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The Structure of the Monomeric Form of Mercury(II) *N*,*N*-Diisopropyldithiocarbamate [Bis(*N*,*N*-diisopropyldithiocarbamato)mercury(II)]

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Abstract. $Hg(C_7H_{14}NS_2)_2$, $C_{14}H_{28}HgN_2S_4$, α form, monoclinic, $C2/c$, $a = 19{\cdot}468$ (1), $b = 8{\cdot}017$ (1), $c = 14{\cdot}832$ (1) Å, $\beta = 119{\cdot}18$ (3)°, $U = 2021{\cdot}3$ Å^3, $D_x = 1{\cdot}818$ Mg m^{-3}, μ(Mo $K\alpha$) = 9·18 mm^{-1}, $Z = 4$. The structure was refined by the block-diagonal least-squares method to $R = 0{\cdot}064$. The molecules exist as monomers with 2 (C_2) symmetry. The two independent Hg—S bond lengths are 2·445 (4) and 2·645 (4) Å.

Introduction. The crystal structure analysis of mercury(II) *N*,*N*-diisopropyldithiocarbamate was undertaken as part of a series of studies on metal complexes with sulfur-containing ligands. The compound was prepared by adding mercury(II) dichloride to an aqueous solution of sodium *N*,*N*-diisopropyldithiocarbamate. Recrystallization from acetone solution gave two types of crystals (the α and β forms), both being plate-like and yellowish. A preliminary report on the structure of the β form has already been published (Iwasaki, Ito & Kobayashi, 1978). The present paper is concerned with the α form, α-$Hg[S_2CN(i\text{-}Pr)_2]_2$ (*i*-Pr = iso-C_3H_7).

A crystal of approximate dimensions 0·20 × 0·18 × 0·07 mm was mounted on a Rigaku automated four-circle diffractometer. Intensities of the reflections up to $2\theta \leq 55°$ were measured with Mo $K\alpha$ radiation monochromatized by a graphite plate. The measurement was made in the ω–2θ scan mode with a scanning speed of 4° min^{-1} in 2θ, and 1360 independent reflections with $|F_o|$ greater than 3·0 times the standard deviation were obtained. The intensities were corrected for Lorentz, polarization and absorption effects.

The structure was solved by the heavy-atom method, and refined by the block-diagonal least-squares method with the weighting scheme $w = 0{\cdot}2$ for $|F_o| \leq 60$, 1·0 for $60 \leq |F_o| \leq 170$ and $8000/|F_o|^2$ for $|F_o| \geq 170$. The atomic scattering factors were taken from *International Tables for X-ray Crystallography* (1974). The

Table 1. *Atomic parameters with estimated standard deviations in parentheses* (× 10^4)

The B_{eq} values are the equivalent isotropic temperature factors (Å^2) (Hamilton, 1959).

	x	*y*	*z*	B_{eq}
Hg	5000 (0)	7441 (2)	2500 (0)	4·7
S(1)	4728 (2)	8238 (4)	3887 (2)	3·6
S(2)	3777 (2)	5728 (4)	2317 (2)	3·6
C(1)	3959 (6)	6782 (13)	3419 (8)	2·5
N	3566 (5)	6530 (13)	3916 (7)	2·8
C(2)	3684 (6)	7443 (20)	4856 (7)	3·4
C(3)	2913 (6)	5240 (14)	3516 (9)	2·9
C(4)	3501 (7)	9301 (16)	4682 (10)	4·0
C(5)	4505 (7)	7079 (17)	5808 (9)	4·7
C(6)	2117 (7)	6090 (19)	3166 (11)	4·7
C(7)	3101 (8)	3856 (17)	4340 (11)	5·1

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final R value was 0·064 excluding the H atoms. The atomic coordinates are given in Table 1.*

Discussion. Crystals of the β form of the title compound were found to consist of both monomeric and dimeric molecules (Iwasaki *et al.*, 1978). However, molecules in crystals of the α form are monomeric and composed of one Hg atom and two chelating carbamate groups. The bond distances and angles are listed in Table 2. A perspective view of the molecule is shown in Fig. 1. The molecule has 2 (C_2) symmetry. The Hg atom is coordinated by four S atoms with two Hg–S bond distances of 2·445 (4) Å and two of 2·645 (4) Å; the configuration around the Hg atom is a strongly distorted tetrahedron. All bond lengths in the carbamate ligand are normal for dithioacid complexes. The molecular geometry is identical with that of the monomeric molecule of the β-form crystal except for a slight change in the orientation of the terminal C–CH_3 bonds. The molecules are isolated and held together only by van der Waals forces.

The molecular structure of the monomeric form of the ethyl analogue, β-$Hg(S_2CNEt_2)_2$ (Iwasaki, 1973), is quite different. The molecule has $\bar{1}$ (C_i) symmetry and the Hg and four S atoms are coplanar. Moreover, relatively short intermolecular Hg–S distances are found between the nearest neighbors, resulting in the formation of a chain-like structure with sixfold coordination of the metal atom. In Lawton's (1971) classification of Hg–S complexes the title compound, α-$Hg[S_2CN(i\text{-}Pr)_2]_2$, belongs to the type *B* group of complexes, in contrast to the ethyl analogue which belongs to type *A*.

* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 34354 (12 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 2. *Interatomic distances* (Å) *and bond angles* (°)

Hg–S(1)	2·445 (4)	S(1)–Hg–S(2)	70·7 (2)
Hg–S(2)	2·645 (4)	S(1)–Hg–S(1′)	149·7 (2)
S(1)–C(1)	1·753 (12)	S(1)–Hg–S(2′)	127·0 (2)
S(2)–C(1)	1·717 (12)	S(2)–Hg–S(2′)	117·5 (2)
C(1)–N	1·311 (15)	Hg–S(1)–C(1)	89·3 (5)
N–C(2)	1·49 (2)	Hg–S(2)–C(1)	83·6 (5)
N–C(3)	1·52 (2)	S(1)–C(1)–S(2)	116·3 (7)
C(2)–C(4)	1·52 (3)	S(1)–C(1)–N	120·1 (9)
C(2)–C(5)	1·56 (3)	S(2)–C(1)–N	123·6 (9)
C(3)–C(6)	1·53 (3)	C(1)–N–C(2)	125·7 (11)
C(3)–C(7)	1·56 (3)	C(1)–N–C(3)	119·5 (10)
		C(2)–N–C(3)	114·8 (10)
		N–C(2)–C(4)	113·9 (13)
		N–C(2)–C(5)	112·2 (12)
		C(4)–C(2)–C(5)	112·8 (13)
		N–C(3)–C(6)	110·2 (11)
		N–C(3)–C(7)	110·3 (11)
		C(6)–C(3)–C(7)	113·2 (12)

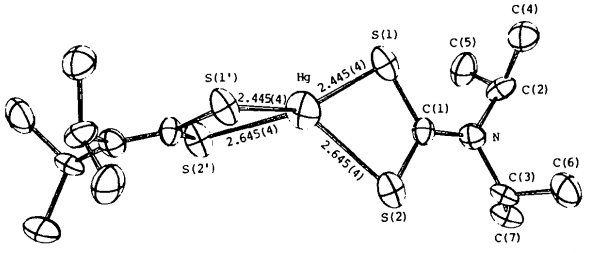

Fig. 1. A perspective view of the molecule. The thermal ellipsoids are at the 50% probability level.

The calculations were performed on a Facom 230-75 computer of this Institute, using the *UNICS* II program system (Sakurai, Iwasaki, Watanabe, Kobayashi, Bando & Nakamichi, 1974). Part of this work was supported by a Grant-in-Aid for Scientific Research No. 354161 from the Ministry of Education, Science and Culture.

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