Structural Studies of Group 5B–Halide–Dithio-ligand Complexes. Part 2.† Crystal Structures of 1 : 1 Adducts of Bis(*NN*-diethyldithiocarbamato)iodoantimony(III) with Chloroform and Molecular Iodine

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Crystallization of $[Sb(S_2CNEt_2)_2I]$ from chloroform gives an adduct $[Sb(S_2CNEt_2)_2I]\cdot CHCl_3$. Single-crystal *X*-ray structure determination shows the complex to be comprised of a polymeric chain of the form $-Sb(S_2CNEt_2)_2-I-Sb(S_2CNEt_2)_2-$ with bridging iodine atoms linking $Sb(S_2CNEt_2)_2$ units; the chloroform molecule is strongly hydrogen bonded to the polymer *via* the iodine atoms. Crystals are orthorhombic, space group *Pcab*, with *a* = 19.237(8), *b* = 12.534(5), *c* = 19.535(7) Å, and *Z* = 8, with the polymer chain parallel to *b*. Crystallization from methylene chloride instead with excess of iodine yields an adduct $[Sb(S_2CNEt_2)_2I]\cdot(0.5\ I_2)_x$, $x \leqslant 1$. The unit-cell dimensions are *a ca.* 22.5, *b ca.* 10.0, *c ca.* 19.2 Å, *Z* = 8, and the space group *Pbcn*. Again the structure is a polymeric unit (similar to the above) running parallel to *b* with adjacent strands linked by I_2 molecules charge-transfer bonded at the iodide units. As the molecular iodine content of the complex rises, its sites become increasingly populated, bonding the polymer strands more compactly and decreasing the cell volume.

COMPLEXES of the type $[M(S_2CNEt_2)_2X]$, M = As, Sb, or Bi; X = Cl, Br, or I, are well known;[1] the structure of $[As(S_2CNEt_2)_2Br]$ has been shown to be pseudo-square-pyramidal,[2] while $[Bi(S_2CNEt_2)_2I]$ is an infinite polymer and $[Bi(S_2CNEt_2)_2Br]$ a tetramer.[3] To date no structural data have been available on any antimony analogues, although the structure of $[Sb(S_2CNBu^n_2)_2]_2[Cd_2I_6]$ [4] has been determined.

In endeavouring to obtain crystals of $[Sb(S_2CNEt_2)_2I]$ for structure determination, $[Sb(S_2CNEt_2)_2I]$, prepared in the usual way [5] by the reaction of methylene chloride solutions of iodine and tris(*NN*-diethyldithiocarbamato)-antimony(III), was dissolved in a number of solvents and evaporated. From chloroform, large crystals were obtained which lost solvent only very slowly (in contrast to a number of other chloroform solvates of dithiocarbamate complexes) and a structure determination was undertaken, showing the complex to be $[Sb(S_2CNEt_2)_2I]\cdot CHCl_3$, (1).

On one occasion an excess of iodine was used in the methylene chloride solution preparation of $[Sb(S_2CNEt_2)_2I]$, and in contrast to the usual pale yellow powder obtained on evaporation, substantial crimson crystals were obtained; it was found that crystallization was straightforward provided a reasonable excess of iodine was present, that the complex varied in colour from a pale brown-crimson to a very intense colour, and that it did not readily lose iodine. Analyses and density determinations suggested the complex to be $[Sb(S_2CNEt_2)_2I]\cdot(0.5\ I_2)_x$ where *x* could be less than 0.5 and as high as 1. Single-crystal photographic examination showed the unit cell and space group to be largely independent of iodine population, although the quality of the intensity data fell off somewhat as the iodine population diminished. Accordingly it was decided to carry out single-crystal structure determinations on a crystal with the iodine population approaching the lower useful limit $[Sb(S_2CNEt_2)_2I]\cdot(0.5\ I_2)_{0.56}$, (2), and another at or near the upper limit, $[Sb(S_2CNEt_2)_2I]\cdot(0.5\ I_2)_{0.84}$, (3), the population of the particular crystal used being established by the refinement of the structure. We report in this paper the structure determinations of (1), (2), and (3) {Found: C, 19.9; H, 3.2; Cl, 16.4; I, 19.3; N, 4.6; S, 19.4. Calc. for (1) $[Sb(S_2CNEt_2)I]\cdot CHCl_3$: C, 19.9; H, 3.2; Cl, 16.0; I, 19.1; N, 4.2; S, 19.3%. Found: C, 17.8; H, 2.9; I, 36.0; N, 3.6; S, 19.4. Calc. for '(3)' $C_{10}H_{20}I_2N_2S_4Sb$: C, 17.9; H, 3.6; I, 37.8; N, 4.2; S, 19.1%}.

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Crystal Data.—(1). $C_{11}H_{21}Cl_3IN_2S_4Sb$, *M* = 664.6, Orthorhombic, space group *Pcab* (D_{2h}^{15}, no. 61), *a* = 19.237(8), *b* = 12.534(5), *c* = 19.535(7) Å, *U* = 4 710(3) Å^3, D_m = 1.89(1), *Z* = 8, D_c = 1.87 g cm^{-3}, *F*(000) = 2 560, μ(Mo) = 29.9 cm^{-1}. Specimen size: 0.20 × 0.30 × 0.25 mm, $2\theta_{max.}$ = 45°, *n* = 3 093, n_o = 2 160. Values of *R*, *R'*, *S* are 0.044, 0.046, 1.3 respectively.

(2). $[Sb(S_2CNEt_2)_2I]\cdot(0.5\ I_2)_{0.56} = C_{10}H_{20}I_{1.56}N_2S_4Sb$, *M* = 616.3, Orthorhombic, space group *Pbcn* (D_{2h}^{14}, no. 60), *a* = 22.49(2), *b* = 9.969(7), *c* = 19.24(1) Å, *U* = 4 313(5) Å^3, *Z* = 8, D_c = 1.90 g cm^{-3}, *F*(000) *ca.* 2 333, μ = 37.0 cm^{-1}. Specimen size: 0.15 × 0.20 × 0.15 mm, $2\theta_{max.}$ = 40°, *n* = 2 019, n_o = 1 269. Values of *R*, *R'* are 0.11, 0.13 respectively.

(3). $[Sb(S_2CNEt_2)_2I]\cdot(0.5\ I_2)_{0.84} = C_{10}H_{20}I_{1.84}N_2S_4Sb$, *M* = 651.8, Orthorhombic, space group *Pbcn* (D_{2h}^{14}, no. 60), *a* = 22.20(1), *b* = 10.038(8), *c* = 19.02(1) Å, *U* = 4 240(5) Å^3, *Z* = 8, D_c = 2.04 g cm^{-3}, *F*(000) *ca.* 2 456, μ = 40.9 cm^{-1}. Specimen size: 0.24 × 0.18 × 0.14 mm, $2\theta_{max.}$ = 45°, *n* = 2 783, n_o = 2 375. Values of *R*, *R'* are 0.037, 0.044 respectively.

Table 1 contains the non-hydrogen atom fractional cell co-ordinates for compounds (1)—(3); see Supplementary Publication No. SUP 22974 (36 pp.)‡ for structure-factor amplitudes, thermal parameters, hydrogen-atom parameters, and least-squares planes.

Abnormal Features.—As outlined above, the data of (2) were of poorer quality than those of the other two structures. Positional parameters were refined for all non-hydrogen

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† Part 1 is ref. 3 and contains general descriptive crystallographic and definitional detail.

‡ For details see Notices to Authors No. 7, *J. Chem. Soc., Dalton Trans.*, 1979, Index issue.

Table 1

Non-hydrogen atom fractional cell co-ordinates for (1), (2), and (3)

	(1)			(2)			(3)		
Atom	*x*	*y*	*z*	*x*	*y*	*z*	*x*	*y*	*z*
Sb	0.200 20(4)	0.449 10(5)	0.246 50(4)	0.288 6(1)	0.634 4(3)	0.469 3(2)	0.287 76(3)	0.626 58(7)	0.466 08(3)
I(1)	0.225 85(4)	0.189 59(6)	0.183 02(4)	0.180 4(2)	0.449 5(4)	0.399 8(2)	0.174 82(3)	0.440 41(7)	0.397 86(4)
(*a*) Ligand 'a'									
S(1)	0.208 2(2)	0.556 4(2)	0.354 4(2)	0.337 0(6)	0.735 3(15)	0.575 7(6)	0.336 0(1)	0.726 5(3)	0.573 3(1)
S(2)	0.250 3(2)	0.331 2(2)	0.347 3(2)	0.221 0(6)	0.604 4(14)	0.580 4(7)	0.218 3(1)	0.594 1(3)	0.577 4(1)
C(1)	0.245 3(5)	0.445 4(8)	0.395 0(5)	0.270(2)	0.707(4)	0.618(2)	0.270 3(4)	0.694 9(9)	0.618 4(5)
N	0.266 9(5)	0.454 2(7)	0.457 8(4)	0.264(2)	0.751(4)	0.684(2)	0.260 5(4)	0.744 1(8)	0.680 5(4)
C(2)	0.266 6(8)	0.556 5(11)	0.496 8(6)	0.313(2)	0.828(4)	0.721(2)	0.307 3(5)	0.820 8(11)	0.720 1(6)
C(3)	0.203 6(9)	0.568 5(11)	0.537 4(7)	0.344(3)	0.741(6)	0.771(3)	0.339 7(6)	0.728 3(13)	0.772 1(7)
C(4)	0.295 1(9)	0.358 5(12)	0.495 1(8)	0.207(2)	0.729(5)	0.722(2)	0.201 9(5)	0.734 6(12)	0.717 1(6)
C(5)	0.372 1(9)	0.354 2(14)	0.498 5(10)	0.171(2)	0.858(6)	0.714(3)	0.164 2(7)	0.855 9(14)	0.708 5(9)
(*b*) Ligand 'b'									
S(1)	0.086 1(2)	0.365 8(2)	0.274 0(2)	0.329 5(6)	0.404 8(15)	0.492 5(8)	0.327 8(1)	0.398 4(3)	0.490 3(2)
S(2)	0.097 4(2)	0.579 4(2)	0.212 8(2)	0.399 1(6)	0.612 3(18)	0.417 5(7)	0.400 2(1)	0.600 2(3)	0.416 2(2)
C(1)	0.045 0(5)	0.476 8(8)	0.238 4(5)	0.394(2)	0.463(5)	0.452(2)	0.394 0(4)	0.442 0(10)	0.449 5(5)
N	−0.022 8(5)	0.479 6(6)	0.232 8(4)	0.438(2)	0.350(4)	0.444(2)	0.438 6(4)	0.355 3(9)	0.446 1(5)
C(2)	−0.066 9(7)	0.386 7(12)	0.252 5(9)	0.431(2)	0.220(6)	0.461(3)	0.431 8(5)	0.212 7(11)	0.470 3(8)
C(3)	−0.090 9(7)	0.388 9(12)	0.325 0(9)	0.456(3)	0.202(7)	0.532(3)	0.457 2(8)	0.184 9(17)	0.538 3(10)
C(4)	−0.059 8(7)	0.575 9(11)	0.211 0(9)	0.500(3)	0.403(7)	0.420(4)	0.499 3(5)	0.392 1(12)	0.417 6(7)
C(5)	−0.075 3(8)	0.574 5(12)	0.135 8(8)	0.500(3)	0.368(7)	0.335(4)	0.503 8(7)	0.364 6(20)	0.340 4(8)
(*c*) Lattice molecules									
I(2) *				0.049 5(3)	0.433 5(8)	0.292 0(4)	0.050 92(4)	0.436 34(10)	0.292 64(5
C	0.060 4(8)	0.205 7(13)	0.071 7(8)						
Cl(1)	0.050 9(2)	0.343 8(3)	0.074 1(2)						
Cl(2)	−0.013 1(3)	0.145 4(4)	0.104 7(3)						
Cl(3)	0.077 1(3)	0.165 0(4)	−0.010 9(3)						
[H	0.102(6)	0.192(10)	0.098(6)]						

* Populations: 0.56(1) for (2); 0.839(3) for (3).

Table 2

Antimony atom environments in (1), (2), and (3): $r_{\mathrm{Sb-X}}$ in Å; the other entries in the matrices are the angles (°) subtended by the relevant atoms *

(*a*) Compound (1)

	$r_{\mathrm{Sb-X}}$	S(a1)	S(a2)	S(b1)	S(b2)	I(1′)
I(1)	3.516(2)	141.71(7)	72.21(7)	79.15(7)	126.00(7)	127.23(4)
S(a1)	2.505(3)		69.57(10)	95.61(11)	85.64(10)	79.27(7)
S(a2)	2.644(3)			85.77(10)	143.34(10)	125.99(7)
S(b1)	2.489(3)				69.75(10)	141.58(7)
S(b2)	2.648(3)					71.90(7)
I(1′)	3.556(1)					

(*b*) Compound (2)

	$r_{\mathrm{Sb-X}}$	S(a1)	S(a2)	S(b1)	S(b2)	I(1′)
I(1)	3.333(5)	149.3(3)	80.9(3)	80.5(3)	118.8(4)	119.4(1)
S(a1)	2.53(1)		68.8(4)	93.5(5)	86.2(4)	82.3(3)
S(a2)	2.64(1)			88.0(4)	145.5(4)	121.9(3)
S(b1)	2.51(1)				69.6(5)	144.8(3)
S(b2)	2.69(1)					75.3(4)
I(1′)	3.485(6)					

(*c*) Compound (3)

	$r_{\mathrm{Sb-X}}$	S(a1)	S(a2)	S(b1)	S(b2)	I(1′)
I(1)	3.386(2)	147.91(7)	78.85(7)	80.13(8)	119.95(7)	121.97(4)
S(a1)	2.512(3)		69.35(9)	93.70(10)	85.96(10)	80.84(8)
S(a2)	2.639(3)			86.97(9)	144.56(9)	123.11(6)
S(b1)	2.500(3)				69.12(9)	143.78(8)
S(b2)	2.684(3)					74.77(7)
I(1′)	3.507(2)					

(*d*) Bismuth environment in $[\mathrm{Bi(S_2CNEt_2)_2I}]$ rearranged for comparison with the present antimony systems, Bi–I–Bi′ is 89.68(4)°

	$r_{\mathrm{Bi-X}}$	S(b1)	S(b2)	S(a2)	S(a1)	I′
I	3.257(2)	142.6(1)	76.5(1)	82.5(1)	131.0(1)	96.09(4)
S(b1)	2.698(5)		66.5(1)	92.9(1)	77.6(1)	86.59(9)
S(b2)	2.691(4)			91.7(1)	135.9(1)	85.38(9)
S(a2)	2.646(4)				64.7(1)	177.0(2)
S(a1)	2.860(5)					118.0(1)
I′	3.354(1)					

* In sections (*a*), (*b*), and (*c*) a prime denotes the transformation $\frac{1}{2} - x, \frac{1}{2} + y, z$; in (*d*) primed atoms are generated by the transformation $x, \frac{1}{2} - y, \frac{1}{2} + z$.

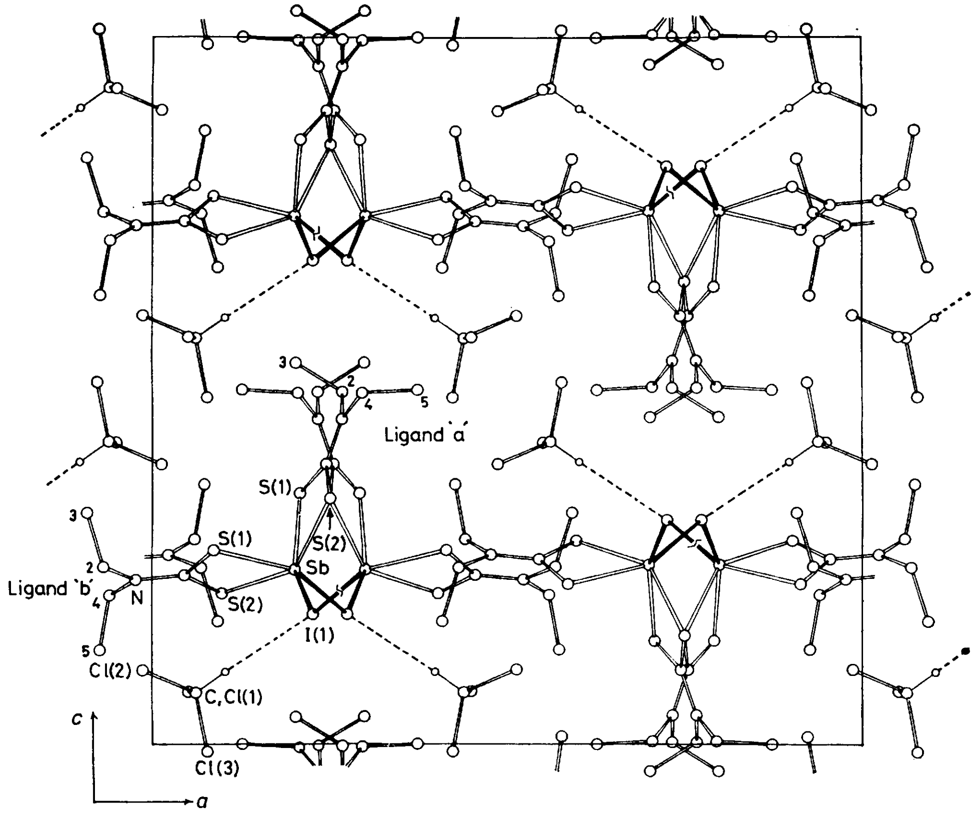


FIGURE 1 Unit-cell contents of (1), projected down *b*

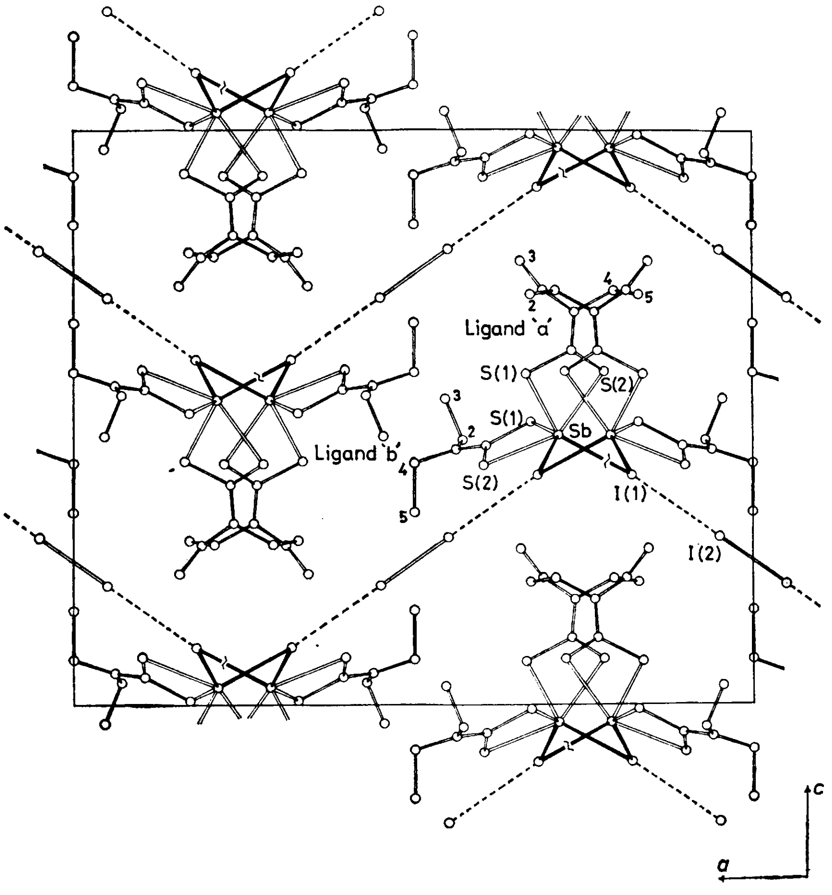
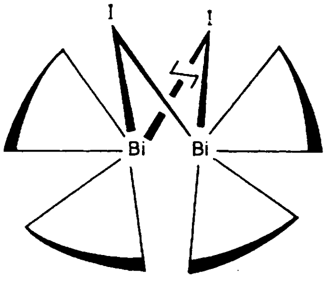


FIGURE 2 Unit-cell contents of (2), projected down *b*

atoms, with anisotropic thermal parameters for Sb, I, and S and isotropic thermal parameters for C and N. Hydrogen atoms were not included in the refinement. Population parameters of the I_2 lattice molecule were refined in both (2) and (3). Although the space groups of (1) and (3) are different, the structures are similar and unit-cell settings have been adopted to bring the two structures into reasonable coincidence in regard to these features to facilitate comparison.

DISCUSSION

Compound (1).—The structure determination confirms the assignment of stoicheiometry as $[Sb(S_2CNEt_2)_2I]\cdot CHCl_3$; there is no evidence from the well behaved refinement that the chloroform molecule is other than fully populated, the quality of the data permitting refinement of the methylene and chloroform hydrogen positional parameters. The structure of the complex comprises an infinite one-dimensional polymeric array of $Sb(S_2CNEt_2)_2$ units linked by iodine bridges, so that the backbone of the polymer is alternating antimony and iodine atoms lying in a string along *b*. The similarity to the structure of $[\{Bi(S_2CNEt_2)_2I\}_\infty]$[3] is striking [Table 2 and Figures 1 and 2, to be compared with Figure 1(*a*) and (*b*) of ref. 3]. Although similar, however, there are nevertheless non-trivial differences in the geometrical parameters of the system (Table 2), the most notable of these being the angle subtended at the bridging iodine atoms by the metal atoms {present compound, (1), 135.82(3)°, *cf.* $[Bi(S_2CNEt_2)_2I]$, 89.68(4)°}. Consideration of the projections of the structures of (1) and $[Bi(S_2CNEt_2)_2I]$ along the polymer show that the bridging iodine atoms lie out of the metal-atom backbone to one side, which is devoid of occupancy by the dithiocarbamate groups which lie disposed on the other three sides (see below). In $[Bi(S_2CNEt_2)_2I]$, the face of the polymer occupied by the iodine atoms confronts the alkyl groups of the next polymer removed by the *a* translation; in (1), however, this face is approached by the chloroform solvent molecules which hydrogen bond quite strongly to the iodine atoms [H $\cdots$ I 2.9(1) Å] (Figure 3). This effective increase in co-ordination number of the iodine is presumably responsible for the change observed in the angle subtended at it by the metal atoms. In contrast to $[Bi(S_2CNEt_2)_2I]$ and other systems derived from the $Bi(S_2CNEt_2)_2$ unit studied in the present series, in which one of the metal–sulphur bonds is longer than the other three, the SbS_4 geometry is very regular and a good approximation to *2* point symmetry, even more so than in $[Sb(S_2CNBu^n{}_2)_2]_2[Cd_2I_6]$ (Figure 4).[4] Relative to the latter, the angular geometry of the unit changes only trivially; the bond lengths, however, are slightly longer and may be indicative that I^- is a more powerful co-ordinating unit than $[Cd_2I_6]^{2-}$; the I $\cdots$ Sb contacts in the $[Sb(S_2CNBu^n{}_2)_2]_2[Cd_2I_6]$ complex are found (at the least) to be 3.659(2), longer than the 3.516(2), 3.556(1) Å found in the present case. Moreover, the structure of the $[Cd_2I_6]^{2-}$ derivative is that of a bridged centro-symmetric dimer rather than the present polymer.



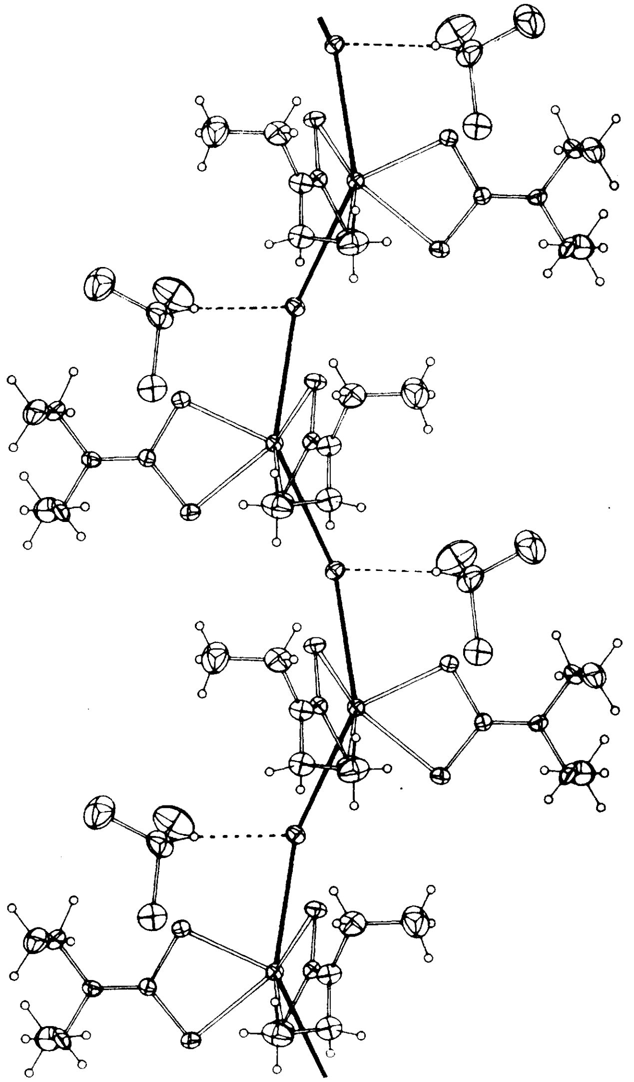


FIGURE 3 Single polymeric strand of (1), showing its interaction with the chloroform solvate molecules

Compound (3).—This complex closely resembles that in (1): infinite polymeric strings of $Sb(S_2CNEt_2)_2$ units interlinked by iodide atoms running parallel to *b*. The geometry of the $Sb(S_2CNEt_2)_2$ unit is very similar to that of (1) (Table 3), but one of the Sb–I distances is appreciably shorter. In this complex we also find inter-

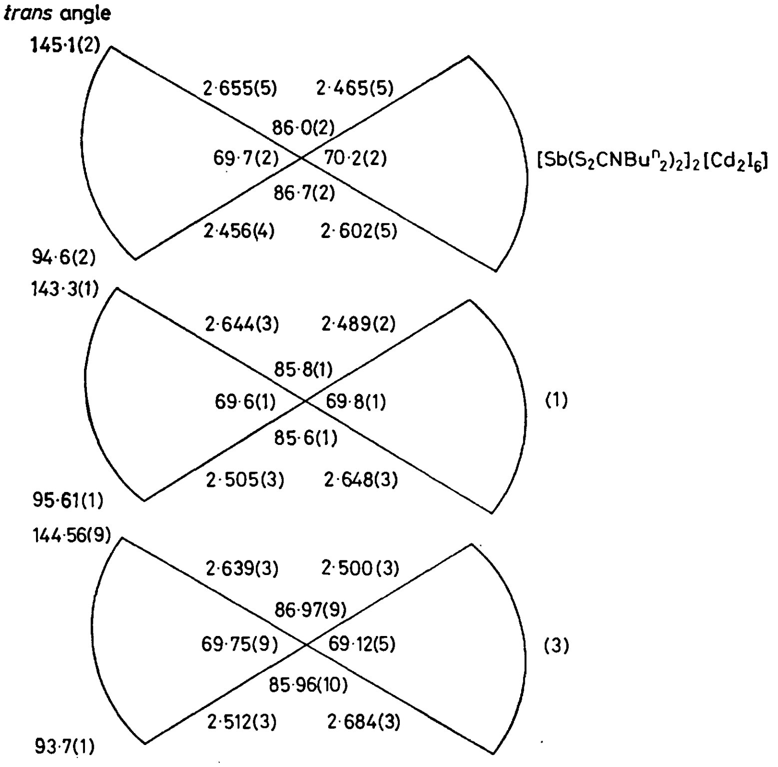


FIGURE 4 Comparative geometries of the SbS_4 unit in $[Sb(S_2CNBu^n{}_2)_2]_2[Cd_2I_6]$, and (1) and (3)

actions with the bridging iodine from lattice molecules; the latter are molecular iodine and these lie about a crystallographic two-fold rotor, and parallel to the *ac* plane. Because of the imposed symmetry they thus act as bridges between diagonally related parallel strands of the polymer. The I–I distance in the iodine molecule is 2.783(2) Å, appreciably longer than the value for free iodine (2.66$_7$ Å) [6] and very similar to the value of 2.779(3) Å observed in $[Fe\{S_2CN(CH_2)_4\}_2I]\cdot 0.5I_2$, where a similar I_2-bridged I interaction is observed.[7] The angle subtended at the bridging iodine by the two metals is substantially diminished relative to that observed in (1), being 100.28(5)°.

Treatment of the antimony environment (Figure 5) in (1) and (3) in terms of the repulsion theory developed by Kepert [8] shows that the structure does not satisfactorily conform to calculations appropriate to the *cis*-[M(bidentate ligand)$_2$(unidentate ligand)$_2$] system, the M-(unidentate)$_2$ plane being grossly twisted relative to the two planes formed at the metal by the bidentate pairs of donor atoms [$b = 1.14$, θ_A(expt.) *ca.* 32°, *cf.* θ_A(calc.) *ca.* 10°]. The location of any stereochemically active lone pair is not obvious but is probably to be found between the two iodine atoms. The description of the co-ordination geometry about the antimony in $[Sb(S_2CNBu^n{}_2)_2]_2[Cd_2I_6]$ has been made without consideration of the long Sb · · · I contacts [3.659(2) Å] as a pseudo-trigonal bipyramid with an equatorial stereochemically active lone pair; [4] such a description seems inappropriate to the present complexes in which the Sb · · · I contacts are shorter. In the structures of both (1) and (3), dithiocarbamate geometries are conventional; metal-atom deviations from the ligand S_2CNC_2 planes are for (1) (a, b); (3) (a, b), respectively 0.19, 0.17, 0.40, and 0.02 Å. Where significant differences in C–S distances are observed these correlate inversely with variations in

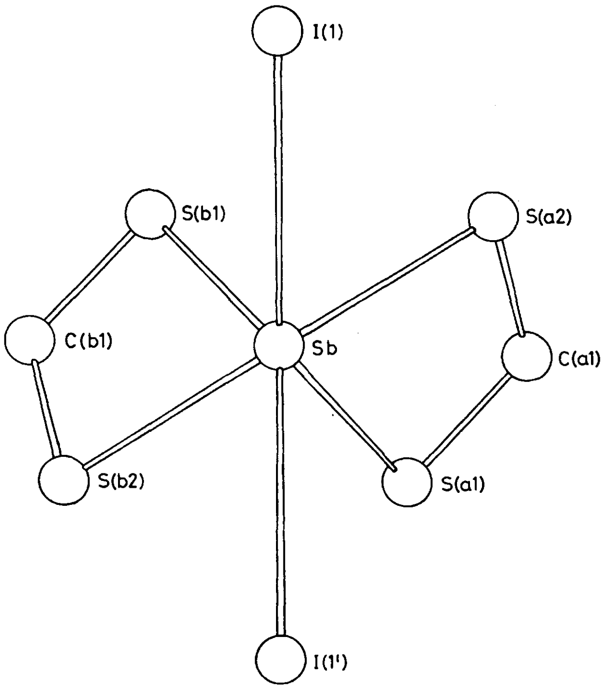


FIGURE 5 Metal-atom environment in (1), projected down the bisector of the I–Sb–I angle

Sb–S distances as is usual. Terminal methyl group dispositions in all cases are to either side of the ligand plane as usual.

The structure determination of (2) is, of course, similar to that of (3) and its precision such that one would only expect to observe significant differences in the heavy-atom geometry. Moreover, because of the considerable diminution in iodine population it should be realised that the observed geometries represent a mean of populated and unpopulated sites. On the unit-cell scale, the most striking observation is the *diminution* of unit-cell volume as the iodine population increases; clearly as the iodine content of the crystal diminishes, the polymer strands (Figure 6) become more loosely bound together and the crystal thermal motion rises (as observed). On the molecular scale we find that the I · · · I contact distance has increased from 3.402(2) [in (3)] to 3.604(8) Å [in (2)], reflecting the looser packing of the polymers in (2). This is accompanied by a reduction in I–Sb distances from 3.386(2), 3.507(2) [in (3)] to 3.333(5), 3.485(6) Å [in (2)], *i.e.* a diminution of the polymer axis, *b*. The gross effect of decreasing the iodine population is thus to increase the I · · · I distance and hence an overall decrease in ' solvation ' of the co-ordinated iodine atom together with enhanced Sb–I interaction, with a decrease in the ' solvent ' I–I distance from 2.783(2) [in (3)] to 2.75(1) Å [in (2)].

TABLE 3

Dithiocarbamate ligand geometries. The two values in each entry correspond to ligands ' a ' and ' b ' respectively

	(1) [a,b]	(2) [b]	(3) [b]
(*a*) Distances/Å			
S(1)–C(1)	1.75(1), 1.74(1)	1.73(4), 1.76(5)	1.722(9), 1.718(10)
S(2)–C(1)	1.71(1), 1.71(1)	1.67(4), 1.63(5)	1.722(9), 1.716(11)
S(1) · · · S(2)	2.940(4), 2.940(4)	2.92(2), 2.97(2)	2.933(7), 2.944(4)
C(1)–N	1.30(1), 1.31(1)	1.35(6), 1.49(6)	1.30(1), 1.32(1)
N–C(2)	1.49(2), 1.49(2)	1.51(6), 1.35(7)	1.50(1), 1.51(1)
N–C(4)	1.50(2), 1.46(2)	1.49(6), 1.57(8)	1.48(1), 1.50(1)
C(2)–C(3)	1.46(2), 1.49(2)	1.47(7), 1.48(8)	1.54(2), 1.44(2)
C(4)–C(5)	1.48(2), 1.50(2)	1.53(8), 1.67(10)	1.49(2), 1.50(2)
(*b*) Angles/°			
Sb–S(1)–C(1)	88.8(4), 88.8(3)	87(1), 86(2)	88.3(3), 89.3(4)
Sb–S(2)–C(1)	85.3(4), 84.5(3)	84(1), 82(2)	84.2(3), 83.5(3)
S(1)–C(1)–S(2)	116.1(7), 116.7(6)	118(2), 122(3)	116.8(5), 118.1(6)
S(1)–C(1)–N	119.3(8), 120.4(7)	118(3), 110(3)	121.7(7), 119.7(8)
S(2)–C(1)–N	124.5(8), 122.9(7)	123(3), 127(3)	121.6(7), 122.2(7)
C(1)–N–C(2)	123.6(9), 121.6(9)	123(4), 128(4)	122.5(9), 122.3(9)
C(1)–N–C(4)	120.3(10), 122.0(9)	120(4), 111(4)	123.4(8), 122.0(9)
C(2)–N–C(4)	116.1(9), 116.2(9)	118(3), 120(4)	114.1(8), 115.7(8)
N–C(2)–C(3)	112(1), 114(1)	110(4), 107(5)	109.8(9), 115(1)
N–C(4)–C(5)	114(1), 112(1)	106(4), 102(5)	113.0(10), 112(1)

[a] Chloroform geometry: H–C, 0.96(12); C–Cl (1,2,3), 1.74(2), 1.73(2), 1.72(2) Å; H–C–Cl(1,2,3), 105(7), 114(7), 107(7); Cl(1)–C–Cl(2,3), 109.8(9), 109.8(9); Cl(2)–C–Cl(3), 111.9(9)°.
[b] Additional iodine geometries: for (1) Sb–I–Sb^{I}, 135.83(3)°; for (2) I(1) · · · I(2), 3.604(8) Å; I(2) · · · I(1)–Sb,Sb^{I}, 148.4(2), 110.2(2); Sb–I–Sb^{I}, 101.4(1)°; I(2)–I(2^{II}), 2.75(1) Å; I(1) · · · I(2)–I(2^{II}), 177.4(3)°; for (3) I(1) · · · I(2), 3.402(2) Å; I(2) · · · I(1)–Sb,Sb^{I}, 146.20(4), 113.48(3); Sb–I–Sb^{I}, 100.28(5)°; I(2)–I(2^{II}), 2.783(2) Å; I(1) · · · I(2)–I(2^{II}), 179.21(4)°. Symmetry transformations are: I $\frac{1}{2} - x, -\frac{1}{2} + y, z$; II $\bar{x}, y, \frac{1}{2} - z$.

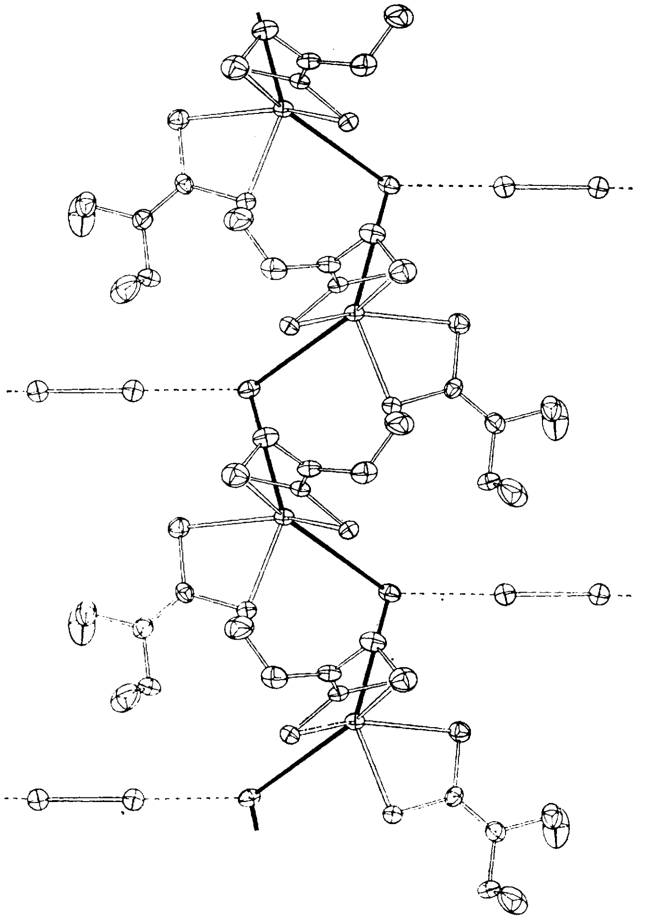


FIGURE 6 Single polymeric strand of (2), showing its interaction with the iodine lattice molecules

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