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Bis(diethylenetriamine)mercury(II) bis(thiocyanate)

M. Linh Tran, Paul V. Bernhardt* and Ian R. Gentle

Department of Chemistry, The University of Queensland, Brisbane, Queensland 4072, Australia

Correspondence e-mail:
bernhardt@chemistry.uq.edu.au

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Key indicators

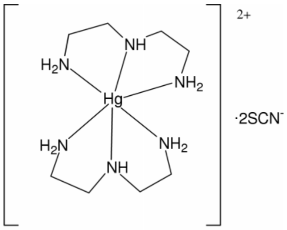
Single-crystal X-ray study
T = 130 K
Mean σ(C–C) = 0.006 Å
R factor = 0.019
wR factor = 0.049
Data-to-parameter ratio = 16.3

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Mercury(II) in the title compound, $[Hg(C_4H_{13}N_3)_2](SCN)_2$, is six-coordinated with two diethylenetriamine (dien) ligands in a *sym*-facial configuration. The complex cation has a twofold axis of symmetry, and the secondary amine groups are in *trans* positions.

Comment

In the search for model compounds in which all the coordination sites of the mercury(II) cation are occupied by N-donor groups from linear polyamines, the title compound, (I), was chosen for crystallographic study. One advantage of using diethylenetriamine, (dien), as a ligand is the ease of synthesis of the complex, which has been reported previously (Cova *et al.*, 1972). Although the crystal structures of many bis-diethylenetriamine–metal complexes have been determined, none has been reported that contains mercury.



(I)

The compound is obtained by reacting dien with mercury(II) thiocyanate in a 2:1 ligand-to-metal ratio in ethanol. The C_2-symmetric complex cation exhibits a distorted trigonal–prismatic geometry. The two dien ligands are coordinated in a *sym*-facial configuration isomer with a twofold axis including the Hg atom bisecting the N1—Hg—N1^{i} angle [symmetry code: (i) $1-x, y, 1/2-z$]. The SCN^- anions are not coordinated. Other metal ions, such as Ni^{II} (Mukherjee *et al.*, 1994), Co^{III} (Kobayashi *et al.*, 1972), and Ir^{III} (Harada, 1993), also form *sym*-facial bis-dien complexes. In the case of Ni^{II} and Co^{III}, the central nitrogen–metal bond is shortened compared with the terminal *M*—N bonds, whereas all Ir—N bonds are similar in length. Unlike these cases, the unique Hg—N bond of the central N atom is slightly longer than the terminal coordinate bonds. Table 1 shows the bond distances and angles of the mercury complex. A survey of the Cambridge Structural Database (Allen & Kennard, 1993) indicates that the bond lengths are similar to the only other published mercury hexaamine structure, namely (1,4,7,10,13,16-hexaazacyclooctadecane)mercury(II) tetrachloromercury(II) (Carrondo *et al.*, 1993).

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Experimental

The title compound was prepared by adding a suspension of mercury(II) thiocyanate dropwise, with stirring, to two equivalents of diethylenetriamine. The colourless crystals used for analysis were obtained by slow evaporation of the ethanol solution. During the acquisition of X-ray data at room temperature, it was observed that the crystal decomposed from a colourless crystalline compound to a dark-brown substance. The identity of this decomposition product is unknown. For this reason, data were collected at the low temperature of 130 K; this obviated the problem.

Crystal data

$[Hg(C_4H_{13}N_3)_2](SCN)_2$
$M_r = 523.1$
Monoclinic, $C2/c$
$a = 7.6710\ (7)$ Å
$b = 13.9712\ (6)$ Å
$c = 16.639\ (2)$ Å
$\beta = 93.809\ (9)^\circ$
$V = 1779.3\ (3)$ Å^3
$Z = 4$
$D_x = 1.953$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 25 reflections
$\theta = 11.3$–14.3°
$\mu = 8.89$ mm^{-1}
$T = 130\ (2)$ K
Prism, colourless
$0.50 \times 0.20 \times 0.07$ mm

Data collection

Enraf–Nonius TurboCAD-4 diffractometer
Non-profiled ω scans
Absorption correction: ψ scan (North *et al.*, 1968)
$T_{min} = 0.089$, $T_{max} = 0.537$
1690 measured reflections
1565 independent reflections
1485 reflections with $I > 2\sigma(I)$
$R_{int} = 0.023$
$\theta_{max} = 25.0^\circ$
$h = 0 \rightarrow 9$
$k = 0 \rightarrow 16$
$l = -19 \rightarrow 19$
3 standard reflections
frequency: 120 min
intensity decay: 9%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.019$
$wR(F^2) = 0.049$
$S = 1.07$
1565 reflections
96 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0341P)^2 + 3.0573P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0$
$\Delta\rho_{max} = 0.95$ e Å^{-3}
$\Delta\rho_{min} = -1.42$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

N1—Hg1	2.396 (3)	N3—Hg1	2.373 (3)
N2—Hg1	2.508 (3)		
N3—Hg1—N3^i	130.19 (17)	N1—Hg1—N2^i	145.46 (11)
N3—Hg1—N1	120.47 (11)	N3—Hg1—N2	72.74 (11)
N3^i—Hg1—N1	96.18 (11)	N1—Hg1—N2	72.85 (11)
N1—Hg1—N1^i	86.24 (15)	N2^i—Hg1—N2	137.52 (15)
N3—Hg1—N2^i	89.52 (11)		

Symmetry code: (i) $1 - x, y, \frac{1}{2} - z$.

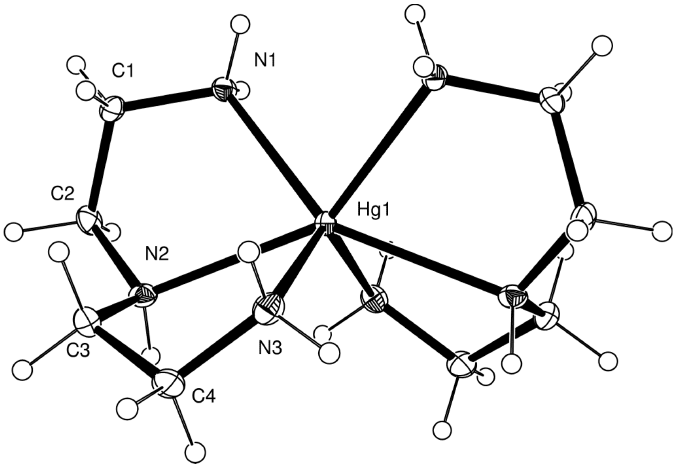

Figure 1
ORTEP-3 plot (Farrugia, 1997) of the title complex cation (30% probability ellipsoids).

Data collection: *CAD*-4 *EXPRESS* (Enraf–Nonius, 1994); cell refinement: *CAD*-4 *EXPRESS*; data reduction: *XCAD*4 (Harms & Wocadlo, 1995); program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1990); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEP*-3 for Windows (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

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