Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

Poly[tetrakis(selenocyanato-κN)bis(methanol-κO)tris(μ-pyrimidine-$\kappa^2 N:N'$)-dicobalt(II)]

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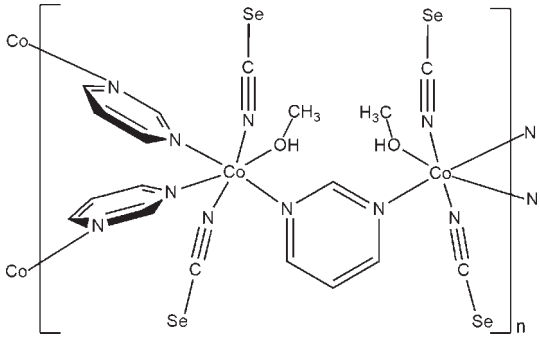
Received 25 May 2010; accepted 31 May 2010

Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.006 Å; R factor = 0.038; wR factor = 0.069; data-to-parameter ratio = 20.7.

In the title compound, $[Co_2(NCSe)_4(C_4H_4N_2)_3(CH_3OH)_2]_n$, the Co^{II} ion is coordinated by three N-bonded pyrimidine ligands, two N-bonded selenocyanate anions and one O-bonded methanol molecule in an octahedral coordination mode. The asymmetric unit consists of one Co^{II} ion, one pyrimidine ligand, two selenocyanate anions and one methanol molecule in general positions as well as one pyrimidine ligand located around a twofold rotation axis. In the crystal structure, the pyrimidine ligands bridge $[Co(CNSe)_2(CH_3OH)]$ units into zigzag-like chains, which are further connected by pyrimidine ligands into layers parallel to (010).

Related literature

For general background, see: Wriedt & Näther (2009*a*,*b*); Wriedt, Sellmer & Näther (2009*a*,*b*). For the isotypic structure of a nickel thiocyanato complex, see: Wriedt *et al.* (2009).



Experimental

Crystal data

$[Co_2(CNSe)_4(C_4H_4N_2)_3(CH_4O)_2]$ M_r = 421.07
Orthorhombic, *Fdd*2
a = 20.4069 (8) Å
b = 33.0633 (15) Å
c = 8.3750 (3) Å
V = 5650.8 (4) Å^3
Z = 16
Mo $K\alpha$ radiation
μ = 6.36 mm^{-1}
T = 293 K
0.16 × 0.11 × 0.02 mm

Data collection

Stoe IPDS-2 diffractometer
Absorption correction: numerical (*X-SHAPE* and *X-RED32*; Stoe & Cie, 2002)
T_{min} = 0.431, T_{max} = 0.885
18545 measured reflections
3391 independent reflections
3067 reflections with $I > 2\sigma(I)$
R_{int} = 0.044

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.038
$wR(F^2)$ = 0.069
S = 1.16
3391 reflections
164 parameters
1 restraint
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.55 e Å^{-3}
$\Delta\rho_{min}$ = −0.35 e Å^{-3}
Absolute structure: Flack (1983), 1579 Friedel pairs
Flack parameter: 0.057 (13)

Table 1
Selected bond lengths (Å).

Co1—N1	2.191 (3)	Co1—N21	2.064 (4)
Co1—N2^{i}	2.188 (3)	Co1—N31	2.059 (4)
Co1—N11	2.184 (3)	Co1—O41	2.142 (3)

Symmetry code: (i) $-x+1, -y+\frac{1}{2}, z+\frac{1}{2}$.

Data collection: *X-AREA* (Stoe & Cie, 2002); cell refinement: *X-AREA*; data reduction: *X-AREA*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXTL*.

MW thanks the Stiftung Stipendien-Fonds des Verbandes der Chemischen Industrie and the Studienstiftung des deutschen Volkes for a PhD scholarship. We gratefully acknowledge financial support by the State of Schleswig-Holstein and the Deutsche Forschungsgemeinschaft (Project 720/3-1). We thank Professor Dr Wolfgang Bensch for the opportunity to use of his experimental facilities.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HY2313).

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supplementary materials

Acta Cryst. (2010). E**66**, m742 [**doi:10.1107/S160053681002060X**]

Poly[tetrakis(selenocyanato-κN)bis(methanol-κO)tris(μ-pyrimidine-$\kappa^2 N:N'$)dicobalt(II)]

M. Wriedt, I. Jess and C. Näther

Comment

Recently, we have shown that thermal decomposition reactions are an elegante route for the discovering and preparation of new ligand-deficient coordination polymers with defined magnetic properties (Wriedt & Näther, 2009a,b; Wriedt, Sellmer & Näther, 2009a,b). In our ongoing investigation in this field, we have reacted cobalt(II) nitrate, potassium selenocyanate and pyrimidine in methanol. The crystals obtained were identified by single crystal X-ray determination.

The title compound (Fig. 1) represents a two-dimensional layered coordination polymer, which is isotypic to its corresponding nickel(II) thiocyanate analogue reproted recently (Wriedt *et al.*, 2009). The crystal structure consists of µ-1,3-(*N*,*N*) pyrimidine bridged zigzag-like cobalt(II) selenocynate chains, which are further linked by µ-1,3-(*N*,*N*) pyrimidine ligands into layers (Fig. 2). Within each layer the Co^{II} ions are bridged by three pyrimidine ligands and are further terminally coordinated by two *N*-bonded selenocyanate anions and one *O*-bonded methanol molecule in an octahedral coordination mode (Fig. 1). The layers are stacked in the direction of the crystallographic *b*-axis (Fig. 3). The CoN_5O octahedron is markedly distorted with three long Co—$N_{pyrimidine}$ distances in the range of 2.184 (3) to 2.191 (3) Å, one long Co—O_{MeOH} distance of 2.142 (3) Å and two short Co—NCSe distances of 2.059 (4) and 2.064 (4) Å (Table 1). The angles arround the metal centers range between 86.37 (13) to 96.00 (13) and 173.71 (13) to 177.16 (14)°. The shortest intra- and interlayer Co···Co distances amount to 6.0723 (6) and 8.5630 (9) Å, respectively.

Experimental

$Co(NO_3)_2.6H_2O$ (72.8 mg, 0.25 mmol), KNCSe (72.0 mg, 0.5 mmol) and pyrimidine (20.0 mg, 0.25 mmol) obtained from Alfa Aesar were transfered in a closed snap-vial with methanol (3 ml). After several days at room temperature without stirring, light pink block-shaped single crystals of the title compound were obtained in a mixture with unknown phases.

Refinement

The O-bound H atom was located in a difference Fourier map and its bond length set to a ideal value and finally refined using a riding model. All other H atoms were positioned with idealized geometry and refined using a riding model, with C—H = 0.93 (CH) and 0.96 (CH_3) Å and with U_{iso}(H) = 1.2(1.5 for methyl)U_{eq}(C). The absolute structure was determined on the basis of 1579 Friedel pairs.

Figures

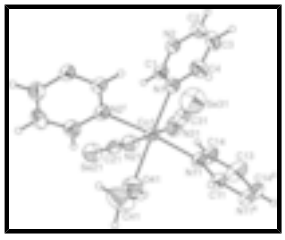


Fig. 1. Structure of the title compound with displacement ellipsoids drawn at the 50% probability level. [Symmetry codes: (i) -*x*+1, -*y*+1/2, *z*+1/2; (ii) -*x*+3/2, -*y*+1/2, *z*.]

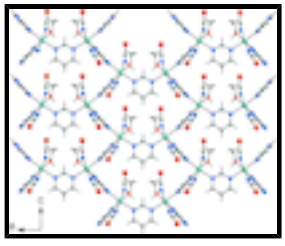


Fig. 2. A single layer in the title compound viewed along the *b* axis.

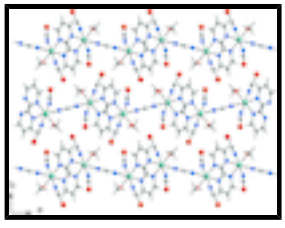


Fig. 3. Packing of the layers in the title compound viewed along the *c* axis.

Poly[tetrakis(selenocyanato-κ*N*)bis(methanol-κ*O*)tris(μ- pyrimidine-κ^2*N*:*N*')dicobalt(II)]

Crystal data

$[Co_2(CNSe)_4(C_4H_4N_2)_3(CH_4O)_2]$	$F(000) = 3232$
$M_r = 421.07$	$D_x = 1.980$ Mg m^{-3}
Orthorhombic, *Fdd*2	Mo *K*α radiation, λ = 0.71073 Å
Hall symbol: F 2 -2d	Cell parameters from 18545 reflections
a = 20.4069 (8) Å	θ = 2.4–28.0°
b = 33.0633 (15) Å	μ = 6.36 mm^{-1}
c = 8.3750 (3) Å	*T* = 293 K
V = 5650.8 (4) Å^3	Block, light pink
Z = 16	0.16 × 0.11 × 0.02 mm

Data collection

Stoe IPDS-2 diffractometer	3391 independent reflections
Radiation source: fine-focus sealed tube	3067 reflections with $I > 2\sigma(I)$
graphite	$R_{int} = 0.044$
ω scans	$\theta_{max} = 28.0°$, $\theta_{min} = 2.4°$
Absorption correction: numerical (*X-SHAPE* and *X-RED32*; Stoe & Cie, 2002)	$h = -26 \rightarrow 26$
$T_{min} = 0.431$, $T_{max} = 0.885$	$k = -43 \rightarrow 43$

18545 measured reflections

$l = -11 \rightarrow 10$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.038$

$wR(F^2) = 0.069$

$S = 1.16$

3391 reflections

164 parameters

1 restraint

Primary atom site location: structure-invariant direct methods

Secondary atom site location: difference Fourier map

Hydrogen site location: inferred from neighbouring sites

H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0286P)^2 + 8.8704P]$ where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{max} < 0.001$

$\Delta\rho_{max} = 0.55$ e Å^{-3}

$\Delta\rho_{min} = -0.35$ e Å^{-3}

Absolute structure: Flack (1983), 1579 Friedel pairs

Flack parameter: 0.057 (13)

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
Co1	0.60574 (2)	0.227474 (17)	0.78759 (6)	0.03270 (12)
N1	0.55104 (15)	0.27269 (10)	0.6504 (4)	0.0360 (8)
N2	0.47521 (15)	0.28919 (10)	0.4458 (5)	0.0382 (7)
C1	0.50711 (17)	0.26316 (13)	0.5388 (6)	0.0374 (9)
H1	0.4979	0.2358	0.5246	0.045*
C2	0.4883 (2)	0.32854 (13)	0.4677 (6)	0.0412 (9)
H2	0.4672	0.3477	0.4048	0.049*
C3	0.5320 (2)	0.34078 (14)	0.5813 (6)	0.0459 (11)
H3	0.5410	0.3681	0.5959	0.055*
C4	0.5623 (2)	0.31226 (13)	0.6730 (6)	0.0434 (10)
H4	0.5912	0.3204	0.7526	0.052*
N11	0.69371 (15)	0.24124 (11)	0.6493 (4)	0.0352 (8)
C11	0.7500	0.2500	0.7228 (7)	0.0372 (12)
H11	0.7500	0.2500	0.8339	0.045*
C13	0.7500	0.2500	0.4059 (8)	0.061 (2)
H13	0.7500	0.2500	0.2948	0.074*
C14	0.6944 (2)	0.24140 (17)	0.4914 (6)	0.0499 (12)
H14	0.6559	0.2355	0.4366	0.060*
N21	0.57994 (17)	0.18287 (12)	0.6270 (5)	0.0434 (8)
C21	0.56005 (17)	0.15562 (13)	0.5553 (5)	0.0381 (9)
Se21	0.53008 (3)	0.114405 (17)	0.44108 (7)	0.06121 (16)
N31	0.63231 (16)	0.26940 (12)	0.9575 (5)	0.0428 (8)
C31	0.63448 (17)	0.29139 (13)	1.0643 (5)	0.0373 (9)
Se31	0.63665 (3)	0.324181 (19)	1.23170 (7)	0.06461 (17)
O41	0.66187 (15)	0.18255 (10)	0.9111 (4)	0.0522 (8)
C41	0.6758 (4)	0.1790 (2)	1.0749 (8)	0.085 (2)
H41A	0.6359	0.1747	1.1328	0.127*
H41B	0.6964	0.2034	1.1118	0.127*

H41C	0.7048	0.1566	1.0918	0.127*
H1O4	0.6862	0.1792	0.8352	0.127*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Co1	0.0252 (2)	0.0341 (3)	0.0388 (3)	−0.0009 (2)	−0.0010 (2)	−0.0043 (2)
N1	0.0287 (15)	0.0336 (18)	0.046 (2)	−0.0015 (13)	−0.0036 (13)	0.0000 (15)
N2	0.0294 (14)	0.0376 (17)	0.048 (2)	−0.0013 (14)	−0.0020 (14)	0.0017 (17)
C1	0.0270 (16)	0.036 (2)	0.049 (2)	0.0006 (15)	−0.0018 (18)	−0.0026 (19)
C2	0.045 (2)	0.033 (2)	0.045 (2)	0.0008 (17)	−0.0001 (19)	0.0031 (19)
C3	0.059 (3)	0.029 (2)	0.049 (3)	−0.0082 (19)	0.003 (2)	−0.0002 (19)
C4	0.048 (2)	0.039 (2)	0.043 (2)	−0.0022 (18)	−0.0087 (19)	−0.0029 (19)
N11	0.0268 (14)	0.0419 (19)	0.037 (2)	−0.0028 (13)	−0.0005 (13)	0.0017 (14)
C11	0.029 (2)	0.050 (3)	0.032 (3)	0.000 (2)	0.000	0.000
C13	0.046 (4)	0.108 (7)	0.030 (4)	−0.001 (4)	0.000	0.000
C14	0.0295 (19)	0.075 (3)	0.045 (3)	−0.005 (2)	−0.0032 (18)	−0.003 (2)
N21	0.0422 (18)	0.040 (2)	0.048 (2)	0.0000 (16)	−0.0047 (16)	−0.0091 (17)
C21	0.0304 (16)	0.043 (2)	0.042 (2)	0.0029 (16)	0.0016 (17)	0.002 (2)
Se21	0.0661 (3)	0.0505 (3)	0.0670 (4)	−0.0056 (2)	−0.0100 (3)	−0.0199 (3)
N31	0.0419 (18)	0.045 (2)	0.042 (2)	−0.0081 (16)	0.0024 (16)	−0.0071 (18)
C31	0.0276 (17)	0.039 (2)	0.046 (3)	−0.0020 (15)	0.0036 (16)	0.003 (2)
Se31	0.0715 (3)	0.0632 (4)	0.0592 (3)	−0.0041 (3)	0.0086 (3)	−0.0256 (3)
O41	0.0456 (16)	0.054 (2)	0.057 (2)	0.0146 (15)	−0.0044 (15)	0.0063 (16)
C41	0.101 (5)	0.071 (4)	0.082 (5)	0.002 (4)	−0.047 (4)	0.007 (3)

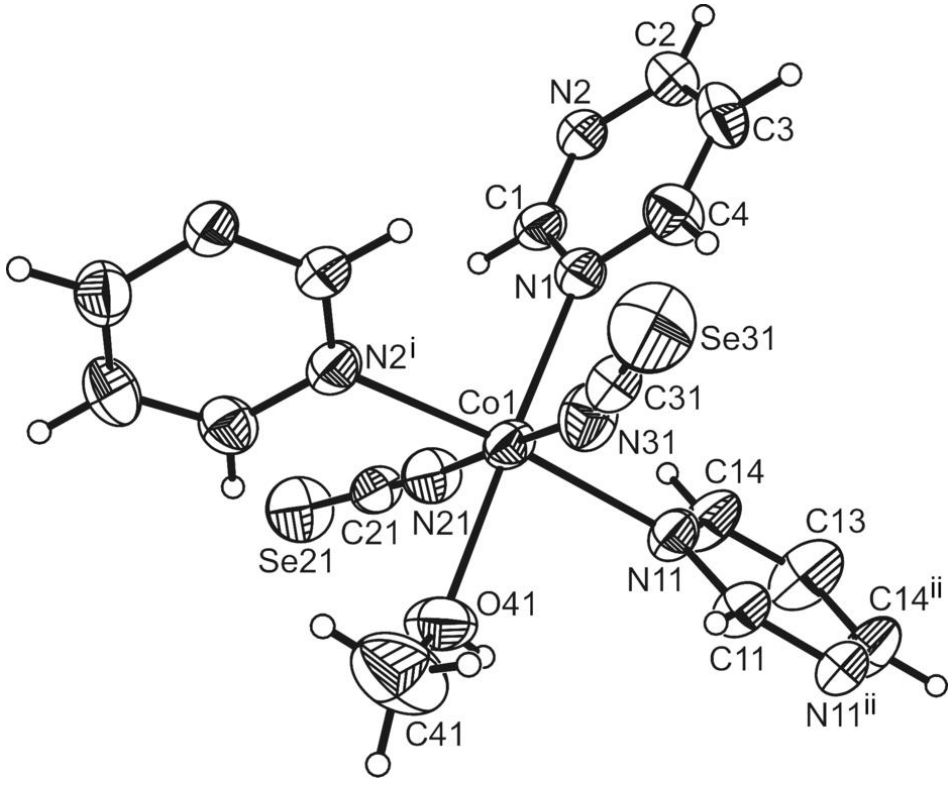
Geometric parameters (Å, °)

Co1—N1	2.191 (3)	N11—C11	1.335 (4)
Co1—N2^{i}	2.188 (3)	C11—N11^{ii}	1.335 (4)
Co1—N11	2.184 (3)	C11—H11	0.9300
Co1—N21	2.064 (4)	C13—C14	1.372 (6)
Co1—N31	2.059 (4)	C13—C14^{ii}	1.372 (6)
Co1—O41	2.142 (3)	C13—H13	0.9300
N1—C1	1.333 (5)	C14—H14	0.9300
N1—C4	1.342 (5)	N21—C21	1.157 (5)
N2—C1	1.331 (5)	C21—Se21	1.774 (5)
N2—C2	1.341 (5)	N31—C31	1.154 (5)
C1—H1	0.9300	C31—Se31	1.773 (4)
C2—C3	1.366 (6)	O41—C41	1.405 (7)
C2—H2	0.9300	O41—H1O4	0.8139
C3—C4	1.364 (7)	C41—H41A	0.9600
C3—H3	0.9300	C41—H41B	0.9600
C4—H4	0.9300	C41—H41C	0.9600
N11—C14	1.323 (6)		
N31—Co1—N21	176.69 (16)	C2—C3—H3	120.6
N31—Co1—O41	89.55 (15)	N1—C4—C3	121.2 (4)
N21—Co1—O41	87.46 (14)	N1—C4—H4	119.4

N31—Co1—N11	90.55 (13)	C3—C4—H4	119.4
N21—Co1—N11	90.75 (14)	C14—N11—C11	116.8 (4)
O41—Co1—N11	87.77 (13)	C14—N11—Co1	122.6 (3)
N31—Co1—N2^{i}	87.12 (14)	C11—N11—Co1	120.5 (3)
N21—Co1—N2^{i}	91.27 (14)	N11—C11—N11^{ii}	125.1 (5)
O41—Co1—N2^{i}	86.37 (13)	N11—C11—H11	117.5
N11—Co1—N2^{i}	173.71 (13)	N11^{ii}—C11—H11	117.5
N31—Co1—N1	92.13 (14)	C14—C13—C14^{ii}	117.1 (6)
N21—Co1—N1	90.91 (14)	C14—C13—H13	121.5
O41—Co1—N1	177.16 (14)	C14^{ii}—C13—H13	121.5
N11—Co1—N1	89.92 (12)	N11—C14—C13	122.1 (4)
N2^{i}—Co1—N1	96.00 (13)	N11—C14—H14	118.9
C1—N1—C4	116.4 (4)	C13—C14—H14	118.9
C1—N1—Co1	123.3 (3)	C21—N21—Co1	169.9 (4)
C4—N1—Co1	120.3 (3)	N21—C21—Se21	178.7 (4)
C1—N2—C2	116.8 (4)	C31—N31—Co1	166.0 (3)
C1—N2—Co1^{iii}	124.1 (3)	N31—C31—Se31	178.4 (4)
C2—N2—Co1^{iii}	118.5 (3)	C41—O41—Co1	129.6 (4)
N2—C1—N1	125.9 (4)	C41—O41—H1O4	128.9
N2—C1—H1	117.0	Co1—O41—H1O4	92.4
N1—C1—H1	117.0	O41—C41—H41A	109.5
N2—C2—C3	120.8 (4)	O41—C41—H41B	109.5
N2—C2—H2	119.6	H41A—C41—H41B	109.5
C3—C2—H2	119.6	O41—C41—H41C	109.5
C4—C3—C2	118.9 (4)	H41A—C41—H41C	109.5
C4—C3—H3	120.6	H41B—C41—H41C	109.5

Symmetry codes: (i) −*x*+1, −*y*+1/2, *z*+1/2; (ii) −*x*+3/2, −*y*+1/2, *z*; (iii) −*x*+1, −*y*+1/2, *z*−1/2.

Fig. 1


C2
N2
C3
C1
C4
N1
Se31
N2i
C31
Co1
N31
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C13
C21
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N11
C14ii
O41
C11
C41
N11ii

Fig. 2

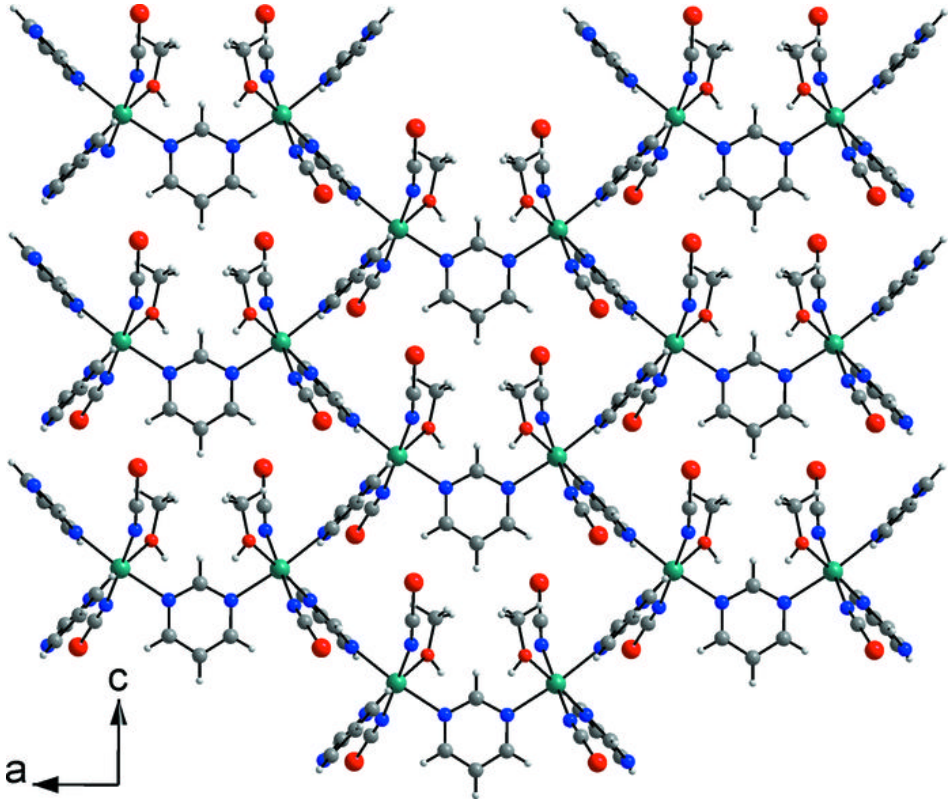

Fig. 3