The sulfate anions are coordinated to metal cations by four oxygen atoms thus linking them to form $\{Cd_2SO_4\}_\infty^{2+}$ chains (Fig. 7a). The chains are connected by isonicotinic acid anions to form a framework with channels directed along a fourfold rotation axis (Fig. 7b). The coordinated DMSO molecules are thus

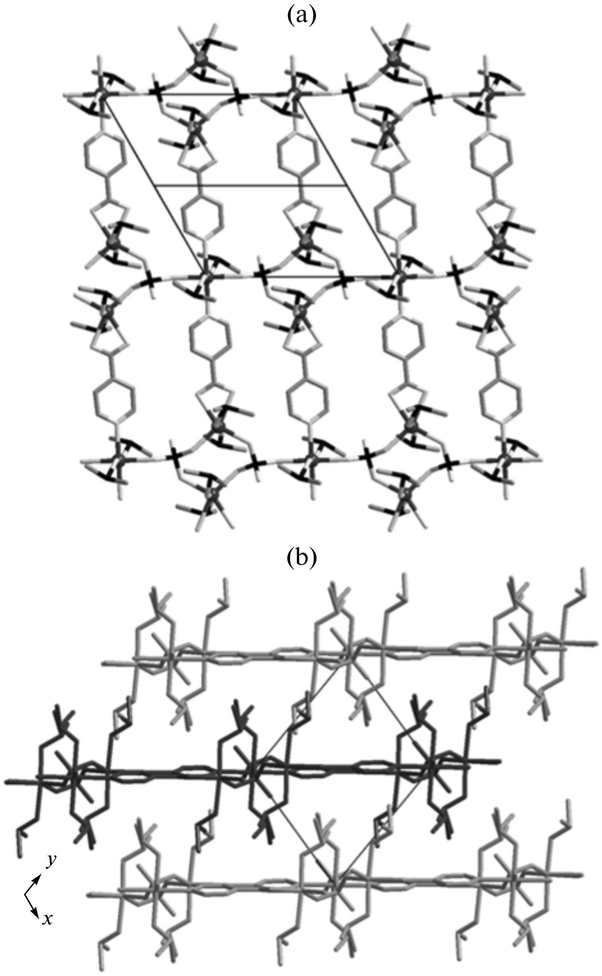

Fig. 5. (a) Structure of a layer in compound **II**; (b) layer packing in **II** (different layers are shown by different shades of gray).

directed inside the channels. According to X-ray diffraction analysis performed at 100 K, the channels accommodate disordered water molecules (two per formula unit). However, according to TGA, elemental analysis, and IR spectroscopy data, these water molecules are completely removed upon drying in air.

The thermal stability of **III** was studied by TGA in a helium atmosphere. The first step in the mass loss curve (below 325°C) corresponds to complete removal of the coordinated DMSO molecules. The framework destruction starts at 360°C.

The IR spectrum of compound **III** exhibits absorption bands inherent in the coordinated carboxyl group of the isonicotinic acid, namely, symmetric bands at 1408 cm^{-1} and asymmetric bands at 1587 cm^{-1} [14]. The coordinated DMSO is responsible for 950 cm^{-1} bands (S=O stretching vibrations) and 2900–3100 cm^{-1} bands (methyl groups). The sulfate anion vibration frequencies occur in the ranges of 1080–1150 cm^{-1} (stretching) and 580–680 cm^{-1} (bending). The lack of absorption bands at 3500–3300 and 1600 cm^{-1} implies that at room temperature no guest water molecules are present in the framework channels.

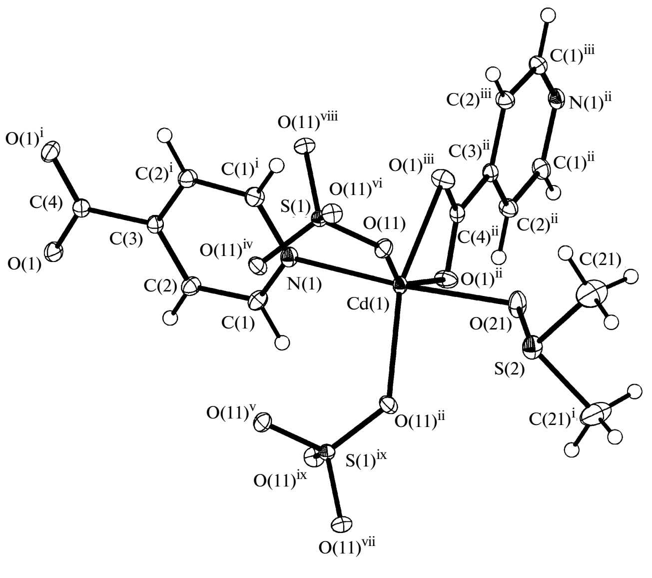

Fig. 6. Coordination environment of cadmium cations in **III**. 50% probability ellipsoids. The coordinates of dependent atoms were obtained by the following symmetry operations: $^{\text{i}}$ $x, y, -z$; $^{\text{ii}}$ $1 - y, x, z$; $^{\text{iii}}$ $1 - y, x, -z$; $^{\text{iv}}$ $1 - x, -y, z$; $^{\text{v}}$ $1 - x, -y, -z$; $^{\text{vi}}$ $1/2 + y, 1/2 - x, 1/2 - z$; $^{\text{vii}}$ $1/2 + y, 1/2 - x, -1/2 + z$; $^{\text{viii}}$ $1/2 - y, -1/2 + x, 1/2 - z$; $^{\text{ix}}$ $1/2 + y, -1/2 + x, -1/2 + z$.

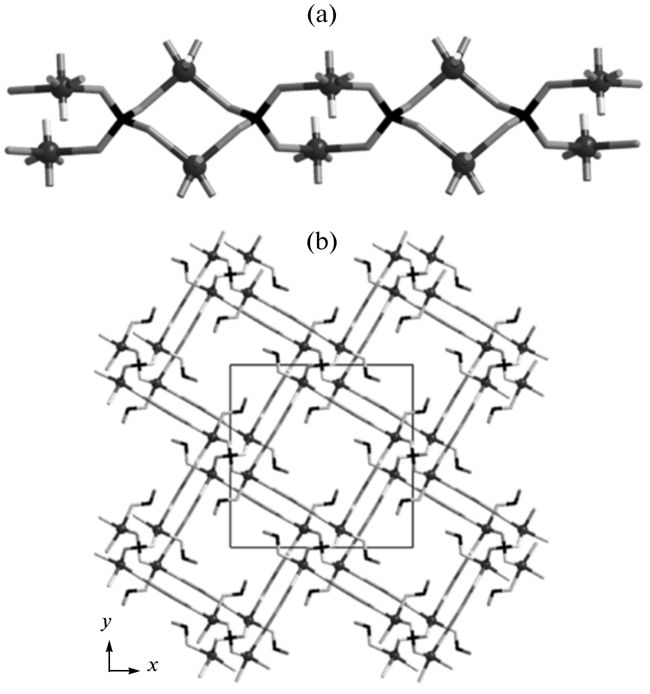

Fig. 7. (a) $\{\text{Cd}_2\text{SO}_4\}_\infty^{2+}$ chains in structure **III**; (b) framework structure of **III** viewed along the channels.

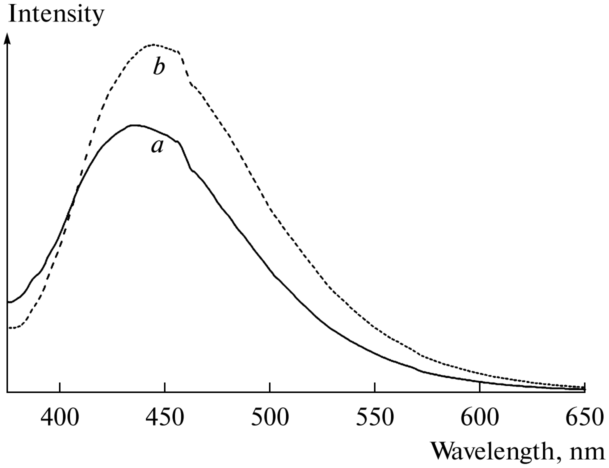

Fig. 8. Luminescence spectra of (*a*) **I** and (*b*) **III**.

Fluorescence spectra at room temperature were recorded for solid compounds **I** and **III** (Fig. 8). The spectra were measured in the 375–650 nm range with λ_{excit} = 360 nm. The spectra of both compounds exhibit a broad strong emission band with a maximum at 435 nm for **I** and 445 nm for **III**, which can be assigned to the intraligand charge transfer. The neutral isonicotinic acid molecule is responsible for very weak luminescence in the same region at λ_{excit} = 360 nm. The enhanced emission in **I** and **III** may be attributed to the coordination of the isonicotinic ligand to cadmium cations [15, 16].

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