

Černák, Chomič, Potočňák & Dunaj-Jurčo 1989). Structures of the general formula $M(en)_2Ni(CN)_4$ have been described by Dunaj-Jurčo, Garaj, Chomič, Haluška & Valach (1976) ($M = Cu$), Jameson, Bachmann, Oswald & Dubler (1981) ($M = Cd$) and Černák *et al.* (1988) ($M = Ni$).

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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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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, $\beta = 104.44$ (3)°, $V = 2791$ (1) Å³, $Z = 4$, $D_x = 2.350$ (1), $D_m = 2.36$ g cm⁻³, $\lambda(Mo K\alpha) = 0.71069$ Å, $\mu = 127.6$ 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 $(C_2H_5)_2PS_2Na \cdot 2H_2O$. 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 ($\lambda = 0.71069$ Å). Cell constants were determined by least-squares method from setting angles of 18 reflections in the range $10 < \theta < 17^\circ$. 5412 reflection intensities were measured with the $\omega/2\theta$ technique, $\Delta\omega = (0.7 + 0.6\tan\theta)^\circ$, scan

speed 0.5° min⁻¹. If $3.0 < I/\sigma(I) < 50.0$, 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 ($R_{int} = 0.035$), 3367 with $I > 3\sigma_c(I)$ used in refinement (σ_c based on counting statistics). Index range $h - 18 \rightarrow 17$, $k - 12 \rightarrow 0$ and $l - 20 \rightarrow 0$, $(\sin\theta)/\lambda < 0.60$ 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, $U_{iso} = 0.08$ Å²). 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, $g = 2.7$ (1) × 10³ [type I, Lorentzian mosaicism (Becker & Coppens, 1974)], $\Delta\rho_{max}/\Delta\rho_{min} = 1.14/-1.10$ 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

Table 1. Fractional atomic coordinates with equivalent isotropic displacement parameters (\AA^2) for the non-H atoms

$$U_{\text{eq}} = (1/3) \sum_i \sum_j U_{ij} a_i^* a_j^* \mathbf{a}_i \cdot \mathbf{a}_j$$

	x	y	z	$U_{\text{eq}} (\text{\AA}^2)$
Hg1	0.24159 (3)	0.02191 (6)	0.44711 (4)	0.0504 (2)
Hg2	0.27194 (3)	0.07653 (6)	0.19231 (4)	0.0498 (2)
I	0.14633 (5)	-0.02912 (10)	0.24432 (6)	0.0527 (3)
S1	0.3965 (2)	0.0611 (4)	0.3148 (2)	0.058 (1)
S2	0.2800 (2)	0.2388 (3)	0.4136 (2)	0.051 (1)
S3	0.3178 (2)	-0.0439 (3)	0.0705 (2)	0.057 (1)
S4	0.3192 (2)	-0.1752 (4)	0.4757 (3)	0.057 (1)
S5	0.2442 (2)	0.3108 (3)	0.1449 (2)	0.046 (1)
S6	0.1141 (2)	0.0281 (4)	0.4908 (2)	0.052 (1)
P1	0.3862 (2)	0.2077 (4)	0.3960 (2)	0.048 (1)
P2	0.2643 (2)	-0.2156 (3)	0.0509 (2)	0.044 (1)
P3	0.1333 (2)	0.1841 (3)	0.5714 (2)	0.039 (1)
C1	0.454 (1)	0.172 (2)	0.502 (1)	0.090 (8)
C2	0.460 (1)	0.273 (2)	0.574 (1)	0.122 (11)
C3	0.408 (1)	0.362 (2)	0.352 (1)	0.094 (9)
C4	0.479 (1)	0.374 (3)	0.325 (2)	0.143 (14)
C5	0.262 (1)	-0.298 (1)	0.154 (1)	0.064 (6)
C6	0.343 (1)	-0.316 (2)	0.219 (1)	0.099 (9)
C7	0.1645 (8)	-0.190 (1)	-0.008 (1)	0.067 (6)
C8	0.153 (1)	-0.135 (2)	-0.100 (1)	0.088 (8)
C9	0.1111 (8)	0.329 (1)	0.5056 (9)	0.055 (5)
C10	0.0312 (9)	0.325 (2)	0.438 (1)	0.096 (8)
C11	0.0665 (8)	0.175 (2)	0.641 (1)	0.059 (6)
C12	0.081 (1)	0.061 (2)	0.707 (1)	0.111 (10)

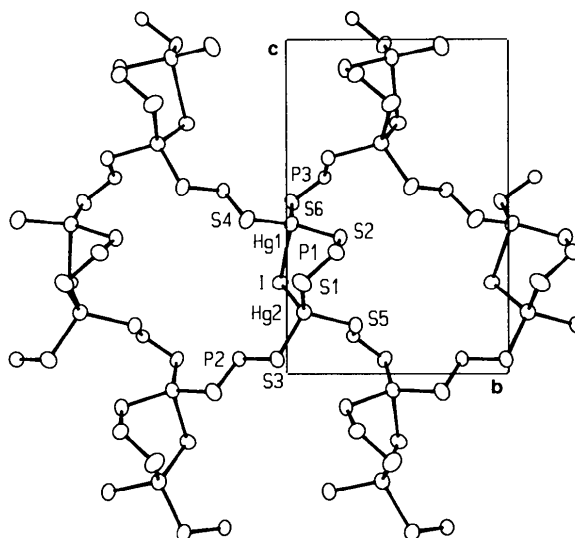


Fig. 1. The two-dimensional network with the numbering scheme. C and H atoms are omitted for clarity.

Table 2. Bond distances (\AA) and angles ($^\circ$) with e.s.d.'s in parentheses

Hg1—I	3.233 (1)	Hg2—I	2.792 (1)
Hg1—S2	2.446 (4)	Hg2—S1	2.548 (4)
Hg1—S4	2.448 (4)	Hg2—S3	2.569 (4)
Hg1—S6	2.536 (4)	Hg2—S5	2.552 (3)
P1—S1	2.014 (6)	P2—S3	2.008 (5)
P1—S2	2.009 (5)	P2—S4'	2.046 (6)
P1—C1	1.82 (2)	P2—C5	1.82 (1)
P1—C3	1.82 (2)	P2—C7	1.81 (2)
P3—S5	2.026 (5) ⁱⁱ	C3—C4	1.44 (4)
P3—S6	2.023 (5)	C5—C6	1.55 (3)
P3—C9	1.81 (1)	C7—C8	1.52 (2)
P3—C11	1.80 (1)	C9—C10	1.55 (2)
C1—C2	1.51 (3)	C11—C12	1.54 (3)
I—Hg1—S2	92.88 (9)	I—Hg2—S1	111.92 (9)
I—Hg1—S4	100.20 (9)	I—Hg2—S3	116.77 (9)
I—Hg1—S6	88.66 (9)	I—Hg2—S5	110.19 (8)
S2—Hg1—S4	129.1 (1)	S1—Hg2—S3	98.1 (1)
S2—Hg1—S6	110.6 (1)	S1—Hg2—S5	110.3 (1)
S4—Hg1—S6	118.6 (1)	S3—Hg2—S5	108.9 (1)
Hg1—I—Hg2	87.31 (4)	P1—C1—C2	116 (1)
S1—P1—S2	116.3 (2)	P1—C3—C4	118 (2)
S3—P2—S4'	107.4 (2)	P2—C5—C6	114 (1)
S5 ⁱⁱ —P3—S6	111.2 (2)	P2—C7—C8	115 (1)
C1—P1—C3	111.4 (9)	P3—C9—C10	113 (1)
C5—P2—C7	106.2 (8)	P3—C11—C12	114 (1)
C9—P3—C11	107.4 (7)		

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

ordered normal deviates $\sigma(|F_o|) = w^{-1/2}$. The result was a slope of 1.223 (3), intercept of 0.287 (3) and a correlation coefficient of 0.9882. The non-zero intercept results from the non-linear appearance of the curve which indicates a systematic error, probably in the intensity measurements. Atomic scattering factors from *International Tables for X-ray Crystallog-*

raphy (1974). The system of computer programs used is described by Lundgren (1982). Fractional atomic coordinates and equivalent isotropic parameters are given in Table 1, selected bond distances and angles in Table 2.* Fig. 1 shows the two-dimensional network and the numbering scheme.

Related literature. Crystal structures of non-transition dithiophosphinates and dithiophosphates reveal many novel and interesting coordination features (Mehrotra, Srivastava & Chauhan, 1984). The only Hg complexes with this type of ligand known to date are $\text{Hg}[\text{S}_2\text{P}(i\text{-C}_3\text{H}_7\text{O})_2]_2$ (Lawton, 1971) and $\text{Hg}[\text{S}_2\text{P}(\text{C}_2\text{H}_5\text{O})_2]_2$ (Watanabe, 1980). In both these structures the complexes form infinite chains. In the investigated complex infinite two-dimensional networks are formed.

* Lists of H-atom coordinates, anisotropic thermal parameters and structure factors have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52543 (47 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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