

## Bis(ferrocenium) bis[tetrachloroantimonate(III)] trichloroantimony(III)

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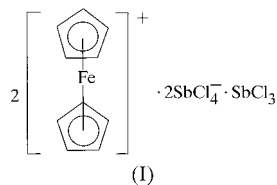
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In the title compound,  $[\text{Fe}(\text{C}_5\text{H}_5)_2]_2[\text{SbCl}_4]_2[\text{SbCl}_3]$ , the cyclopentadienyl rings in both cations are parallel, with a nearly eclipsed conformation. The  $\text{Sb}^{3+}$  ions are coordinated by six  $\text{Cl}^-$  ions to form octahedral arrangements, of which two are slightly distorted. These octahedra form infinite chains along the  $c$  axis through  $\text{Cl}-\text{Sb}-\text{Cl}$  bridges.

### Comment

A simple salt of ferrocenium tetrachloroantimonate,  $[\text{Fe}(\text{C}_5\text{H}_5)_2][\text{SbCl}_4]$ , was obtained by mixing an equimolar mixture of ferrocene and antimony trichloride in chloroform (Yamin *et al.*, 1996). However, the same reaction carried out in benzene yields the title compound, (I), for which the elemental analysis gives different results and in fact does not agree with the structural formula reported by Rheingold *et al.* (1979). Therefore, an X-ray crystallographic study of (I) was carried out and the results are presented herein.



Unlike ferrocenium tetrachloroantimonate, compound (I) is a bis(ferrocenium) salt with two tetrachloroantimonate ions and one molecule of trichloroantimony(III). The conformations of the cyclopentadienyl (Cp) rings are close to eclipsed. The angle of twist of the Cp rings is defined as the torsion angle between a ring C atom, the two ring centres and the corresponding C atom on the opposite ring (Palenik, 1970). The average value of this angle of twist in (I) is  $19.8^\circ$  for Cp1 and Cp2 (rings C1–C5 and C6–C10, respectively), while the angle for Cp3 and Cp4 (rings C11–C15 and C16–C20,

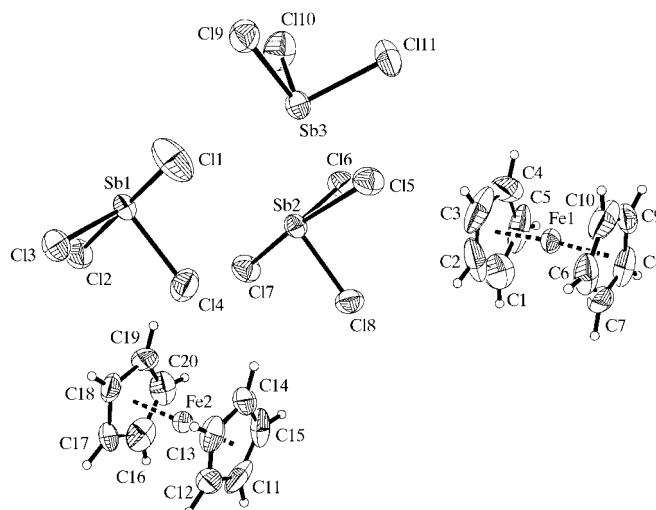


Figure 1

The structure of (I) showing 50% probability displacement ellipsoids and the atom-numbering scheme. H atoms are drawn as spheres of arbitrary radii.

respectively) is  $2.9^\circ$ . The Cp rings in both ferrocenium cations are planar and nearly parallel, with dihedral angles of  $5.1 (7)^\circ$  (Cp1/Cp2) and  $2.9 (5)^\circ$  (Cp3/Cp4). The C–C, Fe–C and Fe–centroid distances in the ferrocenium moieties agree with the reported mean values (Orpen *et al.*, 1989). The Sb–Cl bond lengths range between 2.350 (2) and 2.786 (2) Å. Among the Sb–Cl bonds, the long bonds involving Cl1, Cl2, Cl4, Cl5 and Cl7 are bridging, whereas the short bonds involving the remaining Cl atoms are non-bridging (Porter & Jacobson, 1970; Yamin *et al.*, 1996).

In the crystal of (I), the Sb atoms are connected through Sb–Cl–Sb bridges. The Sb atoms are coordinated to six  $\text{Cl}^-$  ions to form octahedra. The equatorial positions of the coordination around Sb1 are occupied by Cl1, Cl2, Cl4 and Cl7, around Sb2 by Cl5, Cl7, Cl8 and Cl2, and around Sb3 by Cl1, Cl5<sup>ii</sup>, Cl10 and Cl11, while the axial positions are occupied by Cl3 and Cl2<sup>i</sup> for Sb1, Cl6 and Cl4<sup>i</sup> for Sb2, and Cl9 and Cl5<sup>i</sup> for Sb3 [Fig. 2; symmetry codes: (i)  $1 - x, 2 - y, -z$ ; (ii)  $x, y,$

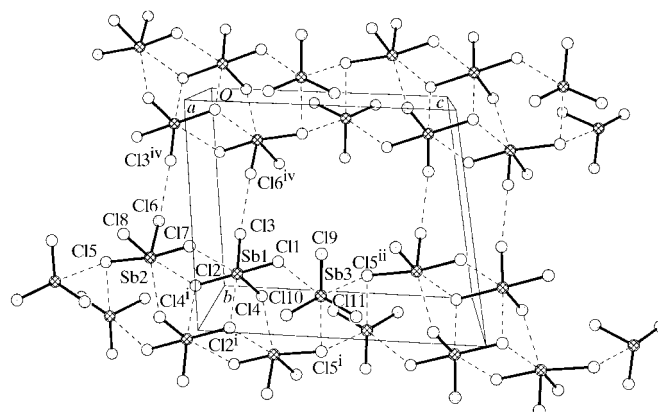


Figure 2

The infinite chains of octahedra in (I) viewed along the  $c$  axis. Symmetry codes are as given in the *Comment*.

1 + z]. Two of the three octahedra, around Sb1 and Sb2, are slightly distorted [Cl4—Sb1—Cl7 = 163.82 (8), Cl8—Sb2—Cl2 = 164.86 (7) and Cl6—Sb2—Cl4<sup>i</sup> = 161.15 (7)°]. The Sb3 atom in the third octahedron acts as a bridge connecting two Cl2—Cl7 edge-shared octahedra through a Cl1—Sb3—Cl5<sup>ii</sup> bridge. These arrangements form infinite chains of octahedra along the *c* axis. A weak Cl···Cl interaction connects the layers of these chains of octahedra along the *b* axis.

Other short contacts observed are: Sb1···Sb1<sup>i</sup> = 4.237 (1), Cl2···Sb1<sup>i</sup> = 3.334 (3), Cl4···Sb2<sup>i</sup> = 3.568 (3), Cl5···Sb3<sup>i</sup> = 3.172 (2), Cl5···Sb3<sup>iii</sup> = 3.096 (3) and Cl3···Cl6<sup>iv</sup> = 3.450 (3) Å [symmetry codes: (iii) *x*, *y*, −1 + *z*, (iv) 1 − *x*, 1 − *y*, −*z*].

## Experimental

The title compound was prepared by mixing equimolar amounts of ferrocene and antimony trichloride in benzene. Dark-blue needle-like crystals were obtained after a few days.

### Crystal data

|  |   |
|--|---|
| [Fe(C <sub>5</sub> H <sub>5</sub> ) <sub>2</sub> ][SbCl <sub>4</sub> ] <sub>2</sub> [SbCl <sub>3</sub> ] | <i>Z</i> = 2                                    |
| <i>M<sub>r</sub></i> = 1127.26   | <i>D<sub>x</sub></i> = 2.222 Mg m <sup>−3</sup> |
| Triclinic, <i>P</i> 1  | Mo <i>K</i> α radiation                         |
| <i>a</i> = 11.615 (2) Å  | Cell parameters from 41 reflections             |
| <i>b</i> = 12.000 (1) Å  | <i>θ</i> = 5.02–12.32°                          |
| <i>c</i> = 13.789 (1) Å  | <i>μ</i> = 4.10 mm <sup>−1</sup>                |
| <i>α</i> = 80.03 (1)°  | <i>T</i> = 293 (2) K                            |
| <i>β</i> = 84.42 (1)°  | Needle, dark blue                               |
| <i>γ</i> = 62.92 (1)°  | 0.36 × 0.18 × 0.08 mm                           |
| <i>V</i> = 1685.0 (3) Å <sup>3</sup>   |   |

**Table 1**

Selected geometric parameters (Å, °).

|                          |             |                            |            |
|--------------------------|-------------|----------------------------|------------|
| Sb1—Cl3                  | 2.367 (2)   | Sb2—Cl5                    | 2.786 (2)  |
| Sb1—Cl4                  | 2.407 (3)   | Sb2—Cl2                    | 3.180 (3)  |
| Sb1—Cl1                  | 2.538 (3)   | Sb2—Cl4 <sup>i</sup>       | 3.568 (3)  |
| Sb1—Cl2                  | 2.683 (2)   | Sb3—Cl9                    | 2.386 (2)  |
| Sb1—Cl7                  | 3.225 (3)   | Sb3—Cl11                   | 2.387 (2)  |
| Sb1—Cl2 <sup>i</sup>     | 3.334 (3)   | Sb3—Cl10                   | 2.395 (3)  |
| Sb2—Cl6                  | 2.350 (2)   | Sb3—Cl5 <sup>ii</sup>      | 3.096 (3)  |
| Sb2—Cl8                  | 2.386 (2)   | Sb3—Cl5 <sup>i</sup>       | 3.172 (2)  |
| Sb2—Cl7                  | 2.523 (2)   | Sb3—Cl1                    | 3.212 (3)  |
| Cl1—Sb1—Cl2              | 179.11 (10) | Cl6—Sb2—Cl4 <sup>i</sup>   | 161.15 (7) |
| Cl4—Sb1—Cl7              | 163.82 (8)  | Cl10—Sb3—Cl5 <sup>ii</sup> | 174.37 (8) |
| Cl3—Sb1—Cl2 <sup>i</sup> | 173.24 (8)  | Cl9—Sb3—Cl5 <sup>i</sup>   | 175.73 (8) |
| Cl7—Sb2—Cl5              | 171.34 (8)  | Cl11—Sb3—Cl1               | 172.73 (8) |
| Cl8—Sb2—Cl2              | 164.86 (7)  |                            |            |

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

### Data collection

|  |  |
|--|--|
| Siemens <i>P4</i> diffractometer   | <i>R</i> <sub>int</sub> = 0.046                                  |
| <i>θ</i> / <i>2θ</i> scans   | <i>θ</i> <sub>max</sub> = 25°                                    |
| Absorption correction: empirical<br>via <i>ψ</i> scans ( <i>XSCANS</i> ; Siemens,<br>1994) | <i>h</i> = −1 → 13<br><i>k</i> = −13 → 14<br><i>l</i> = −16 → 16 |
| <i>T</i> <sub>min</sub> = 0.320, <i>T</i> <sub>max</sub> = 0.735                           | 3 standard reflections   |
| 6898 measured reflections  | every 97 reflections   |
| 5935 independent reflections   | intensity decay: <3%   |
| 3378 reflections with <i>I</i> > 2σ( <i>I</i> )  |  |

### Refinement

|   |  |
|---|--|
| Refinement on <i>F</i> <sup>2</sup>                                     | H-atom parameters constrained  |
| <i>R</i> [ <i>F</i> <sup>2</sup> > 2σ( <i>F</i> <sup>2</sup> )] = 0.040 | <i>w</i> = 1/[σ <sup>2</sup> ( <i>F<sub>o</sub></i> <sup>2</sup> ) + (0.0226 <i>P</i> ) <sup>2</sup> ] |
| <i>wR</i> ( <i>F</i> <sup>2</sup> ) = 0.085                             | where <i>P</i> = ( <i>F<sub>o</sub></i> <sup>2</sup> + 2 <i>F<sub>c</sub></i> <sup>2</sup> )/3         |
| <i>S</i> = 0.81   | (Δ/σ) <sub>max</sub> < 0.001   |
| 5935 reflections  | Δρ <sub>max</sub> = 0.71 e Å <sup>−3</sup>   |
| 325 parameters  | Δρ <sub>min</sub> = −1.04 e Å <sup>−3</sup>  |

After checking their presence in the difference map, all H atoms were fixed geometrically and allowed to ride on their attached atoms. All C—H bonds were fixed at 0.98 Å, with *U*<sub>iso</sub>(H) = 1.2*U*<sub>eq</sub>(C).

Data collection: *XSCANS* (Siemens, 1994); cell refinement: *XSCANS*; data reduction: *XSCANS*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 1997); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL* and *PLATON* (Spek, 1990).

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: SK1355). Services for accessing these data are described at the back of the journal.

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