Table 3. *Values of M—O distances* (Å) *in some metalloporphyrin complexes*

Complex	*M*—O	References
$Os^{VI}(O)_2(oep)$	1·745 (5)	This work
$Os^{VI}(O)_2(ttp)$	1·743 (3)	Che, Chung & Lai (1988)
$Mo^{IV}(O)(ttp)$	1·656 (6)	Diebold, Chevrier & Weiss (1979)
$Mo^{V}(Cl)(O)(tpp)^{a}$	1·714 (3)	Ledon & Mentzen (1978)
$[Mo^{VI}(O)(tpp)]_2O$	1·707 (3)	Johnson & Scheidt (1977)
$Ti^{IV}(O)(oepMe2)^{b}$	1·619 (4)	Dwyer, Puppe, Buchler & Scheidt (1975)
$Ti^{IV}(O)(oep)$	1·613 (5)	Guilard, Latour, Lecomte, Marchon, Protas & Ripoll (1978)
$Cr^{IV}(O)(ttp)$	1·572 (6)	Groves, Kruper, Haushalter & Butler (1982)
$Cr^{IV}(O)(tpp)$	1·62 (2)	Budge, Gatehouse, Nesbit & West (1981)
$V^{IV}(O)(oep)$	1·620 (2)	Molinaro & Ibers (1976)
$V^{IV}(O)(ttp)$	1·625 (16)	Drew, Mitchell & Scott (1984)
$V^{IV}(O)(etp)^{c}$	1·599 (6)	Drew, Mitchell & Scott (1984)
$V^{IV}(O)(dpep)^{d}$	1·582 (3)	Miller, Hambley & Taylor (1984)
$Nb^{V}(F)(O)(oep)$	1·749 (3)	Lecomte, Protas, Richard, Barbe & Guilard (1982)

(*a*) oepMe2 = α,γ-dimethyl-α,γ-dihydrooctaethylporphyrin; (*b*) tpp = tetraphenylporphyrin; (*c*) etp = etioporphyrin; (*d*) dpep = deoxophylloerythroetioporphyrin.

phyrin. In Table 3 are reported the *M*—O distances for several metalloporphyrin complexes; five-coordinate complexes have *M*—O $\simeq$ 1·6 Å and six-coordinate species have *M*—O $\simeq$ 1·7 Å.

Support of this work by the National Institutes of Health (GM-38401) is gratefully acknowledged.

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Acta Cryst. (1990). C**46**, 1098–1100

Structure of *catena*-Poly[bis(ethylenediamine)zinc(II)-μ-cyano-dicyanonickel(II)-μ-cyano]

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(*Received* 17 *July* 1989; *accepted* 8 *January* 1990)

Abstract. $[NiZn(C_2H_8N_2)_2(CN)_4]$, $M_r = 348·35$, monoclinic, $P2_1/n$, $a = 7·173$ (3), $b = 10·606$ (4), $c = 10·091$ (6) Å, $\beta = 115·91$ (4)°, $V = 690·53$ (6) Å^3, $Z = 2$, $D_m = 1·66$ (1), $D_x = 1·675$ Mg m^{-3}, $F(000) = 356$, λ(Mo $K\alpha$) = 0·71069 Å, $\mu = 3·1$ mm^{-1}, $T = 295$ K, $R = 0·058$ for 1048 reflexions. The structure is built up of infinite electroneutral $[—Zn(en)_2—NC—Ni(CN)_2—CN—]_\infty$ chains parallel to the *c* axis (en = $H_2NCH_2CH_2NH_2$). The chains are significantly bent at the N(1) atom, the angle C(1)—N(1)—Zn

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being 148·0 (7)°. The Ni atom is square-planar coordinated by four cyano groups. The Zn atom is coordinated in the form of an elongated tetragonal bipyramid. Two chelate ethylenediamine molecules are bonded in the equatorial plane. The axial positions are occupied by N atoms from bridged cyano groups. The C atoms in the ethylenediamine ligand are disordered in two positions.

Experimental. Preliminary Weissenberg photographs reveal space group $P2_1/n$ and isostructurality with $Ni(en)_2Ni(CN)_4$ (Černák, Chomič, Baloghová & Dunaj-Jurčo, 1988) and $Ni(en)_2Pd(CN)_4$ (Rüegg & Ludi, 1971). D_m by flotation in mixture of bromoform and chloroform. Crystal: yellow parallelepiped 0·6 × 0·4 × 0·3 mm. Data collection: Syntex $P2_1$ diffractometer, graphite-monochromated Mo $K\alpha$ radiation (θ–2θ scan technique, $2\theta_{\max} = 60°$). Unit-cell parameters from 15 reflections with $3° \leq \theta \leq 13°$. 2 standard reflections measured every 100 reflections. Variation 4%. 2014 unique reflections ($0 \leq h \leq 10$, $0 \leq k \leq 14$, $-12 \leq l \leq 12$); of these, 1048 had $F_o \geq 4\sigma(F_o)$. Lp corrections using the program *XP21* (Pavelčík, 1986), absorption corrections after the isotropic model following the method of Walker & Stuart (1983) with the program *ABSORB* (Ugozzoli, 1987). As an initial model the atomic positions from $Ni(en)_2Ni(CN)_4$ (Černák *et al.* 1988) were taken. The structure was subjected to anisotropic full-matrix least-squares refinement except for disordered C atoms which where treated isotropically. The function minimized was $\sum w(F_o - |F_c|)^2$. H atoms included in the model in calculated positions. The final R value was 0·0584, with wR = 0·0583, unit weights; 83 parameters varied in the last cycle; S = 1·74; $(\Delta/\sigma)_{\max} = 0{\cdot}049$, max. $\Delta\rho \pm 0{\cdot}75$ e Å^{-3} near the heavy atoms. Final atomic coordinates are given in Table 1. Bond lengths and angles are given in Table 2.*

Calculations were performed with *SHELX76* (Sheldrick, 1976). Geometrical analysis using *PARST* (Nardelli, 1983). Atomic scattering factors were those in *SHELX76*. A projection of the structure down the x axis is shown in Fig. 1.

Related literature. The crystal structures and properties of $Zn(en)_3Ni(CN)_4.H_2O$ and $Zn(en)_3Ni(CN)_4$ complexes are described in Černák, Chomič, Dunaj-Jurčo & Kappenstein (1984). The preparation of the title compound from solution as well as by thermal decomposition of tris(en) complexes is described in

Table 1. *Fractional coordinates* (× 10^4) *and equivalent or isotropic thermal parameters* (× 10^2 Å^2) *of non*-H *atoms with e.s.d.'s in parentheses*

K (the site occupation factor) is given when it is different from unity. $U_{eq} = (1/3)\sum_i\sum_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j$.

	x	y	z	U/U_{eq}	K
Zn	0	0	5000	2·45 (5)	
Ni	0	0	0	2·53 (5)	
N(1)	−1012 (11)	394 (7)	2564 (7)	3·6 (2)	
N(2)	4028 (12)	−1028 (8)	2264 (9)	5·6 (4)	
N(3)	1168 (10)	1850 (6)	5478 (7)	3·6 (3)	
N(4)	−2733 (10)	882 (6)	4876 (7)	3·1 (2)	
C(1)	−700 (11)	272 (6)	1550 (7)	2·5 (2)	
C(2)	2540 (12)	−647 (8)	1388 (7)	3·2 (3)	
C(3)	−308 (22)	2573 (15)	5909 (17)	4·4 (3)	0·63 (4)
C(31)	−533 (42)	2775 (26)	5170 (29)	5·0 (3)	0·37 (4)
C(4)	−2494 (21)	2209 (15)	4742 (16)	4·0 (3)	0·63 (4)
C(41)	−2059 (39)	2225 (25)	5524 (29)	4·1 (5)	0·37 (4)

Table 2. *Bond lengths* (Å) *and angles* (°)

Zn—N(1)	2·277 (7)	C(31)—C(41)	1·42 (5)
Zn—N(3)	2·105 (6)	C(41)—N(4)	1·55 (3)
Zn—N(4)	2·126 (8)		
N(3)—C(3)	1·52 (2)	Ni—C(1)	1·862 (9)
C(3)—C(4)	1·54 (2)	Ni—C(2)	1·877 (7)
C(4)—N(4)	1·43 (2)		
		N(1)—C(1)	1·146 (12)
N(3)—C(31)	1·49 (3)	N(2)—C(2)	1·122 (10)
N(1)—Zn—N(3)	89·1 (3)	N(3)—C(31)—C(41)	109 (2)
N(1)—Zn—N(4)	90·3 (3)	C(31)—C(41)—N(4)	114 (2)
N(3)—Zn—N(4)	82·4 (3)	C(41)—N(4)—Zn	106 (1)
Zn—N(3)—C(3)	106·2 (7)	C(1)—Ni—C(2)	87·7 (3)
N(3)—C(3)—C(4)	105 (1)		
C(3)—C(4)—N(4)	107 (1)		
C(4)—N(4)—Zn	107·0 (8)	Zn—N(1)—C(1)	148·0 (7)
Zn—N(3)—C(31)	111 (1)	Ni—C(1)—N(1)	175·3 (7)
		Ni—C(2)—N(2)	176·9 (8)

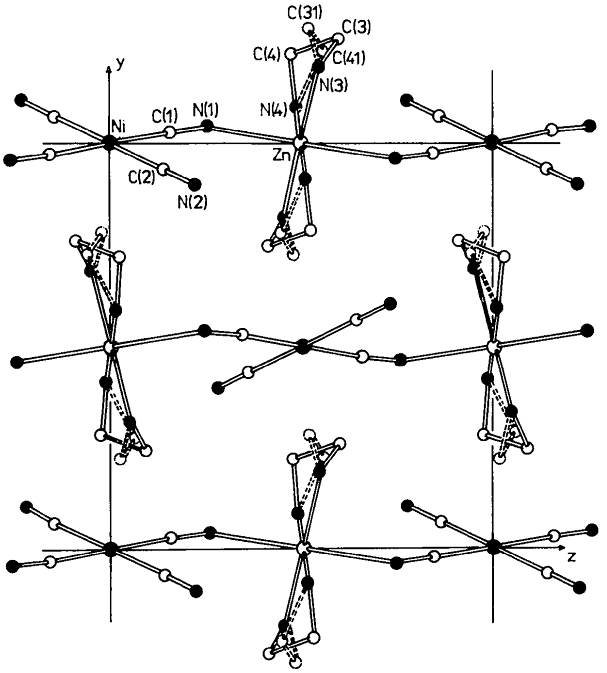

Fig. 1. Projection of the structure of $Zn(en)_2Ni(CN)_4$ down the x axis with the atomic numbering scheme.

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* Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52587 (10 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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We thank the Computer Centre of the Technical University of Košice for the use of the EC 1045 computer.

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Structure of *catena*-Tris(μ-diethyldithiophosphinato)-μ-iodo-dimercury(II), $Hg_2I[S_2P(C_2H_5)_2]_3$

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(*Received* 11 *October* 1989; *accepted* 18 *December* 1989)

Abstract. $[Hg_2(C_4H_{10}PS_2)_3]$, M_r = 987·80, monoclinic, $P2_1/c$, a = 17·865 (5), b = 10·371 (1), c = 15·559 (4) Å, β = 104·44 (3)°, V = 2791 (1) Å^3, Z = 4, D_x = 2·350 (1), D_m = 2·36 g cm^{-3}, λ(Mo $K\alpha$) = 0·71069 Å, μ = 127·6 cm^{-1}, $F(000)$ = 1824, T = 293 K, R = 0·040 for 3367 reflections with $I > 3\sigma(I)$. The crystal is built up from infinite two-dimensional networks. One of the two unique Hg atoms coordinates three S and one I atom in a tetrahedron, while the other Hg also coordinates three S and one I atom but has an almost planar coordination to the three S atoms; the distances from Hg to the planes defined by the three S atoms are 0·9923 (6) and 0·1847 (6) Å, respectively. The diethyldithiophosphinato ligands act as bridges between the Hg atoms.

Experimental. $Hg_2I[S_2P(C_2H_5)_2]_3$ was prepared by dissolving HgI_2 in aqueous NaI solution and then adding an aqueous solution of $(C_2H_5)_2PS_2Na.2H_2O$. Transparent single crystals were grown from dioxane by slow evaporation at room temperature. Dimensions of crystal examined were 0·12 × 0·14 × 0·18 mm. D_m was measured by flotation in a $CHCl_3$/$CHBr_3$ mixture. Huber four-circle X-ray diffractometer (Svensson, 1987) using graphite-monochromated Mo $K\alpha$ radiation (λ = 0·71069 Å). Cell constants were determined by least-squares method from setting angles of 18 reflections in the range $10 < \theta < 17°$. 5412 reflection intensities were measured with the $\omega/2\theta$ technique, $\Delta\omega = (0{\cdot}7 + 0{\cdot}6\tan\theta)°$, scan speed 0·5° min^{-1}. If $3{\cdot}0 < I/\sigma(I) < 50{\cdot}0$, a second scan was performed with a scan speed of 0·2° min^{-1}. Three standard reflections, measured every third hour, decreased by approximately 6% during the data collection; a correction for this decay was made. Data were corrected for Lorentz, polarization and absorption effects. Absorption correction by numerical integration of crystal defined by six faces {100}, $(0\bar{1}\bar{1})$, $(01\bar{1})$, $(1\bar{1}1)$ and (111), grid 12 × 12 × 12, transmission factor 0·10–0·31. 4932 unique reflections ($R_{\rm int}$ = 0·035), 3367 with $I > 3\sigma_c(I)$ used in refinement (σ_c based on counting statistics). Index range h −18→17, k −12→0 and l −20→0, $(\sin\theta)/\lambda < 0{\cdot}60$ Å^{-1}. Structure solved by Patterson methods and subsequent electron-density difference maps. Methylene H atoms placed in calculated positions (C—H = 1·0 Å, $U_{\rm iso}$ = 0·08 Å^2). Non-H atoms refined with anisotropic displacement parameters. Least-squares refinement, function minimized $\sum w(|F_o| - |F_c|)^2$, $w = [\sigma^2(|F_o|) + (c_1|F_o|)^2 + c_2]^{-1}$ with c_1 = 0·017 and c_2 = 7 adjusted to give constant $\langle w(\delta F)^2\rangle$ in different $|F_o|$ and $\sin\theta$ intervals, final R = 0·040, wR = 0·049, S = 1·34, $(\Delta/\sigma)_{\max}$ = 0·20, correction for secondary extinction, $g = 2{\cdot}7(1) \times 10^3$ [type I, Lorentzian mosaicism (Becker & Coppens, 1974)], $\Delta\rho_{\max}/\Delta\rho_{\min} = 1{\cdot}14/-1{\cdot}10$ e Å^{-3}, 218 parameters.

Data and final model were compared by probability plotting (Abrahams & Keve, 1971) of ordered values of $\delta R_i = \Delta F_i/\sigma(|F_o|_i)$ *vs* those expected for

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