

The Structure of *catena*-{Di- μ -chloro-tetrakis[μ -(*N,N*-diethyldithiocarbamato-*S,S'*)]-trimercury(II)}, [Hg₃(C₅H₁₀NS₂)₄Cl₂]

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Abstract

The title compound is one of two substances obtained in a reaction between Hg₂Cl₂ and sodium dithiocarbamate (dtc), Na(C₅H₁₀NS₂). The crystal is triclinic, *P* $\bar{1}$, with $a = 18.53$ (1), $b = 10.174$ (6), $c = 11.016$ (6) Å, $\alpha = 112.82$ (4), $\beta = 97.17$ (5), $\gamma = 91.15$ (6)° and $Z = 2$. The structure was determined from 4706 independent reflections by the heavy-atom method and refined by full-matrix least squares to an R of 0.078, including reflections with zero counts. The structure consists of polymeric chains of which the repeat unit has three mercuric ions linked by two Cl[−] and four dtc unsymmetrically bridged ligands. Two of the three linkages are *via* both a Cl[−] and a dtc on each whereas the third is *via* two dtc groups. The chains pack together with van der Waals forces and they run parallel to the c axis. The eight Hg–S distances in the asymmetric unit range from 2.406 (8) to 2.587 (6) Å. The HgSC angles range from 94.1 to 107.1 (6)°, whereas the Hg–Cl distances of the bridged Cl[−] ions are within the range 2.661 (8)–2.771 (7) Å.

Introduction

The crystal data of the title compound were first reported by Iwasaki (1972) in a communication: however, the atomic coordinates were not given, nor was the detailed structure of this compound available in his subsequent paper that described the crystal structures of the α and β forms of mercury(II) *N,N*-diethyldithiocarbamate, [Hg(dtc)₂]_{*n*} ($n = 2$ for α and 1 for β), (Iwasaki, 1973). Since these crystals repeatedly appeared in many reactions between mercurials and sodium *N,N*-diethyldithiocarbamate, Na(dtc), or tetraethylthiuram disulfide (tetd) (Chieh & Leung, 1976), we have determined the crystal structure of Hg₃(dtc)₄Cl₂. Since the stoichiometry of this compound is different from those of the α and β forms of mercury(II) *N,N*-diethyldithiocarbamate, we do not feel justified to call it the γ form as did Iwasaki.

Experimental

The title compound was prepared from Hg₂Cl₂ and Na(dtc). When Hg₂Cl₂ crystals were added to an ethanolic solution of Na(dtc), a black precipitate formed immediately, indicating disproportionation of mercurous chloride. The filtrate from this reaction gave a mixture of α -[Hg(dtc)₂]₂ and Hg₃(dtc)₄Cl₂ crystals, when the solvent had evaporated at room temperature. The former are deep-yellow rhomboids and the latter are pale-yellow needles elongated along the c axis.

X-ray photographic methods were used to identify the products in the following reactions. In aqueous solution, HgCl₂ reacted with tetd to give Hg₃(dtc)₄Cl₂ crystals along with both the α and β forms of mercury(II) *N,N*-diethyldithiocarbamate, after recrystallization from acetone. All three forms were also present when acetonitrile was used as the solvent for the reaction. When ethanol was used as the solvent, α -[Hg(dtc)₂]₂ and Hg₃(dtc)₄Cl₂ were the products. With *p*-dioxane as the solvent, β -Hg(dtc)₂ and Hg₃(dtc)₄Cl₂ were obtained. The chloro analogue of Hg(tetd)X₂ ($X = \text{Br, I}$) was never observed as a product in these reactions (Chieh, 1978*b*).

The crystal used for intensity measurements was cut from a needle; its dimensions were 0.13, 0.14 and 0.17 mm along the a , b , and c axes respectively. It was mounted on a Syntex P2₁ diffractometer with c approximately along the ϕ axis; however, programs on the machine took a crystal with any orientation. Cell constants along with the orientation matrix were refined by least squares from 15 reflections automatically centered.

Crystal data

C₂₀H₄₀Cl₂Hg₃N₄S₈, FW = 1265.7, triclinic, $a = 18.53$ (1), $b = 10.174$ (6), $c = 11.016$ (6) Å, $\alpha = 112.82$ (4), $\beta = 97.17$ (5), $\gamma = 91.15$ (6)°, $V = 1894$ (2) Å³, $D_o = 2.22$ (by flotation), $Z = 2$, $D_c = 2.219$ Mg m^{−3}, $\mu(\text{Mo } K\alpha) = 12.96$ mm^{−1}, $\mu R = 0.91$, space group *P* $\bar{1}$, $\lambda(\text{Mo } K\alpha) = 0.71069$ Å.

Intensities of 4706 independent reflections with $2\theta < 40^\circ$ were measured using the θ - 2θ scan method at 298 K; graphite-monochromated Mo $K\alpha$ radiation and a scintillation counter equipped with pulse-height analyzer were used. Variable scan speeds in the range 2 – $30^\circ \text{ min}^{-1}$ were used depending on the peak intensities. The time for background measurement was half that used in the scan and equal times were spent at both extremes of the scan range (1° below Mo $K\alpha_1$ and 1° above Mo $K\alpha_2$). Two reflections ($2\bar{2}3$ and 210) were repeatedly measured after every 48 reflections to monitor the stability of the data collection and their intensities fluctuated within 3%. Scale factors based on this fluctuation were used to equalize reflections collected at times after the checked reflections. Reflections with zero or negative net counts were given zero intensities. Of those measured, 569 reflections (12%) had intensities less than $\sigma(I)$. All reflections were included in the least-squares refinement. Lorentz and

polarization factors were applied to derive the structure amplitudes.

Due to overlapping, only six intense peaks were on the Patterson map; however, a solution giving coordinates for the three Hg atoms was obtained. Positions of other non-hydrogen atoms were derived by the heavy-atom method. Atomic scattering curves from *International Tables for X-ray Crystallography* (1962) with an anomalous-dispersion correction for Hg were used. The function minimized was $\sum w(|F_o| - |F_c|)^2$ where $w = (40 - |F_o| + 0.009 F_o^2)^{-1/2}$ in the final stage of refinement using a modified version of *ORFLS* (Busing, Martin & Levy, 1962). This weighting function, which had highest confidence for reflections with $F_o = 55$, gave a small variation of $\sum w(\Delta F)^2/n$ in the ten ranges based on F_o . The shifts of parameters were less than 0.3 of their standard deviations in the last cycle of least squares. A difference Fourier map at the end of refinement showed only ripples (max. $1.9 \text{ e } \text{Å}^{-3}$) around the Hg positions and it did not give us confidence for locating the H atoms. The final R and R_w were 0.069 and 0.087 respectively when reflections with $I < \sigma(I)$ were omitted. If they were included, the R and R_w became 0.078 and 0.092 respectively. Final atomic parameters are given in Table 1.*

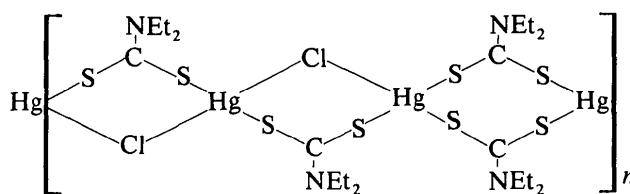
Table 1. Atomic parameters for $\text{Hg}_3(\text{S}_2\text{CNET}_2)_4\text{Cl}_2$

	x	y	z	U^*
Hg(1)	0.27831 (5)	0.0125 (1)	0.36395 (9)	20 (7)
Hg(2)	0.22751 (5)	-0.0322 (1)	0.67548 (9)	21 (5)
Hg(3)	0.24803 (5)	-0.0694 (1)	0.99711 (8)	21 (5)
Cl(1)	0.1537 (4)	0.0041 (9)	0.4689 (7)	23 (9)
Cl(2)	0.2518 (4)	-0.2366 (7)	0.1360 (6)	25 (12)
S(11)	0.3669 (3)	-0.1213 (7)	0.4395 (6)	21 (3)
S(12)	0.3396 (3)	0.1187 (6)	0.6919 (6)	20 (4)
N(1)	0.454 (1)	-0.039 (2)	0.669 (2)	18 (6)
C(11)	0.392 (1)	-0.011 (2)	0.610 (2)	18 (7)
C(12)	0.474 (1)	0.032 (3)	0.816 (2)	19 (8)
C(13)	0.523 (1)	0.169 (3)	0.858 (3)	25 (6)
C(14)	0.504 (1)	-0.144 (3)	0.596 (2)	20 (8)
C(15)	0.485 (2)	-0.296 (2)	0.587 (3)	22 (13)
S(21)	0.1921 (4)	-0.2889 (7)	0.6072 (6)	23 (7)
S(22)	0.3260 (3)	-0.1984 (6)	0.8123 (6)	21 (3)
N(2)	0.255 (1)	-0.458 (2)	0.717 (2)	21 (11)
C(21)	0.257 (1)	-0.326 (3)	0.715 (2)	16 (11)
C(22)	0.313 (2)	-0.494 (3)	0.802 (3)	24 (14)
C(23)	0.376 (2)	-0.541 (4)	0.724 (5)	32 (14)
C(24)	0.193 (2)	-0.565 (3)	0.646 (3)	25 (4)
C(25)	0.133 (2)	-0.548 (4)	0.733 (3)	33 (10)
S(31)	0.1720 (3)	0.1377 (6)	0.8758 (6)	20 (3)
S(32)	0.1163 (3)	-0.1108 (7)	0.9327 (6)	22 (5)
N(3)	0.037 (1)	0.015 (2)	0.796 (2)	21 (6)
C(31)	0.101 (1)	0.010 (2)	0.861 (2)	19 (6)
C(32)	0.020 (1)	0.114 (4)	0.732 (3)	25 (15)
C(33)	-0.009 (2)	0.248 (4)	0.838 (4)	31 (11)
C(34)	-0.024 (1)	-0.096 (3)	0.777 (3)	23 (7)
C(35)	-0.021 (2)	-0.230 (3)	0.653 (3)	27 (4)
S(41)	0.3459 (3)	0.1194 (6)	0.1360 (5)	19 (4)
S(42)	0.2371 (4)	0.2205 (7)	0.3308 (7)	24 (9)
N(4)	0.287 (1)	0.367 (2)	0.202 (2)	18 (11)
C(41)	0.288 (1)	0.246 (2)	0.217 (2)	18 (5)
C(42)	0.320 (2)	0.393 (3)	0.099 (2)	24 (9)
C(43)	0.399 (1)	0.466 (3)	0.159 (3)	26 (10)
C(44)	0.244 (2)	0.484 (3)	0.283 (2)	26 (12)
C(45)	0.169 (1)	0.479 (3)	0.217 (3)	25 (9)

* U is the average of r.m.s. displacements (10^{-2} Å) and values in parentheses are standard deviations of the average, $\{(\sum U_i^2 - (\sum U_i)^2/3)/2\}^{1/2}$; where $i = 1, 2, 3$. They indicate the anisotropy of the thermal parameters.

Results and discussion

The crystal consists of polymeric chains



running in the c direction. A repeat unit in the chain is also a crystallographic asymmetric unit. There are no abnormal thermal parameters and an *ORTEP* (Johnson, 1965) drawing with atomic numbering of this unit is shown in Fig. 1; bond lengths and angles are given in Table 2. All three mercuric ions are strongly bonded to four donor atoms and their degrees of distortion from tetrahedral coordination vary. The largest SHgS angles are $154.3 (2)$, $137.2 (2)$ and $142.4 (2)^\circ$ centered on Hg(1), Hg(2) and Hg(3) respectively. The corresponding smallest ClHgS angles are $84.2 (2)$, $96.1 (2)$ and $93.2 (2)^\circ$. Both Hg(2) and Hg(3) ions are bonded to three S atoms of three dithiocarbamate ligands and one Cl^- ion, whereas Hg(1) is bound to two Cl^- and two S

* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 34820 (24 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

atoms. The environments of Hg(2) and Hg(3) are similar as seen from the bond lengths and angles (Table 2) around them. The distortion from tetrahedral geometry is so great for Hg(1) that the coordination can be viewed as a linear S—Hg—S moiety bent by the approach of two Cl⁻ ions. In contrast, the Hg^{II} ion in a similar polymeric bromide complex bonded to two Br⁻ and two S atoms of two different dithiocarbamates has a value of 106.6 (3)° for the SHgS angle; however, the Br⁻ ions are not bridging (Chieh, 1978*a*).

The eight Hg—S distances in the asymmetric unit range from 2.406 (8) to 2.587 (6) Å. The shortest distances are the two bonds which deviate least from linearity, namely S(11)—Hg(1) and S(42)—Hg(1), whereas other distances are typical for Hg—S bonds in an approximate tetrahedral configuration. Similar trends were also observed for other mercury complexes with sulfur ligands (Lawton, 1971).

The differences between the two Hg—S distances involving S of the same dithiocarbamate are 0.081 (9σ), 0.117 (12σ), 0.132 (16σ) and 0.085 Å (9σ) for the four dithiocarbamates containing C(11), C(21), C(31) and C(41) respectively; however, differences between C—S distances are all less than 3σ. The mean C—S value is 1.74 (3) Å. The SCS angles have expected values for *sp*² C atoms. Bond lengths and angles for the ligands are normal for bridging dithiocarbamate where both S atoms are coordinated to metal ions (Domenicano, Torelli, Vaciago & Zambonelli, 1968; Chieh, 1978*b*).

The mean S₂C—N distance of 1.33 (2) Å is typical for dithiocarbamates or teta (Karle, Estin & Britts, 1967). The short C—N bond is due to the contribution of a canonical form $\begin{matrix} \text{—S} \\ \text{—S} \end{matrix} \text{C}=\text{N} \begin{matrix} \text{Et} \\ \text{Et} \end{matrix}$. The two methyl groups are distributed one on each side of the plane defined by the other non-hydrogen atoms of the ligand and this is the configuration commonly found in structures of their complexes. The equations of least-squares

planes of the four independent dithiocarbamates are given in Table 3. The displacements of mercuric ions from the planes are rather uniform; for one they are in the range 0.2–0.7 Å and for the other 2.2–2.6

Table 2. *Interatomic distances (Å) and bond angles (°) for Hg₃(S₂CNEt₂)₄Cl₂*

(a) Hg—ligand bond lengths

Hg(1)—Cl(1)	2.722 (8)	Hg(3)—Cl(2)	2.690 (7)
Hg(1)—Cl(2)	2.771 (7)	Hg(3)—S(22)	2.587 (6)
Hg(1)—S(11)	2.422 (7)	Hg(3)—S(32)	2.436 (6)
Hg(1)—S(42)	2.406 (8)	Hg(3)—S(41)	2.491 (6)
Hg(2)—Cl(1)	2.661 (8)		
Hg(2)—S(12)	2.503 (6)	Weak interactions	
Hg(2)—S(21)	2.470 (8)	Hg(2)···S(22)	3.143 (6)
Hg(2)—S(31)	2.568 (6)	Hg(3)···S(31)	3.169 (6)

(b) Bond lengths of ligands

C(11)—S(11)	1.77 (2)	C(31)—S(31)	1.78 (2)
C(11)—S(12)	1.69 (2)	C(31)—S(32)	1.71 (2)
C(11)—N(1)	1.34 (3)	C(31)—N(3)	1.32 (3)
N(1)—C(12)	1.49 (3)	N(3)—C(32)	1.46 (5)
N(1)—C(14)	1.49 (3)	N(3)—C(34)	1.52 (3)
C(12)—C(13)	1.52 (4)	C(32)—C(33)	1.57 (5)
C(14)—C(15)	1.54 (4)	C(34)—C(35)	1.52 (4)
C(21)—S(21)	1.73 (2)	C(41)—S(41)	1.73 (2)
C(21)—S(22)	1.73 (2)	C(41)—S(42)	1.75 (2)
C(21)—N(2)	1.35 (4)	C(41)—N(4)	1.30 (3)
N(2)—C(22)	1.48 (4)	N(4)—C(42)	1.46 (3)
N(2)—C(24)	1.48 (4)	N(4)—C(44)	1.50 (4)
C(22)—C(23)	1.51 (5)	C(42)—C(43)	1.57 (4)
C(24)—C(25)	1.53 (5)	C(44)—C(45)	1.48 (4)

(c) Angles around Hg atoms

Cl(1)—Hg(1)—Cl(2)	99.7 (2)	Cl(2)—Hg(1)—S(11)	84.2 (2)
Cl(1)—Hg(1)—S(11)	108.9 (2)	Cl(1)—Hg(1)—S(42)	113.4 (2)
Cl(1)—Hg(1)—S(42)	87.4 (4)	S(11)—Hg(1)—S(42)	154.3 (2)
Cl(1)—Hg(2)—S(12)	96.1 (2)	Cl(2)—Hg(3')—S(32')	93.2 (2)
Cl(1)—Hg(2)—S(21)	98.2 (2)	Cl(2)—Hg(3')—S(41)	100.6 (1)
Cl(1)—Hg(2)—S(31)	103.8 (2)	Cl(2)—Hg(3')—S(22')	102.9 (2)
S(12)—Hg(2)—S(21)	137.2 (2)	S(32')—Hg(3')—S(41)	142.4 (2)
S(12)—Hg(2)—S(31)	99.9 (1)	S(41)—Hg(3')—S(22')	94.8 (1)
S(21)—Hg(2)—S(31)	115.3 (2)	S(32)—Hg(3)—S(22)	116.0 (2)
S(22)···Hg(2)—Cl(1)	154.4 (2)	S(31')···Hg(3')—Cl(2)	153.5 (1)
S(22)···Hg(2)—S(31)	100.3 (1)	S(31)···Hg(3)—S(22)	99.2 (1)
S(22)···Hg(2)—S(21)	63.3 (1)	S(31)···Hg(3)—S(32)	63.7 (1)
S(22)···Hg(2)—S(12)	88.0 (1)	S(31')···Hg(3')—S(41)	91.9 (1)

(d) Angles within ligands

Hg(1)—S(11)—C(11)	103.1 (6)	Hg(2)—S(31)—C(31)	94.1 (6)
Hg(2)—S(12)—C(11)	99.5 (6)	Hg(3)—S(32)—C(31)	100.2 (6)
S(11)—C(11)—S(12)	121 (1)	S(31)—C(31)—S(32)	120 (1)
S(11)—C(11)—N(1)	116 (2)	S(31)—C(31)—N(3)	118 (2)
S(12)—C(11)—N(1)	123 (2)	S(32)—C(31)—N(3)	122 (2)
C(11)—N(1)—C(12)	120 (1)	C(31)—N(3)—C(32)	126 (2)
C(11)—N(1)—C(14)	124 (1)	C(31)—N(3)—C(34)	119 (2)
C(12)—N(1)—C(14)	117 (1)	C(32)—N(3)—C(34)	116 (2)
N(1)—C(12)—C(13)	112 (2)	N(3)—C(32)—C(33)	107 (2)
N(1)—C(14)—C(15)	113 (2)	N(3)—C(34)—C(35)	111 (2)
Hg(2)—S(21)—C(21)	98.9 (8)	Hg(3)···S(31)—Hg(2)	79.1 (6)
Hg(3)—S(22)—C(21)	94.4 (8)	Hg(3)···S(31)—C(31)	75.2 (6)
S(21)—C(21)—S(22)	120 (1)	Hg(3')—S(41)—C(41)	96.1 (6)
S(21)—C(21)—N(2)	119 (2)	Hg(1)—S(42)—C(41)	107.1 (6)
S(22)—C(21)—N(2)	121 (2)	S(41)—C(41)—S(42)	121 (1)
C(21)—N(2)—C(22)	120 (2)	S(41)—C(41)—N(4)	120 (2)
C(21)—N(2)—C(24)	122 (2)	S(42)—C(41)—N(4)	119 (2)
C(22)—N(2)—C(24)	118 (2)	C(41)—N(4)—C(42)	124 (2)
N(2)—C(22)—C(23)	108 (2)	C(41)—N(4)—C(44)	120 (2)
N(2)—C(24)—C(25)	111 (2)	C(42)—N(4)—C(44)	116 (2)
Hg(2)···S(22)—Hg(3)	79.3 (1)	N(4)—C(42)—C(43)	109 (3)
Hg(2)···S(22)—C(21)	77.1 (8)	N(4)—C(44)—C(45)	113 (2)

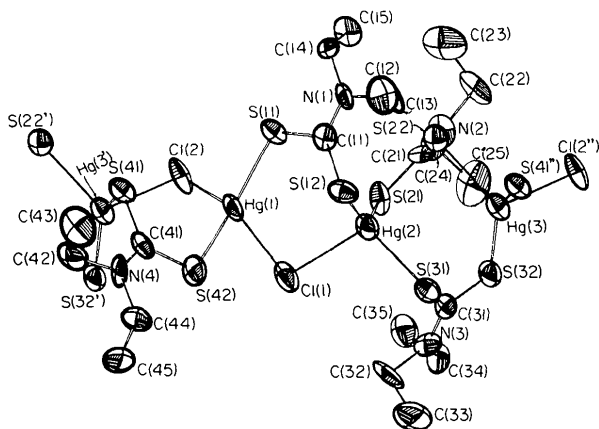


Fig. 1. An ORTEP drawing with atomic numbering.

Table 3. Equations of least-squares planes $\begin{matrix} S \\ \diagup \\ C-N \\ \diagdown \\ S \end{matrix}$ in the form $lX + mY + nZ = d$, where X , Y and Z are orthogonal axes in Å

Plane	l	m	n	d (Å)	Displacements from plane (Å)		
					Max.	Hg	C(CH ₃)
>C-N(1)<	0.5269	0.8281	-0.1912	-0.06	0.10	0.63, -2.17	1.25, -1.30
>C-N(2)<	-0.6586	0.0494	0.7508	2.53	0.07	0.24, 2.61	1.53, -1.50
>C-N(3)<	-0.4262	0.3282	0.8430	5.82	0.02	-0.40, -2.57	1.50, -1.45
>C-N(4)<	0.6855	0.1412	0.7142	5.23	0.09	0.40, -2.18	1.33, -1.24

Å. The shorter of the two Hg-S distances of each Hg-dtc-Hg link is always between an S and the Hg atom that deviates least from the >C-N< plane. The HgSC angles containing one of these bonds lie between 98.9 (6) and 107.1 (6)° with -Hg bent toward the other S of the same ligand. The S(12)-Hg(2), S(22)-Hg(3), S(31)-Hg(2) and S(41)-Hg(3) bonds tilt out of the planes with angles of 64 (1),

81 (1), 84 (1) and 66 (1)° respectively; HgSC angles containing one of these bonds lie in the range 94.1-99.5°.

Viewed from the c axis, the four dithiocarbamate groups of the asymmetric unit spread evenly in four directions around the chain. A packing diagram is shown in Fig. 2. The chains are held together by van der Waals forces. Selective shortest distances between interchain atoms are given in Table 4. Aside from the expected van der Waals distances of the types CH₂...CH₂, CH₂...CH₃ and CH₃...CH₃, for these groups are on the exterior of the chains, there are also N...Cl, S...CH₂, C(sp²)...CH₂ etc. short contacts indicating that groups from neighboring chains interlock tightly. The closest approach of a group from another chain to the Hg is 4.14 (2) Å between Hg(1) and C(14).

Table 4. Short distances between interchain atoms

Type	Distance (Å)	Between atoms at positions*
CH ₃ ...CH ₃	3.87 (6)	C(25)...C(33) (i)
CH ₂ ...CH ₃	3.99 (6)	C(32)...C(25) (ii)
CH ₂ ...CH ₂	3.85 (4)	C(22)...C(42) (iii)
Cl...N	4.24 (2)	Cl(1)...N(3) (iv)
Cl...CH ₂	3.56 (2)	Cl(1)...C(32) (iv)
Cl...CH ₃	3.70 (3)	Cl(2)...C(45) (i)
Hg...CH ₂	4.14 (2)	Hg(1)...C(14) (v)
S...N	4.17 (2)	S(11)...N(1) (v)
S...CH ₂	3.52 (3)	S(42)...C(24) (ii)
S...CH ₃	3.39 (5)	S(12)...C(23) (ii)
N...CH ₂	4.16 (3)	N(1)...C(14) (v)
C(sp ²)...CH ₂	3.89 (3)	C(11)...C(14) (v)
C(sp ²)...CH ₃	4.41 (4)	C(41)...C(15) (v)

* Positions refer to the second atoms with: (i) $x, -1 + y, z$; (ii) $x, 1 + y, z$; (iii) $x, -1 + y, 1 + z$; (iv) $-x, -y, 1 - z$; (v) $1 - x, -y, 1 - z$.

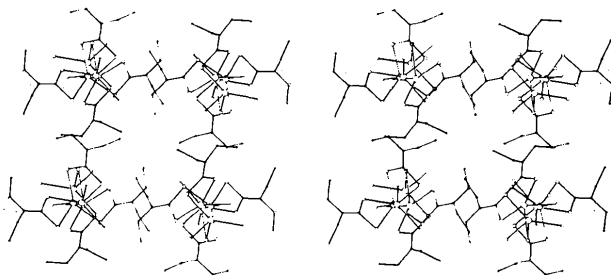


Fig. 2. Packing diagram viewed from the c axis.

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References

- BUSING, W. R., MARTIN, K. O. & LEVY, H. A. (1962). *ORFLS*. Report ORNL-TM-305. Oak Ridge National Laboratory, Tennessee.
- CHIEH, C. (1978a). *Can. J. Chem.* **56**, 564-566.
- CHIEH, C. (1978b). *Can. J. Chem.* **56**, 974-975.
- CHIEH, C. & LEUNG, L. P. C. (1976). *Can. J. Chem.* **54**, 3077-3084.
- DOMENICANO, A., TORELLI, L., VACIAGO, A. & ZAMBONELLI, L. (1968). *J. Chem. Soc. A*, pp. 1351-1361.
- International Tables for X-ray Crystallography* (1962). Vol. III, pp. 202-207. Birmingham: Kynoch Press.
- IWASAKI, H. (1972). *Chem. Lett.* pp. 1105-1106.
- IWASAKI, H. (1973). *Acta Cryst.* **B29**, 2115-2124.
- JOHNSON, C. K. (1965). *ORTEP*. Report ORNL-3794. Oak Ridge National Laboratory, Tennessee.
- KARLE, I. L., ESTIN, J. A. & BRITTS, K. (1967). *Acta Cryst.* **22**, 273-280.
- LAWTON, S. L. (1971). *Inorg. Chem.* **10**, 328-335.