

12-Chloro-6-cyclohexyl-5,6,7,12-tetrahydrodibenzo[c,f][1,5]azastibocene

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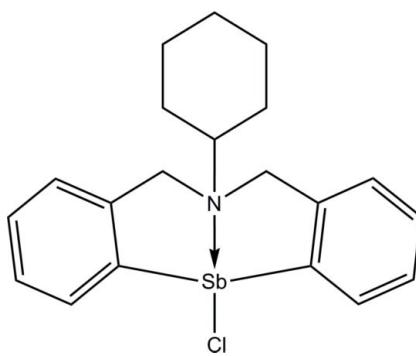
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Key indicators: single-crystal X-ray study; $T = 293\text{ K}$; mean $\sigma(\text{C}-\text{C}) = 0.006\text{ \AA}$; R factor = 0.032; wR factor = 0.092; data-to-parameter ratio = 17.4.

In the title organometallic complex, $[\text{Sb}(\text{C}_{20}\text{H}_{23}\text{N})\text{Cl}]$, the central antimony-containing part of the complex exhibits a pseudo-trigonal-bipyramidal geometry, where two C atoms and a lone electron pair of the Sb atom exist at the equatorial positions, while the N and Cl atoms are located at the apical positions, and a transannular interaction exists between the Sb and N atoms on 1,5-azastibocene. Intermolecular C–H···Cl hydrogen bonds are also observed.

Related literature

For general background, see: Yin *et al.* (2008); Chovancová *et al.* (2009); Opris *et al.* (2009); Svoboda *et al.* (2010); Tan & Zhang (2011). For related structures, see: Kakusawa *et al.* (2006); Xia *et al.* (2010).



Experimental

Crystal data

| | |
|--|--|
| $[\text{Sb}(\text{C}_{20}\text{H}_{23}\text{N})\text{Cl}]$ | $V = 1859.7(2)\text{ \AA}^3$ |
| $M_r = 434.59$ | $Z = 4$ |
| Monoclinic, $P2_1/c$ | Mo $K\alpha$ radiation |
| $a = 10.0771(7)\text{ \AA}$ | $\mu = 1.63\text{ mm}^{-1}$ |
| $b = 16.2881(12)\text{ \AA}$ | $T = 293\text{ K}$ |
| $c = 12.2040(9)\text{ \AA}$ | $0.37 \times 0.35 \times 0.21\text{ mm}$ |
| $\beta = 111.812(1)^{\circ}$ | |

Data collection

| | |
|--|--|
| Bruker SMART CCD area-detector diffractometer | 10058 measured reflections |
| Absorption correction: multi-scan (<i>SADABS</i> ; Sheldrick, 1999) | 3644 independent reflections |
| $T_{\min} = 0.653$, $T_{\max} = 1.000$ | 3107 reflections with $I > 2\sigma(I)$ |
| | $R_{\text{int}} = 0.047$ |

Refinement

| | |
|---------------------------------|---|
| $R[F^2 > 2\sigma(F^2)] = 0.032$ | 209 parameters |
| $wR(F^2) = 0.092$ | H-atom parameters constrained |
| $S = 1.05$ | $\Delta\rho_{\max} = 0.78\text{ e \AA}^{-3}$ |
| 3644 reflections | $\Delta\rho_{\min} = -0.55\text{ e \AA}^{-3}$ |

Table 1
Hydrogen-bond geometry (\AA , $^{\circ}$).

| $D-\text{H}\cdots A$ | $D-\text{H}$ | $\text{H}\cdots A$ | $D\cdots A$ | $D-\text{H}\cdots A$ |
|-----------------------------------|--------------|--------------------|-------------|----------------------|
| $C7-\text{H7A}\cdots\text{Cl}1^i$ | 0.97 | 2.80 | 3.695 (4) | 154 |

Symmetry code: (i) $-x + 1, -y + 2, -z$.

Data collection: *SMART* (Bruker, 1997); cell refinement: *SAINT* (Bruker, 1997); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXL97*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: VM2099).

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supplementary materials

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12-Chloro-6-cyclohexyl-5,6,7,12-tetrahydrodibenzo[*c,f*][1,5]azastibocene

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Comment

The chemistry of hypervalent compounds bearing heavier pnictogens (in particular Sb, Bi) has been studied intensively in recent years (Yin *et al.*, 2008; Chovancová *et al.*, 2009; Svoboda *et al.*, 2010; Tan & Zhang, 2011). Intramolecular interactions between antimony and sp^3 -nitrogen atoms have been widely reported (Kakusawa *et al.*, 2006; Opris *et al.*, 2009; Xia *et al.*, 2010). Here, we reported the crystal structure of the title organometallic complex (Fig. 1). The central antimony-containing part of the complex shows a distorted pseudo trigonal-bipyramidal structure. The C1, C8 atoms along with a lone electron pair of the Sb atom exist at the equatorial positions while the N1 and Cl1 atoms are located at the apical positions. The Sb—C1 and Sb—C8 distance is 2.144 (4) Å and 2.134 (3) Å, respectively. The C1—Sb—C8 angle is 98.17 (12) $^\circ$, while the N1—Sb—Cl1 angle is 162.92 (7) $^\circ$ (rather than 180 $^\circ$). The Sb—N1 distance (2.397 (3) Å) is shorter than the sum of the van der Waals radii of nitrogen and antimony atoms (3.74 Å) (Kakusawa *et al.*, 2006), indicating that coordination exists between the two atoms. The complex also displays intermolecular hydrogen-bonding interaction between the CH₂ groups and chlorine atom Cl1 (Table 1).

Experimental

N,N-bis(2-bromobenzyl)cyclohexanamine (2.186 g, 5.0 mmol) was allowed to react with n-BuLi (2.5 M, 4.0 ml, 10 mmol) at -50 °C, and the resulting solution was added to a mixture of SbCl₃ (1.141 g, 5.0 mmol) in Et₂O (80 ml) at -78 °C. The obtained mixture was gradually warmed to room temperature and stirred for 12 h. Then the solvent was removed under vacuum and the residue was extracted with toluene, and the insoluble material was removed by filtration. The organic layer was washed with de-ionized H₂O and dried over anhydrous Na₂SO₄. After the solvent was removed under reduced pressure, the residue was recrystallized from CH₂Cl₂/hexane to obtain the title compound in the form of colorless crystals.

Refinement

All H atoms were positioned geometrically and refined using a riding model, with C—H = 0.93 Å for aryl, 0.98 Å for methine and 0.97 Å for methylene H atoms, respectively. $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$ for all H atoms.

supplementary materials

Figures

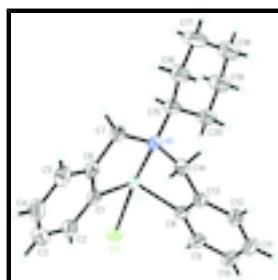


Fig. 1. The molecular structure of the title compound with atomic numbering scheme. Displacement ellipsoids are drawn at the 30% probability level.

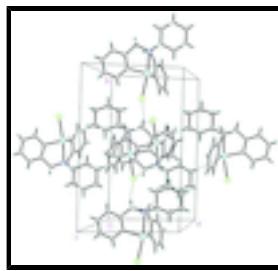


Fig. 2. A packing diagram of the title compound viewed down the a axis.

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Crystal data

| | |
|---|---|
| [Sb(C ₂₀ H ₂₃ N)Cl] | $F(000) = 872$ |
| $M_r = 434.59$ | $D_x = 1.552 \text{ Mg m}^{-3}$ |
| Monoclinic, $P2_1/c$ | Melting point: 527.15 K |
| Hall symbol: -P 2ybc | Mo $K\alpha$ radiation, $\lambda = 0.71073 \text{ \AA}$ |
| $a = 10.0771 (7) \text{ \AA}$ | Cell parameters from 5285 reflections |
| $b = 16.2881 (12) \text{ \AA}$ | $\theta = 4.4\text{--}55.7^\circ$ |
| $c = 12.2040 (9) \text{ \AA}$ | $\mu = 1.63 \text{ mm}^{-1}$ |
| $\beta = 111.812 (1)^\circ$ | $T = 293 \text{ K}$ |
| $V = 1859.7 (2) \text{ \AA}^3$ | Prismatic, colorless |
| $Z = 4$ | $0.37 \times 0.35 \times 0.21 \text{ mm}$ |

Data collection

| | |
|--|---|
| Bruker SMART CCD area-detector diffractometer | 3644 independent reflections |
| Radiation source: fine-focus sealed tube graphite | 3107 reflections with $I > 2\sigma(I)$ |
| Detector resolution: 10.00 pixels mm^{-1} | $R_{\text{int}} = 0.047$ |
| φ and ω scans | $\theta_{\text{max}} = 26.0^\circ, \theta_{\text{min}} = 2.2^\circ$ |
| Absorption correction: multi-scan (<i>SADABS</i> ; Sheldrick, 1999) | $h = -12 \rightarrow 7$ |
| $T_{\text{min}} = 0.653, T_{\text{max}} = 1.000$ | $k = -20 \rightarrow 19$ |
| 10058 measured reflections | $l = -15 \rightarrow 14$ |

Refinement

| | |
|--|---|
| Refinement on F^2 | Secondary atom site location: difference Fourier map |
| Least-squares matrix: full | Hydrogen site location: inferred from neighbouring sites |
| $R[F^2 > 2\sigma(F^2)] = 0.032$ | H-atom parameters constrained |
| $wR(F^2) = 0.092$ | $w = 1/[\sigma^2(F_o^2) + (0.0544P)^2 + 0.1065P]$ where $P = (F_o^2 + 2F_c^2)/3$ |
| $S = 1.05$ | $(\Delta/\sigma)_{\max} = 0.020$ |
| 3644 reflections | $\Delta\rho_{\max} = 0.78 \text{ e \AA}^{-3}$ |
| 209 parameters | $\Delta\rho_{\min} = -0.55 \text{ e \AA}^{-3}$ |
| 0 restraints | Extinction correction: <i>SHELXL97</i> (Sheldrick, 2008), $F_c^* = kF_c[1 + 0.001xF_c^2\lambda^3/\sin(2\theta)]^{1/4}$ |
| Primary atom site location: structure-invariant direct methods | Extinction coefficient: 0.0030 (4) |

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s 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 $F^2 > \sigma(F^2)$ 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 (\AA^2)

| | <i>x</i> | <i>y</i> | <i>z</i> | $U_{\text{iso}}^*/U_{\text{eq}}$ |
|-----|--------------|---------------|---------------|----------------------------------|
| Sb | 0.38578 (2) | 1.037779 (13) | 0.117005 (18) | 0.04278 (12) |
| Cl1 | 0.43763 (12) | 1.19188 (5) | 0.12783 (8) | 0.0597 (3) |
| N1 | 0.3869 (3) | 0.89526 (15) | 0.1662 (2) | 0.0400 (6) |
| C1 | 0.6082 (4) | 1.0063 (2) | 0.1912 (3) | 0.0459 (8) |
| C2 | 0.7215 (5) | 1.0616 (3) | 0.2281 (4) | 0.0615 (10) |
| H2 | 0.7033 | 1.1177 | 0.2246 | 0.074* |
| C3 | 0.8613 (5) | 1.0335 (3) | 0.2700 (5) | 0.0772 (14) |
| H3 | 0.9367 | 1.0707 | 0.2947 | 0.093* |
| C4 | 0.8882 (5) | 0.9511 (3) | 0.2750 (5) | 0.0734 (13) |
| H4 | 0.9822 | 0.9324 | 0.3034 | 0.088* |
| C5 | 0.7772 (4) | 0.8953 (3) | 0.2383 (4) | 0.0647 (11) |
| H5 | 0.7968 | 0.8393 | 0.2424 | 0.078* |
| C6 | 0.6365 (4) | 0.9224 (2) | 0.1953 (3) | 0.0475 (8) |
| C7 | 0.5159 (4) | 0.8623 (2) | 0.1497 (3) | 0.0505 (9) |
| H7A | 0.4943 | 0.8521 | 0.0666 | 0.061* |
| H7B | 0.5436 | 0.8107 | 0.1917 | 0.061* |

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|------|-------------|--------------|-------------|-------------|
| C8 | 0.3459 (3) | 1.04120 (18) | 0.2769 (3) | 0.0405 (7) |
| C9 | 0.3038 (4) | 1.1103 (2) | 0.3214 (3) | 0.0544 (9) |
| H9 | 0.2980 | 1.1605 | 0.2836 | 0.065* |
| C10 | 0.2703 (4) | 1.1057 (3) | 0.4213 (4) | 0.0662 (11) |
| H10 | 0.2449 | 1.1530 | 0.4515 | 0.079* |
| C11 | 0.2746 (4) | 1.0317 (3) | 0.4757 (4) | 0.0641 (11) |
| H11 | 0.2484 | 1.0286 | 0.5410 | 0.077* |
| C12 | 0.3175 (4) | 0.9621 (2) | 0.4344 (3) | 0.0530 (10) |
| H12 | 0.3233 | 0.9124 | 0.4733 | 0.064* |
| C13 | 0.3522 (3) | 0.96613 (19) | 0.3339 (3) | 0.0413 (7) |
| C14 | 0.4045 (4) | 0.89040 (19) | 0.2921 (3) | 0.0445 (7) |
| H14A | 0.5048 | 0.8823 | 0.3398 | 0.053* |
| H14B | 0.3523 | 0.8431 | 0.3033 | 0.053* |
| C15 | 0.2520 (4) | 0.8556 (2) | 0.0804 (3) | 0.0546 (9) |
| H15 | 0.2451 | 0.8713 | 0.0009 | 0.066* |
| C16 | 0.2535 (5) | 0.7640 (2) | 0.0841 (5) | 0.0772 (13) |
| H16A | 0.2598 | 0.7455 | 0.1615 | 0.093* |
| H16B | 0.3362 | 0.7435 | 0.0702 | 0.093* |
| C17 | 0.1141 (5) | 0.7300 (3) | -0.0122 (5) | 0.1008 (18) |
| H17A | 0.1129 | 0.7441 | -0.0898 | 0.121* |
| H17B | 0.1127 | 0.6706 | -0.0065 | 0.121* |
| C18 | -0.0157 (5) | 0.7648 (3) | 0.0023 (5) | 0.0968 (17) |
| H18A | -0.1006 | 0.7448 | -0.0607 | 0.116* |
| H18B | -0.0193 | 0.7461 | 0.0767 | 0.116* |
| C19 | -0.0150 (5) | 0.8545 (3) | 0.0004 (5) | 0.0885 (15) |
| H19A | -0.0989 | 0.8749 | 0.0125 | 0.106* |
| H19B | -0.0197 | 0.8731 | -0.0765 | 0.106* |
| C20 | 0.1202 (4) | 0.8898 (2) | 0.0964 (4) | 0.0636 (10) |
| H20A | 0.1197 | 0.9492 | 0.0907 | 0.076* |
| H20B | 0.1217 | 0.8751 | 0.1739 | 0.076* |

Atomic displacement parameters (\AA^2)

| | U^{11} | U^{22} | U^{33} | U^{12} | U^{13} | U^{23} |
|-----|--------------|--------------|--------------|--------------|--------------|--------------|
| Sb | 0.05674 (19) | 0.03541 (16) | 0.03702 (16) | 0.00115 (9) | 0.01840 (11) | -0.00007 (8) |
| Cl1 | 0.0872 (7) | 0.0321 (4) | 0.0647 (6) | -0.0061 (4) | 0.0338 (5) | -0.0033 (4) |
| N1 | 0.0432 (15) | 0.0373 (14) | 0.0405 (14) | 0.0032 (11) | 0.0168 (11) | -0.0007 (11) |
| C1 | 0.051 (2) | 0.0490 (19) | 0.0467 (19) | -0.0022 (16) | 0.0285 (16) | -0.0007 (15) |
| C2 | 0.067 (3) | 0.059 (2) | 0.067 (3) | -0.007 (2) | 0.035 (2) | -0.007 (2) |
| C3 | 0.057 (3) | 0.096 (4) | 0.084 (3) | -0.022 (2) | 0.032 (2) | -0.017 (3) |
| C4 | 0.051 (2) | 0.095 (4) | 0.081 (3) | 0.005 (2) | 0.034 (2) | -0.002 (3) |
| C5 | 0.058 (2) | 0.075 (3) | 0.069 (3) | 0.009 (2) | 0.033 (2) | 0.001 (2) |
| C6 | 0.053 (2) | 0.050 (2) | 0.048 (2) | 0.0037 (16) | 0.0287 (16) | 0.0017 (15) |
| C7 | 0.061 (2) | 0.0412 (18) | 0.059 (2) | 0.0029 (16) | 0.0332 (18) | -0.0030 (16) |
| C8 | 0.0390 (17) | 0.0445 (18) | 0.0372 (17) | 0.0020 (13) | 0.0134 (13) | -0.0059 (13) |
| C9 | 0.053 (2) | 0.058 (2) | 0.050 (2) | 0.0065 (17) | 0.0155 (16) | -0.0105 (16) |
| C10 | 0.056 (2) | 0.084 (3) | 0.061 (3) | 0.010 (2) | 0.0237 (19) | -0.026 (2) |
| C11 | 0.056 (2) | 0.094 (3) | 0.049 (2) | -0.003 (2) | 0.0279 (19) | -0.015 (2) |

| | | | | | | |
|-----|-------------|-------------|-------------|--------------|-------------|--------------|
| C12 | 0.044 (2) | 0.076 (3) | 0.040 (2) | -0.0035 (16) | 0.0162 (16) | 0.0037 (16) |
| C13 | 0.0347 (17) | 0.052 (2) | 0.0355 (17) | -0.0007 (13) | 0.0118 (13) | -0.0021 (13) |
| C14 | 0.0472 (18) | 0.0436 (18) | 0.0433 (18) | 0.0026 (14) | 0.0177 (14) | 0.0068 (14) |
| C15 | 0.057 (2) | 0.0428 (19) | 0.059 (2) | -0.0008 (16) | 0.0163 (18) | -0.0080 (16) |
| C16 | 0.069 (3) | 0.048 (2) | 0.105 (4) | -0.0050 (19) | 0.020 (2) | -0.012 (2) |
| C17 | 0.085 (4) | 0.065 (3) | 0.131 (5) | -0.017 (3) | 0.016 (3) | -0.041 (3) |
| C18 | 0.066 (3) | 0.080 (3) | 0.128 (5) | -0.020 (3) | 0.018 (3) | -0.021 (3) |
| C19 | 0.062 (3) | 0.076 (3) | 0.106 (4) | -0.004 (2) | 0.007 (3) | -0.016 (3) |
| C20 | 0.051 (2) | 0.057 (2) | 0.075 (3) | -0.0014 (18) | 0.0144 (19) | -0.0096 (19) |

Geometric parameters (\AA , $^\circ$)

| | | | |
|------------|-------------|---------------|-----------|
| Sb—C8 | 2.134 (3) | C10—H10 | 0.9300 |
| Sb—C1 | 2.144 (4) | C11—C12 | 1.374 (5) |
| Sb—N1 | 2.397 (3) | C11—H11 | 0.9300 |
| Sb—Cl1 | 2.5573 (9) | C12—C13 | 1.396 (5) |
| N1—C14 | 1.481 (4) | C12—H12 | 0.9300 |
| N1—C7 | 1.487 (4) | C13—C14 | 1.503 (4) |
| N1—C15 | 1.518 (4) | C14—H14A | 0.9700 |
| C1—C2 | 1.391 (5) | C14—H14B | 0.9700 |
| C1—C6 | 1.393 (5) | C15—C16 | 1.493 (5) |
| C2—C3 | 1.385 (6) | C15—C20 | 1.518 (5) |
| C2—H2 | 0.9300 | C15—H15 | 0.9800 |
| C3—C4 | 1.367 (6) | C16—C17 | 1.560 (6) |
| C3—H3 | 0.9300 | C16—H16A | 0.9700 |
| C4—C5 | 1.381 (6) | C16—H16B | 0.9700 |
| C4—H4 | 0.9300 | C17—C18 | 1.495 (7) |
| C5—C6 | 1.389 (5) | C17—H17A | 0.9700 |
| C5—H5 | 0.9300 | C17—H17B | 0.9700 |
| C6—C7 | 1.497 (5) | C18—C19 | 1.462 (6) |
| C7—H7A | 0.9700 | C18—H18A | 0.9700 |
| C7—H7B | 0.9700 | C18—H18B | 0.9700 |
| C8—C9 | 1.383 (4) | C19—C20 | 1.541 (6) |
| C8—C13 | 1.397 (4) | C19—H19A | 0.9700 |
| C9—C10 | 1.382 (6) | C19—H19B | 0.9700 |
| C9—H9 | 0.9300 | C20—H20A | 0.9700 |
| C10—C11 | 1.369 (6) | C20—H20B | 0.9700 |
| C8—Sb—C1 | 98.17 (12) | C11—C12—C13 | 119.9 (4) |
| C8—Sb—N1 | 77.37 (10) | C11—C12—H12 | 120.0 |
| C1—Sb—N1 | 75.86 (11) | C13—C12—H12 | 120.0 |
| C8—Sb—Cl1 | 91.80 (8) | C8—C13—C12 | 119.9 (3) |
| C1—Sb—Cl1 | 92.95 (10) | C8—C13—C14 | 120.4 (3) |
| N1—Sb—Cl1 | 162.92 (7) | C12—C13—C14 | 119.6 (3) |
| C14—N1—C7 | 110.3 (3) | N1—C14—C13 | 112.7 (3) |
| C14—N1—C15 | 115.1 (3) | N1—C14—H14A | 109.0 |
| C7—N1—C15 | 110.9 (3) | C13—C14—H14A | 109.0 |
| C14—N1—Sb | 107.40 (18) | N1—C14—H14B | 109.0 |
| C7—N1—Sb | 103.79 (19) | C13—C14—H14B | 109.0 |
| C15—N1—Sb | 108.64 (19) | H14A—C14—H14B | 107.8 |

supplementary materials

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|---------------|------------|-----------------|------------|
| C2—C1—C6 | 119.4 (4) | C16—C15—C20 | 111.2 (3) |
| C2—C1—Sb | 125.8 (3) | C16—C15—N1 | 114.1 (3) |
| C6—C1—Sb | 114.7 (2) | C20—C15—N1 | 110.9 (3) |
| C3—C2—C1 | 120.4 (4) | C16—C15—H15 | 106.7 |
| C3—C2—H2 | 119.8 | C20—C15—H15 | 106.7 |
| C1—C2—H2 | 119.8 | N1—C15—H15 | 106.7 |
| C4—C3—C2 | 119.9 (4) | C15—C16—C17 | 109.6 (4) |
| C4—C3—H3 | 120.0 | C15—C16—H16A | 109.7 |
| C2—C3—H3 | 120.0 | C17—C16—H16A | 109.7 |
| C3—C4—C5 | 120.6 (4) | C15—C16—H16B | 109.7 |
| C3—C4—H4 | 119.7 | C17—C16—H16B | 109.7 |
| C5—C4—H4 | 119.7 | H16A—C16—H16B | 108.2 |
| C4—C5—C6 | 120.2 (4) | C18—C17—C16 | 111.0 (4) |
| C4—C5—H5 | 119.9 | C18—C17—H17A | 109.4 |
| C6—C5—H5 | 119.9 | C16—C17—H17A | 109.4 |
| C5—C6—C1 | 119.5 (3) | C18—C17—H17B | 109.4 |
| C5—C6—C7 | 120.4 (3) | C16—C17—H17B | 109.4 |
| C1—C6—C7 | 120.0 (3) | H17A—C17—H17B | 108.0 |
| N1—C7—C6 | 110.1 (3) | C19—C18—C17 | 111.5 (4) |
| N1—C7—H7A | 109.6 | C19—C18—H18A | 109.3 |
| C6—C7—H7A | 109.6 | C17—C18—H18A | 109.3 |
| N1—C7—H7B | 109.6 | C19—C18—H18B | 109.3 |
| C6—C7—H7B | 109.6 | C17—C18—H18B | 109.3 |
| H7A—C7—H7B | 108.2 | H18A—C18—H18B | 108.0 |
| C9—C8—C13 | 118.7 (3) | C18—C19—C20 | 111.6 (4) |
| C9—C8—Sb | 124.7 (3) | C18—C19—H19A | 109.3 |
| C13—C8—Sb | 116.3 (2) | C20—C19—H19A | 109.3 |
| C10—C9—C8 | 120.9 (4) | C18—C19—H19B | 109.3 |
| C10—C9—H9 | 119.5 | C20—C19—H19B | 109.3 |
| C8—C9—H9 | 119.5 | H19A—C19—H19B | 108.0 |
| C11—C10—C9 | 120.0 (4) | C15—C20—C19 | 109.5 (4) |
| C11—C10—H10 | 120.0 | C15—C20—H20A | 109.8 |
| C9—C10—H10 | 120.0 | C19—C20—H20A | 109.8 |
| C10—C11—C12 | 120.4 (4) | C15—C20—H20B | 109.8 |
| C10—C11—H11 | 119.8 | C19—C20—H20B | 109.8 |
| C12—C11—H11 | 119.8 | H20A—C20—H20B | 108.2 |
| C8—Sb—N1—C14 | -17.0 (2) | C1—Sb—C8—C13 | -68.2 (3) |
| C1—Sb—N1—C14 | 84.9 (2) | N1—Sb—C8—C13 | 5.3 (2) |
| Cl1—Sb—N1—C14 | 34.7 (4) | Cl1—Sb—C8—C13 | -161.4 (2) |
| C8—Sb—N1—C7 | -133.8 (2) | C13—C8—C9—C10 | 0.8 (5) |
| C1—Sb—N1—C7 | -31.9 (2) | Sb—C8—C9—C10 | 175.3 (3) |
| Cl1—Sb—N1—C7 | -82.1 (3) | C8—C9—C10—C11 | -1.9 (6) |
| C8—Sb—N1—C15 | 108.1 (2) | C9—C10—C11—C12 | 2.6 (6) |
| C1—Sb—N1—C15 | -150.0 (2) | C10—C11—C12—C13 | -2.2 (6) |
| Cl1—Sb—N1—C15 | 159.8 (2) | C9—C8—C13—C12 | -0.5 (5) |
| C8—Sb—C1—C2 | -91.1 (3) | Sb—C8—C13—C12 | -175.4 (3) |
| N1—Sb—C1—C2 | -165.8 (3) | C9—C8—C13—C14 | -176.9 (3) |
| Cl1—Sb—C1—C2 | 1.2 (3) | Sb—C8—C13—C14 | 8.2 (4) |
| C8—Sb—C1—C6 | 92.5 (2) | C11—C12—C13—C8 | 1.2 (5) |

| | | | |
|--------------|------------|-----------------|------------|
| N1—Sb—C1—C6 | 17.8 (2) | C11—C12—C13—C14 | 177.6 (3) |
| Cl1—Sb—C1—C6 | -175.2 (2) | C7—N1—C14—C13 | 137.9 (3) |
| C6—C1—C2—C3 | -0.9 (6) | C15—N1—C14—C13 | -95.7 (3) |
| Sb—C1—C2—C3 | -177.2 (3) | Sb—N1—C14—C13 | 25.4 (3) |
| C1—C2—C3—C4 | 0.1 (7) | C8—C13—C14—N1 | -24.4 (4) |
| C2—C3—C4—C5 | 0.2 (8) | C12—C13—C14—N1 | 159.2 (3) |
| C3—C4—C5—C6 | 0.3 (7) | C14—N1—C15—C16 | -72.1 (4) |
| C4—C5—C6—C1 | -1.0 (6) | C7—N1—C15—C16 | 54.0 (4) |
| C4—C5—C6—C7 | 176.9 (4) | Sb—N1—C15—C16 | 167.5 (3) |
| C2—C1—C6—C5 | 1.3 (5) | C14—N1—C15—C20 | 54.4 (4) |
| Sb—C1—C6—C5 | 178.0 (3) | C7—N1—C15—C20 | -179.5 (3) |
| C2—C1—C6—C7 | -176.6 (3) | Sb—N1—C15—C20 | -66.0 (3) |
| Sb—C1—C6—C7 | 0.0 (4) | C20—C15—C16—C17 | 56.9 (5) |
| C14—N1—C7—C6 | -74.4 (3) | N1—C15—C16—C17 | -176.8 (4) |
| C15—N1—C7—C6 | 156.9 (3) | C15—C16—C17—C18 | -55.8 (6) |
| Sb—N1—C7—C6 | 40.4 (3) | C16—C17—C18—C19 | 56.2 (7) |
| C5—C6—C7—N1 | 151.3 (3) | C17—C18—C19—C20 | -57.1 (7) |
| C1—C6—C7—N1 | -30.7 (4) | C16—C15—C20—C19 | -57.3 (5) |
| C1—Sb—C8—C9 | 117.3 (3) | N1—C15—C20—C19 | 174.6 (3) |
| N1—Sb—C8—C9 | -169.3 (3) | C18—C19—C20—C15 | 57.0 (6) |
| Cl1—Sb—C8—C9 | 24.0 (3) | | |

Hydrogen-bond geometry (\AA , $^\circ$)

| $D\cdots H$ | $D—H$ | $H\cdots A$ | $D\cdots A$ | $D—H\cdots A$ |
|----------------------------------|-------|-------------|-------------|---------------|
| C7—H7A \cdots Cl1 ⁱ | 0.97 | 2.80 | 3.695 (4) | 154 |

Symmetry codes: (i) $-x+1, -y+2, -z$.

supplementary materials

Fig. 1

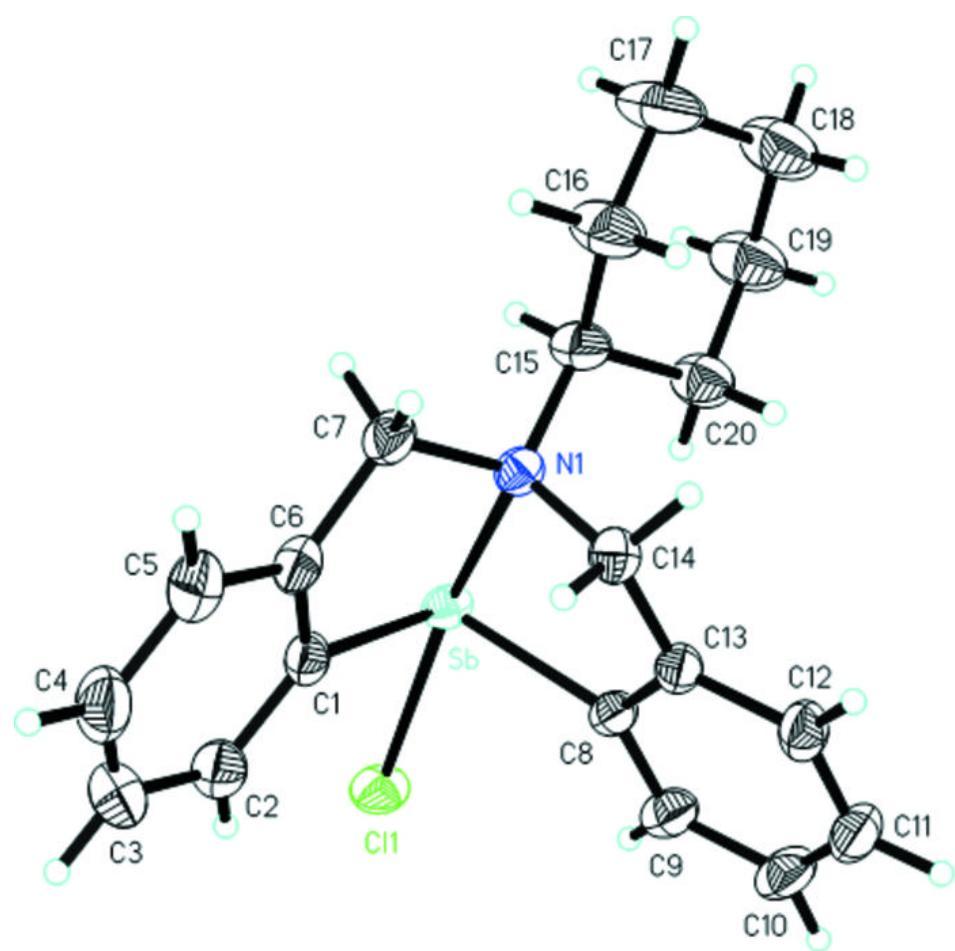


Fig. 2

