Table 2. *Unit-cell dimensions* (Å) *and space-group data*

	a	*b*	*c*	Space group	Reference
Cl_2	6·29	4·50	8·21	*Cmca*	Collin (1952)
CH_3Cl	6·495 (10)	5·139 (10)	7·523 (10)	$Cmc2_1$	Burbank (1953)
SiH_3Cl	7·119 (15)	5·568 (9)	8·453 (11)	$Cmc2_1$	This work
GeH_3Cl	7·146 (3)	5·5587 (21)	8·383 (14)	$Cmc2_1$	This work

From Guinier powder films of SiH_3Cl recorded at 148 K, we have established that it is isostructural with GeH_3Cl, and the close structural similarities between methyl chloride and elementary chlorine have been noted previously (Burbank, 1953): selected crystallographic data for these four compounds are listed in Table 2. Whereas the similarities between CH_3Cl and Cl_2 may be explained in terms of the similar sizes of Cl and CH_3 (van der Waals radii 1·8 and 2·0 Å respectively) and those between silyl and germyl chlorides in terms of the similar sizes of the silyl and germyl groups and the analogous Cl(3*p*)→Si(3*d*) and Cl(3*p*)→Ge(4*d*) supplementary bonding involved in the intermolecular interactions, the reasons for the similarity between the former and latter pair of compounds are less obvious. We believe this can be explained as follows: although the angles at C (~180°) and Cl (~90°) in solid CH_3Cl are similar to those at Ge and Cl in GeH_3Cl, the C···Cl non-bonded distance is 3·51 Å, well outside the sum of the van der Waals radii for C and Cl (3·3 Å) and indicative of essentially isolated molecules.

In the larger GeH_3Cl molecules, the existence of strong Ge···Cl intermolecular interactions allows a closer approach than would be possible for non-interacting molecules. The combination of these factors – increasing molecular size compensated by closer approach of molecules – has the result that the CH_3Cl structure is retained in GeH_3Cl and SiH_3Cl.

New studies of the vibrational spectra of solid GeH_3Cl and other silyl and germyl halides, using both IR and Raman spectroscopy, are under way in this Department and will be reported independently (Cradock & Gillespie, 1987).

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Structures of the Isomorphous Compounds Dimeric Tris[2(1*H*)-pyridine-2-thione]copper(I) Chloride and Tris[2(1*H*)-pyridine-2-thione]copper(I) Bromide

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Abstract. $[Cu(C_5H_5NS)_3]_2^{2+}.2Cl^-$, $M_r = 865·0$, monoclinic, $P2_1/n$, $a = 7·593\ (1)$, $b = 18·995\ (2)$, $c = 13·038\ (2)$ Å, $\beta = 102·13\ (2)°$, $Z = 2$, $V = 1838·5$ Å^3, $D_x = 1·562$, $D_m = 1·556$ Mg m^{-3}, λ(Mo $K\alpha$) = 0·71069 Å, $\mu = 1·70$ mm^{-1}, $F(000) = 880$, $T = 295$ K, final $R = 0·050$ for 1243 unique observed reflections.

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$[Cu(C_5H_5NS)_3]_2^{2+}.2Br^-$, $M_r = 953·9$, monoclinic, $P2_1/n$, $a = 7·617\ (1)$, $b = 19·005\ (1)$, $c = 13·045\ (1)$ Å, $\beta = 102·35\ (1)°$, $Z = 2$, $V = 1844·7$ Å^3, $D_x = 1·716$, $D_m = 1·710$ Mg m^{-3}, $\lambda(\mathrm{Mo}\ K\alpha) = 0·71069$ Å, $\mu = 3·84$ mm^{-1}, $F(000) = 952$, $T = 295$ K, final $R = 0·049$ for 1199 unique observed reflections. The isomorphous structures consist of centrosymmetrical cationic dimers $[Cu(C_5H_5NS)_3]_2^{2+}$ bridged by S atoms and connected to the X^- (X = Cl or Br) anions through N—H···X hydrogen bonds. In each dimer the two monomeric units share a tetrahedral Cu—S edge so that each Cu atom is surrounded by four S atoms in a distorted tetrahedral coordination. The Cu···Cu separation is 2·950 (2) Å in the Cl^- and 2·907 (2) Å in the Br^- compound.

Introduction. Copper(I) complexes show a variety of coordination geometries (Gagné, Kreh, Dodge, Marsh & McCool, 1982, and literature cited therein). Since the prediction of a structure for the title compounds (hereafter TPTCCl and TPTCBr) was by no means straightforward, it was deemed worthwhile to carry out their X-ray crystal structure determination.

Experimental. A solution of pyridine-2-thione (0·005 mol) in ethanol (30 ml) was added to a solution of copper chloride or bromide (0·001 mol) in water (15 ml). The mixture was stirred for 20 min, filtered and kept in a refrigerator for a few days. Orange prismatic crystals were collected by filtration, washed with cold ethanol and dried at room temperature. Philips PW 1100 computer-controlled four-circle single-crystal diffractometer, graphite-monochromated Mo $K\alpha$ radiation, ω scan, three reference reflections without significant intensity variation, space group from systematic absences, cell parameters and their standard deviations by least-squares analysis of the 41 strongest reflections, no absorption correction. Experimental data and structure-solution parameters are summarized in Table 1. Structure of TPTCCl solved with *MULTAN*77 (Main, Lessinger, Woolfson, Germain & Declercq, 1977), all non-H atoms located on E map; the refined coordinates of TPTCCl used as the starting point for TPTCBr; full-matrix least-squares refinement using F with *XRAY*76 (Stewart, Machin, Dickinson, Ammon, Heck & Flack, 1976), positional and anisotropic thermal parameters refined but U_{ij} of C(1), C(3), C(11) for TPTCBr fixed during last cycles; atomic scattering factors and anomalous-dispersion corrections for Cu, Cl, Br from *International Tables for X-ray Crystallography* (1974).

H atoms at calculated positions very close to those from a difference Fourier map, with isotropic temperature factors as those of bonded C or N atoms, were not refined.

Table 1. *Experimental data and structure refinement parameters*

	TPTCCl	TPTCBr
Crystal size	0·60 × 0·40 × 0·30	0·55 × 0·35 × 0·25
D_m by flotation in	$CCl_4/CHCl_3$	CCl_4/1,3-dibromobutane
$[(\sin\theta)/\lambda]_{\max}$(Å^{-1})	0·5938	0·5914
Range of hkl	$0 \leq h \leq 8$ $0 \leq k \leq 21$ $-15 \leq l \leq 15$	$0 \leq h \leq 8$ $0 \leq k \leq 20$ $-15 \leq l \leq 14$
Standard reflections	$04\bar{1}$; $06\bar{1}$; 101	101
Number of reflections measured	3226	3128
Number of unique reflections	3080	2917
R_{int}	0·042	0·046
Number of unique observed reflections $\lvert I \geq 2\sigma(I) \rvert$	1243	1199
Number of parameters refined	208	190
Quantity minimized	$w(\lvert F_o\rvert - \lvert F_c\rvert)^2$ $w = 1/[(1+(F-B)^2/A]$	
	$A = 3, B = 33$	$A = 8, B = 49$
R	0·050	0·049
wR	0·053	0·054
S	1·220	1·118
$(\Delta/\sigma)_{\max}$; $(\Delta/\sigma)_{\mathrm{mean}}$	0·0012; 0·0002	0·0004; 0·0001
$(\Delta\rho)_{\max}$; $(\Delta\rho)_{\min}$(e Å^{-3})	0·55; −0·38	0·57; −0·60

Discussion. Final atomic coordinates and equivalent isotropic temperature factors are given in Table 2.* Bond lengths and angles are given in Table 3. A clinographic projection of the dimer (two asymmetric units) with atom labelling is shown in Fig. 1. The Cu atom is surrounded in the form of a distorted tetrahedron, by the three S atoms of one cationic unit plus an additional S atom from an adjacent symmetry-equivalent unit. Thus, a centrosymmetrical dimer is formed in which two CuS_4 tetrahedra share one edge. This is quite different from the case of tris(thiourea)-copper(I) chloride (Okaya & Knobler, 1964), the structure of which contains infinite spiral chains of $\mathrm{Cu(thiourea)}_3^+$ cations with CuS_4 tetrahedra formed by sharing one S atom but having only one corner in common.

As in the structure of tetrakis[2(1*H*)-pyridinethione-*S*]copper(I) perchlorate–2-pyridinethione (1/2) (Kokkou, Schramm & Karagiannidis, 1985) three S—Cu—S bond angles in the CuS_4 group are nearly those of a regular tetrahedron while the rest deviate considerably from 109·5°. The Cu—S bond lengths are not all equal, ranging from 2·298 (3) to 2·538 (4) Å in TPTCCl and from 2·274 (3) to 2·534 (3) Å in TPTCBr. As expected, the longest distance is to the shared S atom. The Cu—S—Cu and S—Cu—S angles deviate from the ideal values (70·5 and 109·5°) for a symmetrical tetrahedral dimer, thus indicating an elongation of the Cu_2S_2 bridging group (*cf.* Summerville

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* Lists of structure amplitudes, anisotropic thermal parameters, H-atom parameters, bond angles and least-squares planes have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 43969 (23 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 2. *Atomic coordinates and equivalent isotropic temperature factors for the non-hydrogen atoms in TPTC* Cl *and TPTC* Br

Here and throughout this paper the e.s.d.'s are given in parentheses and refer to the last digit.

$$B_{eq} = \tfrac{8}{3}\pi^2 \textstyle\sum_i \sum_j a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	B_{eq}(Å^2)
TPTCCl				
Cu	0·0658 (2)	0·0433 (6)	−0·07807 (9)	4·9
S(1)	0·2371 (4)	−0·0420 (1)	0·0249 (2)	4·4
S(2)	0·1825 (5)	0·1554 (1)	−0·0609 (2)	5·0
S(3)	−0·0264 (4)	0·0266 (1)	−0·2572 (2)	4·1
C(1)	0·231 (1)	−0·1218 (5)	−0·0407 (7)	4·0
C(2)	0·317 (2)	−0·1836 (6)	0·0094 (8)	5·3
C(3)	0·298 (2)	−0·2460 (6)	−0·052 (1)	7·8
C(4)	0·222 (2)	−0·2477 (6)	−0·1545 (9)	6·3
C(5)	0·150 (2)	−0·1885 (5)	−0·2005 (8)	6·6
N(1)	0·160 (1)	−0·1265 (4)	−0·1424 (6)	5·3
C(6)	0·251 (2)	0·1767 (5)	0·0687 (8)	4·7
C(7)	0·309 (2)	0·1282 (5)	0·1502 (8)	5·7
C(8)	0·374 (2)	0·1509 (6)	0·2533 (8)	6·8
C(9)	0·386 (2)	0·2216 (7)	0·2760 (10)	7·7
C(10)	0·334 (2)	0·2698 (6)	0·1959 (10)	6·6
N(2)	0·275 (1)	0·2470 (4)	0·0952 (6)	5·6
C(11)	0·158 (2)	0·0407 (4)	−0·3088 (7)	4·1
C(12)	0·339 (2)	0·0374 (5)	−0·2530 (8)	5·1
C(13)	0·480 (2)	0·0502 (7)	−0·3057 (10)	6·5
C(14)	0·455 (2)	0·0653 (6)	−0·412 (1)	6·6
C(15)	0·277 (2)	0·0693 (5)	−0·4624 (9)	5·6
N(3)	0·135 (1)	0·0581 (4)	−0·4131 (6)	3·6
Cl	0·2010 (4)	−0·1248 (1)	−0·4460 (2)	5·1
TPTCBr				
Cu	0·0660 (2)	0·04395 (7)	−0·07481 (9)	4·3
S(1)	0·2369 (4)	−0·0416 (1)	0·0239 (2)	3·5
S(2)	0·1774 (5)	0·1553 (1)	−0·0543 (2)	4·4
S(3)	−0·0207 (4)	0·0265 (2)	−0·2512 (2)	4·0
C(1)	0·231 (1)	−0·1191 (6)	−0·0388 (7)	4·3
C(2)	0·312 (2)	−0·1824 (5)	0·0049 (9)	4·8
C(3)	0·296 (2)	−0·2441 (5)	−0·049 (1)	6·7
C(4)	0·209 (2)	−0·2482 (8)	−0·152 (1)	8·6
C(5)	0·140 (2)	−0·1875 (7)	−0·1947 (9)	5·0
N(1)	0·154 (1)	−0·1256 (4)	−0·1392 (6)	4·3
C(6)	0·257 (1)	0·1777 (5)	0·0724 (7)	3·4
C(7)	0·307 (2)	0·1267 (6)	0·1518 (8)	4·5
C(8)	0·375 (2)	0·1515 (7)	0·2520 (8)	5·3
C(9)	0·396 (2)	0·2214 (6)	0·2784 (9)	5·0
C(10)	0·342 (2)	0·2701 (6)	0·1957 (10)	5·3
N(2)	0·283 (1)	0·2435 (4)	0·0987 (7)	3·9
C(11)	0·174 (1)	0·0433 (5)	−0·3014 (7)	2·4
C(12)	0·351 (2)	0·0419 (6)	−0·2458 (9)	4·6
C(13)	0·489 (2)	0·0501 (6)	−0·2894 (10)	5·1
C(14)	0·471 (2)	0·0661 (9)	−0·396 (1)	8·2
C(15)	0·294 (3)	0·0658 (5)	−0·453 (1)	7·7
N(3)	0·147 (1)	0·0592 (4)	−0·4064 (6)	3·9
Br	0·1957 (2)	−0·12329 (6)	−0·44261 (8)	4·4

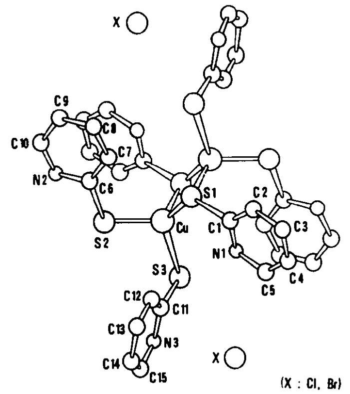

Fig. 1. Clinographic projection of the TPTCCl (or TPTCBr) dimer.

& Hoffmann, 1976). The Cu···Cu distances are 2·950 (2) Å for TPTCCl and 2·907 (2) Å for TPTCBr. The S—C bond distances are normal and the Cu—S—C angles are not far from the tetrahedral values. The geometrical features of the pyridine rings in both compounds are close to those of an aromatic ring.

The Cl^- (or Br^-) anions bridge the cationic dimers through hydrogen bonds of the type N—H···X (X = Cl or Br), thus forming infinite chains along the diagonal [1$\bar{1}$1]. An intramolecular N—H···S hydrogen bond is also important for the orientation of the pyridinethione ligands (Fig. 2).

The IR spectra of the complexes in KBr discs do not contain the $\tilde{\nu}$(SH) band in the 2500–2600 cm^{-1} region. The presence of broad bands in the range 3400–3150 cm^{-1} is in accordance with the fact that the thione form of the ligand contributes predominantly to the formation of the complexes.

Thanks are due to the University of Thessaloniki for the use of its computing facilities.

Table 3. *Interatomic distances* (Å) *and selected bond angles* (°)

	TPTCCl	TPTCBr
Cu–S(1)	2·320 (3)	2·297 (3)
Cu–S(2)	2·298 (3)	2·274 (3)
Cu–S(3)	2·326 (3)	2·278 (3)
Cu–S(1^{iii})	2·538 (4)	2·534 (3)
S(1)–C(1)	1·737 (9)	1·68 (1)
S(2)–C(6)	1·709 (10)	1·688 (9)
S(3)–C(11)	1·71 (1)	1·777 (10)
C(1)–C(2)	1·43 (1)	1·42 (2)
C(1)–N(1)	1·33 (1)	1·32 (1)
C(2)–C(3)	1·42 (2)	1·36 (2)
C(3)–C(4)	1·34 (2)	1·37 (2)
C(4)–C(5)	1·33 (2)	1·34 (2)
C(5)–N(1)	1·39 (1)	1·37 (1)
C(6)–C(7)	1·41 (1)	1·41 (1)
C(6)–N(2)	1·38 (1)	1·30 (1)
C(7)–C(8)	1·40 (1)	1·38 (1)
C(8)–C(9)	1·38 (2)	1·37 (2)
C(9)–C(10)	1·38 (2)	1·41 (2)
C(10)–N(2)	1·37 (1)	1·35 (1)
C(11)–C(12)	1·41 (2)	1·38 (1)
C(11)–N(3)	1·38 (1)	1·37 (1)
C(12)–C(13)	1·41 (2)	1·31 (2)
C(13)–C(14)	1·40 (2)	1·40 (2)
C(14)–C(15)	1·37 (2)	1·40 (2)
C(15)–N(3)	1·38 (2)	1·40 (2)
Hydrogen bonds		
N(1)–H(N1)···S(3)	3·372 (8)	3·382 (9)
N(2)–H(N2)···X^{i}	3·146 (9)	3·275 (9)
N(3)–H(N3)···X^{ii}	3·088 (8)	3·159 (9)
S(1)–Cu–S(2)	115·6 (1)	116·1 (1)
S(1)–Cu–S(3)	118·5 (1)	118·1 (1)
S(1)–Cu–S(1^{iii})	105·4 (1)	106·2 (1)
S(2)–Cu–S(3)	106·4 (1)	105·9 (1)
S(2)–Cu–S(1^{iii})	109·6 (1)	108·9 (1)
S(3)–Cu–S(1^{iii})	100·0 (1)	100·3 (1)
Cu–S(1)–C(1)	111·7 (3)	113·2 (3)
Cu–S(2)–C(6)	110·4 (4)	113·4 (4)
Cu–S(3)–C(11)	105·2 (3)	105·2 (3)
Cu–S(1^{iii})–C(1^{iii})	101·2 (4)	100·4 (4)
Cu–S(1)–Cu^{iii}	74·6 (1)	73·8 (1)

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

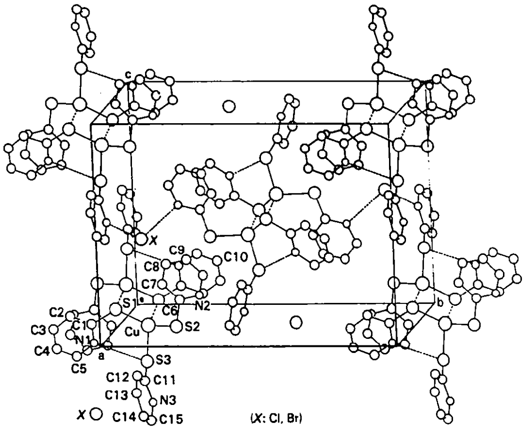

Fig. 2. Clinographic projection of the unit-cell contents of the title compounds.

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Structure of 1,1-Dichloro-1-methyl-1λ^5-stibacyclohexane

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Abstract. $C_6H_{13}Cl_2Sb$, $M_r = 277{\cdot}8$, orthorhombic, *Pbcn*, $a = 10{\cdot}624$ (8), $b = 9{\cdot}094$ (6), $c = 10{\cdot}281$ (9) Å, $V = 993$ (1) Å^3, $Z = 4$, $D_x = 1{\cdot}858$ g cm^{-3}, λ(Mo $K\alpha$) = 0·71069 Å, $\mu = 32{\cdot}7$ cm^{-1}, $F(000) = 536$, $T =$ 295 K. $R = 0{\cdot}025$ for 709 observed reflections with $I > 2{\cdot}5\sigma(I)$. The six-membered stibacyclohexane ring is puckered into a chair conformation. The coordination of Sb is slightly distorted trigonal bipyramidal with the three C atoms in equatorial positions and the two Cl atoms in axial positions. The endocyclic C—Sb—C bond angle is 106·0 (2)° and significantly smaller than the exocyclic C—Sb—C bond angles of 127·0 (1)°.

Introduction. The present structure determination was carried out in relation to a study of the correlation of C—Sb—C bond angles in *cis*-diorganoantimony(V) compounds with ^{121}Sb Mössbauer spectra (Barbieri, Bertazzi, Gibb, Meinema & Noltes, 1979).

Experimental. Colorless needle-shaped crystal (0·12 × 0·12 × 0·82 mm) glued on top of a glass fiber. Enraf–Nonius CAD-4*F* diffractometer, Zr-filtered Mo$K\alpha$ radiation. 1344 reflections scanned, $\omega/2\theta$ scan, $\Delta\omega = (0{\cdot}60 + 0{\cdot}35\tan\theta)°$; $2{\cdot}2 < \theta < 27{\cdot}5°$; h: 0→13, k: 0→11, l 0→13. Two reference reflections (232: e.s.d. 1·4%; $\bar{2}3\bar{2}$: e.s.d. 1·0%), linear decay of 20% during 31 h of X-ray exposure time. Cell dimensions from the setting angles of eight reflections ($\theta \simeq 10{\cdot}5°$). An analysis of the lattice symmetry (Le Page, 1982) did not reveal any symmetry higher than *oP*. Space group *Pbcn* from the observed extinctions ($0kl$: $k = 2n+1$; $h0l$: $l = 2n+1$; $hk0$: $h+k = 2n+1$). Correction for Lp and linear decay but not for absorption. 1140 unique reflections of which 431 had $I < 2{\cdot}5\sigma(I)$. The structure was solved by standard Patterson and Fourier methods. Full-matrix least-squares refinement on F (excluding H atoms) with anisotropic thermal parameters converged to $R = 0{\cdot}0309$. An analysis of the resulting essentially planar six-membered-ring geometry showed some unsatisfactory short bonds (1·41–1·44 Å) involving

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