Cl(5)—Cd(3)—O(3W)	95.3 (2)	Cl(5)—Cd(3)—Cl(6^{ii})	91.7 (1)
Cl(6)—Cd(3)—O(3W)	168.2 (2)	Cl(6)—Cd(3)—Cl(6^{ii})	88.8 (1)
Cl(6)—Cd(3)—O(4W)	82.8 (2)	O(3W)—Cd(3)—Cl(6^{ii})	83.8 (1)
O(3W)—Cd(3)—O(4W)	87.8 (2)	Cd(1)—Cl(1)—Cd(1^{i})	91.5 (1)
O(4W)—Cd(3)—Cl(6^{ii})	87.8 (1)	Cd(1)—Cl(3)—Cd(2)	91.0 (1)
Cd(1)—Cl(2)—Cd(2)	92.4 (1)	Cd(2)—Cl(5)—Cd(3)	91.7 (1)
Cd(2)—Cl(4)—Cd(3)	91.5 (1)	Cd(2)—O(5)—C(19)	128.3 (8)
Cd(3)—Cl(6)—Cd(3^{ii})	91.2 (1)	O(5)—C(19)—C(20)	111.9 (11)
Cd(2)—O(5)—C(19′)	126 (2)	Cd(2)—O(6)—C(21)	128.2 (6)
O(5)—C(19′)—C(20)	113 (4)	O(6)—C(21)—C(22′)	103.5 (13)
O(6)—C(21)—C(22)	103.7 (16)	C(2)—N(1)—C(9)	119.9 (6)
C(2)—N(1)—C(5)	121.4 (6)	C(7)—N(2)—C(10)	123.0 (7)
C(5)—N(1)—C(9)	118.5 (7)	C(10)—N(2)—C(11)	114.7 (8)
C(7)—N(2)—C(11)	122.3 (7)	C(3)—N(3)—C(16)	122.0 (6)
C(3)—N(3)—C(12)	119.9 (6)	C(14)—N(4)—C(17)	121.7 (7)
C(14)—N(4)—C(18)	122.7 (7)	C(17)—N(4)—C(18)	115.5 (8)
O(1)—C(1)—O(2)	126.9 (10)	O(1)—C(1)—C(2)	116.6 (8)
O(2)—C(1)—C(2)	116.4 (9)	N(1)—C(2)—C(1)	117.1 (7)
N(1)—C(2)—C(3)	120.3 (8)	C(1)—C(2)—C(3)	122.4 (8)
N(3)—C(3)—C(2)	122.7 (8)	N(3)—C(3)—C(4)	113.3 (6)
C(2)—C(3)—C(4)	123.9 (8)	O(3)—C(4)—O(4)	126.2 (8)
O(3)—C(4)—C(3)	115.3 (7)	O(4)—C(4)—C(3)	117.7 (7)
N(1)—C(5)—C(6)	121.5 (7)	C(5)—C(6)—C(7)	121.6 (7)
N(2)—C(7)—C(6)	122.6 (7)	N(2)—C(7)—C(8)	122.6 (8)
C(6)—C(7)—C(8)	114.7 (8)	C(7)—C(8)—C(9)	120.6 (7)
N(1)—C(9)—C(8)	123.0 (7)	N(3)—C(12)—C(13)	121.3 (7)
C(12)—C(13)—C(14)	122.2 (7)	N(4)—C(14)—C(13)	121.6 (7)
N(4)—C(14)—C(15)	123.0 (7)	C(13)—C(14)—C(15)	115.4 (8)
C(14)—C(15)—C(16)	120.7 (7)	N(3)—C(16)—C(15)	122.6 (7)
Cl(1)—Cd(1)—Cl(3)	92.9 (1)		
O(1)—C(1)—C(2)—C(3)	4.0 (9)	O(3)—C(4)—C(3)—C(2)	89.9 (9)
C(5)—N(1)—C(2)—C(3)	119.6 (8)	C(12)—N(3)—C(3)—C(2)	127.4 (8)

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

Table 3. *Hydrogen-bond parameters (Å, °)*

The donor atom is given first in each hydrogen bond.

O(1W)···O(1)	2.689 (10)	O(1W)···Cl(4)	3.175 (6)
O(2W)···O(2)	2.718 (11)	O(3W)···O(3)	2.748 (10)
O(3W)···Cl(2)	3.189 (7)	O(4W)···O(4)	2.754 (10)
O(5W)···O(3^{i})	2.773 (8)	O(5W)···Cl(6)	3.228 (9)
O(5)···O(5W)	2.629 (12)	O(6)···(O4^{ii})	2.775 (8)
O(1)···O(1W)···Cl(4)	101.4 (2)	O(3)···O(3W)···Cl(2)	134.7 (2)
O(5)···O(5W)···O(3^{i})	121.3 (3)	O(5)···O(5W)···Cl(6)	118.0 (3)
O(3^{i})···O(5W)···Cl(6)	116.1 (3)	O(3W)···O(3)···O(5W^{iii})	107.1 (3)

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

The structure was solved by direct methods and all non-H atoms were refined anisotropically except the disordered C atoms of the ethanol molecules [C(19), C(19′), C(22) and C(22′)]. The H atoms of the double-betaine molecule were generated geometrically (C—H = 0.96 Å), and the H atoms of the water and ethanol molecules were located in difference Fourier maps. All H atoms were assigned displacement parameters and included in the structure-factor calculations. All computations were performed on a PC 486 computer.

Data collection: *P3/PC Diffractometer Control Program* (Siemens, 1989). Cell refinement: *P3/PC Diffractometer Control Program*. Data reduction: *SHELXTL-Plus XDISK* (Sheldrick, 1987). Program(s) used to solve structure: *SHELXTL/PC XS* (Sheldrick, 1990). Program(s) used to refine structure: *SHELXTL/PC XLS*. Molecular graphics: *SHELXTL/PC XP*. Software used to prepare material for publication: *SHELXTL/PC XPUBL*.

This work is supported by a Hong Kong Research Grants Council Earmarked Grant (ref. CUHK 89/93E).

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: KH1054). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

References

Chen, X.-M. & Mak, T. C. W. (1991*a*). *J. Crystallogr. Spectrosc. Res.* **21**, 27–32.

Chen, X.-M. & Mak, T. C. W. (1991*b*). *Aust. J. Chem.* **44**, 639–643.

Chen, X.-M., Mak, T. C. W., Huang, W.-Y. & Lü, L. (1992). *Acta Cryst.* C**48**, 57–59.

Kopfmann, G. & Huber, R. (1968). *Acta Cryst.* A**24**, 348–351.

Prince, R. H. (1987). *Comprehensive Coordination Chemistry*, Vol. 5, edited by G. Wilkinson, R. D. Gillard & J. A. McCleverty, pp. 925–1045. Oxford: Pergamon Press.

Sheldrick, G. M. (1987). *SHELXTL-Plus*. PC version. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.

Sheldrick, G. M. (1990). *SHELXTL/PC User's Manual*. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.

Siemens (1989). *P3/PC Diffractometer Control Program*. Version 3.13. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.

Weiss, R., Roth, R., Lowack, R. H. & Bremer, M. (1990). *Angew. Chem. Int. Ed. Engl.* **29**, 1132–1134.

Wu, D.-D. & Mak, T. C. W. (1994*a*). *J. Chem. Crystallogr.* **24**, 689–694.

Wu, D.-D. & Mak, T. C. W. (1994*b*). *Polyhedron*, **13**, 3333–3339.

Wu, D.-D. & Mak, T. C. W. (1995). *Inorg. Chim. Acta*. In the press.

Acta Cryst. (1996). C**52**, 529–532

Polymeric Cadmium(II) Nitrate Adduct of a Flexible Double Betaine: $[Cd_2(L)_2(NO_3)_2(H_2O)_3]_n(NO_3)_{2n}$ [L = *meso*-2,5-Bis(trimethylammonio)adipate]

De-Dong Wu and Thomas C. W. Mak

Department of Chemistry, The Chinese University of Hong Kong, Shatin, New Territories, Hong Kong. E-mail: tcwmak@cuhk.hk

(*Received 14 July 1995; accepted 30 August 1995*)

Abstract

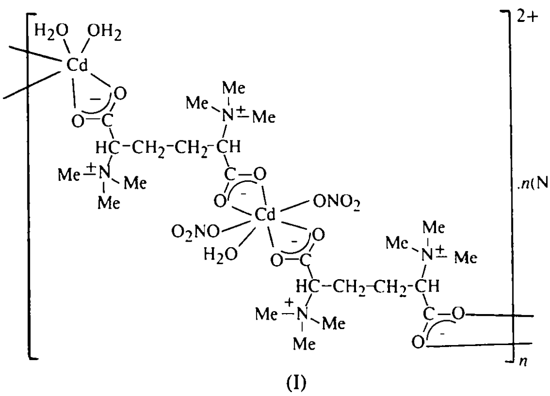
The title complex, *catena*-poly[(diaquacadmium)-μ-[*meso*-2,5-bis(trimethylammonio)adipato-$O,O':O'',O'''$]-(aquadinitratocadmium)-μ-[*meso*-2,5-bis(trimethylammonio)adipato-$O,O':O'',O'''$] dinitrate], $[Cd_2(L)_2(NO_3)_2(H_2O)_3]_n(NO_3)_{2n}$ [L = $^-O_2CCH(Me_3N^+)CH_2CH_2CH(Me_3N^+)CO_2^-$], consists of an infinite zigzag chain in which the two independent Cd^{II} atoms, lying on separate crystallographic C_2 axes, have approximately pentagonal bipyramidal and very dis-

Acta Crystallographica Section C
ISSN 0108-2701 ©1996

torted octahedral coordination geometries, respectively. The formation of hydrogen bonds between the aqua ligands and other O atoms leads to a layer network.

Comment

Betaine ($Me_3N^+CH_2CO_2^-$; trimethylammonioacetate) and its derivatives, by virtue of their permanent dipolarity, can form soluble cadmium(II) carboxylate complexes (Chen & Mak, 1991*a*,*b*; Chen, Mak, Huang & Lü, 1992). Our recent study of three Cd^{II} halide complexes containing a flexible double betaine with the adipic acid skeleton, namely $[Cd_4(L)_2X_8(H_2O)_2]_n$ [X = Cl, Br, I; L = *meso*-2,5-bis(trimethylammonio)adipate, $^-O_2CCH(Me_3N^+)CH_2CH_2CH(Me_3N^+)CO_2^-$], has shown that variation of the halide anions results in the adoption of different polymeric structures (Wu & Mak, 1995*a*). As a sequel to this work, we now report the preparation and crystal structure of a cadmium(II) nitrate adduct of this flexible double betaine, namely *catena*-poly[(diaquacadmium)-μ-[*meso*-2,5-bis(trimethylammonio)adipato-*O*,*O*′:*O*″,*O*‴]-(aquadinitratocadmium)-μ-[*meso*-2,5-bis(trimethylammonio)adipato-*O*,*O*′:*O*″,*O*‴] dinitrate], (I).



(I)

The title complex contains two independent Cd^{II} atoms in different coordination environments, both being located at special positions of crystallographic symmetry 2, as shown in Fig. 1. Atom Cd(1) is coordinated by an aqua ligand lying on the C_2 axis [Cd(1)—O(1*W*) 2.194 (5) Å], a pair of unidentate nitrate groups [Cd(1)—O(5) 2.421 (3) Å] and a pair of asymmetrically chelated carboxylate groups [Cd(1)—O(1) 2.353 (3), Cd(1)—O(2) 2.503 (3) Å]. The coordination geometry about Cd(1) can be best described as approximately pentagonal bipyramidal, with the two nitrato O atoms in axial positions [O(5)—Cd(1)—O(5^i) 167.6 (2)°] and the pair of carboxylate groups and aqua ligand in the equatorial plane [the largest bond angle at Cd(1) involving the axial O atom with an atom in the equatorial plane is 103.9 (1)°], which is similar to that found in cadmium(II) nicotinate, $Cd[(C_5H_4N)CO_2]_2(H_2O)$ (Clegg, Cressey, McCamley & Straughan, 1995). Cd(2) is coordinated by a pair of *cis*-related aqua ligands [Cd(2)—O(2*W*) 2.297 (3) Å] and a pair of asymmetrically chelated carboxylate groups from the remaining betaine moieties [Cd(2)—O(3) 2.299 (3), Cd(2)—O(4) 2.511 (3) Å] in a very distorted octahedral six-coordinate environment, which commonly occurs in cadmium(II) complexes (Prince, 1987). The double-betaine ligand almost possesses an inversion centre at the midpoint of the C(3)—C(4) bond with maximum separation of the pair of ammonio groups; it thus adopts an all-*trans* conformation similar to that in its cadmium(II) halide complexes (Wu & Mak, 1995*a*), as well as in its silver(I) (Wu & Mak, 1995*b*) and mercury(II) complexes (Wu & Mak, 1995*c*). The dihedral angles formed by the two carboxylate groups with the principal skeletal plane containing atoms C(7), N(1), C(2), C(3), C(4), C(5), N(2) and C(10) [the mean deviation from coplanarity is 0.0832 Å] are 79.4 (6) and 89.5 (6)°.

The crystal structure of the title complex exhibits an infinite zigzag chain running in the *a* direction, which is constructed from the linkage of Cd^{II} atoms

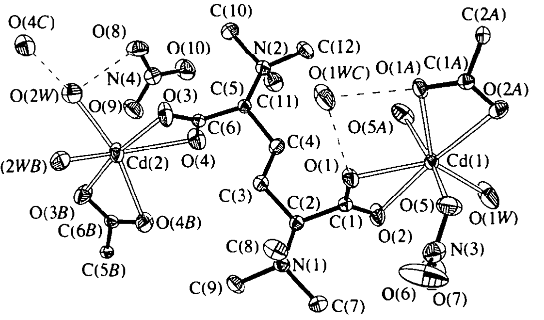


Fig. 1. Perspective view (35% probability displacement ellipsoids) of the asymmetric unit and coordination geometries in (I) with the atom-numbering scheme. H atoms have been omitted for clarity and hydrogen bonds are indicated by broken lines.

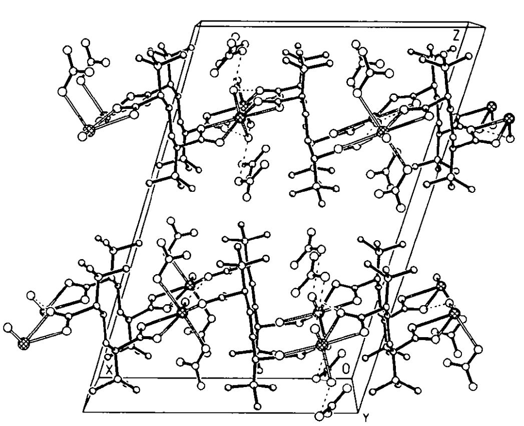


Fig. 2. Projection of the crystal structure of (I) along the *b* axis showing the hydrogen-bonded layer structure corresponding to the planes *z* = 1/4 and 3/4.

by the flexible double-betaine ligands in the *cis-trans-cis* conformation. The formation of donor hydrogen bonds by the aqua ligand $O(1W^{\rm iii})$ of an adjacent chain with two O atoms from a pair of carboxylate groups [$O(1W^{\rm iii})\cdots O(1)$ 2.683 (5) Å, $O(1)\cdots O(1W^{\rm iii})\cdots O(1^{\rm i})$ 72.4 (2)°], and by the second aqua ligand $O(2W)$ with a carboxylate O atom in the adjacent chain and an O atom of the uncoordinated nitrate ion [$O(2W)\cdots O(4^{\rm iii})$ 2.771 (5), $O(2W)\cdots O(8)$ 2.744 (5) Å, $O(4^{\rm iii})\cdots O(2W)\cdots O(8)$ 113.4 (2)°], leads to further cross-linkage between neighbouring chains to generate a layer network (Table 2).

Experimental

The flexible double betaine in its hydrated form, $L.2H_2O$, was synthesized by the reaction of aqueous trimethylamine (45%) with *meso*-2,5-dibromoadipic acid as described previously (Wu & Mak, 1995*b*). The title complex was prepared by mixing a solution of $Cd(NO_3)_2.4H_2O$ (314.1 mg, 1.02 mmol) in distilled water (2.0 ml) and a solution of $L.2H_2O$ (81.5 mg, 0.28 mmol) in distilled water (2.0 ml) at *ca* 333 K with stirring. After the resulting clear solution was cooled to room temperature and allowed to stand in air for *ca* two weeks, colourless polyhedral crystals were formed.

Crystal data

$[Cd_2(C_{12}H_{24}N_2O_4)_2(NO_3)_2(H_2O)_3](NO_3)_2$
$M_r = 1047.55$
Monoclinic
$P2/n$
$a = 15.298$ (4) Å
$b = 6.097$ (1) Å
$c = 22.037$ (4) Å
$\beta = 106.72$ (2)°
$V = 1968.5$ (7) Å^3
$Z = 2$
$D_x = 1.767$ Mg m^{-3}

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 25 reflections
$\theta = 7.5$–15.0°
$\mu = 1.176$ mm^{-1}
$T = 293$ (2) K
Prism
0.35 × 0.20 × 0.10 mm
Colourless

Data collection

Siemens $R3m/V$ diffractometer
ω scans
Absorption correction: ψ scan (Kopfmann & Huber, 1968)
$T_{\min} = 0.784$, $T_{\max} = 0.895$
3627 measured reflections
3488 independent reflections
2384 observed reflections [$I > 2\sigma(I)$]

$R_{\rm int} = 0.0176$
$\theta_{\max} = 25.05°$
$h = 0 \rightarrow 18$
$k = 0 \rightarrow 7$
$l = -26 \rightarrow 25$
2 standard reflections monitored every 98 reflections
intensity decay: <1.1%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.0384$
$wR(F^2) = 0.0871$

$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.418$ e Å^{-3}
$\Delta\rho_{\min} = -0.959$ e Å^{-3}

$S = 1.063$
3488 reflections
259 parameters
H-atom parameters not refined
$w = 1/[\sigma^2(F_o^2) + (0.0291P)^2 + 1.8792P]$
where $P = (F_o^2 + 2F_c^2)/3$

Extinction correction: none
Atomic scattering factors from *International Tables for Crystallography* (1992, Vol. C, Tables 4.2.6.8 and 6.1.1.4)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters (Å^2)*

$$U_{\rm eq} = (1/3)\Sigma_i\Sigma_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	$U_{\rm eq}$
Cd(1)	3/4	−0.25836 (7)	1/4	0.02970 (13)
Cd(2)	1/4	0.72955 (9)	1/4	0.0517 (2)
O(1)	0.6876 (2)	0.0268 (5)	0.29629 (15)	0.0408 (8)
O(2)	0.6140 (2)	−0.2842 (5)	0.2912 (2)	0.0465 (8)
O(3)	0.3819 (2)	0.6938 (5)	0.2206 (2)	0.0581 (10)
O(4)	0.2963 (2)	0.4053 (6)	0.1961 (2)	0.0531 (9)
N(1)	0.5902 (2)	0.0761 (6)	0.40066 (14)	0.0275 (8)
N(2)	0.4242 (2)	0.3473 (6)	0.1108 (2)	0.0329 (8)
C(1)	0.6231 (3)	−0.0863 (7)	0.3039 (2)	0.0278 (9)
C(2)	0.5509 (3)	0.0276 (6)	0.3292 (2)	0.0256 (9)
C(3)	0.5142 (3)	0.2345 (7)	0.2913 (2)	0.0315 (9)
C(4)	0.4753 (3)	0.1802 (7)	0.2209 (2)	0.0333 (10)
C(5)	0.4475 (3)	0.3905 (7)	0.1823 (2)	0.0274 (9)
C(6)	0.3679 (3)	0.5052 (7)	0.1999 (2)	0.0289 (10)
C(7)	0.6214 (3)	−0.1338 (8)	0.4354 (2)	0.0445 (12)
C(8)	0.6689 (3)	0.2352 (8)	0.4154 (2)	0.0445 (12)
C(9)	0.5154 (3)	0.1676 (8)	0.4254 (2)	0.0422 (12)
C(10)	0.3946 (3)	0.5592 (8)	0.0754 (2)	0.0451 (12)
C(11)	0.3487 (3)	0.1808 (9)	0.0871 (2)	0.0496 (13)
C(12)	0.5088 (3)	0.2670 (8)	0.0955 (2)	0.0460 (12)
N(3)	0.8568 (3)	−0.2548 (8)	0.3947 (2)	0.0509 (11)
O(5)	0.8771 (2)	−0.2153 (6)	0.34460 (14)	0.0575 (10)
O(6)	0.9013 (4)	−0.1694 (11)	0.4433 (2)	0.124 (2)
O(7)	0.7965 (3)	−0.3818 (8)	0.3959 (2)	0.092 (2)
N(4)	0.1433 (3)	0.6210 (7)	0.0841 (2)	0.0405 (9)
O(8)	0.1844 (3)	0.7949 (6)	0.0754 (2)	0.0556 (10)
O(9)	0.1088 (3)	0.6177 (6)	0.1292 (2)	0.0711 (12)
O(10)	0.1397 (3)	0.4626 (6)	0.0491 (2)	0.0656 (11)
O(1W)	3/4	−0.6182 (8)	1/4	0.112 (3)
O(2W)	0.1950 (2)	1.0227 (6)	0.18469 (15)	0.0535 (9)

Table 2. *Selected geometric parameters (Å, °)*

Cd(1)—O(1W)	2.194 (5)	N(2)—C(12)	1.510 (5)
Cd(1)—O(1)	2.353 (3)	N(2)—C(10)	1.510 (5)
Cd(1)—O(5)	2.421 (3)	N(2)—C(11)	1.513 (6)
Cd(1)—O(2)	2.503 (3)	N(2)—C(5)	1.535 (5)
Cd(2)—O(2W)	2.297 (3)	C(1)—C(2)	1.539 (5)
Cd(2)—O(3)	2.299 (3)	C(2)—C(3)	1.528 (5)
Cd(2)—O(4)	2.511 (3)	C(3)—C(4)	1.530 (6)
O(1)—C(1)	1.254 (5)	C(4)—C(5)	1.529 (5)
O(2)—C(1)	1.238 (5)	C(5)—C(6)	1.547 (5)
O(3)—C(6)	1.234 (5)	N(3)—O(6)	1.209 (6)
O(4)—C(6)	1.236 (5)	N(3)—O(7)	1.210 (5)
N(1)—C(7)	1.497 (5)	N(3)—O(5)	1.253 (5)
N(1)—C(8)	1.507 (5)	N(4)—O(10)	1.227 (5)
N(1)—C(9)	1.509 (5)	N(4)—O(9)	1.253 (5)
N(1)—C(2)	1.543 (5)	N(4)—O(8)	1.274 (5)
O(1W)—Cd(1)—O(1)	137.65 (7)	C(8)—N(1)—C(2)	113.3 (3)
O(1$^{\rm i}$)—Cd(1)—O(1)	84.71 (14)	C(9)—N(1)—C(2)	108.9 (3)
O(1W)—Cd(1)—O(5)	96.22 (9)	C(12)—N(2)—C(10)	108.1 (3)
O(1$^{\rm i}$)—Cd(1)—O(5)	88.16 (12)	C(12)—N(2)—C(11)	108.9 (4)
O(1)—Cd(1)—O(5)	82.65 (12)	C(10)—N(2)—C(11)	108.1 (4)
O(5)—Cd(1)—O(5$^{\rm i}$)	167.6 (2)	C(12)—N(2)—C(5)	108.9 (3)
O(1)—Cd(1)—O(2$^{\rm i}$)	133.40 (10)	C(10)—N(2)—C(5)	109.3 (3)
O(5)—Cd(1)—O(2$^{\rm i}$)	76.91 (11)	C(11)—N(2)—C(5)	113.5 (3)
O(1W)—Cd(1)—O(2)	86.39 (7)	O(2)—C(1)—O(1)	123.1 (4)
O(1)—Cd(1)—O(2)	53.56 (9)	O(2)—C(1)—C(2)	118.5 (4)

O(5)—Cd(1)—O(2)	103.90 (11)	O(1)—C(1)—C(2)	118.4 (4)
O(2$^{\rm i}$)—Cd(1)—O(2)	172.77 (14)	C(3)—C(2)—C(1)	111.3 (3)
O(2$W^{\rm ii}$)—Cd(2)—O(2W)	77.8 (2)	C(3)—C(2)—N(1)	112.0 (3)
O(2$W^{\rm ii}$)—Cd(2)—O(3)	93.28 (12)	C(1)—C(2)—N(1)	110.2 (3)
O(2W)—Cd(2)—O(3)	95.18 (13)	C(2)—C(3)—C(4)	110.5 (3)
O(3)—Cd(2)—O(3$^{\rm ii}$)	169.1 (2)	C(5)—C(4)—C(3)	110.3 (3)
O(2$W^{\rm ii}$)—Cd(2)—O(4)	143.77 (11)	C(4)—C(5)—N(2)	111.9 (3)
O(2W)—Cd(2)—O(4)	114.76 (11)	C(4)—C(5)—C(6)	110.6 (3)
O(3)—Cd(2)—O(4)	53.32 (11)	N(2)—C(5)—C(6)	111.5 (3)
O(3)—Cd(2)—O(4$^{\rm ii}$)	116.62 (12)	O(3)—C(6)—O(4)	122.6 (4)
O(4)—Cd(2)—O(4$^{\rm ii}$)	76.1 (2)	O(3)—C(6)—C(5)	116.8 (4)
C(1)—O(1)—Cd(1)	94.9 (2)	O(4)—C(6)—C(5)	120.5 (4)
C(1)—O(2)—Cd(1)	88.3 (3)	O(6)—N(3)—O(7)	119.7 (5)
C(6)—O(3)—Cd(2)	97.0 (3)	O(6)—N(3)—O(5)	118.6 (5)
C(6)—O(4)—Cd(2)	86.9 (3)	O(7)—N(3)—O(5)	121.6 (5)
C(7)—N(1)—C(8)	108.8 (3)	N(3)—O(5)—Cd(1)	113.4 (3)
C(7)—N(1)—C(9)	107.2 (3)	O(10)—N(4)—O(9)	122.2 (4)
C(8)—N(1)—C(9)	109.1 (3)	O(10)—N(4)—O(8)	119.7 (4)
C(7)—N(1)—C(2)	109.3 (3)	O(9)—N(4)—O(8)	118.1 (4)

$X\cdots Y\cdots Z$	$X\cdots Y$	$Y\cdots Z$	$X\cdots Y\cdots Z$
O(1)$\cdots$O(1$W^{\rm iii}$)$\cdots$O(1$^{\rm i}$)	2.683 (5)	–	72.4 (2)
O(8)$\cdots$O(2W)$\cdots$O(4$^{\rm iii}$)	2.744 (5)	2.771 (5)	113.4 (2)

Symmetry codes: (i) $\frac{3}{2}-x, y, \frac{1}{2}-z$; (ii) $\frac{1}{2}-x, y, \frac{1}{2}-z$; (iii) $x, 1+y, z$.

The structure was solved with the Patterson superposition method. H atoms of the double-betaine molecule were generated geometrically (C—H = 0.96 Å) and the aqua H atoms were located in difference Fourier maps. All H atoms were assigned isotropic displacement parameters and included in the structure-factor calculations. All computations were performed on a PC 486 computer.

Data collection: *P3/PC Diffractometer Control Program* (Siemens, 1989). Cell refinement: *P3/PC Diffractometer Control Program*. Data reduction: *SHELXTL-Plus XDISK* (Sheldrick, 1987). Program(s) used to solve structure: *SHELXTL/PC* (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL93* (Sheldrick, 1993). Molecular graphics: *SHELXTL/PC XP*. Software used to prepare material for publication: *SHELXL93*.

This work was supported by a Hong Kong Research Grants Council Earmarked Grant (ref. CUHK 89/93E).

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: JZ1080). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

References

Chen, X.-M. & Mak, T. C. W. (1991*a*). *J. Crystallogr. Spectrosc. Res.* **21**, 27–32.
Chen, X.-M. & Mak, T. C. W. (1991*b*). *Aust. J. Chem.* **44**, 639–643.
Chen, X.-M., Mak, T. C. W., Huang, W.-Y. & Lü, L. (1992). *Acta Cryst.* **C48**, 57–59.
Clegg, W., Cressey, J. T., McCamley, A. & Straughan, B. P. (1995). *Acta Cryst.* **C51**, 234–235.
Kopfmann, G. & Huber, R. (1968). *Acta Cryst.* **A24**, 348–351.
Prince, R. H. (1987). *Comprehensive Coordination Chemistry*, Vol. 5, edited by G. Wilkinson, R. D. Gillard & J. A. McCleverty, pp. 925–1045. Oxford: Pergamon Press.
Sheldrick, G. M. (1987). *SHELXTL-Plus*. PC version. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.
Sheldrick, G. M. (1990). *SHELXTL/PC User's Manual*. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.
Sheldrick, G. M. (1993). *SHELXL93. Program for the Refinement of Crystal Structures*. University of Göttingen, Germany.
Siemens (1989). *P3/PC Diffractometer Control Program*. Version 3.13. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.
Wu, D.-D. & Mak, T. C. W. (1995*a*). *Polyhedron*. In the press.
Wu, D.-D. & Mak, T. C. W. (1995*b*). *J. Chem. Soc. Dalton Trans.* pp. 2671–2678.
Wu, D.-D. & Mak, T. C. W. (1995*c*). *Inorg. Chim. Acta*. In the press.

Acta Cryst. (1996). **C52**, 532–535

Isothiocyanatobis(1,10-phenanthroline)-copper(II) Tricyanomethanide

Ivan Potočňák,[a] Michal Dunaj-Jurčo,[a] Dušan Mikloš[a] and Lothar Jäger[b]

[a]*Department of Inorganic Chemistry, Slovak Technical University, Radlinského 9, 812 37 Bratislava, Slovakia, and* [b]*Fachbereich Chemie, Martin-Luther-University, Halle-Wittenberg, Weinbergweg 16, 06120 Halle/Saale, Germany*

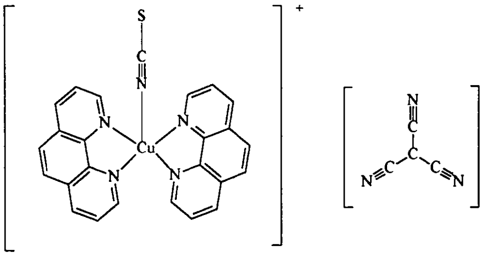
(*Received 15 March 1995; accepted 6 June 1995*)

Abstract

The crystal structure of $[Cu(NCS)(C_{12}H_8N_2)_2][C(CN)_3]$ is presented here. The structure comprises discrete $[Cu(phen)_2(NCS)]^+$ cations and $[C(CN)_3]^-$ anions. The coordination polyhedron of Cu^{II} is a distorted trigonal bipyramid with a CuN$_5$ chromophore.

Comment

The title compound, $[Cu(phen)_2(NCS)][C(CN)_3]$, (I), where phen = 1,10-phenanthroline, was prepared and its crystal structure solved as part of our study concerning changes in the shape of the $[Cu(phen)_2X]^+$ cation [X^- = pseudohalide anion (1–)] resulting from alteration of the out-of-sphere anion.



(I)

Recently, we have published the structure of $[Cu(phen)_2(NCS)][(ON)C(CN)_2]$, (II), with NCS$^-$ = X^- and $(ON)C(CN)_2^-$ (dicyanonitrosomethanide) as the out-of-sphere anion (Potočňák, Dunaj-Jurčo, Mikloš, Kabešová & Jäger, 1995). We decided to prepare a com-