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# *catena*-Poly[1,10-phenanthrolin-1-ium [[dichloridoantimonate(III)]-di-$\mu$-chlorido] methanol solvate]

**Jun Zhai, Handong Yin,* Feng Li and Daqi Wang**

College of Chemistry and Chemical Engineering, Liaocheng University, Shandong 252059, People's Republic of China
Correspondence e-mail: handongyin@163.com

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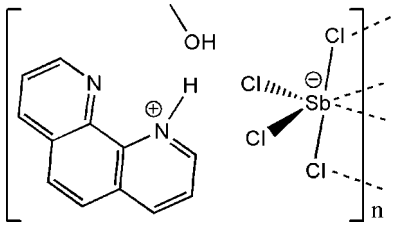
Key indicators: single-crystal X-ray study; $T$ = 298 K; mean $\sigma$(C–C) = 0.006 Å; $R$ factor = 0.028; $wR$ factor = 0.077; data-to-parameter ratio = 16.0.

The asymmetric unit of the title compound, $\{(C_{12}H_9N_2)[SbCl_4]\cdot CH_3OH\}_n$, comprises a 1,10-phenanthrolinium cation, a tetrachloridoantimonate anion and a methanol solvent molecule. Centrosymmetrically-related anions are linked by short Sb···Cl intermolecular contacts into $[Sb_2Cl_8]^{2-}$ dimeric units to form one-dimensional polymeric chains running parallel to the $a$ axis. The crystal structure is stabilized by O—H···Cl and N—H···O hydrogen-bonding interactions.

## Related literature

For the corresponding complex of $SbF_3$ with 1,10-phenanthroline, see: Bertazzi *et al.* (1983).




## Experimental

### *Crystal data*

$(C_{12}H_9N_2)[SbCl_4]\cdot CH_4O$
$M_r$ = 476.80
Triclinic, $P\overline{1}$
$a$ = 7.5228 (10) Å
$b$ = 10.0340 (14) Å
$c$ = 12.3933 (19) Å
$\alpha$ = 78.242 (3)°
$\beta$ = 73.266 (2)°
$\gamma$ = 73.751 (2)°
$V$ = 852.3 (2) Å$^3$
$Z$ = 2
Mo $K\alpha$ radiation
$\mu$ = 2.24 mm$^{-1}$
$T$ = 298 (2) K
0.43 × 0.18 × 0.16 mm

### *Data collection*

Bruker SMART CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{min}$ = 0.436, $T_{max}$ = 0.701
4451 measured reflections
2947 independent reflections
2584 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.017

### *Refinement*

$R[F^2 > 2\sigma(F^2)]$ = 0.028
$wR(F^2)$ = 0.077
$S$ = 1.01
2947 reflections
184 parameters
1 restraint
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.74 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.64 e Å$^{-3}$

**Table 1**
Hydrogen-bond geometry (Å, °).

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| N1—H1$A$···O1$^{i}$ | 0.86 | 1.88 | 2.685 (4) | 155 |
| O1—H1···Cl2 | 0.82 | 2.34 | 3.143 (4) | 166 |

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

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*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: RZ2155).

## References

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Siemens (1996). *SMART* and *SAINT*. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.

  

# supplementary materials

*Acta Cryst.* (2007). E**63**, m2165 [ **doi:10.1107/S1600536807034344** ]

# *catena*-Poly[1,10-phenanthrolin-1-ium [[dichloridoantimonate(III)]-di-$\mu$-chlorido] methanol solvate]

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

## Comment

The acceptor ability of $SbF_3$ towards neutral ligands has been previously investigated. Adducts with bi- and tri-dentate nitrogen donor atoms such as 1,10-phenanthroline have been obtainted (Bertazzi *et al.*, 1983), but the corresponding complex of $SbCl_3$ has not yet been reported. As a contribution to the chemistry of antimony complexes with neutral ligands, we report here the synthesis and crystal structure of the title compound.

The title compound, $\{C_{12}H_9N_2^+\cdot SbCl_4^-\cdot CH_3OH\}_n$ (Fig.1), consists of tetrachloroantimonate(III) anions, 1,10-phenanthrolinium cations and methanol solvent molecules. In the crystal, centrosymmetrically related anions are linked by short intermolecular Sb1···Cl2$^{i}$ and Cl1···Sb1$^{ii}$ contacts [symmetry codes: (i) $1-x, 1-y, 1-z$; (ii) $2-x, 1-y, 1-z$] into $[Sb_2Cl_8]^{2-}$ dianions made up from two octahedra sharing a common edge, forming one-dimensional polymeric chains running parallel to the *a* axis. The *cis* and *trans* Cl—Sb—Cl bond angles within the distorted octahedral coordination geometry about Sb are in the range 81.90 (3)–99.14 (3)° and 171.36 (4)–178.83 (3)°, respectively. The Sb—Cl distances also vary with the role they play in the structure, the terminal Sb1—Cl3 [2.3970 (11) Å] and Sb1—Cl4 [2.4248 (11) Å] bonds being shorter than those involving the bridging Cl atoms which range from 2.5375 (11) Å to 2.7890 (12) Å. The crystal structure is further stabilized by O—H···Cl and N—H···O hydrogen bonds (Fig. 2, Table 1).

## Experimental

Antimony trichloride (0.5 mmol) was dissolved in methanol (20 ml) and 1,10-phenanthroline (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. Orange crystals suitable for X-ray analysis were obtained by slow evaporation of a methanol/dichloromethane (1:2 *v*/*v*) solution over a period of two weeks (yield 85%. m.p. 400k). Anal. Calcd (%) for $C_{13}H_{13}Cl_4N_2OSb$ (Mr = 476.80): C, 32.75; H, 2.75; N, 5.88. Found (%): C, 32.81; H, 2.71; N, 5.81.

## Refinement

The H atom bound to N1 was located in a difference map and refined with a distance restraint (N—H = 0.86 (2) Å). All other H atoms were placed in calculated positions, with C—H = 0.93–0.96 Å, O—H = 0.82 Å, and treated as riding on their parent atoms, with $U_{iso}$(H) = 1.2 $U_{eq}$(C, O) or 1.5 $U_{eq}$(C) for the methyl group.

**Figures**

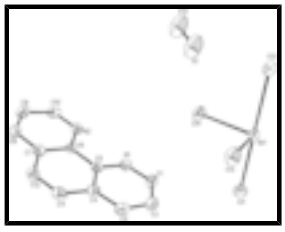

Fig. 1. The molecular structure of the title compound, with atom labels and 50% probability displacement ellipsoids. H atoms are omitted for clarity.

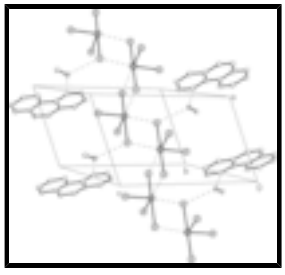

Fig. 2. Crystal packing of the title compound, showing a one-dimensional polymeric chain formed by O—H···Cl, N—H···O hydrogen bonds and intermolecular Sb···Cl contacts (dashed lines). H atoms are omitted for clarity.

## *catena*-Poly[1,10-phenanthrolin-1-ium [[dichloridoantimonate(III)]-di- μ-chlorido] methanol solvate]

*Crystal data*

| | |
|---|---|
| $(C_{12}H_9N_2)[SbCl_4]\cdot CH_4O$ | $Z = 2$ |
| $M_r = 476.80$ | $F_{000} = 464$ |
| Triclinic, $P\overline{1}$ | $D_x = 1.858$ Mg m$^{-3}$ |
| Hall symbol: -P 1 | Mo $K\alpha$ radiation<br>$\lambda = 0.71073$ Å |
| $a = 7.5228$ (10) Å | Cell parameters from 3120 reflections |
| $b = 10.0340$ (14) Å | $\theta = 2.6$–$27.8$º |
| $c = 12.3933$ (19) Å | $\mu = 2.24$ mm$^{-1}$ |
| $\alpha = 78.242$ (3)º | $T = 298$ (2) K |
| $\beta = 73.266$ (2)º | Block, orange |
| $\gamma = 73.751$ (2)º | 0.43 × 0.18 × 0.16 mm |
| $V = 852.3$ (2) Å$^3$ | |

*Data collection*

| | |
|---|---|
| Bruker SMART CCD area-detector diffractometer | 2947 independent reflections |
| Radiation source: fine-focus sealed tube | 2584 reflections with $I > 2\sigma(I)$ |
| Monochromator: graphite | $R_{int} = 0.017$ |
| $T = 298(2)$ K | $\theta_{max} = 25.0$º |
| φ and ω scans | $\theta_{min} = 1.7$º |
| Absorption correction: multi-scan (SADABS; Sheldrick, 1996) | $h = -8\rightarrow8$ |
| $T_{min} = 0.436$, $T_{max} = 0.701$ | $k = -11\rightarrow11$ |
| 4451 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 |
| $R[F^2 > 2\sigma(F^2)] = 0.028$ | H-atom parameters constrained |
| $wR(F^2) = 0.077$ | $w = 1/[\sigma^2(F_o^2) + (0.043P)^2 + 0.4963P]$ where $P = (F_o^2 + 2F_c^2)/3$ |
| $S = 1.01$ | $(\Delta/\sigma)_{max} = 0.001$ |
| 2947 reflections | $\Delta\rho_{max} = 0.74$ e Å$^{-3}$ |
| 184 parameters | $\Delta\rho_{min} = -0.63$ e Å$^{-3}$ |
| 1 restraint | 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 | 0.68935 (3) | 0.57643 (2) | 0.559783 (19) | 0.03694 (11) |
| Cl1 | 0.95856 (16) | 0.69961 (11) | 0.46610 (9) | 0.0530 (3) |
| Cl2 | 0.38832 (17) | 0.44568 (12) | 0.66090 (9) | 0.0555 (3) |
| Cl3 | 0.46181 (17) | 0.79796 (12) | 0.55715 (10) | 0.0607 (3) |
| Cl4 | 0.71999 (18) | 0.58182 (13) | 0.74824 (9) | 0.0602 (3) |
| N1 | 0.9207 (4) | 0.8500 (3) | 0.8227 (3) | 0.0417 (7) |
| H1A | 0.9810 | 0.7763 | 0.8582 | 0.050* |
| N2 | 0.8913 (5) | 0.8228 (3) | 1.0520 (3) | 0.0415 (7) |
| O1 | 0.1745 (6) | 0.6117 (4) | 0.8691 (3) | 0.0868 (10) |
| H1 | 0.2104 | 0.5678 | 0.8146 | 0.130* |
| C1 | 0.9356 (6) | 0.8526 (5) | 0.7135 (4) | 0.0513 (10) |
| H1B | 1.0100 | 0.7761 | 0.6767 | 0.062* |
| C2 | 0.8398 (6) | 0.9697 (5) | 0.6539 (4) | 0.0528 (11) |
| H2 | 0.8466 | 0.9710 | 0.5776 | 0.063* |
| C3 | 0.7359 (6) | 1.0829 (5) | 0.7076 (4) | 0.0495 (10) |
| H3 | 0.6746 | 1.1624 | 0.6670 | 0.059* |
| C4 | 0.7203 (5) | 1.0806 (4) | 0.8233 (3) | 0.0394 (8) |
| C5 | 0.8147 (5) | 0.9581 (4) | 0.8822 (3) | 0.0350 (8) |
| C6 | 0.8008 (5) | 0.9454 (4) | 1.0008 (3) | 0.0339 (8) |
| C7 | 0.6897 (5) | 1.0596 (4) | 1.0593 (3) | 0.0392 (9) |
| C8 | 0.6761 (6) | 1.0441 (4) | 1.1762 (3) | 0.0468 (10) |
| H8 | 0.6064 | 1.1172 | 1.2180 | 0.056* |
| C9 | 0.7660 (6) | 0.9209 (5) | 1.2281 (4) | 0.0532 (11) |
| H9 | 0.7571 | 0.9083 | 1.3058 | 0.064* |
| C10 | 0.8719 (6) | 0.8133 (4) | 1.1624 (3) | 0.0478 (10) |
| H10 | 0.9325 | 0.7298 | 1.1990 | 0.057* |
| C11 | 0.6112 (6) | 1.1946 (4) | 0.8862 (4) | 0.0469 (10) |
| H11 | 0.5492 | 1.2769 | 0.8489 | 0.056* |
| C12 | 0.5970 (6) | 1.1848 (4) | 0.9975 (4) | 0.0463 (10) |

| | | | | |
|---|---|---|---|---|
| H12 | 0.5256 | 1.2605 | 1.0359 | 0.056* |
| C13 | 0.2317 (9) | 0.5266 (5) | 0.9613 (4) | 0.0868 (10) |
| H13A | 0.3662 | 0.5159 | 0.9511 | 0.130* |
| H13B | 0.2060 | 0.4364 | 0.9678 | 0.130* |
| H13C | 0.1628 | 0.5681 | 1.0292 | 0.130* |

*Atomic displacement parameters (Å$^2$)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| Sb1 | 0.03766 (17) | 0.04080 (16) | 0.03077 (16) | −0.00679 (11) | −0.00700 (11) | −0.00671 (10) |
| Cl1 | 0.0555 (7) | 0.0497 (6) | 0.0502 (6) | −0.0168 (5) | −0.0071 (5) | −0.0011 (5) |
| Cl2 | 0.0637 (7) | 0.0637 (7) | 0.0374 (5) | −0.0188 (5) | −0.0062 (5) | −0.0066 (5) |
| Cl3 | 0.0650 (7) | 0.0510 (6) | 0.0553 (7) | 0.0096 (5) | −0.0156 (5) | −0.0148 (5) |
| Cl4 | 0.0736 (8) | 0.0784 (8) | 0.0364 (6) | −0.0285 (6) | −0.0178 (5) | −0.0041 (5) |
| N1 | 0.0424 (19) | 0.0375 (17) | 0.0427 (19) | −0.0106 (14) | −0.0055 (15) | −0.0053 (14) |
| N2 | 0.0410 (19) | 0.0375 (17) | 0.0441 (19) | −0.0092 (14) | −0.0100 (15) | −0.0018 (14) |
| O1 | 0.100 (2) | 0.0662 (18) | 0.0658 (19) | 0.0277 (16) | −0.0156 (17) | −0.0179 (15) |
| C1 | 0.051 (3) | 0.059 (3) | 0.043 (2) | −0.016 (2) | −0.0009 (19) | −0.016 (2) |
| C2 | 0.056 (3) | 0.067 (3) | 0.039 (2) | −0.021 (2) | −0.008 (2) | −0.010 (2) |
| C3 | 0.048 (2) | 0.055 (2) | 0.045 (2) | −0.018 (2) | −0.0137 (19) | 0.006 (2) |
| C4 | 0.032 (2) | 0.042 (2) | 0.041 (2) | −0.0125 (16) | −0.0052 (16) | −0.0007 (17) |
| C5 | 0.0283 (19) | 0.0358 (19) | 0.041 (2) | −0.0110 (15) | −0.0042 (15) | −0.0060 (16) |
| C6 | 0.0317 (19) | 0.0330 (18) | 0.037 (2) | −0.0116 (15) | −0.0051 (15) | −0.0049 (15) |
| C7 | 0.031 (2) | 0.041 (2) | 0.044 (2) | −0.0099 (16) | −0.0040 (17) | −0.0081 (17) |
| C8 | 0.046 (2) | 0.055 (2) | 0.040 (2) | −0.0126 (19) | −0.0055 (18) | −0.0148 (19) |
| C9 | 0.055 (3) | 0.071 (3) | 0.037 (2) | −0.024 (2) | −0.0076 (19) | −0.006 (2) |
| C10 | 0.047 (2) | 0.050 (2) | 0.043 (2) | −0.0146 (19) | −0.0115 (19) | 0.0056 (19) |
| C11 | 0.041 (2) | 0.039 (2) | 0.056 (3) | −0.0044 (17) | −0.0129 (19) | 0.0009 (18) |
| C12 | 0.041 (2) | 0.038 (2) | 0.055 (3) | −0.0020 (17) | −0.0085 (19) | −0.0127 (18) |
| C13 | 0.100 (2) | 0.0662 (18) | 0.0658 (19) | 0.0277 (16) | −0.0156 (17) | −0.0179 (15) |

*Geometric parameters (Å, °)*

| | | | |
|---|---|---|---|
| Sb1—Cl3 | 2.3970 (11) | C3—C4 | 1.402 (6) |
| Sb1—Cl4 | 2.4248 (11) | C3—H3 | 0.9300 |
| Sb1—Cl1 | 2.5375 (11) | C4—C5 | 1.411 (5) |
| Sb1—Cl2 | 2.7890 (12) | C4—C11 | 1.435 (5) |
| Sb1—Cl2$^{i}$ | 3.0184 (12) | C5—C6 | 1.425 (5) |
| Sb1—Cl1$^{ii}$ | 3.2492 (12) | C6—C7 | 1.411 (5) |
| Cl1—Sb1$^{ii}$ | 3.2492 (12) | C7—C8 | 1.402 (5) |
| Cl2—Sb1$^{i}$ | 3.0184 (12) | C7—C12 | 1.441 (5) |
| N1—C1 | 1.322 (5) | C8—C9 | 1.363 (6) |
| N1—C5 | 1.363 (5) | C8—H8 | 0.9300 |
| N1—H1A | 0.8600 | C9—C10 | 1.402 (6) |
| N2—C10 | 1.320 (5) | C9—H9 | 0.9300 |
| N2—C6 | 1.357 (5) | C10—H10 | 0.9300 |
| O1—C13 | 1.378 (6) | C11—C12 | 1.338 (6) |
| O1—H1 | 0.8200 | C11—H11 | 0.9300 |

| | | | |
|---|---|---|---|
| C1—C2 | 1.388 (6) | C12—H12 | 0.9300 |
| C1—H1B | 0.9300 | C13—H13A | 0.9600 |
| C2—C3 | 1.362 (6) | C13—H13B | 0.9600 |
| C2—H2 | 0.9300 | C13—H13C | 0.9600 |
| Cl3—Sb1—Cl4 | 93.24 (4) | C3—C4—C11 | 123.9 (4) |
| Cl3—Sb1—Cl1 | 89.95 (4) | C5—C4—C11 | 118.0 (3) |
| Cl4—Sb1—Cl1 | 91.93 (4) | N1—C5—C4 | 118.4 (3) |
| Cl3—Sb1—Cl2 | 88.98 (4) | N1—C5—C6 | 120.0 (3) |
| Cl4—Sb1—Cl2 | 88.61 (4) | C4—C5—C6 | 121.7 (3) |
| Cl1—Sb1—Cl2 | 178.83 (3) | N2—C6—C7 | 123.5 (3) |
| Cl3—Sb1—Cl2$^{i}$ | 86.39 (4) | N2—C6—C5 | 118.2 (3) |
| Cl4—Sb1—Cl2$^{i}$ | 173.22 (4) | C7—C6—C5 | 118.3 (3) |
| Cl1—Sb1—Cl2$^{i}$ | 94.84 (3) | C8—C7—C6 | 117.3 (3) |
| Cl2—Sb1—Cl2$^{i}$ | 84.62 (3) | C8—C7—C12 | 123.4 (4) |
| Cl3—Sb1—Cl1$^{ii}$ | 171.36 (4) | C6—C7—C12 | 119.3 (3) |
| Cl4—Sb1—Cl1$^{ii}$ | 89.92 (4) | C9—C8—C7 | 119.4 (4) |
| Cl1—Sb1—Cl1$^{ii}$ | 81.90 (3) | C9—C8—H8 | 120.3 |
| Cl2—Sb1—Cl1$^{ii}$ | 99.14 (3) | C7—C8—H8 | 120.3 |
| Cl2$^{i}$—Sb1—Cl1$^{ii}$ | 91.41 (3) | C8—C9—C10 | 118.9 (4) |
| Sb1—Cl1—Sb1$^{ii}$ | 98.10 (3) | C8—C9—H9 | 120.6 |
| Sb1—Cl2—Sb1$^{i}$ | 95.38 (3) | C10—C9—H9 | 120.6 |
| C1—N1—C5 | 123.4 (3) | N2—C10—C9 | 124.2 (4) |
| C1—N1—H1A | 118.3 | N2—C10—H10 | 117.9 |
| C5—N1—H1A | 118.3 | C9—C10—H10 | 117.9 |
| C10—N2—C6 | 116.7 (3) | C12—C11—C4 | 121.2 (4) |
| C13—O1—H1 | 109.5 | C12—C11—H11 | 119.4 |
| N1—C1—C2 | 119.7 (4) | C4—C11—H11 | 119.4 |
| N1—C1—H1B | 120.1 | C11—C12—C7 | 121.6 (4) |
| C2—C1—H1B | 120.1 | C11—C12—H12 | 119.2 |
| C3—C2—C1 | 119.7 (4) | C7—C12—H12 | 119.2 |
| C3—C2—H2 | 120.1 | O1—C13—H13A | 109.5 |
| C1—C2—H2 | 120.1 | O1—C13—H13B | 109.5 |
| C2—C3—C4 | 120.6 (4) | H13A—C13—H13B | 109.5 |
| C2—C3—H3 | 119.7 | O1—C13—H13C | 109.5 |
| C4—C3—H3 | 119.7 | H13A—C13—H13C | 109.5 |
| C3—C4—C5 | 118.1 (4) | H13B—C13—H13C | 109.5 |

Symmetry codes: (i) −*x*+1, −*y*+1, −*z*+1; (ii) −*x*+2, −*y*+1, −*z*+1.

*Hydrogen-bond geometry (Å, °)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| N1—H1A···O1$^{iii}$ | 0.86 | 1.88 | 2.685 (4) | 155 |
| O1—H1···Cl2 | 0.82 | 2.34 | 3.143 (4) | 166 |

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

**Fig. 1**

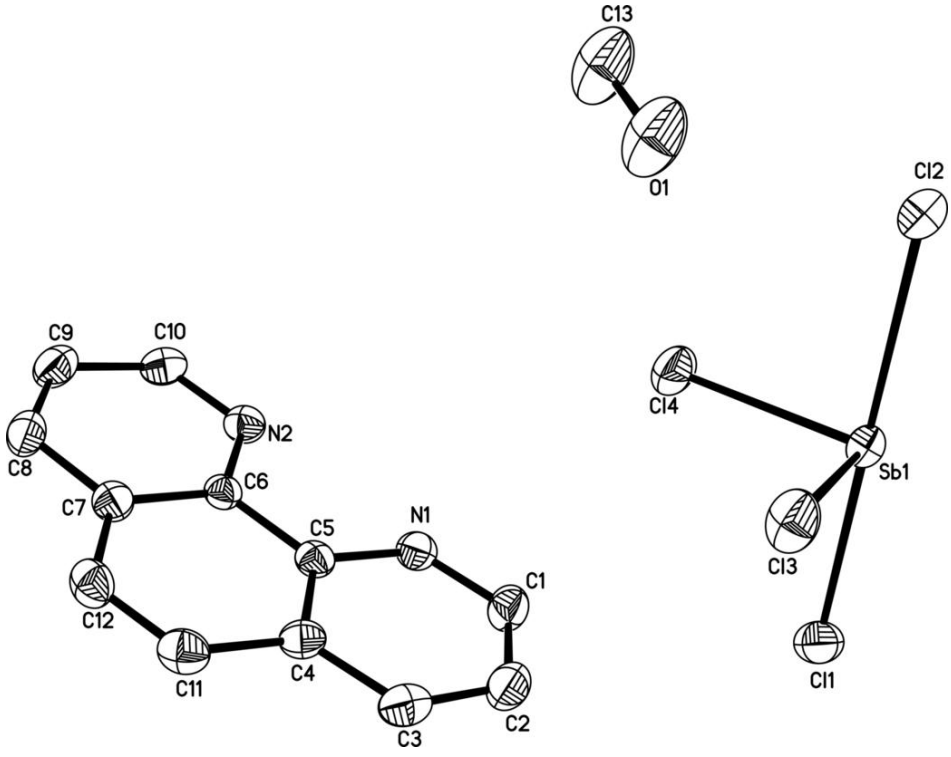
C13
O1
Cl2
Cl4
Sb1
Cl3
Cl1
C9
C10
N2
C8
C6
C7
C5
N1
C1
C12
C11
C4
C3
C2

**Fig. 2**