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Bromo(*N*,*N*-dimethyldithiocarbamato)dimethylgermanium and (*N*,*N*-Dimethyldithiocarbamato)iododimethylgermanium

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Abstract. $[Ge\{S_2CN(CH_3)_2\}Br(CH_3)_2]$, $M_r = 302{\cdot}8$, monoclinic, $P2_1$, $a = 9{\cdot}471$ (4), $b = 10{\cdot}777$ (5), $c = 11{\cdot}383$ (4) Å, $\beta = 106{\cdot}99$ (3)°, $V = 1111$ (1) Å^3, $Z = 4$, $D_x = 1{\cdot}81$, $D_m = 1{\cdot}79$ Mg m^{-3}, λ(Mo $K\alpha$) = 0·71069 Å, $\mu = 650{\cdot}1$ mm^{-1}, $F(000) = 592$, $T = 291$ K, final $R = 0{\cdot}0519$, $wR = 0{\cdot}0543$ for 1407 unique observed reflections. $[Ge\{S_2CN(CH_3)_2\}(CH_3)_2I]$, $M_r = 349{\cdot}8$, monoclinic, $P2_1/c$, $a = 9{\cdot}935$ (3), $b = 10{\cdot}434$ (3), $c = 12{\cdot}425$ (2) Å, $\beta = 117{\cdot}71$ (2)°, $V = 1140$ (1) Å^3, $Z = 4$, $D_x = 2{\cdot}04$, $D_m = 2{\cdot}00$ Mg m^{-3}, $\mu = 543{\cdot}4$ mm^{-1}, $F(000) = 664$, $T = 291$ K, final $R = 0{\cdot}0468$, $wR = 0{\cdot}0498$ for 1235 unique observed reflections. The structure of $(CH_3)_2GeBr[S_2CN(CH_3)_2]$ contains two independent molecules in which the Ge atom is at the centre of a distorted trigonal bipyramid. Two Ge—C bonds and one Ge—S bond occupy the equatorial positions with the Ge—Br bond accounting for one of the axial positions. The other axial position is occupied by the second S atom of the dithiocarbamate ligand but at a distance much greater than in the Ge—S equatorial bond. The structure of $(CH_3)_2GeI[S_2CN(CH_3)_2]$ is similar to that just described for the bromo analogue with two notable exceptions. There is only one independent molecule and the axial Ge—S distance is considerably shorter.

Introduction. In an earlier publication (Chadha, Drake & Sarkar, 1984) we demonstrated that the structure of $(CH_3)_2GeCl[S_2CN(CH_3)_2]$ provided an example of a distorted trigonal bipyramidal germanium compound with an anisobidentate ligand. The Ge—Cl bond occupying the axial position was considerably longer and weaker than those typically found for four-coordinate germanium chlorides (Drake, Hencher & Shen, 1977; Li & Durig, 1973; Morino, Nakamaru & Iijima, 1960) and was comparable with the axial Ge—Cl bond in $[N(CH_3)_3][GeCl_4]$ (Beattie & Ozin, 1970; Bilton & Webster, 1972). A comparison of the spectroscopic data of the three compounds $(CH_3)_2GeX[S_2CN(CH_3)_2]$, X = Cl, Br, I (Chadha, Drake & Sarkar, 1986), indicated that similar distorted trigonal bipyramidal structures might be expected for the bromo and iodo derivatives. In particular, the values of the Ge—Br and Ge—I stretching vibrations were of the order of 80 and 100 cm^{-1} respectively lower than in the related four-coordinate germanium halide derivatives $(CH_3)_2GeBr_2$ and $(CH_3)_2GeI_2$ (Anderson, Barker, Drake & Hemmings, 1971; Griffiths, 1964; Van de Vondel, Van der Kelen & Van Hooydonk, 1970). We have completed the crystal structures of bromo(*N*,*N*-dimethyldithiocarbamato)dimethylgermanium, $(CH_3)_2GeBr[S_2CN(CH_3)_2]$, and (*N*,*N*-dimethyldithiocarbamato)iododimethylgermanium, $(CH_3)_2GeI[S_2CN(CH_3)_2]$, which not only confirm the predictions based on spectroscopy but also draw attention to some interesting trends.

Experimental. Preparation as described by Chadha, Drake & Sarkar (1986) followed by slow evaporation of CS_2 solution, density measured by flotation in C_2H_5I/ligroin or CH_3I/ligroin for the bromo and iodo

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Table 1. *Final fractional coordinates and equivalent isotropic temperature factors* ($Å^2 \times 10^3$)

	$(CH_3)_2GeBr[S_2CN(CH_3)_2]$					$(CH_3)_2GeI[S_2(CN(CH_3)_2]$			
	x	y	z	U_{eq}*		x	y	z	U_{eq}*
Ge1	0·5106 (2)	0·8984†	0·4131 (2)	48·7 (2)	Ge	0·3491 (1)	0·6869 (1)	0·6340 (1)	43·0 (2)
Br1	0·4166 (3)	0·8316 (3)	0·5788 (2)	76 (1)	I	0·2486 (1)	0·4411 (1)	0·6005 (1)	64·7 (1)
S1	0·4752 (5)	0·7080 (4)	0·3350 (4)	49 (1)	S1	0·1337 (2)	0·7414 (2)	0·4667 (2)	46 (1)
S2	0·6089 (5)	0·8832 (4)	0·2005 (4)	62 (1)	S2	0·3588 (3)	0·9390 (2)	0·5952 (2)	62 (1)
N1	0·523 (1)	0·654 (1)	0·124 (1)	48 (1)	N	0·1009 (8)	0·9836 (6)	0·3926 (6)	53 (1)
C1	0·360 (2)	1·020 (2)	0·332 (2)	60 (2)	C1	0·354 (1)	0·7174 (8)	0·7877 (8)	63 (1)
C2	0·709 (2)	0·955 (2)	0·503 (2)	69 (2)	C2	0·532 (1)	0·6586 (9)	0·6209 (8)	57 (1)
C3	0·536 (2)	0·740 (1)	0·208 (1)	40 (1)	C3	0·1885 (8)	0·9010 (7)	0·4758 (6)	41 (1)
C4	0·461 (2)	0·531 (2)	0·135 (2)	70 (2)	C4	0·143 (1)	1·1162 (8)	0·3943 (9)	76 (1)
C5	0·574 (2)	0·673 (2)	0·016 (2)	71 (3)	C5	−0·044 (1)	0·9478 (8)	0·2899 (9)	77 (1)
Ge2	0·0444 (2)	0·8212 (2)	0·8743 (1)	45·1 (2)					
Br2	0·1236 (3)	0·6169 (2)	0·8288 (2)	89 (1)					
S3	0·0983 (5)	0·9043 (5)	0·7111 (4)	56 (1)					
S4	−0·0271 (6)	1·0756 (4)	0·8539 (4)	63 (1)					
N2	0·039 (2)	1·140 (1)	0·650 (1)	62 (2)					
C6	0·182 (2)	0·858 (2)	1·029 (2)	68 (2)					
C7	−0·155 (2)	0·788 (2)	0·862 (2)	65 (2)					
C8	0·035 (2)	1·057 (2)	0·732 (2)	50 (1)					
C9	0·094 (2)	1·116 (2)	0·546 (2)	78 (3)					
C10	−0·020 (3)	1·261 (2)	0·661 (3)	96 (3)					

* $U_{eq} = \frac{1}{3}[U_{22} + 1/\sin^2\beta(U_{11} + U_{33} + 2U_{13}\cos\beta)]$.
† Arbitrarily assigned to fix y axis.

compounds respectively, crystals 0·39 × 0·50 × 0·61 mm (bromo) and 0·30 × 0·40 × 0·50 mm (iodo), Syntex $P2_1$ diffractometer, highly oriented graphite monochromator, cell parameters from 15 strong reflections ($15 < 2\theta < 30°$), data collected and processed as described earlier (Chadha, Drake & Sarkar, 1986); intensities of three monitor reflections changed by less than 3% (bromo), 7% (iodo) during data collection, corrected by applying a scale factor; 2194 reflections ($4 < 2\theta < 50°$; h −11–10, k 0–12, l 0–13), 1407 [$I > 3\sigma(I)$] unique (bromo) and 1688 reflections ($4 < 2\theta < 45°$; h −10–9, k 0–11, l 0–13), 1235 [$I > 3\sigma(I)$] unique (iodo), Lorentz–polarization and empirical absorption corrections [transmission factors 0·825–0·875 (bromo), 0·917–0·949 (iodo)]; positions of the Ge atoms obtained by sharpened Patterson synthesis, positions of remaining non-H atoms determined from a difference Fourier map; full-matrix least-squares complete anisotropic refinement minimizing $\sum w(|F_o| - |F_c|)^2$, 182 (bromo) or 91 (iodo) parameters in each cycle, H atoms included in ideal positions (C—H = 0·95 Å, HCH = 109·5°), methyl H atoms refined as a group with isotropic U values assigned 0·01 $Å^2$ greater than the atoms to which they are attached, $R = 0·0519$ for 1407 reflections (bromo) or 0·0468 for 1235 reflections (iodo), $wR = 0·0543$ (bromo) or 0·0498 (iodo); in final cycle of refinement largest shift/e.s.d. 0·001 (bromo) or 0·003 (iodo), final difference maps had no features of chemical significance, largest peaks 1·1 e $Å^{-3}$, 1·03 Å from Ge (bromo) or 1·1 e $Å^{-3}$, 1·01 Å from Ge (iodo); $w = 1/\sum[\sigma^2(F) + pF^2]$, final $p = 0·0013$ (bromo) or 0·0001 (iodo); scattering factors for all non-H atoms, including anomalous-dispersion corrections for Br and I, obtained from Ibers & Hamilton (1974), for H from Stewart, Davidson & Simpson (1965); programs used include *SHELX* (Fourier and least-squares calculations; Sheldrick, 1977), *XANADU* (Roberts & Sheldrick, 1975) and *ORTEP* (Johnson, 1965).

Discussion. The final atomic coordinates for non-H atoms are given in Table 1 for both compounds and important distances and angles are combined in Table 2* along with those of the corresponding chloride derivative for ease of comparison.

The crystal structure analyses of $(CH_3)_2GeBr[S_2CN(CH_3)_2]$ and $(CH_3)_2GeI[S_2CN(CH_3)_2]$ confirm the predictions made from spectroscopic evidence, namely that in both compounds the Ge atom is inside a distorted trigonal bipyramid. This can be seen clearly in the *ORTEP* diagram of one of the two independent molecules of the bromo compound (Fig. 1) and that of the iodo analogue (Fig. 2). The Ge—C bond lengths vary less in the chloro, bromo and iodo derivatives than they do in the two independent molecules of the bromo compound although if the *average* bond length is taken from the bromo derivative, then all Ge—C bond lengths fall in the 1·92–1·93 Å range found in the gas-phase studies of $(CH_3)_2GeCl_2$ and $(CH_3)_2GeBr_2$ (Drake, Hencher & Shen, 1977; Drake, Hemmings, Hencher, Mustoe & Shen, 1976). The C—Ge—C bond angles are also similar in all three compounds and comparable with the values of 121 (4) and 124 (7)° reported for

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* Lists of thermal parameters of non-H atoms, final fractional coordinates and thermal parameters for H atoms, structure factors, and unit-cell diagrams have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51314 (39 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 2. *Bond lengths* (Å) *and angles* (°) *with standard deviations in parentheses*

	$(CH_3)_2GeI$-$[S_2CN(CH_3)_2]$	$(CH_3)_2GeBr[S_2CN(CH_3)_2]$		$(CH_3)_2GeCl$-$[S_2CN(CH_3)_2]$*
Ge–*X*	2·712 (1)	2·418 (3)	2·430 (3)†	2·251 (1)
Ge–S1	2·255 (2)	2·222 (4)	2·252 (4)	2·254 (1)
Ge–S2	2·685 (2)	2·840 (5)	2·817 (5)	2·896 (1)
Ge–C1	1·916 (8)	1·96 (2)	1·90 (2)	1·929 (2)
Ge–C2	1·920 (8)	1·96 (2)	1·89 (2)	1·925 (2)
S1–C3	1·739 (8)	1·74 (2)	1·79 (2)	1·750 (2)
S2–C3	1·699 (8)	1·70 (2)	1·68 (2)	1·690 (2)
N–C3	1·32 (1)	1·31 (2)	1·30 (2)	1·317 (2)
N–C4	1·44 (1)	1·46 (2)	1·45 (2)	1·463 (2)
N–C5	1·46 (1)	1·46 (2)	1·44 (3)	1·453 (2)
X–Ge–S1	88·5 (1)	89·4 (1)	91·5 (1)	90·7 (1)
X–Ge–S2	160·1 (1)	159·1 (1)	161·1 (1)	159·2 (1)
S1–Ge–S2	71·6 (1)	69·8 (1)	69·6 (2)	68·5 (1)
X–Ge–C1	98·5 (3)	100·6 (6)	102·3 (5)	101·2 (1)
S1–Ge–C1	116·7 (3)	115·2 (5)	114·8 (6)	115·6 (1)
S2–Ge–C1	91·7 (3)	90·7 (6)	87·6 (5)	87·8 (1)
X–Ge–C2	97·9 (3)	101·5 (6)	100·2 (6)	101·7 (1)
S1–Ge–C2	119·4 (3)	120·6 (6)	118·5 (6)	116·7 (1)
S2–Ge–C2	91·2 (3)	87·8 (6)	88·1 (6)	89·1 (1)
C1–Ge–C2	121·6 (4)	119·4 (9)	120·8 (9)	121·9 (1)
Ge–S1–C3	92·7 (3)	95·8 (5)	95·4 (6)	96·4 (1)
Ge–S2–C3	79·8 (3)	76·8 (5)	79·5 (6)	76·9 (1)
C3–N–C4	122·4 (7)	123 (1)	124 (2)	123·2 (2)
C3–N–C5	122·8 (7)	122 (1)	118 (2)	121·2 (2)
C4–N–C5	114·7 (7)	116 (2)	117 (2)	115·6 (2)
S1–C3–S2	115·8 (4)	117·2 (9)	115·5 (9)	117·9 (2)
S1–C3–N	120·2 (6)	119 (1)	118 (1)	117·6 (1)
S2–C3–N	124·0 (6)	124 (1)	127 (1)	124·5 (1)

* Chadha, Drake & Sarkar (1984).

† This column refers to the second independent molecule.

$(CH_3)_2GeCl_2$ and $(CH_3)_2GeBr_2$. The length of the Ge—S bond that constitutes the third member of the equatorial plane is close to the sum of the covalent radii of Ge and S (2·25 Å) (Cotton & Wilkinson, 1980). The Ge atom sits slightly [0·26 and 0·21 (bromo), 0·18 Å (iodo)] out of the plane formed by the two methyl C atoms and the equatorial S atom.

However, as was observed for the Ge—Cl bond in $(CH_3)_2GeCl[S_2CN(CH_3)_2]$, the Ge—Br bond lengths in the two modifications are appreciably longer than those reported previously for the related molecule $(CH_3)_2$-$GeBr_2$ [2·418 (3) and 2·430 (3) compared with 2·303 (2) Å]. Similarly the Ge—I bond is appreciably longer than that reported for CH_3GeI_3 [2·712 (1) compared with 2·500 (2) Å]; the only previously reported structure of a methylgermanium iodide (Chadha, Drake & Neo, 1985). These halogen atoms all occupy one of the axial positions with the *X*—Ge—S1 angle being close to 90° in all three compounds. The second S atom of the dithiocarbamate ligand occupies the second axial site but at a distance much greater than that found for the Ge—S(equatorial) bond and in all cases making an angle close to 160° rather than 180° with the Ge—*X* bond. However, the Ge—S(axial) distances in the three compounds differ considerably, the longest 2·896 (1) Å being seen in the chloro derivative which obviously has the shortest Ge—*X*(Cl) bond of 2·251 (1) Å. The Ge—S(axial) distances in the two modifications of the bromo compound are significantly shorter 2·840 (5) and 2·817 (5) Å with the shorter Ge—S(axial) distance being formed with the longer Ge—Br bond. In the iodo compound which contains the long Ge—I bond of 2·712 (1) Å, the Ge—S(axial) distance is significantly shorter still at 2·685 (2) Å.

Finally, the bond lengths and bond angles within the dithiocarbamate remain reasonably constant in all of the halogen derivatives despite the significant change in the Ge—S(axial) distances. Thus the ligand is consistently asymmetric with the longer S—C bond being associated with the shorter Ge—S(equatorial) bond and the shorter S—C bond with the longer Ge—S(axial) distance. However, the bite angle, S—Ge—S, shows a gradual increase along the series chloride, bromide, iodide, namely 68·5 (1), 69·7 (2), 71·6 (1)° respectively.

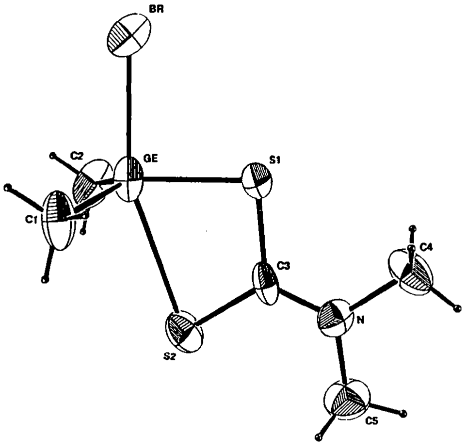

Fig. 1. *ORTEP* plot of one of the two independent molecules of $(CH_3)_2GeBr[S_2CN(CH_3)_2]$. The atoms are drawn with 20% probability ellipsoids. H atoms are drawn arbitrarily small.

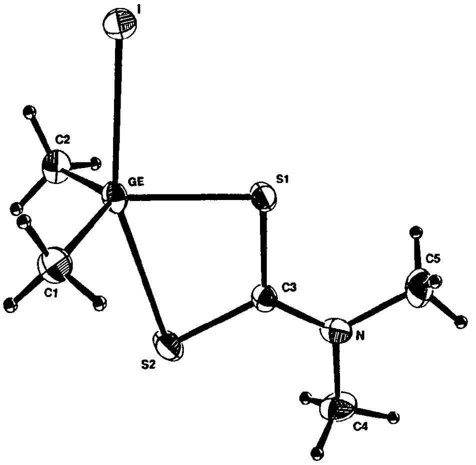

Fig. 2. *ORTEP* plot of $(CH_3)_2GeI[S_2CN(CH_3)_2]$. The atoms are drawn with 20% probability ellipsoids. H atoms are drawn arbitrarily small.

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Structure of Chloro-*N*,*N*,*N'*,*N'*-tetramethylformamidinium Bis[dichloromercury(II)] Chloride

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Abstract. $C_5H_{12}ClN_2^+.2HgCl_2.Cl^-$, $M_r = 714\cdot06$, triclinic, $P\bar{1}$, $a = 10\cdot840$ (3), $b = 10\cdot409$ (3), $c = 7\cdot422$ (2) Å, $\alpha = 96\cdot59$ (5), $\beta = 100\cdot87$ (5), $\gamma = 105\cdot73$ (5)°, $V = 779\cdot4$ (1) Å^3, $Z = 2$, $D_x = 3\cdot05$ Mg m^{-3}, λ(Mo $K\alpha$) = 0·71069 Å, $\mu = 199\cdot8$ cm^{-1}, $F(000) = 636$, room temperature, $R = 0\cdot040$, $wR = 0\cdot042$ for 2904 observed reflections [$F_o \geq 3\sigma(F_o)$]. The chlorotetramethylformamidinium cation assumes a non planar conformation as a result of non-bonded intramolecular repulsion between methyl groups. There are two independent Hg atoms in the asymmetric unit. The coordination around Hg(1) can be described as a distorted octahedron of six chlorines with two short, two long and two very long Hg—Cl bonds. The coordination around Hg(2) is a distorted trigonal bipyramid with two short and one intermediate Hg—Cl bond in the equatorial plane, the coordination sphere being completed by two very long Hg—Cl contacts. The anionic framework can be described in terms of $Hg_2Cl_5^-$ units. If Hg—Cl contacts up to 3·30 Å are considered the structure can be described as being formed by layers of interconnected $Hg_2Cl_5^-$ units parallel to (010) with the cations located between the layers.

Introduction. Information concerning reactions of chloro-*N*,*N*,*N'*,*N'*-tetramethylformamidinium chloride with metal complexes is very scarce (Petz, 1975; Hartshorn, Lappert & Turner, 1978). Some time ago the synthesis of chloro-*N*,*N*,*N'*,*N'*-tetramethylformamidinium bis[dichloromercury(II)] chloride was reported (Altman & Welcman, 1980); we now report the crystal structure of this compound.

Experimental. Transparent plate (0·28 × 0·15 × 0·07 mm). Cell parameters refined using the setting angles of 25 strong reflections having $5\cdot6 < 2\theta < 33\cdot0°$. Intensity collected on a Philips PW 1100 four-circle diffractometer, graphite-monochromatized Mo $K\alpha$ radiation, $\omega/2\theta$ scan technique, scan speed 3·0° ω min^{-1}, scan width 1·15 in ω, $5° \leq 2\theta \leq 55°$, 3570 unique reflections (h −13→13, k −13→13, l 0→9) of which 2904 with $F_o \geq 3\sigma(F_o)$. No significant intensity variation for three standards ($2\bar{1}\bar{1}$, $\bar{2}21$, $21\bar{1}$) measured every two hours. Corrections for Lorentz, polarization and absorption (Busing & Levy, 1957). Structure solved by Patterson and refined using *SHELX*77 (Sheldrick, 1977) system of programs; hydrogen atoms were not located. Full-matrix least squares, 136 parameters refined (scale factor, atomic coordinates and anisotropic displacement factors for non-H atoms). Function minimized $\sum w(|F_o| - |F_c|)^2$. Final $R = 0\cdot040$, $wR = 0\cdot042$, weighting scheme $a/[\sigma^2(F_o) + b(F_o)^2]$ with $a = 1\cdot658$ and $b = 0$, $(\Delta/\sigma)_{\max} = 0\cdot11$.

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