Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

1,10-Phenanthrolin-1-ium tetrabromido(1,10-phenanthroline-κ^2N,N')-antimonate(III) dichloromethane solvate

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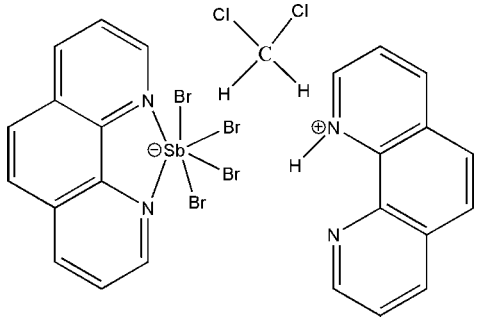
Received 13 November 2007; accepted 18 November 2007

Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.012 Å; R factor = 0.040; wR factor = 0.112; data-to-parameter ratio = 15.8.

In the title compound, $(C_{12}H_9N_2)[SbBr_4(C_{12}H_8N_2)]\cdot CH_2Cl_2$, the Sb atom is six-coordinated by two N atoms of 1,10-phenanthroline and four Br atoms with a distorted cis-SbN_2Br_4 octahedral geometry. An uncoordinated phenanthrolinium cation interacts with the anion via an N—H···Br hydrogen bond.

Related literature

The corresponding SbF_3 complex with 1,10-phenanthroline was reported by Bertazzi *et al.* (1983).



Experimental

Crystal data

$(C_{12}H_9N_2)[SbBr_4(C_{12}H_8N_2)]\cdot CH_2Cl_2$
M_r = 887.73
Triclinic, $P\overline{1}$
a = 11.471 (8) Å
b = 11.582 (9) Å
c = 12.154 (9) Å
α = 65.296 (8)°
β = 84.535 (9)°
γ = 88.244 (9)°
V = 1460.1 (19) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 6.62 mm^{-1}
T = 298 (2) K
0.18 × 0.14 × 0.08 mm

Data collection

Siemens SMART CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.382, T_{max} = 0.619
7508 measured reflections
5056 independent reflections
3378 reflections with $I > 2\sigma(I)$
R_{int} = 0.025

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.040
$wR(F^2)$ = 0.112
S = 1.01
5056 reflections
319 parameters
9 restraints
H-atom parameters constrained
$\Delta\rho_{max}$ = 1.81 e Å^{-3}
$\Delta\rho_{min}$ = −1.12 e Å^{-3}

Table 1
Selected bond lengths (Å).

Sb1—N1	2.312 (5)	Sb1—Br4	2.746 (2)
Sb1—N2	2.386 (6)	Sb1—Br2	2.784 (2)
Sb1—Br3	2.742 (2)	Sb1—Br1	3.035 (2)

Table 2
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
N4—H4···Br1^{i}	0.86	2.61	3.327 (6)	141

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

Data collection: *SMART* (Siemens, 1996); cell refinement: *SAINT* (Siemens, 1996); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997*a*); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997*a*); molecular graphics: *SHELXTL* (Sheldrick, 1997*b*); software used to prepare material for publication: *SHELXTL*.

The authors acknowledge the National Natural Science Foundation of China (grant No. 20771053), Shandong Province Science Foundation and the State Key Laboratory of Crystalline Materials, Shandong University, China.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HB2661).

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supplementary materials

Acta Cryst. (2007). E**63**, m3131 [**doi:10.1107/S1600536807060485**]

1,10-Phenanthrolin-1-ium tetrabromido(1,10-phenanthroline-κ^2N,N')antimonate(III) dichloromethane solvate

J. Zhai, H. Yin, F. Li and D. Wang

Comment

The adducts of SbF_3 with bi-and tri-dentate nitrogen donors such as 1,10-phenanthroline have been reported (Bertazzi *et al.*, 1983). As a continuation of this work, we report here the synthesis and crystal structure of the title compound, (I), (Fig. 1). In this complex, the antimony atom is six-coordinated by two N atoms of phen and four Br atoms with a distorted *cis*-SbN_2Br_4 octahedral geometry (Table 1).

The charge on the anion is balanced by a phenanthrolinium cation, which bonds to the anion *via* an N—H···Br hydrongen bond (Table 2, Fig. 2). A dichloromethane solvent molecule completes the structure.

Experimental

Antimony tribromide (180.7 mg, 0.5 mmol) was dissolved in methanol (20 ml) and 1,10-phenanthroline (99.1 mg, 0.5 mmol) was added with stirring at room temperature. The resulting orange–red solution was allowed to react for five hours and was then filtered. Yellow blocks of (I) were obtained by slow evaporation of an ethanol/dichloromethane (1:2 *v*/*v*) solution over a period of two weeks (yield 88%. m.p. 430 K). Anal. Calcd (%) for $C_{25}H_{19}Br_4C_{l2}N_4Sb$: C 33.82; H 2.16; N 6.31. Found: C 33.94; H 2.05; N 6.45.

Refinement

The H atom bound to N1 was located in a difference map, relocated in an idealized position (N—H = 0.86 Å) and refined as riding with U_{iso}(H) = 1.2U_{eq}(N). The C-bound H atoms were placed in calculated positions (C—H = 0.93–0.97 Å) and refined as riding with U_{iso}(H) = 1.2U_{eq}(C) or 1.5U_{eq}(C) for the methyl group.

Figures

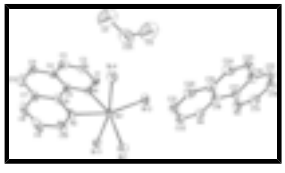

Fig. 1. The molecular structure of (I), with 50% probability displacement ellipsoids for non-H atoms.

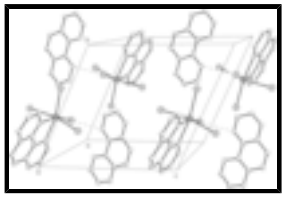

Fig. 2. Crystal packing of (I), showing the donor···acceptor contacts for the N—H···Br hydrogen bonds as dashed lines.

1,10-Phenanthrolin-1-ium tetrabromido(1,10-phenanthroline-κ^2*N*,*N*')antimonate(III) dichloromethane solvate

Crystal data

$(C_{12}H_9N_2)[SbBr(C_{12}H_8N_2)_4]\cdot CH_2Cl_2$	$Z = 2$
$M_r = 887.73$	$F_{000} = 844$
Triclinic, $P\overline{1}$	$D_x = 2.019$ Mg m^{-3}
Hall symbol: -P 1	Mo $K\alpha$ radiation $\lambda = 0.71073$ Å
$a = 11.471$ (8) Å	Cell parameters from 2399 reflections
$b = 11.582$ (9) Å	$\theta = 2.5$–25.3º
$c = 12.154$ (9) Å	$\mu = 6.62$ mm^{-1}
$\alpha = 65.296$ (8)º	$T = 298$ (2) K
$\beta = 84.535$ (9)º	Block, yellow
$\gamma = 88.244$ (9)º	0.18 × 0.14 × 0.08 mm
$V = 1460.1$ (19) Å^3	

Data collection

Siemens SMART CCD diffractometer	5056 independent reflections
Radiation source: fine-focus sealed tube	3378 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{int} = 0.025$
$T = 298$(2) K	$\theta_{max} = 25.0$º
ω scans	$\theta_{min} = 1.9$º
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	$h = -13\rightarrow12$
$T_{min} = 0.382$, $T_{max} = 0.619$	$k = -12\rightarrow13$
7508 measured reflections	$l = -11\rightarrow14$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.040$	H-atom parameters constrained
$wR(F^2) = 0.112$	$w = 1/[\sigma^2(F_o^2) + (0.0567P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.01$	$(\Delta/\sigma)_{max} = 0.004$
5056 reflections	$\Delta\rho_{max} = 1.81$ e Å^{-3}
319 parameters	$\Delta\rho_{min} = -1.11$ e Å^{-3}
9 restraints	Extinction correction: none
Primary atom site location: structure-invariant direct methods	

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

	x	*y*	*z*	U_{iso}*/U_{eq}
Sb1	0.74351 (4)	0.63084 (4)	0.10677 (4)	0.03882 (16)
Br1	0.49189 (7)	0.70645 (7)	0.05598 (7)	0.0551 (2)
Br2	0.77617 (8)	0.37772 (7)	0.14655 (8)	0.0652 (3)
Br3	0.83075 (7)	0.73103 (7)	−0.13286 (7)	0.0577 (2)
Br4	0.69659 (9)	0.56828 (8)	0.35088 (8)	0.0731 (3)
Cl1	1.0701 (5)	0.3451 (6)	0.5116 (5)	0.2246 (18)
Cl2	0.9780 (4)	0.1461 (6)	0.4665 (5)	0.2246 (18)
N1	0.9366 (4)	0.6463 (5)	0.1393 (5)	0.0398 (14)
N2	0.7796 (5)	0.8350 (5)	0.1013 (5)	0.0457 (15)
N3	0.4363 (5)	−0.0210 (6)	0.2689 (6)	0.0569 (17)
N4	0.4445 (5)	0.2270 (6)	0.2372 (5)	0.0500 (16)
H4	0.4677	0.2034	0.1804	0.060*
C1	1.0167 (6)	0.5575 (7)	0.1477 (7)	0.0488 (19)
H1	0.9951	0.4868	0.1363	0.059*
C2	1.1303 (7)	0.5663 (8)	0.1728 (7)	0.061 (2)
H2	1.1838	0.5025	0.1788	0.073*
C3	1.1625 (7)	0.6716 (9)	0.1886 (8)	0.065 (2)
H3	1.2386	0.6791	0.2055	0.078*
C4	1.0815 (6)	0.7671 (7)	0.1794 (6)	0.0477 (19)
C5	0.9690 (6)	0.7522 (6)	0.1522 (6)	0.0397 (17)
C6	0.8850 (6)	0.8488 (6)	0.1358 (6)	0.0405 (17)
C7	0.9154 (7)	0.9599 (6)	0.1483 (6)	0.052 (2)
C8	0.8308 (9)	1.0549 (7)	0.1263 (8)	0.069 (3)
H8	0.8469	1.1290	0.1344	0.083*
C9	0.7264 (9)	1.0383 (7)	0.0936 (8)	0.072 (3)
H9	0.6695	1.1006	0.0795	0.087*
C10	0.7036 (7)	0.9268 (7)	0.0809 (7)	0.059 (2)
H10	0.6313	0.9174	0.0568	0.071*
C11	1.1071 (8)	0.8766 (8)	0.1957 (8)	0.067 (2)
H11	1.1808	0.8848	0.2177	0.080*
C12	1.0297 (8)	0.9689 (8)	0.1808 (8)	0.071 (3)
H12	1.0506	1.0401	0.1918	0.085*
C13	0.4339 (8)	−0.1411 (8)	0.2883 (8)	0.069 (2)
H13	0.4627	−0.1633	0.2256	0.082*
C14	0.3901 (7)	−0.2374 (8)	0.3986 (9)	0.068 (2)
H14	0.3891	−0.3211	0.4074	0.081*
C15	0.3489 (7)	−0.2098 (8)	0.4932 (8)	0.064 (2)
H15	0.3213	−0.2738	0.5674	0.076*
C16	0.3490 (7)	−0.0823 (7)	0.4765 (7)	0.053 (2)
C17	0.3943 (6)	0.0078 (6)	0.3625 (6)	0.0425 (17)
C18	0.3990 (6)	0.1376 (7)	0.3458 (7)	0.0456 (18)
C19	0.3614 (6)	0.1753 (7)	0.4382 (7)	0.0499 (19)
C20	0.3712 (7)	0.3039 (8)	0.4138 (8)	0.062 (2)
H20	0.3445	0.3316	0.4732	0.075*
C21	0.4194 (7)	0.3887 (8)	0.3042 (8)	0.060 (2)

H21	0.4285	0.4734	0.2901	0.072*
C22	0.4549 (7)	0.3494 (7)	0.2141 (7)	0.055 (2)
H22	0.4857	0.4076	0.1380	0.066*
C23	0.3076 (7)	−0.0432 (8)	0.5711 (7)	0.060 (2)
H23	0.2756	−0.1032	0.6457	0.072*
C24	0.3148 (7)	0.0804 (8)	0.5525 (7)	0.064 (2)
H24	0.2890	0.1037	0.6151	0.076*
C25	0.9717 (14)	0.3019 (13)	0.4409 (15)	0.207 (7)
H25A	0.9846	0.3534	0.3541	0.248*
H25B	0.8937	0.3197	0.4679	0.248*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Sb1	0.0378 (3)	0.0331 (3)	0.0476 (3)	0.0008 (2)	−0.0008 (2)	−0.0195 (2)
Br1	0.0486 (5)	0.0630 (5)	0.0542 (5)	0.0059 (4)	−0.0029 (4)	−0.0257 (4)
Br2	0.0822 (6)	0.0437 (4)	0.0748 (6)	−0.0054 (4)	0.0056 (5)	−0.0319 (4)
Br3	0.0639 (5)	0.0546 (5)	0.0564 (5)	−0.0063 (4)	−0.0001 (4)	−0.0256 (4)
Br4	0.1065 (7)	0.0592 (5)	0.0570 (5)	−0.0105 (5)	0.0125 (5)	−0.0310 (4)
Cl1	0.173 (3)	0.312 (5)	0.205 (4)	−0.014 (3)	−0.007 (3)	−0.125 (4)
Cl2	0.173 (3)	0.312 (5)	0.205 (4)	−0.014 (3)	−0.007 (3)	−0.125 (4)
N1	0.037 (3)	0.042 (3)	0.051 (4)	0.004 (3)	−0.005 (3)	−0.029 (3)
N2	0.044 (4)	0.032 (3)	0.056 (4)	0.005 (3)	0.009 (3)	−0.016 (3)
N3	0.063 (4)	0.057 (4)	0.059 (4)	−0.003 (3)	0.004 (4)	−0.034 (3)
N4	0.051 (4)	0.054 (4)	0.049 (4)	−0.006 (3)	0.005 (3)	−0.028 (3)
C1	0.049 (5)	0.052 (4)	0.059 (5)	0.011 (4)	−0.013 (4)	−0.035 (4)
C2	0.046 (5)	0.071 (6)	0.079 (6)	0.021 (4)	−0.015 (4)	−0.043 (5)
C3	0.031 (4)	0.091 (7)	0.076 (6)	0.001 (4)	−0.004 (4)	−0.038 (5)
C4	0.043 (5)	0.059 (5)	0.044 (4)	−0.010 (4)	−0.002 (4)	−0.024 (4)
C5	0.044 (4)	0.037 (4)	0.036 (4)	−0.002 (3)	0.004 (3)	−0.015 (3)
C6	0.044 (4)	0.038 (4)	0.041 (4)	−0.003 (3)	0.009 (3)	−0.020 (3)
C7	0.069 (6)	0.036 (4)	0.046 (5)	−0.012 (4)	0.017 (4)	−0.018 (3)
C8	0.105 (8)	0.034 (4)	0.064 (6)	−0.011 (5)	0.029 (6)	−0.024 (4)
C9	0.088 (7)	0.033 (4)	0.081 (7)	0.011 (5)	0.018 (6)	−0.015 (4)
C10	0.044 (5)	0.040 (4)	0.076 (6)	0.005 (4)	0.010 (4)	−0.010 (4)
C11	0.063 (6)	0.071 (6)	0.070 (6)	−0.020 (5)	−0.011 (5)	−0.030 (5)
C12	0.085 (7)	0.056 (5)	0.081 (7)	−0.027 (5)	0.008 (6)	−0.040 (5)
C13	0.077 (6)	0.068 (6)	0.077 (6)	0.004 (5)	−0.005 (5)	−0.046 (5)
C14	0.071 (6)	0.051 (5)	0.084 (7)	−0.002 (4)	−0.002 (5)	−0.032 (5)
C15	0.069 (6)	0.052 (5)	0.057 (5)	−0.003 (4)	−0.010 (5)	−0.009 (4)
C16	0.054 (5)	0.055 (5)	0.047 (5)	−0.002 (4)	0.000 (4)	−0.020 (4)
C17	0.039 (4)	0.044 (4)	0.044 (4)	0.001 (3)	−0.001 (4)	−0.018 (4)
C18	0.042 (4)	0.049 (4)	0.046 (5)	0.006 (4)	−0.004 (4)	−0.022 (4)
C19	0.046 (5)	0.059 (5)	0.050 (5)	0.002 (4)	−0.001 (4)	−0.029 (4)
C20	0.076 (6)	0.066 (6)	0.060 (6)	0.014 (5)	−0.010 (5)	−0.042 (5)
C21	0.066 (6)	0.051 (5)	0.072 (6)	−0.001 (4)	−0.010 (5)	−0.033 (5)
C22	0.055 (5)	0.047 (5)	0.059 (5)	−0.001 (4)	−0.007 (4)	−0.017 (4)
C23	0.064 (5)	0.058 (5)	0.041 (5)	−0.009 (4)	0.009 (4)	−0.008 (4)

C24	0.072 (6)	0.075 (6)	0.046 (5)	0.005 (5)	0.007 (4)	−0.029 (5)
C25	0.173 (10)	0.174 (10)	0.200 (10)	−0.040 (8)	−0.051 (8)	0.004 (8)

Geometric parameters (Å, °)

Sb1—N1	2.312 (5)	C8—H8	0.9300
Sb1—N2	2.386 (6)	C9—C10	1.399 (11)
Sb1—Br3	2.742 (2)	C9—H9	0.9300
Sb1—Br4	2.746 (2)	C10—H10	0.9300
Sb1—Br2	2.784 (2)	C11—C12	1.332 (11)
Sb1—Br1	3.035 (2)	C11—H11	0.9300
Cl1—C25	1.686 (13)	C12—H12	0.9300
Cl2—C25	1.698 (13)	C13—C14	1.396 (11)
N1—C1	1.336 (8)	C13—H13	0.9300
N1—C5	1.366 (8)	C14—C15	1.357 (11)
N2—C10	1.310 (8)	C14—H14	0.9300
N2—C6	1.352 (8)	C15—C16	1.403 (10)
N3—C13	1.311 (10)	C15—H15	0.9300
N3—C17	1.356 (8)	C16—C17	1.402 (10)
N4—C22	1.332 (9)	C16—C23	1.440 (10)
N4—C18	1.359 (9)	C17—C18	1.433 (10)
N4—H4	0.8600	C18—C19	1.394 (9)
C1—C2	1.382 (10)	C19—C20	1.397 (10)
C1—H1	0.9300	C19—C24	1.429 (10)
C2—C3	1.378 (11)	C20—C21	1.358 (11)
C2—H2	0.9300	C20—H20	0.9300
C3—C4	1.398 (10)	C21—C22	1.376 (10)
C3—H3	0.9300	C21—H21	0.9300
C4—C5	1.395 (9)	C22—H22	0.9300
C4—C11	1.406 (10)	C23—C24	1.356 (11)
C5—C6	1.416 (9)	C23—H23	0.9300
C6—C7	1.414 (9)	C24—H24	0.9300
C7—C8	1.400 (11)	C25—H25A	0.9700
C7—C12	1.425 (11)	C25—H25B	0.9700
C8—C9	1.340 (12)		
N1—Sb1—N2	70.23 (19)	N2—C10—C9	123.0 (8)
N1—Sb1—Br3	83.16 (14)	N2—C10—H10	118.5
N2—Sb1—Br3	86.42 (14)	C9—C10—H10	118.5
N1—Sb1—Br4	85.43 (14)	C12—C11—C4	122.5 (8)
N2—Sb1—Br4	82.19 (14)	C12—C11—H11	118.8
Br3—Sb1—Br4	166.06 (3)	C4—C11—H11	118.8
N1—Sb1—Br2	90.30 (13)	C11—C12—C7	120.8 (7)
N2—Sb1—Br2	160.06 (15)	C11—C12—H12	119.6
Br3—Sb1—Br2	95.76 (3)	C7—C12—H12	119.6
Br4—Sb1—Br2	92.28 (3)	N3—C13—C14	123.1 (8)
N1—Sb1—Br1	159.61 (13)	N3—C13—H13	118.5
N2—Sb1—Br1	89.39 (15)	C14—C13—H13	118.5
Br3—Sb1—Br1	95.34 (4)	C15—C14—C13	120.5 (8)
Br4—Sb1—Br1	92.57 (4)	C15—C14—H14	119.8

Br2—Sb1—Br1	110.07 (3)	C13—C14—H14	119.8
C1—N1—C5	118.8 (6)	C14—C15—C16	118.5 (7)
C1—N1—Sb1	123.8 (4)	C14—C15—H15	120.8
C5—N1—Sb1	117.3 (4)	C16—C15—H15	120.8
C10—N2—C6	118.6 (6)	C17—C16—C15	117.0 (7)
C10—N2—Sb1	126.1 (5)	C17—C16—C23	120.3 (7)
C6—N2—Sb1	114.9 (4)	C15—C16—C23	122.8 (7)
C13—N3—C17	116.9 (7)	N3—C17—C16	124.1 (7)
C22—N4—C18	123.4 (6)	N3—C17—C18	118.4 (6)
C22—N4—H4	118.3	C16—C17—C18	117.5 (6)
C18—N4—H4	118.3	N4—C18—C19	118.6 (7)
N1—C1—C2	122.7 (7)	N4—C18—C17	119.1 (6)
N1—C1—H1	118.7	C19—C18—C17	122.3 (7)
C2—C1—H1	118.7	C18—C19—C20	118.1 (7)
C3—C2—C1	118.7 (7)	C18—C19—C24	118.2 (7)
C3—C2—H2	120.7	C20—C19—C24	123.6 (7)
C1—C2—H2	120.7	C21—C20—C19	120.7 (7)
C2—C3—C4	120.3 (7)	C21—C20—H20	119.7
C2—C3—H3	119.8	C19—C20—H20	119.7
C4—C3—H3	119.8	C20—C21—C22	120.0 (7)
C5—C4—C3	117.7 (7)	C20—C21—H21	120.0
C5—C4—C11	118.6 (7)	C22—C21—H21	120.0
C3—C4—C11	123.8 (7)	N4—C22—C21	119.1 (7)
N1—C5—C4	121.8 (6)	N4—C22—H22	120.4
N1—C5—C6	118.1 (6)	C21—C22—H22	120.4
C4—C5—C6	120.1 (6)	C24—C23—C16	120.6 (7)
N2—C6—C7	121.8 (6)	C24—C23—H23	119.7
N2—C6—C5	118.4 (6)	C16—C23—H23	119.7
C7—C6—C5	119.7 (7)	C23—C24—C19	121.1 (7)
C8—C7—C6	117.5 (8)	C23—C24—H24	119.4
C8—C7—C12	124.2 (8)	C19—C24—H24	119.4
C6—C7—C12	118.3 (7)	Cl1—C25—Cl2	113.9 (9)
C9—C8—C7	119.6 (8)	Cl1—C25—H25A	108.8
C9—C8—H8	120.2	Cl2—C25—H25A	108.8
C7—C8—H8	120.2	Cl1—C25—H25B	108.8
C8—C9—C10	119.6 (8)	Cl2—C25—H25B	108.8
C8—C9—H9	120.2	H25A—C25—H25B	107.7
C10—C9—H9	120.2		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N4—H4···Br1^{i}	0.86	2.61	3.327 (6)	141

Symmetry codes: (i) −*x*+1, −*y*+1, −*z*.

Fig. 1

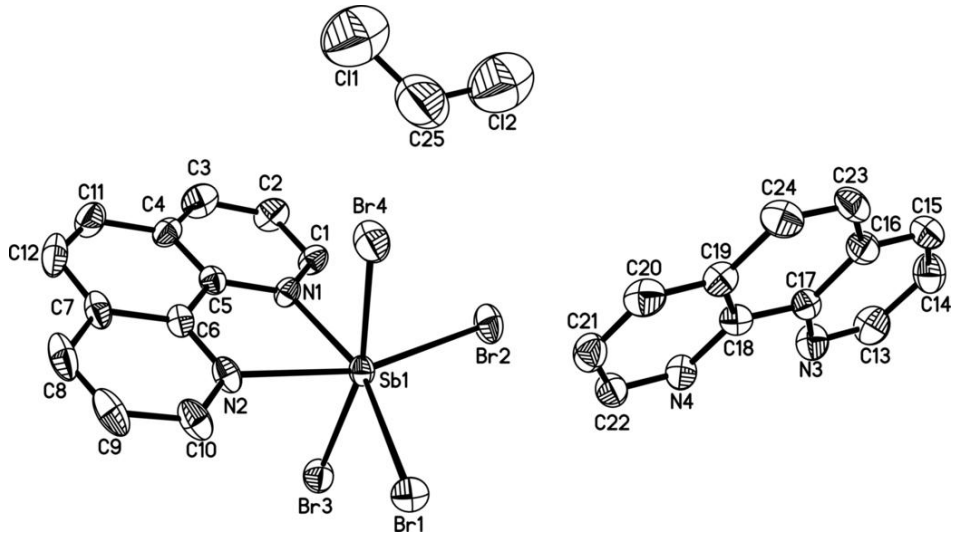
Cl1
C25
Cl2
C3
C11
C4
C2
Br4
C1
C12
C5
N1
C7
C6
Br2
C8
Sb1
N2
C9
C10
Br3
Br1
C24
C23
C15
C16
C19
C20
C17
C14
C21
C18
N3
C13
N4
C22

Fig. 2