

O4—C14	1.414 (12)	C14—C15	1.499 (13)
O4—C13	1.418 (11)	C16—C17	1.49 (2)
O5—C15	1.416 (11)		
C11—Hg—C12	175.71 (9)	C4—C5—C6	126.5 (11)
C7—S1—C8	103.7 (5)	C7—C6—C5	129.8 (12)
C2—S2—C17	102.9 (5)	C6—C7—C1	129.9 (11)
C10—O2—C9	114.1 (7)	C6—C7—S1	123.1 (9)
C11—O3—C12	114.5 (8)	C1—C7—S1	106.9 (8)
C14—O4—C13	112.4 (8)	C9—C8—S1	109.8 (9)
C15—O5—C16	113.6 (7)	O2—C9—C8	109.1 (8)
O1—C1—C7	119.9 (11)	O2—C10—C11	109.1 (8)
O1—C1—C2	117.6 (11)	O3—C11—C10	109.3 (8)
C7—C1—C2	122.3 (10)	O3—C12—C13	110.4 (8)
C3—C2—C1	124.6 (10)	O4—C13—C12	107.4 (8)
C3—C2—S2	117.1 (9)	O4—C14—C15	109.1 (9)
C1—C2—S2	118.0 (7)	O5—C15—C14	108.5 (7)
C4—C3—C2	132.1 (11)	O5—C16—C17	106.7 (9)
C3—C4—C5	129.1 (10)	C16—C17—S2	112.1 (9)
C7—S1—C8—C9	-160.1 (8)	O3—C12—C13—O4	70.0 (11)
C10—O2—C9—C8	176.1 (10)	C13—O4—C14—C15	-177.0 (8)
S1—C8—C9—O2	71.2 (12)	C16—O5—C15—C14	169.3 (10)
C9—O2—C10—C11	-158.2 (10)	O4—C14—C15—O5	-70.1 (11)
C12—O3—C11—C10	178.9 (8)	C15—O5—C16—C17	150.6 (10)
O2—C10—C11—O3	-69.1 (11)	O5—C16—C17—S2	169.5 (8)
C11—O3—C12—C13	176.9 (8)	C2—S2—C17—C16	-76.7 (10)
C14—O4—C13—C12	-178.2 (8)		

All H atoms were located at ideal positions and were included in the refinement, but restrained to ride on the atom to which they are bonded. The isotropic displacement parameter of each H atom was held fixed at 1.2 times U_{eq} of the parent atom.

Data collection: *CAD-4 Software* (Enraf-Nonius, 1989). Cell refinement: *CAD-4 Software*. Data reduction: *MolEN* (Fair, 1990). Program(s) used to solve structure: *SIR92* (Altomare *et al.*, 1994). Program(s) used to refine structure: *SHELXL93* (Sheldrick, 1993). Molecular graphics: *Xtal.GX* (Hall & du Boulay, 1995). Software used to prepare material for publication: *SHELXL93*.

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: TA1067). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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cis-Dichlorobis(1,10-phenanthroline-*N*¹,*N*¹⁰)cadmium(II)

HONG WANG,^a REN-GEN XIONG,^b HONG-YUAN CHEN^a,
XIAO-YING HUANG^c AND XIAO-ZENG YOU^b

^aDepartment of Chemistry, Nanjing University, Nanjing 210008, People's Republic of China, ^bInstitute and State Key Laboratory of Coordination Chemistry, Nanjing University, Nanjing 210008, People's Republic of China, and ^cState Key Laboratory of Structural Chemistry, Fujian Institute of Research on the Structure of Matter, Academia Sinica, Fuzhou 350002, People's Republic of China

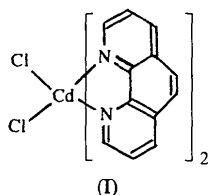
(Received 4 January 1996; accepted 14 February 1996)

Abstract

The title complex, [CdCl₂(C₁₂H₈N₂)₂], exhibits slightly distorted octahedral geometry; the Cd atom is coordinated to four N atoms from two phenanthroline ligands and to two *cis* Cl atoms. The Cd—N bond lengths are in the range 2.387 (3)–2.486 (4) Å and the Cd—Cl distances are 2.505 (1) and 2.528 (1) Å. The Cl—Cd—Cl angle is 106.19 (5)° and the chelate ring N—Cd—N bond angles are 68.4 (1) and 68.5 (1)°.

Comment

Potential models for cysteine-containing zinc metallo-proteins that mimic both the structure and reactivity of the metal ion sites have attracted increasing attention (Rijn, Reedijk, Dartmann & Krebs, 1987; Santos, Gruff, Koch & Harbison, 1990). Cadmium has been substituted for zinc in the native proteins to aid the study of the spectroscopic features since zinc is a spectroscopically invisible metal, whereas with cadmium the coordination environment of the metal can be probed by ^{113}Cd NMR (Summers, 1988). The chemical-shift range of ^{113}Cd NMR exceeds 900 p.p.m. and the technique is regarded as one of the most useful for the determination of the nature of the coordination in a wide range of cadmium compounds. On the other hand, phenanthroline (phen) and related bidentate ligands form water-soluble neutral chelate complexes such as $M(\text{phen})_2\text{X}_2$, which have found practical applications in medicine as, for example, potential antitumor agents. We report here the crystal structure of dichlorobis(phenanthroline)cadmium(II), (I), which displays *cis*-octahedral coordination very similar to that found in the pseudo-halide complexes *cis*- $[\text{Mn}(\text{phen})_2(\text{NCS})_2]$, *cis*- $[\text{Mn}(\text{bipy})_2(\text{NCS})_2]$ and *cis*- $[\text{Co}(\text{bipy})_2(\text{NCS})_2]$ (bipy = 2,2'-bipyridine), but quite different from that of $[\text{Cu}(\text{phen})(\text{NCS})_2]$ (Holleman, Parker & Breneman, 1994; Veidis, Dockum, Charron, Reiff & Brennam, 1981; Lumme & Lindell, 1988; Breneman & Parker, 1993).



The Cd—N bond distances [2.387 (3)–2.486 (4) Å] are much longer than the Mn—N_{phen} distances in *cis*- $[\text{Mn}(\text{phen})_2(\text{NCS})_2]$ [2.294 (2)–2.301 (3) Å] (Holleman *et al.*, 1994). They are also somewhat longer than the corresponding Cd—N bonds in the tetrahedral species $[\text{Cd}(\text{S}-2,4,6\text{-}^i\text{Pr}_3\text{-C}_6\text{H}_2)_2(\text{bipy})]$ [2.334 (6)–2.337 (6) Å], in $[\text{Cd}(\text{S}-2,4,6\text{-}^i\text{Pr}_3\text{-C}_6\text{H}_2)_2(\text{phen})]$ [2.364 (8)–2.395 (8) Å], where the Cd atom is trigonal bipyramidal, and in the non-chelate complex $[\text{Cd}(\text{S}-2,4,6\text{-}^i\text{Pr}_3\text{-C}_6\text{H}_2)_2(1\text{-Me-imid})_2]$ [2.259 (9)–2.268 (9) Å] (S-2,4,6-*i*Pr₃-C₆H₂ is 2,4,6-triisopropylbenzenethiolate and 1-Me-imid is 1-methylimidazole) (Santos *et al.*, 1990). It is worth noting that in the title complex the Cd—N bonds *trans* to N are slightly shorter than those *trans* to Cl. Similar trends have been observed in MnQ₃ and CrQ₃ (Q is 8-quinolinol) (Xiong, You, Wu & Huang, 1995; Foltz, Cox, Moore & Merrit, 1968). The N—Cd—N bond angles, however, appear to agree with those found in $[\text{Cd}(\text{S}-2,4,6\text{-}^i\text{Pr}_3\text{C}_6\text{H}_2)_2(\text{phen})]$ [69.6 (3)°] (Santos *et al.*, 1990). The Cd—Cl bond dis-

tances [2.505 (1)–2.528 (1) Å] are slightly shorter than those found in both $[(\text{C}_3\text{H}_7\text{NH}_2)_2\text{CdCl}_4]$ [2.61–2.65 Å] and $[(\text{C}_4\text{H}_{12}\text{N})_2\text{CdCl}_4]$ [2.528 (2)–2.660 (2) Å], where the corner-sharing CdCl₆ octahedra form chessboard-like layers and the presence of hydrogen bonds perturbs the metal coordination (Doudin & Chapuis, 1988, 1992), but they are in agreement with the values found in $[\text{CdCl}_2\{\mu\text{-S}(\text{CH}_2)_3\text{NH}(\text{CH}_3)_2\}]$ [2.455 (1)–2.507 (1) Å] (Casals, Gonzalez-Duarte, Sola, Font-Bardia, Solans & Solans, 1987). Even shorter Cd—Cl bonds are found in $\text{RH}_2(\text{CdCl}_4)$ [2.444 (2)–2.466 (1) Å] (RH is the rhodamine 6G cation), where the metal displays slightly distorted tetrahedral coordination (Wang, Xiong, Chen, You, Chen, Zhou & Mak, 1996). The C—C and C—N bond lengths are unexceptional.

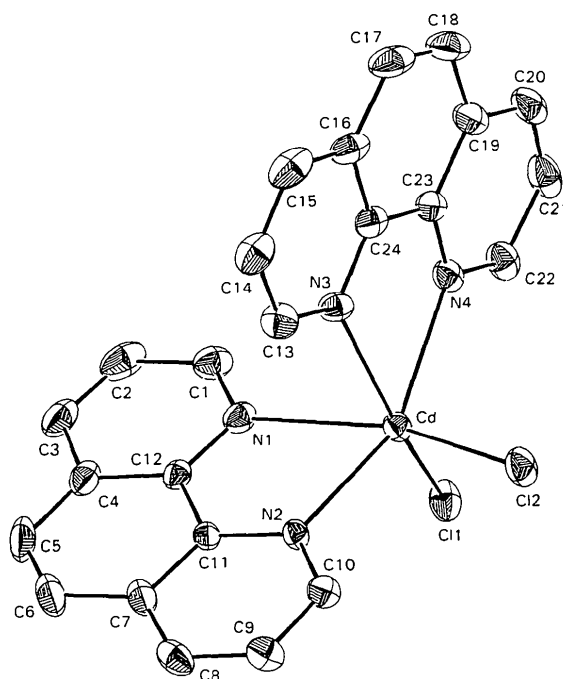


Fig. 1. The structure of $[\text{CdCl}_2(\text{phen})_2]$ shown with 30% probability displacement ellipsoids. H atoms have been omitted for clarity.

Experimental

The title complex was prepared by heating $\text{CdCl}_2 \cdot 6\text{H}_2\text{O}$, 8-quinolinol and phenanthroline under reflux in $\text{CHCl}_3/\text{EtOH}$ solution for 2 h. The resulting solution was evaporated at room temperature for a few weeks. Pale yellow column-shaped crystals were obtained.

Crystal data

$[\text{CdCl}_2(\text{C}_{12}\text{H}_8\text{N}_2)_2]$
 $M_r = 543.73$

Mo K α radiation
 $\lambda = 0.71069 \text{ \AA}$

Monoclinic	Cell parameters from 25 reflections
$P2_1/c$	$\theta = 13.98\text{--}14.81^\circ$
$a = 9.570(2) \text{ \AA}$	$\mu = 1.318 \text{ mm}^{-1}$
$b = 15.150(4) \text{ \AA}$	$T = 296 \text{ K}$
$c = 14.602(2) \text{ \AA}$	Column
$\beta = 99.21(2)^\circ$	$0.70 \times 0.50 \times 0.45 \text{ mm}$
$V = 2089.8(8) \text{ \AA}^3$	Pale yellow
$Z = 4$	
$D_x = 1.73 \text{ Mg m}^{-3}$	
Data collection	
Enraf-Nonius CAD-4 diffractometer	3198 observed reflections
$\omega/2\theta$ scans	$[I > 3\sigma(I)]$
Absorption correction: ψ scans (TEXSAN; Molecular Structure Corporation, 1985)	$R_{\text{int}} = 0.061$
$T_{\text{min}} = 0.804$, $T_{\text{max}} = 0.916$	$\theta_{\text{max}} = 25^\circ$
4073 measured reflections	$h = 0 \rightarrow 11$
3835 independent reflections	$k = -18 \rightarrow 0$
	$l = -17 \rightarrow 17$
	3 standard reflections monitored every 400 reflections
	intensity decay: 2.2%

Refinement

Refinement on F	$w = 1/\sigma^2(F)$
$R = 0.040$	$(\Delta/\sigma)_{\text{max}} = 0.32$
$wR = 0.053$	$\Delta\rho_{\text{max}} = 0.53 \text{ e \AA}^{-3}$
$S = 1.63$	$\Delta\rho_{\text{min}} = -1.63 \text{ e \AA}^{-3}$
3198 reflections	(near Cd)
328 parameters	Extinction correction: none
H-atom coordinates refined; $U(\text{H}) \approx 1.2U_{\text{eq}}(\text{C})$	Atomic scattering factors from Cromer & Waber (1974)

Table 1. Fractional atomic coordinates and equivalent isotropic displacement parameters (\AA^2)

	$B_{\text{eq}} = (4/3)\sum_i \sum_j \beta_{ij} a_i \cdot a_j$			
	x	y	z	B_{eq}
Cd	0.24108 (3)	0.22633 (2)	0.04869 (2)	2.56 (2)
Cl(1)	0.1835 (1)	0.13003 (9)	0.17697 (9)	3.82 (5)
Cl(2)	0.0377 (1)	0.20965 (8)	-0.08376 (9)	3.86 (5)
N(1)	0.4902 (4)	0.2226 (2)	0.1294 (3)	3.0 (2)
N(2)	0.3873 (3)	0.1389 (2)	-0.0337 (2)	2.5 (1)
N(3)	0.3252 (4)	0.3570 (2)	-0.0240 (3)	3.0 (1)
N(4)	0.1692 (4)	0.3598 (2)	0.1168 (3)	3.0 (1)
C(1)	0.5393 (6)	0.2613 (3)	0.2088 (4)	3.8 (2)
C(2)	0.6808 (7)	0.2566 (4)	0.2511 (4)	4.8 (3)
C(3)	0.7726 (6)	0.2103 (3)	0.2081 (4)	4.4 (2)
C(4)	0.7259 (5)	0.1678 (3)	0.1233 (4)	3.6 (2)
C(5)	0.8160 (5)	0.1183 (4)	0.0746 (5)	4.7 (3)
C(6)	0.7652 (5)	0.0778 (4)	-0.0037 (5)	4.4 (3)
C(7)	0.6211 (5)	0.0819 (3)	-0.0451 (3)	3.2 (2)
C(8)	0.5617 (6)	0.0380 (3)	-0.1275 (3)	3.7 (2)
C(9)	0.4214 (5)	0.0465 (3)	-0.1607 (3)	3.5 (2)
C(10)	0.3369 (5)	0.0966 (3)	-0.1111 (3)	3.1 (2)
C(11)	0.5271 (4)	0.1322 (3)	0.0008 (3)	2.5 (2)
C(12)	0.5808 (4)	0.1765 (3)	0.0867 (3)	2.6 (2)
C(13)	0.4036 (6)	0.3564 (3)	-0.0900 (3)	3.7 (2)
C(14)	0.4353 (6)	0.4312 (4)	-0.1366 (4)	4.5 (3)
C(15)	0.3823 (7)	0.5096 (4)	-0.1144 (4)	4.8 (3)
C(16)	0.2996 (6)	0.5149 (3)	-0.0451 (4)	3.9 (2)
C(17)	0.2383 (8)	0.5936 (4)	-0.0174 (5)	5.2 (3)
C(18)	0.1590 (7)	0.5956 (4)	0.0488 (5)	5.2 (3)
C(19)	0.1308 (5)	0.5161 (3)	0.0972 (4)	4.0 (2)
C(20)	0.0482 (6)	0.5145 (4)	0.1675 (4)	4.8 (3)
C(21)	0.0300 (6)	0.4380 (4)	0.2115 (4)	4.9 (3)

Table 2. Selected geometric parameters (\AA , $^\circ$)

Cd—N(2)	2.387 (3)	N(1)—C(12)	1.341 (6)
Cd—N(4)	2.401 (4)	N(2)—C(10)	1.322 (6)
Cd—N(3)	2.444 (4)	N(2)—C(11)	1.356 (5)
Cd—N(1)	2.486 (4)	N(3)—C(13)	1.313 (6)
Cd—Cl(1)	2.505 (1)	N(3)—C(24)	1.350 (6)
Cd—Cl(2)	2.528 (1)	N(4)—C(22)	1.315 (6)
N(1)—C(1)	1.317 (7)	N(4)—C(23)	1.346 (6)
N(2)—Cd—N(4)	154.9 (1)	N(4)—Cd—Cl(2)	99.5 (1)
N(2)—Cd—N(3)	88.3 (1)	N(3)—Cd—N(1)	82.3 (1)
N(2)—Cd—N(1)	68.4 (1)	N(3)—Cd—Cl(1)	157.6 (1)
N(2)—Cd—Cl(1)	105.73 (9)	N(3)—Cd—Cl(2)	90.9 (1)
N(2)—Cd—Cl(2)	90.02 (8)	N(1)—Cd—Cl(1)	86.7 (1)
N(4)—Cd—N(3)	68.5 (1)	N(1)—Cd—Cl(2)	157.4 (1)
N(4)—Cd—N(1)	98.0 (1)	Cl(1)—Cd—Cl(2)	106.19 (5)
N(4)—Cd—Cl(1)	93.9 (1)		

Data collection used *CONTROL* software (Molecular Structure Corporation, 1986). The structure was solved by direct methods using *MITHRIL* (Gilmore, 1983) and *DIRDIF* (Beurskens, 1984). The structure was refined by full-matrix least-squares techniques, with anisotropic displacement parameters for all non-H atoms. All calculations were performed on a VAX3100 computer using the *TEXSAN* (Molecular Structure Corporation, 1985) program package.

This work was supported by a Major Project grant from the State Science and Technology Commission and the National Science Foundation of China.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry, including least-squares-planes data, intermolecular contact distances and torsion angles, have been deposited with the IUCr (Reference: MU1246). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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(5-Benzylidibenzob[*b,d*]phosphole 5-oxide)-trimethylindium

WENJIE WANG,^a YI PAN,^{a*} XIAOYING HUANG,^b HONGSUI SUN,^a ZHIBING LU^a AND XIANGZHEN SUN^{a*}

^aState Key Laboratory of Coordination Chemistry, Department of Chemistry, Nanjing University, Nanjing 210093, People's Republic of China, and ^bState Key Laboratory of Structural Chemistry, Fuzhou 250003, People's Republic of China

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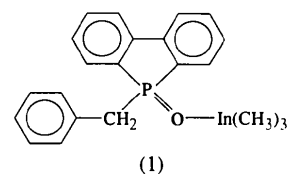
Abstract

The X-ray crystal structure determination of the title compound, [In(CH₃)₃(C₁₉H₁₅OP)], shows that the In atom has a four-coordinate distorted tetrahedral environment with an In—O bond length of 2.261 (3) Å. The In—O—P linkage is bent with a bond angle of 135.9 (2)°.

Comment

High-purity indium trialkyls have been used extensively in the growth of compound semiconductors by metal-organic chemical vapour deposition (MOCVD). One of the most successful purification methods for indium trialkyls may be called adduct purification, which involves the formation, purification and dissociation of non-volatile trialkylindium adducts. Donor ligands applied hitherto have mainly been N- and P-containing Lewis bases (Bradley, Chudzynska & Frigo, 1988; Bradley *et al.*, 1988; Foster, Rushworth, Cole-Hamilton, Jones & Stagg, 1988; Moore *et al.*, 1986). In the course of our work on searching for new useful adducts for the purification of indium trialkyls, we isolated the title

compound, (1). The X-ray structure of (1) is presented in Fig. 1.



In contrast to indium halide phosphine oxide complexes, in which indium is six-coordinate with distorted octahedral geometry (Robinson, Wilkins & Zhang, 1990), indium in (1) is four-coordinate in a distorted tetrahedron. The In—O bond length of 2.261 (3) Å is longer than those in InCl₃(Me₃PO)₃ [2.164 (11) Å], InCl₃(Me₃PO)₂(MeOH) [2.155 (4) Å] and [InCl₃(Me₃PO)₂(H₂O)]H₂O [2.157 (5) Å]. The trigonal Me₃In molecule undergoes only slight pyramidalization upon coordination of the phosphine oxide [C—In—C = 116.1 (2)–120.7 (2)°], and therefore no significant change in In—C distances [2.16 (4)–2.25 (6) Å for Me₃In (Vranka & Amma, 1967) and 2.158 (5)–2.168 (5) Å for (1)] was observed. These data suggest weak interaction between the indium centre and the O atom. Although there are no crystal data available for 5-benzylidibenzob[*b,d*]phosphole 5-oxide, the P—O bond distance for the oxide should be comparable with that of Ph₃PO. The P—O distance in (1) [1.481 (3) Å] is almost the same as the P—O bond distance for free Ph₃PO [1.484 (1) Å] (Spek, 1987), which should imply only weak π delocalization over P—O—In, if any. In stark contrast to the Al—O—P and Ga—O—P linear linkages in AlMe₃.OPPh₃ (Feher, Budzichowski & Weller, 1993), AlCl₃.OPPh₃ and GaCl₃.OPPh₃ (Burford *et al.*, 1990), In—O—P in (1) is bent with an In—O—P bond angle of 135.9 (2)°. This agrees well with the idea of weak π delocalization over the In—O—P linkage. The P centre in (1) has distorted tetrahedral geometry. Although the P-containing five-membered ring is nearly planar, normal P—C(2) and P—C(12) single-bond distances indicate non-aromatic character of the ring.

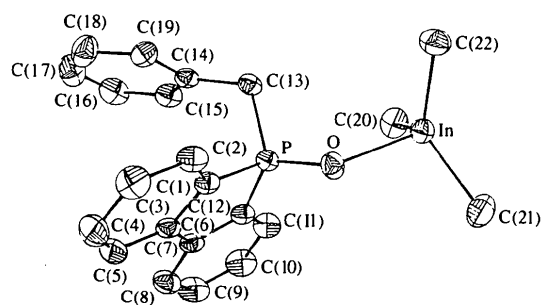


Fig. 1. Molecular structure showing 50% probability displacement ellipsoids. H atoms are omitted for clarity.