

Table 1. Selected geometric parameters (Å, °)

Co1—O2	1.885 (3)	Co1—N2	1.945 (3)
Co1—O1	1.901 (3)	O1—C7	1.293 (5)
Co1—N3	1.922 (3)	O2—C7	1.294 (5)
Co1—N1	1.930 (3)	O3—C7	1.224 (5)
Co1—N4	1.937 (3)		
O2—Co1—O1	68.18 (12)	O1—Co1—N2	90.60 (13)
O2—Co1—N3	166.06 (12)	N3—Co1—N2	91.07 (14)
O1—Co1—N3	97.91 (13)	N1—Co1—N2	89.49 (14)
O2—Co1—N1	98.65 (13)	N4—Co1—N2	178.18 (13)
O1—Co1—N1	166.83 (12)	C1—N1—Co1	115.6 (3)
N3—Co1—N1	95.25 (14)	C3—N2—Co1	117.4 (2)
O2—Co1—N4	90.12 (13)	C4—N3—Co1	115.9 (3)
O1—Co1—N4	91.11 (13)	C6—N4—Co1	117.0 (2)
N3—Co1—N4	89.31 (13)	C7—O1—Co1	90.4 (2)
N1—Co1—N4	88.70 (13)	C7—O2—Co1	91.1 (2)
O2—Co1—N2	89.94 (13)		

Table 2. Hydrogen-bonding geometry (Å, °)

D—H...A	D—H	H...A	D...A	D—H...A
N1—H1A...Cl1 ⁱ	0.90	2.33	3.215 (4)	168
N1—H1B...O3 ⁱⁱ	0.88	2.38	2.982 (5)	126
N2—H2A...Cl1 ⁱⁱⁱ	0.95	2.49	3.317 (4)	146
N2—H2B...Cl1	0.93	2.95	3.459 (3)	116
N3—H3A...Cl1 ⁱ	0.81	2.72	3.226 (4)	123
N3—H3B...Cl1	0.83	2.44	3.202 (4)	154
N4—H4A...O3 ⁱⁱ	0.88	2.04	2.894 (4)	164
N4—H4B...O2 ^{iv}	0.91	2.00	2.865 (4)	159
O1W—H1WB...Cl1	1.00	2.28	3.263 (5)	168
O1W—H1WA...O1	0.98	1.88	2.867 (5)	180

Symmetry codes: (i) $\frac{1}{2}-x, \frac{1}{2}+y, \frac{1}{2}-z$; (ii) $x, 1+y, z$; (iii) $\frac{1}{2}-x, y-\frac{1}{2}, \frac{1}{2}-z$; (iv) $1-x, -y, -z$.

The H atoms of the water molecule and of the amine groups were located from a difference map but not refined. The H atoms of the methylene groups were placed at calculated positions and allowed to ride on their respective parent atoms. All H atoms were assigned fixed isotropic displacement parameters.

Data collection: *SHELXTL-Plus* (Sheldrick, 1990a). Cell refinement: *SHELXTL-Plus*. Data reduction: *SHELXTL-Plus*. Program(s) used to solve structure: *SHELXS90* (Sheldrick, 1990b). Program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997). Molecular graphics: *ORTEP* (Johnson, 1965) in *SHELXTL-Plus*. Software used to prepare material for publication: *SHELXL97*.

This work was supported by the NSFC. We are also indebted to the Chemistry Department of the Chinese University of Hong Kong for donation of the diffractometer. HLZ thanks the Ma Can-an Foundation for a scholarship award.

Supplementary data for this paper are available from the IUCr electronic archives (Reference: HA1264). Services for accessing these data are described at the back of the journal.

References

- Ardon, M. & Bino, A. (1985). *Inorg. Chem.* **24**, 1343–1344.
 Baxter, K. E., Hanton, L. R., Simpson, J., Vincent, B. R. & Blackman, A. G. (1995). *Inorg. Chem.* **34**, 2795–2796.
 Behr, A. (1988). *Angew. Chem. Int. Ed. Engl.* **27**, 661–678.
 Bernal, I., Myrczek, J., Cetrullo, J. & Massoud, S. S. (1993). *J. Coord. Chem.* **29**, 319–324.
 Egan, T. J., Baldwin, D. A., Denner, L., Levendis, D. C. & Marques, H. M. (1995). *Acta Cryst.* **C51**, 1994–1995.

Johnson, C. K. (1965). *ORTEP*. Report ORNL-3794. Oak Ridge National Laboratory, Tennessee, USA.

- Kitajima, N., Hikichi, S., Tanaka, M. & Moro-oka, Y. (1993). *J. Am. Chem. Soc.* **115**, 5496–5508.
 Kopfman, G. & Huber, R. (1968). *Acta Cryst.* **A24**, 348–351.
 Masuda, H., Masuda, C., Jitsukawa, K. & Einaga, H. (1994). *Bull. Chem. Soc. Jpn.* **67**, 3000–3003.
 Sheldrick, G. M. (1990a). *SHELXTL-Plus*. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.
 Sheldrick, G. M. (1990b). *Acta Cryst.* **A46**, 467–473.
 Sheldrick, G. M. (1997). *SHELXL97. Program for the Refinement of Crystal Structures*. University of Göttingen, Germany.
 Tsuboyama, S., Fujimoto, J., Hanabira, S., Yasuda, N., Kobayashi, K., Sakurai, T. & Tsuboyama, K. (1996). *J. Chem. Soc. Dalton Trans.* pp. 45–51.

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catena-Poly[[bis(imidazole-*N*³)cadmium(II)]-bis(μ -thiocyanato)-*S*:*N*; *N*:*S*]

HONG-JI CHEN, GUANG YANG AND XIAO-MING CHEN

School of Chemistry and Chemical Engineering, Zhongshan University, Guangzhou 510275, People's Republic of China.
E-mail: cescxm@zsu.edu.cn

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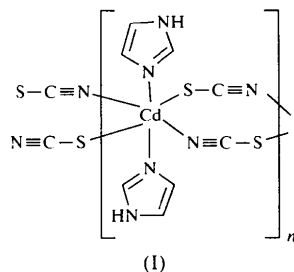
Abstract

The title polymeric complex, [Cd(SCN)₂(C₃H₄N₂)₂]_n, exhibits an infinite chain structure in which each pair of Cd atoms is bridged by two η -1,3-SCN[−] groups. The unique Cd atom lies on an inversion centre and the coordination sphere is completed by two imidazole N atoms to form a CdS₂N₄ octahedron. The polymeric chains are further extended into two-dimensional sheets via N—H...S hydrogen bonds between the uncoordinated imidazole N atoms and the SCN[−] groups.

Comment

The structures of a few polymeric Lewis-base adducts of cadmium(II) thiocyanate, [Cd(SCN)₂(L)₂] (where L is 2-, 3- or 4-methylpyridine, benzylamine, dibenzylamine, tri-*m*-tolylphosphine or 1*H*-1,2,4-triazole), have been reported (Ram *et al.*, 1981; Taniguchi & Ouchi, 1987; Taniguchi *et al.*, 1987). In these compounds, the Lewis base (L) is a monofunctional ligand, which gives a one-dimensional structure with thiocyanate as a bridging ligand. We report here a similar Lewis-base adduct of Cd(SCN)₂, [Cd(SCN)₂(ImH)₂]_n, (I), in which imidazole (ImH) is a bifunctional ligand capable of both coordinating with a metal ion and donating a hydrogen bond (Andrew *et al.*, 1994a,b; Chow *et al.*,

1993; Chowdhry *et al.*, 1996). The hydrogen bonds may extend the one-dimensional chains into a higher dimensional structure.



In the title compound, the Cd^{II} ion, being located at an inversion centre, is hexacoordinated by a pair of S atoms [Cd1—S1 2.8510 (13) Å] from two SCN⁻ groups, a pair of N atoms [Cd1—N3a 2.322 (2) Å; symmetry code: (a) $-x, 1-y, -z$; Fig. 1] from another two SCN⁻ groups, and two imidazole N atoms [Cd1—N1 2.274 (2) Å] in an octahedral geometry, with the most distorted bond angle S1—Cd1—N3a being 92.33 (7)°.

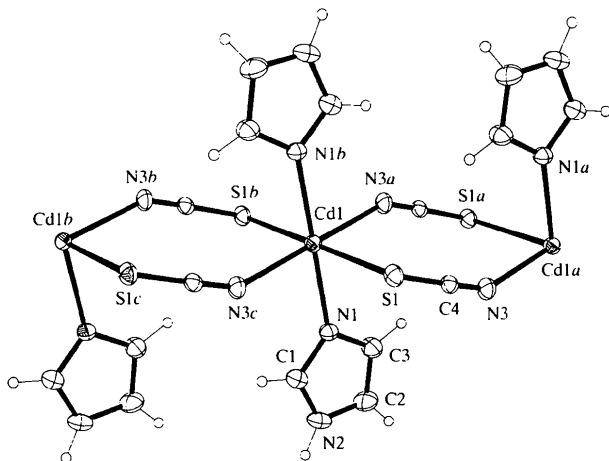


Fig. 1. ORTEP (Johnson, 1976) plot (30% probability) of the coordination environment in (I). [Symmetry codes: (a) $-x, 1-y, -z$; (b) $-x, -y, -z$; (c) $x, y-1, z$.]

Each pair of adjacent metal atoms are bridged by a pair of SCN⁻ groups through both ends, resulting in a chain-like structure comprising $(-N-C-S-Cd)_2$ eight-membered rings in a chair conformation. Similar eight-membered rings have been documented (Ram *et al.*, 1981; Taniguchi & Ouchi, 1987; Taniguchi *et al.*, 1987). As expected, adjacent linear chains are interlinked by hydrogen bonds [3.470 (3) Å] between the protonated imidazole N atoms and the coordinated S atoms of the SCN⁻ groups, resulting in two-dimensional sheets perpendicular to the *c* axis in the lattice, as shown in Fig. 2.

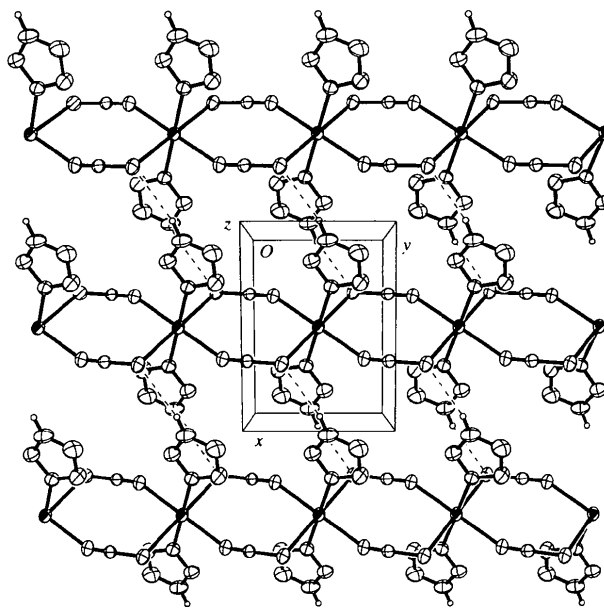


Fig. 2. The molecular packing of (I) in the lattice.

Experimental

A hot ethanol–water (1:1 *v/v*) solution (3 ml) of imidazole (0.17 g, 2.5 mmol) was added to a hot aqueous solution (5 ml) of Cd(SCN)₂ (0.75 g, 2.5 mmol). After cooling to room temperature, the resulting solution was allowed to stand for *ca* one week to give colourless crystals of (I). The yield was 86% based on Cd(SCN)₂. IR data (cm⁻¹): 3296 (*m*), 3128 (*m*), 2938 (*w*), 2860 (*w*), 2101 (*vs*), 1532 (*m*), 1489 (*w*), 1426 (*m*), 1384 (*w*), 1321 (*m*), 1060 (*s*), 934 (*w*), 920 (*w*), 842 (*w*), 765 (*m*), 716 (*m*), 646 (*s*), 610 (*s*), 463 (*m*). Elemental analysis, found: C 26.08, H 2.25, N 22.60, S 17.39%; calculated for C₁₆H₁₆Cd₂N₁₂S₄: C 26.35, H 2.21, N 23.04, S 17.58%.

Crystal data

[Cd(SCN)₂(C₃H₄N₂)₂]

M_r = 364.72

Monoclinic

*P*2₁/*n*

a = 7.804 (4) Å

b = 5.765 (2) Å

c = 14.164 (9) Å

β = 102.37 (1)°

V = 622.4 (6) Å³

Z = 2

D_x = 1.946 Mg m⁻³

D_m not measured

Mo *K*α radiation

λ = 0.71073 Å

Cell parameters from 25

reflections

θ = 7–14°

μ = 2.075 mm⁻¹

T = 293 (2) K

Polyhedral

0.60 × 0.40 × 0.34 mm

Colourless

Data collection

Siemens *R3m* diffractometer

ω scans

Absorption correction:

semi-empirical based on ψ

scan (Kopfmán & Huber,

1968)

T_{min} = 0.372, *T_{max}* = 0.494

1095 reflections with

I > 2σ(*I*)

R_{int} = 0.015

θ_{max} = 26°

h = 0 → 9

k = 0 → 7

l = -17 → 17

1302 measured reflections
1212 independent reflections

2 standard reflections
every 200 reflections
intensity decay: none

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.023$
 $wR(F^2) = 0.059$
 $S = 1.114$
1212 reflections
79 parameters
H atoms constrained
 $w = 1/[\sigma^2(F_o^2) + (0.0317P)^2 + 0.3616P]$
where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} < 0.001$
 $\Delta\rho_{\max} = 0.289 \text{ e } \text{Å}^{-3}$
 $\Delta\rho_{\min} = -0.392 \text{ e } \text{Å}^{-3}$
Extinction correction: none
Scattering factors from
International Tables for Crystallography (Vol. C)

Ram, G. G., William, P. H., Marc, J. O. & Andre, L. B. (1981). *Inorg. Chem.* **20**, 3924–3928.
Sheldrick, G. M. (1990a). *SHELXTL-Plus User's Manual*. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.
Sheldrick, G. M. (1990b). *Acta Cryst.* **A46**, 467–473.
Sheldrick, G. M. (1997). *SHELXL97. Program for the Refinement of Crystal Structures*. University of Göttingen, Germany.
Taniguchi, M. & Ouchi, A. (1987). *Bull. Chem. Soc. Jpn.* **60**, 4172–4174.
Taniguchi, M., Sugita, Y. & Ouchi, A. (1987). *Bull. Chem. Soc. Jpn.* **60**, 1321–1326.

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Copper(I) cyanide–thiomorpholine (3/1)

DOYLE BRITTON,^a FRED B. STOCKER^b AND TEODORA P. STAEVA^b

^aDepartment of Chemistry, University of Minnesota, Minneapolis, MN 55455-0431, USA, and ^bDepartment of Chemistry, Macalester College, St Paul, MN 55105, USA.
E-mail: britton@chemsun.chem.umn.edu

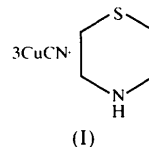
(Received 14 June 1999; accepted 9 September 1999)

Abstract

In the polymeric 3/1 complex of copper(I) cyanide and thiomorpholine, $3\text{CuCN}\cdot\text{C}_4\text{H}_9\text{NS}$ or $[\text{Cu}_3(\text{CN})_3(\text{C}_4\text{H}_9\text{NS})]_n$, the CuCN forms two-dimensional sheets, which are held together by the thiomorpholine. Each thiomorpholine molecule bonds to three different sheets.

Comment

In the course of studies of complexes of copper(I) cyanide with thioureas (Stocker *et al.*, 1996), diamines (Stocker, Staeva *et al.*, 1999) and imidazoles (Stocker & Britton, 1999), the title compound, (I), was prepared.



The anisotropic displacement ellipsoids and the labeling are shown in Fig. 1. The atoms in the CN groups were treated as a mixture of C and N atoms at each position, constrained to have the same coordinates and anisotropic displacement parameters, with rationally related occupancies. When the refinement of the occupancies showed that C1—N2 was completely ordered and that C3—N4 was completely disordered within experimental error, the occupancies were fixed at 1.00

Table 1. Selected geometric parameters and hydrogen-bonding geometry (Å, °)

Cd1—N1	2.274 (2)	N1—C1	1.320 (4)
Cd1—N3 ⁱ	2.322 (2)	N1—C3	1.375 (4)
Cd1—S1	2.8510 (13)	N2—C1	1.329 (4)
S1—C4	1.653 (3)	N2—C2	1.347 (5)
C4—N3	1.149 (3)	C2—C3	1.344 (5)
N1—Cd1—N3 ⁱ	88.93 (9)	C1—N1—Cd1	125.6 (2)
N1—Cd1—S1	90.04 (7)	C3—N1—Cd1	128.9 (2)
N3 ⁱ —Cd1—S1	87.67 (7)	C1—N2—C2	108.7 (3)
C4—S1—Cd1	94.39 (10)	N1—C1—N2	110.3 (3)
N3—C4—S1	178.6 (3)	C3—C2—N2	106.0 (3)
C4—N3—Cd1 ⁱⁱ	149.9 (2)	C2—C3—N1	109.5 (3)
C1—N1—C3	105.4 (3)		

D—H...A	D—H	H...A	D...A	D—H...A
N2—H2A...S1 ⁱⁱⁱ	0.90	2.60	3.470 (3)	162

Symmetry codes: (i) $x, y - 1, z$; (ii) $x, 1 + y, z$; (iii) $1 - x, -y, -z$.

Data collection: *SHELXTL-Plus* (Sheldrick, 1990a). Cell refinement: *SHELXTL-Plus*. Data reduction: *SHELXTL-Plus*. Program(s) used to solve structure: *SHELXS90* (Sheldrick, 1990b). Program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997). Molecular graphics: *ORTEPII* (Johnson, 1976) in *SHELXTL-Plus*. Software used to prepare material for publication: *SHELXL97*.

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: OB1018). Services for accessing these data are described at the back of the journal.

References

- Andrew, H. D., Michael, P. M. & David, J. W. (1994a). *Transition Met. Chem.* **19**, 653–656.
Andrew, H. D., Michael, P. M. & David, J. W. (1994b). *J. Chem. Soc. Chem. Commun.* pp. 503–504.
Chow, M.-Y., Chen, X.-M. & Mak, T. C. W. (1993). *J. Chem. Soc. Dalton Trans.* pp. 3413–3420.
Chowdhry, M. M., Mingos, D. M. P., White, A. J. P. & Williams, D. J. (1996). *J. Chem. Soc. Chem. Commun.* pp. 899–900.
Johnson, C. K. (1976). *ORTEPII*. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.
Kopfmann, G. & Huber, R. (1968). *Acta Cryst.* **A24**, 348–351.