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Tri-*tert*-butylgallane–(*tert*-butyl)diethylstibine adduct

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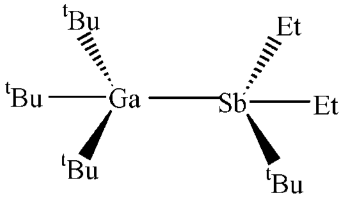
Key indicators: single-crystal X-ray study; T = 120 K; mean σ(C–C) = 0.005 Å; R factor = 0.037; wR factor = 0.074; data-to-parameter ratio = 28.6.

Geometric parameters of the title compound, tri-*tert*-butyldiethylgalliumantimony(Ga—Sb), [^{t}Bu$_3$Ga—Sb(^{t}Bu)Et$_2$] or [GaSb(C$_2$H$_5$)$_2$(C$_4$H$_9$)$_4$], reflect the influence of the larger steric demand of the ^{t}Bu ligand compared with that of the ethyl groups.

Related literature

For related literature, see Coates (1951); Kuczkowski *et al.* (2001); Kuczkowski *et al.* (2005); Schulz & Nieger (2000) and Schuchmann *et al.* (2007).



Experimental

Crystal data

[GaSb(C$_2$H$_5$)$_2$(C$_4$H$_9$)$_4$]
M_r = 478.04
Monoclinic, $P2_1/n$
a = 9.1138 (16) Å
b = 23.698 (4) Å
c = 11.027 (2) Å
β = 91.479 (4)°
V = 2380.7 (7) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 2.27 mm^{-1}
T = 120 (2) K
0.22 × 0.20 × 0.16 mm

Data collection

Bruker SMART APEX diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2002)
$T_{\min}$ = 0.624, $T_{\max}$ = 0.699
20988 measured reflections
5684 independent reflections
4418 reflections with $I > 2\sigma(I)$
$R_{\rm int}$ = 0.082

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.037
$wR(F^2)$ = 0.074
S = 0.95
5684 reflections
199 parameters
H-atom parameters constrained
$\Delta\rho_{\max}$ = 0.95 e Å^{-3}
$\Delta\rho_{\min}$ = −0.95 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Sb1—C17	2.152 (3)	Ga1—C9	2.034 (3)
Sb1—C19	2.158 (4)	Ga1—C1	2.039 (3)
Sb1—C13	2.197 (3)	Ga1—C5	2.039 (3)
Sb1—Ga1	2.9243 (5)		
C17—Sb1—C19	98.05 (15)	C13—Sb1—Ga1	126.75 (8)
C17—Sb1—C13	100.69 (14)	C9—Ga1—C1	117.05 (13)
C19—Sb1—C13	98.53 (15)	C9—Ga1—C5	116.50 (13)
C17—Sb1—Ga1	117.47 (11)	C1—Ga1—C5	115.65 (13)
C19—Sb1—Ga1	110.36 (12)		

Data collection: *SMART* (Bruker, 2002); cell refinement: *SAINT* (Bruker, 2002); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Bruker, 2002); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BT2352).

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supplementary materials

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Tri-*tert*-butylgallane-(*tert*-butyl)diethylstibine adduct

D. Schuchmann, S. Schulz and U. Flörke

Comment

Lewis acidic trialkylgallanes GaR_3 generally react with Lewis basic group 15 organyles ER'_3 (E = N, P, As, Sb, Bi) with formation of adducts of the type R_3Ga—ER'_3. The acid–base interaction within the adducts becomes weaker and the thermodynamic stability decreases with increasing atomic number of the central group 15 element owing to an increase in *s* character of the electron lone pair. The same is true for distibine adducts with trialkylgallanes of the general type $[R_3\text{Ga}]_2[\text{Sb}_2R'_4]$. In solution, these adducts are stable at low temperature (253 K), whereas at ambient temperature, Sb—Sb bond cleavage occurs with subsequent formation of heterocyclic stibinogallanes $[R_2\text{GaSb}R'_2]_x$ and the corresponding Lewis acid–base adduct R_3Ga–SbRR'_2, containing a mixed substituted trialkylstibine. The thermodynamic stability of Me_3Ga–$E\text{Me}_3$ adducts (E = N, P, As, Sb) decreases from NMe_3 to SbMe_3, while BiMe_3 did not react (Coates, 1951). Since then, numerous adducts with strong Lewis basic amines and phosphines have been prepared and structurally characterized, whereas the first completely alkyl-substituted gallane–stibine adducts $^t\text{Bu}_3$Ga—SbR_3 [R = Me (Kuczkowski *et al.*, 2005), Et, ^{i}Pr (Schulz & Nieger, 2000)] became only recently available. In addition, a very few gallane–distibine adducts of the type $[^t\text{Bu}_3\text{Ga}]_2[\text{Sb}_2\text{R}'_4]$ [R = Me, Et (Kuczkowski *et al.*, 2001), ^{n}Pr (Schuchmann *et al.*, 2007)], with the distibine serving as bidentate ligand, were prepared at low temperatures and structurally characterized. These adducts tend to undergo Sb–Sb bond cleavage reactions in solution at ambient temperature, yielding four- and six-membered heterocycles of the general type $[R_2\text{GaSb}R'_2]_2$ and $[R_2\text{GaSb}R'_2]_3$ (Kuczkowski *et al.*, 2001; Schuchmann *et al.*, 2007), and the corresponding adduct R_3Ga—E(R)R'_2. The Ga and Sb atoms show distorted tetrahedral environments with the organic substituents adopting a staggered conformation related to one other as was previously observed for such adducts. The mean Ga–C [2.037 (3) Å] and Sb–C bond lengths [2.169 (4) Å] and C–Ga–C [116.4 (1)°] and C–Sb–C bond angles [99.1 (1)°] are within typical ranges. However, the larger steric demand of the ^{t}Bu substituent bound to the Sb atom is clearly reflected by the significantly larger Ga–Sb–C13 bond angle [126.75 (8)°] compared to those of the Et substituents [Ga–Sb–C17 117.5 (2), Ga–Sb–C19 110.4 (2)°]. In addition, the sum of the C–Sb–C bond angles in the title compound [297.3 (1)°] is bigger than that in the corresponding adduct $^t\text{Bu}_3$Ga-SbEt_3 (292.8°). The influence of bulky organic substituents on the Ga—Sb distance becomes obvious when comparing the title compound, which shows a Ga—Sb bond length of 2.9243 (5) Å, with the Ga—Sb distances reported for $^t\text{Bu}_3$Ga-SbMe_3 [2.8435 (3) Å], $^t\text{Bu}_3$Ga-SbEt_3 [2.8479 (5) Å] and $^t\text{Bu}_3$Ga—Sb($^i\text{Pr})_3$ [2.9618 (2) Å]. Sb($^i\text{Pr})_3$ is sterically more demanding than $^t\text{BuSbEt}_2$, leading to a larger sum of the C–Sb–C bond angles, whereas SbMe_3 and SbEt_3 are sterically less hindered.

Experimental

A solution of Sb_2Et_4 (1.5 mmol, 0.54 g) and $^t\text{Bu}_3$Ga (3 mmol, 0.72 g) in 30 ml of n-hexane was stirred for 5 d at ambient temperature and then stored for 12 h at -30°C. The resulting colorless solid ($[^t\text{Bu}_2\text{GaSbEt}_2]_2$) was isolated by filtration, the filtrate concentrated to 5 ml and again stored for 12 h at -30°C. Colorless crystals of $^t\text{Bu}_3$Ga—Sb(Et)$_2{}^t$Bu were obtained

(0.56 g, 78%. m.p. 65°C), which can be further purified by sublimation at 55°C at 10^{-3} mbar. Spectroscopic analysis: ^{1}H NMR (500 MHz; C_6D_5H; 25°C) δ = 1.13 [9H, s, $(CH_3)_3CSb$], 1.19 - 1.28 [10H, m, CH_3CH_2Sb], 1.24 [27H, s, $(CH_3)_3CGa$] ppm. $^{13}C\{^1H\}$ NMR (125 MHz; C_6D_5H; 25°C) δ = 7.4 [s, CH_3CH_2Sb), 12.0 [s, CH_3CH_2Sb], 29.3 [s, $(CH_3)_3CGa$], 30.4 [s, $(CH_3)_3CSb$], 31.6 [s, $(CH_3)_3CGa$] ppm.

Refinement

Hydrogen atoms were located in Fourier difference maps and refined at idealizeded positions, riding on their parent C atoms, with isotropic displacement parameters $U_{iso}(H) = 1.2U_{eq}(C)$ and $1.5U_{eq}(C\text{-methyl})$.

Figures

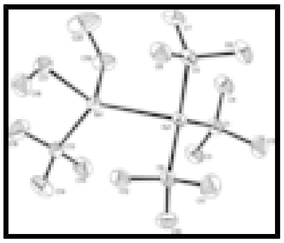

Fig. 1. Molecular structure with hydrogen atoms omitted. Displacement ellipsoids are drawn at the 50% probability level.

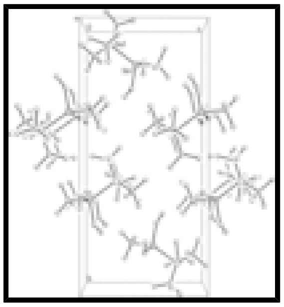

Fig. 2. Crystal packing viewed along [100].

Fig. 3. Reaction pathway.

tri-*tert*-butyldiethylgalliumantimony(Ga—Sb)

Crystal data

$[GaSb(C_2H_5)_2(C_4H_9)_4]$

$M_r = 478.04$

Monoclinic, $P2_1/n$

Hall symbol: -P 2yn

$a = 9.1138 (16)$ Å

$b = 23.698 (4)$ Å

$c = 11.027 (2)$ Å

$β = 91.479 (4)$º

$F_{000} = 992$

$D_x = 1.334$ Mg m^{-3}

Mo $Kα$ radiation

$λ = 0.71073$ Å

Cell parameters from 986 reflections

$θ = 2.5–26.7$º

$μ = 2.27$ mm^{-1}

$T = 120 (2)$ K

Block, colourless

V = 2380.7 (7) Å^3	0.22 × 0.20 × 0.16 mm
Z = 4	

Data collection

Bruker SMART APEX diffractometer	5684 independent reflections
Radiation source: sealed tube	4418 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.082
T = 120(2) K	θ_{max} = 27.9°
φ and ω scans	θ_{min} = 1.7°
Absorption correction: multi-scan (SADABS; Bruker, 2002)	$h = -11 \rightarrow 11$
T_{min} = 0.624, T_{max} = 0.699	$k = -31 \rightarrow 31$
20988 measured reflections	$l = -14 \rightarrow 14$

Refinement

Refinement on F^2	H-atom parameters constrained
Least-squares matrix: full	$w = 1/[\sigma^2(F_o^2) + (0.0303P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)]$ = 0.037	$(\Delta/\sigma)_{\text{max}}$ = 0.001
$wR(F^2)$ = 0.074	$\Delta\rho_{\text{max}}$ = 0.95 e Å^{-3}
S = 0.95	$\Delta\rho_{\text{min}}$ = −0.95 e Å^{-3}
5684 reflections	Extinction correction: none
199 parameters	
Primary atom site location: structure-invariant direct methods	
Secondary atom site location: difference Fourier map	
Hydrogen site location: difference Fourier map	

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted R-factor wR and goodness of fit S are based on F^2, conventional R-factors R are based on F, with F set to zero for negative F^2. The threshold expression of F^2 > 2sigma(F^2) is used only for calculating R-factors(gt) etc. and is not relevant to the choice of reflections for refinement. R-factors based on F^2 are statistically about twice as large as those based on F, and R- factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	y	z	$U_{\text{iso}}*/U_{\text{eq}}$
Sb1	0.71254 (2)	0.158353 (8)	0.431944 (19)	0.02535 (7)
Ga1	0.78052 (4)	0.087011 (13)	0.22442 (3)	0.02383 (9)

C1	0.7108 (4)	0.01031 (13)	0.2837 (3)	0.0325 (7)
C2	0.7744 (5)	−0.03402 (14)	0.1981 (4)	0.0502 (10)
H2A	0.7433	−0.0717	0.2233	0.075*
H2B	0.8818	−0.0318	0.2014	0.075*
H2C	0.7385	−0.0268	0.1150	0.075*
C3	0.7617 (4)	−0.00311 (15)	0.4125 (3)	0.0420 (9)
H3A	0.7249	−0.0403	0.4355	0.063*
H3B	0.7240	0.0255	0.4677	0.063*
H3C	0.8693	−0.0032	0.4173	0.063*
C4	0.5432 (4)	0.00410 (15)	0.2780 (4)	0.0433 (9)
H4A	0.5160	−0.0334	0.3073	0.065*
H4B	0.5076	0.0088	0.1940	0.065*
H4C	0.4990	0.0330	0.3291	0.065*
C5	0.6503 (4)	0.12448 (13)	0.0946 (3)	0.0310 (7)
C6	0.6332 (5)	0.08250 (15)	−0.0105 (3)	0.0450 (9)
H6A	0.5707	0.0990	−0.0748	0.068*
H6B	0.5881	0.0477	0.0187	0.068*
H6C	0.7300	0.0739	−0.0427	0.068*
C7	0.4968 (4)	0.13883 (16)	0.1399 (3)	0.0414 (9)
H7A	0.4388	0.1566	0.0742	0.062*
H7B	0.5057	0.1649	0.2087	0.062*
H7C	0.4479	0.1042	0.1656	0.062*
C8	0.7166 (4)	0.17842 (14)	0.0457 (3)	0.0388 (8)
H8A	0.6499	0.1948	−0.0160	0.058*
H8B	0.8110	0.1699	0.0093	0.058*
H8C	0.7317	0.2054	0.1121	0.058*
C9	1.0008 (3)	0.09786 (13)	0.2096 (3)	0.0294 (7)
C10	1.0882 (4)	0.06349 (15)	0.3047 (4)	0.0447 (9)
H10A	1.1934	0.0697	0.2944	0.067*
H10B	1.0659	0.0233	0.2943	0.067*
H10C	1.0611	0.0755	0.3861	0.067*
C11	1.0439 (4)	0.07810 (17)	0.0837 (4)	0.0515 (11)
H11A	1.1500	0.0828	0.0749	0.077*
H11B	0.9916	0.1006	0.0219	0.077*
H11C	1.0179	0.0382	0.0734	0.077*
C12	1.0490 (4)	0.15945 (13)	0.2228 (3)	0.0342 (7)
H12A	1.1557	0.1620	0.2152	0.051*
H12B	1.0216	0.1736	0.3026	0.051*
H12C	1.0007	0.1822	0.1592	0.051*
C13	0.8331 (4)	0.16198 (13)	0.6069 (3)	0.0325 (7)
C14	0.9914 (4)	0.17754 (19)	0.5847 (4)	0.0559 (11)
H14A	1.0460	0.1793	0.6624	0.084*
H14B	0.9951	0.2144	0.5448	0.084*
H14C	1.0356	0.1490	0.5327	0.084*
C15	0.8294 (5)	0.10403 (16)	0.6651 (4)	0.0538 (11)
H15A	0.8829	0.1051	0.7434	0.081*
H15B	0.8757	0.0765	0.6118	0.081*
H15C	0.7273	0.0930	0.6778	0.081*
C16	0.7661 (5)	0.20555 (18)	0.6891 (4)	0.0656 (13)

H16A	0.8212	0.2066	0.7666	0.098*
H16B	0.6637	0.1955	0.7035	0.098*
H16C	0.7700	0.2427	0.6505	0.098*
C17	0.6764 (4)	0.24674 (14)	0.3980 (4)	0.0439 (9)
H17A	0.5912	0.2506	0.3411	0.053*
H17B	0.6499	0.2650	0.4752	0.053*
C18	0.8048 (5)	0.27849 (14)	0.3462 (4)	0.0504 (10)
H18A	0.8904	0.2749	0.4013	0.076*
H18B	0.7790	0.3184	0.3369	0.076*
H18C	0.8281	0.2627	0.2669	0.076*
C19	0.4982 (4)	0.13805 (17)	0.4982 (4)	0.0548 (11)
H19A	0.4616	0.1046	0.4529	0.066*
H19B	0.5105	0.1267	0.5843	0.066*
C20	0.3854 (5)	0.1806 (3)	0.4913 (6)	0.103 (2)
H20A	0.4240	0.2161	0.5244	0.154*
H20B	0.3013	0.1684	0.5383	0.154*
H20C	0.3543	0.1861	0.4064	0.154*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Sb1	0.02265 (11)	0.02651 (10)	0.02678 (12)	0.00206 (8)	−0.00157 (8)	0.00041 (9)
Ga1	0.02381 (18)	0.02428 (16)	0.02328 (18)	−0.00316 (14)	−0.00149 (14)	−0.00031 (14)
C1	0.0339 (19)	0.0278 (15)	0.0354 (19)	−0.0046 (14)	−0.0043 (15)	0.0041 (14)
C2	0.061 (3)	0.0312 (18)	0.058 (3)	−0.0046 (18)	−0.004 (2)	−0.0080 (17)
C3	0.047 (2)	0.0367 (18)	0.042 (2)	−0.0112 (16)	−0.0090 (18)	0.0092 (16)
C4	0.045 (2)	0.0392 (19)	0.046 (2)	−0.0183 (17)	−0.0044 (18)	0.0081 (17)
C5	0.0309 (18)	0.0373 (17)	0.0244 (16)	−0.0037 (14)	−0.0061 (14)	0.0018 (13)
C6	0.055 (3)	0.049 (2)	0.0304 (19)	−0.0116 (18)	−0.0093 (18)	−0.0045 (16)
C7	0.031 (2)	0.054 (2)	0.039 (2)	−0.0018 (17)	−0.0083 (16)	0.0111 (17)
C8	0.042 (2)	0.0387 (17)	0.035 (2)	−0.0038 (16)	−0.0075 (17)	0.0076 (15)
C9	0.0228 (16)	0.0322 (16)	0.0334 (18)	−0.0038 (13)	0.0008 (13)	−0.0040 (14)
C10	0.0277 (19)	0.0413 (19)	0.065 (3)	0.0013 (15)	−0.0022 (18)	0.0065 (18)
C11	0.041 (2)	0.061 (2)	0.053 (3)	−0.0050 (19)	0.015 (2)	−0.019 (2)
C12	0.0272 (17)	0.0366 (17)	0.0390 (19)	−0.0053 (14)	0.0008 (15)	0.0046 (15)
C13	0.0357 (19)	0.0355 (17)	0.0261 (17)	0.0045 (15)	−0.0028 (14)	−0.0049 (14)
C14	0.045 (3)	0.084 (3)	0.039 (2)	−0.014 (2)	−0.0093 (19)	−0.002 (2)
C15	0.069 (3)	0.048 (2)	0.043 (2)	−0.005 (2)	−0.020 (2)	0.0085 (18)
C16	0.082 (3)	0.073 (3)	0.041 (2)	0.035 (3)	−0.009 (2)	−0.019 (2)
C17	0.052 (2)	0.0342 (17)	0.045 (2)	0.0094 (17)	−0.0066 (18)	0.0023 (16)
C18	0.062 (3)	0.0332 (18)	0.055 (2)	−0.0087 (18)	−0.015 (2)	0.0036 (17)
C19	0.028 (2)	0.061 (2)	0.076 (3)	−0.0029 (18)	0.012 (2)	0.013 (2)
C20	0.038 (3)	0.119 (5)	0.153 (6)	0.023 (3)	0.030 (3)	0.025 (4)

Geometric parameters (Å, °)

Sb1—C17	2.152 (3)	C10—H10A	0.9800
Sb1—C19	2.158 (4)	C10—H10B	0.9800
Sb1—C13	2.197 (3)	C10—H10C	0.9800

Sb1—Ga1	2.9243 (5)	C11—H11A	0.9800
Ga1—C9	2.034 (3)	C11—H11B	0.9800
Ga1—C1	2.039 (3)	C11—H11C	0.9800
Ga1—C5	2.039 (3)	C12—H12A	0.9800
C1—C3	1.516 (5)	C12—H12B	0.9800
C1—C4	1.534 (5)	C12—H12C	0.9800
C1—C2	1.536 (5)	C13—C16	1.513 (5)
C2—H2A	0.9800	C13—C14	1.515 (5)
C2—H2B	0.9800	C13—C15	1.517 (5)
C2—H2C	0.9800	C14—H14A	0.9800
C3—H3A	0.9800	C14—H14B	0.9800
C3—H3B	0.9800	C14—H14C	0.9800
C3—H3C	0.9800	C15—H15A	0.9800
C4—H4A	0.9800	C15—H15B	0.9800
C4—H4B	0.9800	C15—H15C	0.9800
C4—H4C	0.9800	C16—H16A	0.9800
C5—C8	1.519 (4)	C16—H16B	0.9800
C5—C6	1.532 (4)	C16—H16C	0.9800
C5—C7	1.536 (5)	C17—C18	1.516 (5)
C6—H6A	0.9800	C17—H17A	0.9900
C6—H6B	0.9800	C17—H17B	0.9900
C6—H6C	0.9800	C18—H18A	0.9800
C7—H7A	0.9800	C18—H18B	0.9800
C7—H7B	0.9800	C18—H18C	0.9800
C7—H7C	0.9800	C19—C20	1.440 (6)
C8—H8A	0.9800	C19—H19A	0.9900
C8—H8B	0.9800	C19—H19B	0.9900
C8—H8C	0.9800	C20—H20A	0.9800
C9—C11	1.527 (5)	C20—H20B	0.9800
C9—C12	1.530 (4)	C20—H20C	0.9800
C9—C10	1.534 (5)		

C17—Sb1—C19	98.05 (15)	C9—C10—H10A	109.5
C17—Sb1—C13	100.69 (14)	C9—C10—H10B	109.5
C19—Sb1—C13	98.53 (15)	H10A—C10—H10B	109.5
C17—Sb1—Ga1	117.47 (11)	C9—C10—H10C	109.5
C19—Sb1—Ga1	110.36 (12)	H10A—C10—H10C	109.5
C13—Sb1—Ga1	126.75 (8)	H10B—C10—H10C	109.5
C9—Ga1—C1	117.05 (13)	C9—C11—H11A	109.5
C9—Ga1—C5	116.50 (13)	C9—C11—H11B	109.5
C1—Ga1—C5	115.65 (13)	H11A—C11—H11B	109.5
C9—Ga1—Sb1	102.65 (9)	C9—C11—H11C	109.5
C1—Ga1—Sb1	100.97 (10)	H11A—C11—H11C	109.5
C5—Ga1—Sb1	99.58 (9)	H11B—C11—H11C	109.5
C3—C1—C4	107.4 (3)	C9—C12—H12A	109.5
C3—C1—C2	108.7 (3)	C9—C12—H12B	109.5
C4—C1—C2	107.5 (3)	H12A—C12—H12B	109.5
C3—C1—Ga1	113.4 (2)	C9—C12—H12C	109.5
C4—C1—Ga1	113.0 (2)	H12A—C12—H12C	109.5
C2—C1—Ga1	106.7 (2)	H12B—C12—H12C	109.5

C1—C2—H2A	109.5	C16—C13—C14	109.3 (3)
C1—C2—H2B	109.5	C16—C13—C15	110.5 (3)
H2A—C2—H2B	109.5	C14—C13—C15	108.7 (3)
C1—C2—H2C	109.5	C16—C13—Sb1	110.6 (2)
H2A—C2—H2C	109.5	C14—C13—Sb1	109.0 (2)
H2B—C2—H2C	109.5	C15—C13—Sb1	108.7 (2)
C1—C3—H3A	109.5	C13—C14—H14A	109.5
C1—C3—H3B	109.5	C13—C14—H14B	109.5
H3A—C3—H3B	109.5	H14A—C14—H14B	109.5
C1—C3—H3C	109.5	C13—C14—H14C	109.5
H3A—C3—H3C	109.5	H14A—C14—H14C	109.5
H3B—C3—H3C	109.5	H14B—C14—H14C	109.5
C1—C4—H4A	109.5	C13—C15—H15A	109.5
C1—C4—H4B	109.5	C13—C15—H15B	109.5
H4A—C4—H4B	109.5	H15A—C15—H15B	109.5
C1—C4—H4C	109.5	C13—C15—H15C	109.5
H4A—C4—H4C	109.5	H15A—C15—H15C	109.5
H4B—C4—H4C	109.5	H15B—C15—H15C	109.5
C8—C5—C6	108.1 (3)	C13—C16—H16A	109.5
C8—C5—C7	107.7 (3)	C13—C16—H16B	109.5
C6—C5—C7	108.3 (3)	H16A—C16—H16B	109.5
C8—C5—Ga1	112.7 (2)	C13—C16—H16C	109.5
C6—C5—Ga1	107.1 (2)	H16A—C16—H16C	109.5
C7—C5—Ga1	112.8 (2)	H16B—C16—H16C	109.5
C5—C6—H6A	109.5	C18—C17—Sb1	115.6 (3)
C5—C6—H6B	109.5	C18—C17—H17A	108.4
H6A—C6—H6B	109.5	Sb1—C17—H17A	108.4
C5—C6—H6C	109.5	C18—C17—H17B	108.4
H6A—C6—H6C	109.5	Sb1—C17—H17B	108.4
H6B—C6—H6C	109.5	H17A—C17—H17B	107.4
C5—C7—H7A	109.5	C17—C18—H18A	109.5
C5—C7—H7B	109.5	C17—C18—H18B	109.5
H7A—C7—H7B	109.5	H18A—C18—H18B	109.5
C5—C7—H7C	109.5	C17—C18—H18C	109.5
H7A—C7—H7C	109.5	H18A—C18—H18C	109.5
H7B—C7—H7C	109.5	H18B—C18—H18C	109.5
C5—C8—H8A	109.5	C20—C19—Sb1	118.5 (3)
C5—C8—H8B	109.5	C20—C19—H19A	107.7
H8A—C8—H8B	109.5	Sb1—C19—H19A	107.7
C5—C8—H8C	109.5	C20—C19—H19B	107.7
H8A—C8—H8C	109.5	Sb1—C19—H19B	107.7
H8B—C8—H8C	109.5	H19A—C19—H19B	107.1
C11—C9—C12	107.4 (3)	C19—C20—H20A	109.5
C11—C9—C10	108.5 (3)	C19—C20—H20B	109.5
C12—C9—C10	107.4 (3)	H20A—C20—H20B	109.5
C11—C9—Ga1	108.2 (2)	C19—C20—H20C	109.5
C12—C9—Ga1	113.2 (2)	H20A—C20—H20C	109.5
C10—C9—Ga1	112.0 (2)	H20B—C20—H20C	109.5

C17—Sb1—Ga1—C9	83.96 (15)	Sb1—Ga1—C5—C7	−43.5 (2)

C19—Sb1—Ga1—C9	−164.86 (15)	C1—Ga1—C9—C11	87.7 (3)
C13—Sb1—Ga1—C9	−46.52 (14)	C5—Ga1—C9—C11	−55.2 (3)
C17—Sb1—Ga1—C1	−154.82 (15)	Sb1—Ga1—C9—C11	−162.8 (2)
C19—Sb1—Ga1—C1	−43.65 (15)	C1—Ga1—C9—C12	−153.4 (2)
C13—Sb1—Ga1—C1	74.69 (15)	C5—Ga1—C9—C12	63.7 (3)
C17—Sb1—Ga1—C5	−36.15 (15)	Sb1—Ga1—C9—C12	−43.9 (2)
C19—Sb1—Ga1—C5	75.02 (15)	C1—Ga1—C9—C10	−31.8 (3)
C13—Sb1—Ga1—C5	−166.63 (14)	C5—Ga1—C9—C10	−174.7 (2)
C9—Ga1—C1—C3	64.0 (3)	Sb1—Ga1—C9—C10	77.7 (2)
C5—Ga1—C1—C3	−152.7 (2)	C17—Sb1—C13—C16	41.6 (3)
Sb1—Ga1—C1—C3	−46.4 (3)	C19—Sb1—C13—C16	−58.3 (3)
C9—Ga1—C1—C4	−173.5 (2)	Ga1—Sb1—C13—C16	178.2 (2)
C5—Ga1—C1—C4	−30.2 (3)	C17—Sb1—C13—C14	−78.6 (3)
Sb1—Ga1—C1—C4	76.1 (2)	C19—Sb1—C13—C14	−178.6 (3)
C9—Ga1—C1—C2	−55.6 (3)	Ga1—Sb1—C13—C14	58.0 (3)
C5—Ga1—C1—C2	87.6 (3)	C17—Sb1—C13—C15	163.1 (3)
Sb1—Ga1—C1—C2	−166.1 (2)	C19—Sb1—C13—C15	63.2 (3)
C9—Ga1—C5—C8	−30.7 (3)	Ga1—Sb1—C13—C15	−60.3 (3)
C1—Ga1—C5—C8	−174.2 (2)	C19—Sb1—C17—C18	−178.9 (3)
Sb1—Ga1—C5—C8	78.7 (2)	C13—Sb1—C17—C18	80.7 (3)
C9—Ga1—C5—C6	88.1 (2)	Ga1—Sb1—C17—C18	−60.9 (3)
C1—Ga1—C5—C6	−55.3 (3)	C17—Sb1—C19—C20	7.2 (5)
Sb1—Ga1—C5—C6	−162.5 (2)	C13—Sb1—C19—C20	109.4 (5)
C9—Ga1—C5—C7	−152.9 (2)	Ga1—Sb1—C19—C20	−116.1 (4)
C1—Ga1—C5—C7	63.7 (3)		

Fig. 1

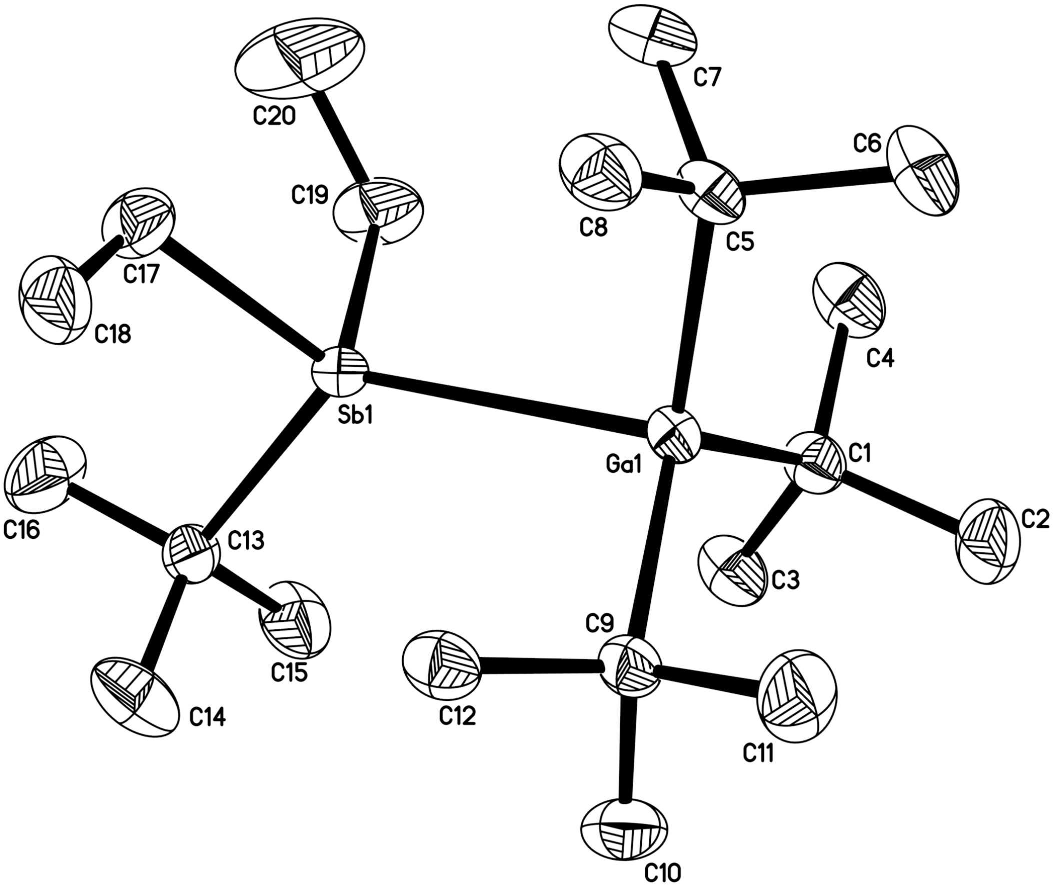
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Fig. 2

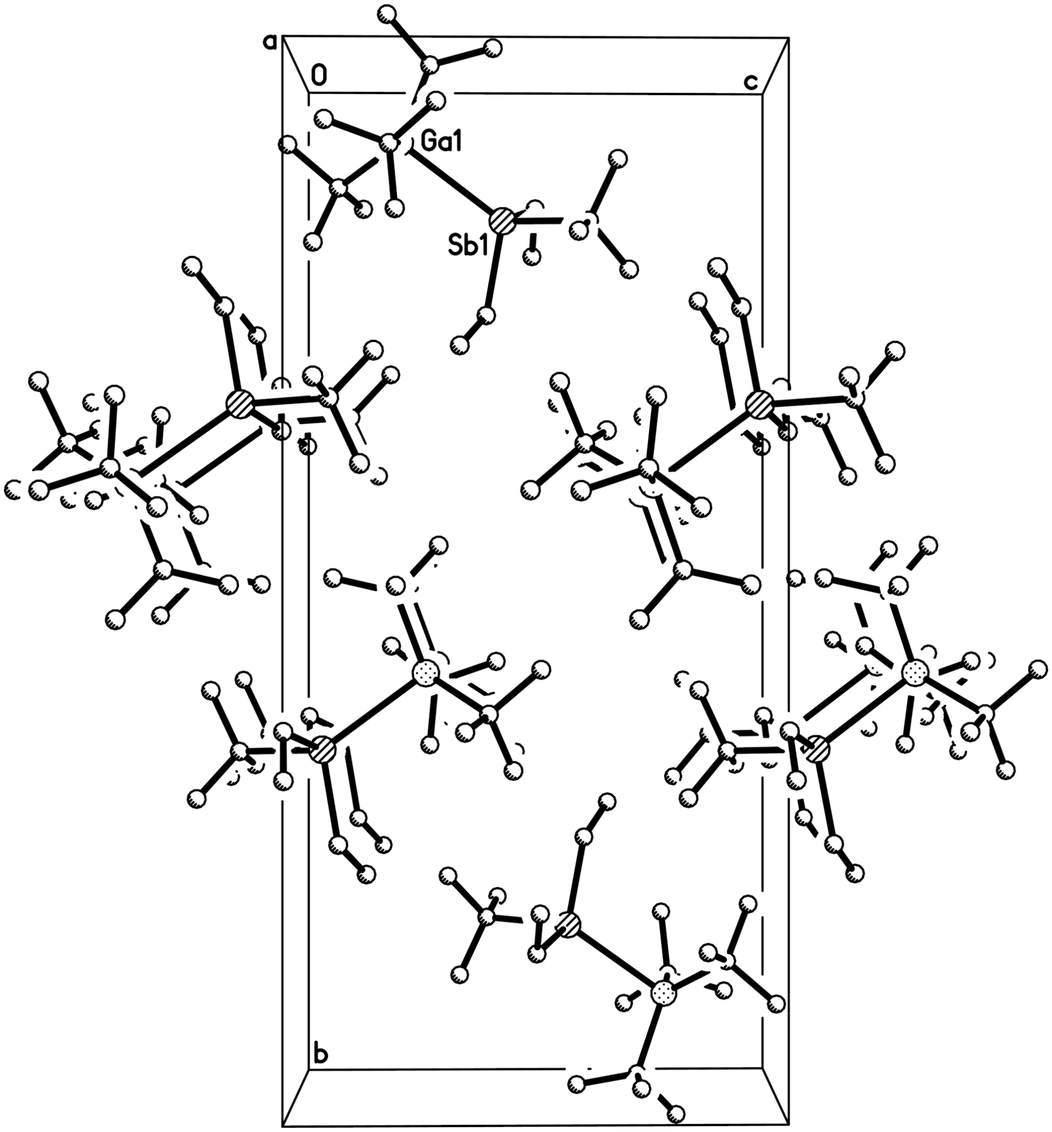
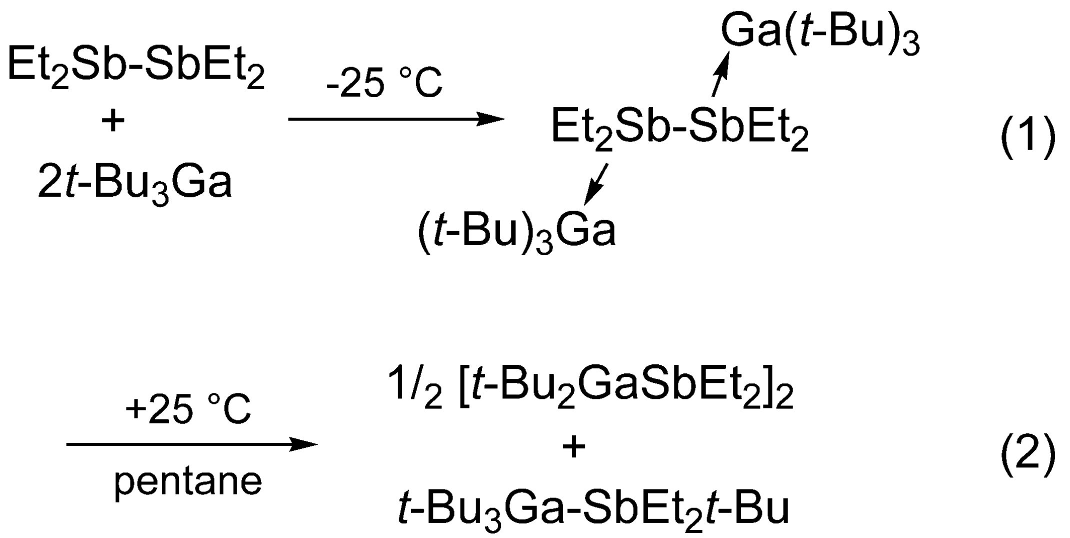


Fig. 3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-25 °C
Ga(t-Bu)3
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(t-Bu)3Ga
(1)
+25 °C
pentane
1/2 [t-Bu2GaSbEt2]2
+
t-Bu3Ga-SbEt2t-Bu
(2)