

## The Crystal and Molecular Structure of [Dicarbonyl( $\eta$ -cyclopentadienyl)ferrio]triphenylantimony Hexafluorophosphate

BY R. E. COBBLEDICK AND F. W. B. EINSTEIN

*Department of Chemistry, Simon Fraser University, Burnaby, British Columbia V5A 1S6, Canada*

(Received 11 October 1977; accepted 19 November 1977)

$[(C_6H_5)_3Sb\{Fe(CO)_2(h^5-C_5H_5)\}][PF_6]$  is monoclinic, space group  $P2_1/c$ , with  $a = 9.561$  (2),  $b = 26.330$  (5),  $c = 10.747$  (2) Å,  $\beta = 104.48$  (1)°,  $Z = 4$ . The structure was refined by full-matrix least-squares calculations to an  $R$  of 0.035 for 2102 counter reflexions. It consists of discrete  $[(C_6H_5)_3Sb\{Fe(CO)_2(h^5-C_5H_5)\}]^+$  cations and  $[PF_6]^-$  anions. In the cation there is irregular tetrahedral geometry about the Sb atom with an Sb–Fe distance of 2.477 (1) Å and an average Sb–C(phenyl) distance of 2.109 (12) Å. The Fe atom can be considered to have octahedral coordination, with the cyclopentadienyl ligand occupying three sites. In the  $[PF_6]^-$  anion the average corrected P–F length is 1.627 (6) Å.

### Introduction

Recently there has been interest in transition-metal compounds containing Fe–Sb bonds (Cullen, Patmore, Sams & Scott, 1974). Mössbauer spectroscopic studies of the cationic species  $[X_pSb\{Fe(CO)_2(h^5-C_5H_5)\}_{4-p}]^+$  (where  $X = Cl, Br, I, CF_3, C_6H_5, n-C_4H_9$ ;  $p = 1 \rightarrow 3$ ) in comparison with the essentially isoelectronic neutral Sn compounds indicated that the positive charge is mainly localized on the Sb atom and that the Fe–Sb bonding is primarily  $\sigma$  in character. Many of the neutral Sn compounds have been studied by X-ray diffraction but few compounds with Fe–Sb bonds have been studied. Two structures containing cations of the type  $[X_pSb\{Fe(CO)_2(h^5-C_5H_5)\}_{4-p}]^+$ , namely  $[Cl_2Sb\{Fe(CO)_2(h^5-C_5H_5)\}_2][Sb_4Cl_{14}]$  (Einstein & Jones, 1973) and  $[ClSb\{Fe(CO)_2(h^5-C_5H_5)\}_3][FeCl_4] \cdot CH_2Cl_2$  (Trinh-Toan & Dahl, 1971) have been described.  $[(C_6H_5)_3Sb\{Fe(CO)_2(h^5-C_5H_5)\}][PF_6]$  was chosen for investigation to yield information regarding the Fe–Sb bonding and for comparison with the neutral Sn compound (Bryan, 1967).

### Experimental

Recrystallization from a dichloromethane/benzene mixture gave yellow, plate-shaped crystals. Photographs (Cu  $K\alpha$  radiation) showed the crystals to be monoclinic and gave approximate cell dimensions. Systematically absent reflexions  $h0l$  for  $l = 2n + 1$  and  $0k0$  for  $k = 2n + 1$  uniquely determined the space group as  $P2_1/c$ . A plate-shaped crystal ( $0.25 \times 0.15 \times 0.25$  mm) mounted on the end of a fibre with shellac was used to measure the cell dimensions and the intensities with a Picker FACS-I computer-controlled diffractometer (Mo  $K\alpha$  radiation). Accurate cell dimensions

were obtained from least-squares refinement of the setting angles of 12 reflections ( $2\theta > 35^\circ$ ). Crystal data are given in Table 1. Intensities were measured by the  $\theta$ – $2\theta$  scan method with graphite-monochromated Mo  $K\alpha$  radiation. Data were collected in two shells; for the inner shell ( $0^\circ < 2\theta \leq 35^\circ$ ) a base width of  $1.3^\circ$  was used and background counts of 4 s were made on either side of the reflexions; for the outer shell ( $35 < 2\theta \leq 40^\circ$ ) a width of  $0.8^\circ$  and background counts of 10 s were employed. In both instances the scan rate was  $2^\circ \text{ min}^{-1}$  in  $2\theta$  and the scan width was increased for dispersion. Two standard reflexions were measured at frequent intervals. Intensities for 2460 unique reflexions were measured and those 2102 with net intensity  $I > 2.3\sigma(I)$  were regarded as observed, where  $\sigma(I) = \{S + (t_s^2/t_b^2)B + (0.03I)^2\}^{1/2}$ ;  $S$  is the total scan count,  $B$  the total background count and  $t_s$  and  $t_b$  are the scan and background count times respectively. Intensities were corrected for Lorentz and polarization effects. No correction for absorption was applied and this could give a maximum error of 5% in  $F$  in extreme cases.

### Structure determination and refinement

A Patterson function gave positional coordinates for the Sb and Fe atoms. A structure factor calculation followed by an electron density synthesis located the

Table 1. *Crystal data*

$[(C_6H_5)_3Sb\{Fe(CO)_2(C_5H_5)\}][PF_6]$ , $M_r = 675.0$	
Space group: $P2_1/c$	$Z = 4$
$a = 9.561$ (2) Å	$D_o = 1.70 \text{ g cm}^{-3}$
$b = 26.330$ (5)	$D_c = 1.711$
$c = 10.747$ (2)	$\mu(\text{Mo } K\alpha) = 10.2 \text{ cm}^{-1}$
$\beta = 104.48$ (1)°	Temperature: $23^\circ\text{C}$

Table 2. Final atomic coordinates and isotropic thermal parameters

	x	y	z	U ( $\text{\AA}^2 \times 10^3$ )
Sb	0.88085 (5)	0.35131 (2)	0.29549 (4)	
Fe	0.71909 (9)	0.42621 (3)	0.26501 (9)	
P	0.7415 (2)	0.0267 (1)	0.2917 (2)	
F(1)	0.8801 (5)	-0.0012 (2)	0.3740 (6)	
F(2)	0.6529 (5)	-0.0028 (2)	0.3741 (5)	
F(3)	0.6016 (5)	0.0550 (2)	0.2200 (6)	
F(4)	0.7760 (6)	0.0709 (2)	0.3944 (5)	
F(5)	0.7129 (8)	-0.0171 (2)	0.1938 (6)	
F(6)	0.8321 (6)	0.0560 (2)	0.2121 (5)	
O(1)	0.5359 (5)	0.3968 (2)	0.0155 (5)	
O(2)	0.5269 (6)	0.3785 (2)	0.4036 (6)	
C(20)	0.7800 (7)	0.2801 (3)	0.2867 (7)	
C(21)	0.8298 (8)	0.2419 (3)	0.3712 (8)	
C(22)	0.7687 (10)	0.1938 (3)	0.3580 (9)	
C(23)	0.6534 (11)	0.1857 (3)	0.2603 (11)	
C(24)	0.6035 (10)	0.2214 (4)	0.1669 (11)	
C(25)	0.6667 (10)	0.2695 (3)	0.1829 (9)	
C(1)	0.6088 (7)	0.4079 (3)	0.1117 (7)	53 (2)
C(2)	0.6025 (8)	0.3967 (3)	0.3492 (7)	66 (2)
C(3)	0.6879 (8)	0.5036 (3)	0.2234 (7)	64 (2)
C(4)	0.6931 (8)	0.4954 (3)	0.3505 (7)	69 (2)
C(5)	0.8297 (8)	0.4742 (3)	0.4100 (7)	68 (2)
C(6)	0.9087 (7)	0.4712 (3)	0.3167 (6)	57 (2)
C(7)	0.8217 (7)	0.4881 (3)	0.2012 (6)	56 (2)
C(8)	1.0283 (7)	0.3881 (2)	0.1813 (6)	48 (2)
C(9)	1.0756 (7)	0.3773 (3)	0.1159 (7)	60 (2)
C(10)	1.1873 (8)	0.3687 (3)	0.0562 (7)	72 (2)
C(11)	1.2468 (8)	0.3218 (3)	0.0598 (8)	75 (2)
C(12)	1.2007 (9)	0.2828 (3)	0.1221 (8)	77 (2)
C(13)	1.0897 (8)	0.2912 (3)	0.1839 (7)	66 (2)
C(14)	1.0311 (7)	0.3535 (2)	0.4767 (6)	46 (2)
C(15)	1.1732 (7)	0.3643 (2)	0.4849 (6)	54 (2)
C(16)	1.2701 (8)	0.3711 (3)	0.6054 (7)	65 (2)
C(17)	1.2244 (8)	0.3666 (3)	0.7140 (7)	66 (2)
C(18)	1.0817 (8)	0.3557 (3)	0.7078 (8)	72 (2)
C(19)	0.9826 (8)	0.3495 (3)	0.5894 (7)	62 (2)
H(1)	0.622	0.514	0.154	58
H(2)	0.633	0.500	0.391	128
H(3)	0.861	0.461	0.484	63
H(4)	0.996	0.455	0.335	52
H(5)	0.838	0.489	0.131	49
H(6)	1.019	0.409	0.109	57
H(7)	1.223	0.402	0.022	77
H(8)	1.330	0.314	0.012	131
H(9)	1.239	0.254	0.133	182
H(10)	1.071	0.265	0.233	100
H(11)	1.220	0.366	0.412	54
H(12)	1.364	0.377	0.597	136
H(13)	1.308	0.380	0.804	162
H(14)	1.038	0.351	0.781	115
H(15)	0.868	0.344	0.580	91
H(16)	0.920	0.250	0.443	105
H(17)	0.831	0.163	0.413	101
H(18)	0.620	0.158	0.258	57
H(19)	0.521	0.218	0.086	120
H(20)	0.633	0.292	0.118	91

anisotropic thermal motion of the Sb, Fe and F atoms. With anisotropic parameters for these atoms  $R$  was reduced to 0.080. All the H atoms were located on a subsequent difference map and were included in the structure factors without refinement. C atoms of one of the phenyl groups and the carbonyl atoms showed evidence of anisotropic motion and further refinement with anisotropic parameters for these atoms and for the P atom reduced  $R$  to a final value of 0.035. The H atom parameters were allowed to refine on the penultimate cycle of refinement. There was no evidence of disorder or large vibrational motion of the cyclopentadienyl ring on a final difference map and the largest peaks ( $<0.5 \text{ e \AA}^{-3}$ ) were in the neighbourhood of the Sb and F atoms. On the final cycle no coordinate shift was more than  $0.01\sigma$ . Weights determined by  $w = 1/\sigma^2(F_o)$  were used in the later stages. Analysis of  $\sum w(|F_o| - |F_c|)^2$  as a function of  $\sin \theta/\lambda$  and  $|F_o|$  showed no significant trends and confirmed the reasonableness of the weighting scheme. Scattering factors (including real and imaginary components of the anomalous dispersion for Sb and Fe) were those listed in *International Tables for X-ray Crystallography* (1974) with the exception of those for H which were taken from Stewart, Davidson & Simpson (1965). The final positional parameters are given in Table 2.\*

\* Lists of observed structure amplitudes and calculated structure factors, and anisotropic thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 33255 (18 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 13 White Friars, Chester CH1 1NZ, England.

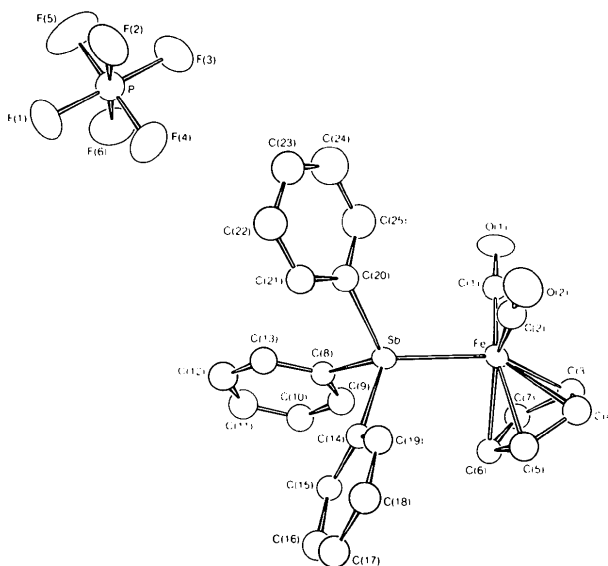


Fig. 1. A perspective view of the cation and anion in  $[(C_6H_5)_3Sb\{Fe(CO)_2(h^5-C_5H_5)\}][PF_6]$ , drawn by ORTEP (Johnson, 1965).

remaining non-hydrogen atoms. Full-matrix least-squares refinement with individual isotropic thermal parameters and unit weights for the observable reflexions gave an  $R$  of 0.098. A difference map indicated

## Results and discussion

Bond distances and angles are listed in Table 3. The structure consists of discrete  $[(C_6H_5)_3Sb\{Fe(CO)_2(h^5-C_5H_5)\}]^+$  cations and  $[PF_6]^-$  anions. The molecular configuration of the ions and the atom-numbering scheme are depicted in Fig. 1.

Table 3. *Interatomic distances (Å) and angles (°)*

Asterisks denote values corrected for thermal motion, assuming the second atom to ride on the first.

Sb—Fe	2.477 (1)	C(10)—C(11)	1.356 (10)
Sb—C(8)	2.116 (6)	C(11)—C(12)	1.359 (10)
Sb—C(14)	2.112 (6)	C(12)—C(13)	1.403 (10)
Sb—C(20)	2.100 (7)	C(14)—C(15)	1.369 (8)
Fe—C(1)	1.786 (7)	C(14)—C(19)	1.404 (9)
Fe—C(2)	1.781 (8)	C(15)—C(16)	1.403 (9)
Fe—C(3)	2.092 (7)	C(16)—C(17)	1.350 (10)
Fe—C(4)	2.084 (8)	C(17)—C(18)	1.380 (10)
Fe—C(5)	2.078 (8)	C(18)—C(19)	1.393 (10)
Fe—C(6)	2.120 (7)	C(20)—C(25)	1.375 (10)
Fe—C(7)	2.101 (7)	C(20)—C(21)	1.360 (9)
C(1)—O(1)	1.131 (7)	C(21)—C(22)	1.385 (10)
C(2)—O(2)	1.141 (8)	C(22)—C(23)	1.336 (11)
C(3)—C(4)	1.372 (9)	C(23)—C(24)	1.371 (12)
C(3)—C(7)	1.417 (9)	C(24)—C(25)	1.395 (12)
C(4)—C(5)	1.418 (9)	P—F(1)	1.579 (5) 1.625*
C(5)—C(6)	1.400 (9)	P—F(2)	1.574 (5) 1.622*
C(6)—C(7)	1.385 (9)	P—F(3)	1.557 (5) 1.629*
C(8)—C(9)	1.387 (9)	P—F(4)	1.582 (5) 1.624*
C(8)—C(13)	1.365 (9)	P—F(5)	1.537 (5) 1.639*
C(9)—C(10)	1.394 (9)	P—F(6)	1.564 (5) 1.625*
		C—H <sub>av</sub>	0.96 (10)
Fe—Sb—C(8)	122.6 (2)	C(15)—C(16)—C(17)	120.3 (7)
Fe—Sb—C(14)	111.0 (2)	C(16)—C(17)—C(18)	120.4 (7)
Fe—Sb—C(20)	116.1 (2)	C(17)—C(18)—C(19)	120.5 (7)
C(8)—Sb—C(14)	98.1 (2)	C(14)—C(19)—C(18)	118.9 (7)
C(8)—Sb—C(20)	101.1 (3)	Sb—C(20)—C(21)	123.4 (5)
C(14)—Sb—C(20)	105.3 (3)	Sb—C(20)—C(25)	118.6 (6)
Sb—Fe—C(1)	96.4 (2)	C(21)—C(20)—C(25)	117.6 (7)
Sb—Fe—C(2)	92.0 (2)	C(20)—C(21)—C(22)	122.6 (7)
C(1)—Fe—C(2)	92.9 (3)	C(21)—C(22)—C(23)	118.1 (8)
Fe—C(1)—O(1)	178.2 (9)	C(22)—C(23)—C(24)	122.2 (8)
Fe—C(2)—O(2)	178.8 (8)	C(23)—C(24)—C(25)	118.1 (8)
C(4)—C(3)—C(7)	108.2 (6)	C(20)—C(25)—C(24)	120.9 (8)
C(3)—C(4)—C(5)	108.1 (7)	F(1)—P—F(2)	87.2 (3)
C(4)—C(5)—C(6)	107.5 (6)	F(1)—P—F(3)	175.7 (4)
C(5)—C(6)—C(7)	108.2 (6)	F(1)—P—F(4)	87.3 (3)
C(3)—C(7)—C(6)	108.0 (6)	F(1)—P—F(5)	90.9 (4)
Sb—C(8)—C(9)	121.4 (5)	F(1)—P—F(6)	91.9 (3)
Sb—C(8)—C(13)	118.9 (5)	F(2)—P—F(3)	89.8 (3)
C(9)—C(8)—C(13)	119.3 (6)	F(2)—P—F(4)	91.0 (3)
C(8)—C(9)—C(10)	119.9 (7)	F(2)—P—F(5)	89.3 (4)
C(9)—C(10)—C(11)	119.9 (7)	F(2)—P—F(6)	178.9 (7)
C(10)—C(11)—C(12)	121.2 (8)	F(3)—P—F(4)	89.7 (3)
C(11)—C(12)—C(13)	119.4 (7)	F(3)—P—F(5)	92.1 (4)
C(8)—C(13)—C(12)	120.4 (7)	F(3)—P—F(6)	91.0 (3)
Sb—C(14)—C(15)	120.0 (5)	F(4)—P—F(5)	178.1 (6)
Sb—C(14)—C(19)	119.9 (5)	F(4)—P—F(6)	88.4 (3)
C(15)—C(14)—C(19)	119.7 (6)	F(5)—P—F(6)	91.3 (4)
C(14)—C(15)—C(16)	120.1 (6)		

(a) *The  $[(C_6H_5)_3Sb\{Fe(CO)_2(h^5-C_5H_5)\}]^+$  cation*

A C atom from each of the three phenyl rings and the Fe atom are bonded to the Sb atom in a distorted tetrahedral arrangement. The three Fe—Sb—C angles range from 111.0 (2) to 122.6 (2)°. The Sb—Fe length of 2.477 (1) Å is intermediate between values of 2.422–2.446 (2) Å and 2.527–2.563 (3) Å in the related derivatives  $[Cl_2Sb\{Fe(CO)_2(h^5-C_5H_5)\}_2]_2$ — $[Sb_4Cl_{14}]$  (Einstein & Jones, 1973) and  $[ClSb\{Fe(CO)_2(h^5-C_5H_5)\}_3]_2[FeCl_4] \cdot CH_2Cl_2$  (Trinh-Toan & Dahl, 1971). X-ray structural work on the neutral Sn analogue  $[(C_6H_5)_3Sn\{Fe(CO)_2(h^5-C_5H_5)\}]$  (Bryan, 1967) indicates Fe—Sn covalent bonds of 2.533 and 2.540 Å in the two molecules of the asymmetric unit.

Table 4. *Deviations of atoms (Å) from least-squares planes*

The equations of the planes are referred to orthogonal axes with *X* along *a*, *Y* along *b*, and *Z* along *c*\*.

(a) *Plane through C(3), C(4), C(5), C(6) and C(7)*

$$-0.3133X - 0.9232Y - 0.2227Z - 14.6298 = 0$$

C(3)	-0.002	H(1)	0.04
C(4)	-0.007	H(2)	0.01
C(5)	0.013	H(3)	0.13
C(6)	-0.012	H(4)	0.09
C(7)	0.009	H(5)	0.03
Fe	1.724		
$\chi^2$	8.68		

(b) *Plane through C(8), C(9), C(10), C(11), C(12) and C(13)*

$$-0.4821X - 0.2485Y - 0.8401Z + 8.2974 = 0$$

C(8)	-0.004	H(6)	0.11
C(9)	0.008	H(7)	-0.13
C(10)	-0.006	H(8)	0.03
C(11)	0.000	H(9)	-0.07
C(12)	0.004	H(10)	-0.11
C(13)	0.000		
Sb	-0.262		
$\chi^2$	2.72		

(c) *Plane through C(14), C(15), C(16), C(17), C(18) and C(19)*

$$-0.2044X + 0.9782Y - 0.0363Z - 7.1751 = 0$$

C(14)	-0.004	Sb	0.203
C(15)	-0.001	H(11)	-0.07
C(16)	0.005	H(12)	-0.03
C(17)	-0.002	H(13)	0.18
C(18)	-0.006	H(14)	-0.03
C(19)	0.009	H(15)	0.09
$\chi^2$	3.23		

(d) *Plane through C(20), C(21), C(22), C(23), C(24) and C(25)*

$$0.7924X - 0.2928Y - 0.5351Z - 1.5566 = 0$$

C(20)	-0.013	Sb	0.134
C(21)	0.008	H(16)	0.08
C(22)	0.017	H(17)	0.30
C(23)	-0.041	H(18)	-0.06
C(24)	0.024	H(19)	0.05
C(25)	0.009	H(20)	0.08
$\chi^2$	29.17		

The Sb—Fe bond is thus only  $\sim 0.06$  Å shorter than the Sn—Fe bond. Allowing for the difference in covalent radii between Sb and Sn [1.36 and 1.40 Å respectively (Pauling, 1960)] this indicates that there is not a large difference in bonding in the Sb and Sn analogues, *i.e.* both types of bonds are essentially pure  $\sigma$  in character. If there were  $d-d_\pi$  bonding between the Sb and Fe atoms a shorter Sb—Fe length would be expected. This is consistent with Mössbauer studies (Cullen, Patmore, Sams & Scott, 1974) which suggested that the electron distributions in the Sb—Fe and Sn—Fe bonds were similar with very little  $\pi$  back-donation from the occupied  $3d$  orbitals of Fe to the empty  $5d$  orbitals in the Sb compound.

The carbonyl Fe—C distances of 1.786 (7) and 1.781 (8) Å are longer than those in the neutral Sn compound [ $Fe-C_{av} = 1.713$  (13) Å] and closer to the values observed in other Fe carbonyl compounds. The Fe—C—O groups show no significant deviation from linearity and the C—Fe—C angle is  $92.9$  (3)°. In contrast to the Sn compound the carbonyl groups are bent away from the metal atom with an average Sb—Fe—C angle of  $94.2^\circ$  compared with an average  $86.8^\circ$  Sn—Fe—C angle. The tridentate cyclopentadienyl ring completes the approximate octahedral geometry about the Fe atom. The average Fe—C (cyclopentadienyl) distance is  $2.095$  (8)° which is comparable with values in similar Fe cyclopentadienyl compounds (Einstein & Jones, 1973; Trinh-Toan & Dahl, 1976). Although the cyclopentadienyl C—C lengths are not statistically equivalent within the limits of the errors there is no reason to propose a chemically significant difference. Unsatisfactory positional refinement of C(4) involving the shortest and longest bonds would account for the entire deviation. The mean C—C of  $1.398$  (9) Å is comparable with values found in similar compounds. There is no large deviation of the ring from planarity, the largest displacement being  $0.013$  (8) Å and the Fe atom is  $1.724$  Å out of this plane. Least-squares-planes results are shown in Table 4. The arrangement of ligands about the Fe—cyclopentadienyl centroid axis is such that the ring is rotated by approximately  $8^\circ$  from a position having mirror symmetry.

The mean Sb—C(phenyl) distance is  $2.109$  (12) Å, which is close to the average of  $2.119$  (6) Å observed in bis(isocyanato)triphenylantimony (Ferguson, Goel & Ridley, 1975). The Fe—Sb—C angles are all greater than the regular tetrahedral value with an average of  $116.6^\circ$ . The average C—C length in the three phenyl rings is  $1.377$  (20) Å and the rings do not deviate

markedly from planarity. Maximum displacements are  $0.008$ ,  $0.009$  and  $0.041$  Å for the rings defined by C(8) to C(13), C(14) to C(19), C(20) to C(25) respectively and the corresponding displacements of the Sb atom from these planes are  $0.262$ ,  $0.203$  and  $0.134$  Å.

#### (b) The $[PF_6]^-$ anion

The  $[PF_6]^-$  ion was reasonably well-behaved and there was no evidence for gross disordering although there may be a small degree of rotational disorder. The F and P atoms form an almost regular octahedron with a mean P—F distance of  $1.565$  (17) Å. Correction for thermal motion, assuming riding motion of the F atoms on the P atom, increased the average length and consistency to  $1.627$  (6) Å.

#### (c) Molecular packing

Each ion has four closest neighbours of the opposite type. Closest approaches involving the  $[PF_6]^-$  ion and the cation are  $F(4)\cdots O(1) = 3.03$ ,  $F(3)\cdots C(4) = 3.15$  and  $F(4)\cdots H(17) = 2.48$  Å, so that only normal van der Waals forces are involved. There are no abnormally short intermolecular contacts between the cations.

We thank Dr W. R. Cullen for the crystals and for his interest throughout the work, and the National Research Council of Canada for financial support.

#### References

- BRYAN, R. F. (1967). *J. Chem. Soc. A*, pp. 192–202.  
 CULLEN, W. R., PATMORE, D. J., SAMS, J. R. & SCOTT, J. C. (1974). *Inorg. Chem.* **13**, 649–655.  
 EINSTEIN, F. W. B. & JONES, R. D. G. (1973). *Inorg. Chem.* **12**, 1690–1696.  
 FERGUSON, G., GOEL, R. G. & RIDLEY, D. R. (1975). *J. Chem. Soc. Dalton Trans.* pp. 1288–1290.  
*International Tables for X-ray Crystallography* (1974). Vol. IV, pp. 72–97, 149–150. Birmingham: Kynoch Press.  
 JOHNSON, C. K. (1965). *ORTEP*. Report ORNL-3794. Oak Ridge National Laboratory, Tennessee.  
 PAULING, L. (1960). *The Nature of the Chemical Bond*, p. 246. Ithaca: Cornell Univ. Press.  
 STEWART, R. F., DAVIDSON, E. R. & SIMPSON, W. T. (1965). *J. Chem. Phys.* **42**, 3175–3187.  
 TRINH-TOAN & DAHL, L. F. (1971). *J. Am. Chem. Soc.* **93**, 2654–2666.  
 TRINH-TOAN & DAHL, L. F. (1976). *J. Am. Chem. Soc.* **98**, 2953–2959.