

fois à un composé oxydé, la distance O—O trouvée très courte semble donc nécessaire à l'arrangement structural décrit.

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## Structure and Mössbauer Spectrum of Bis(tetraethylammonium) 1,1'-commo-Bis(decahydro-1-ferra-2-selena-closo-decaborate)(2-)

BY GEORGE FERGUSON AND BARBARA L. RUHL

*Chemistry Department, University of Guelph, Guelph, Ontario, Canada N1G 2W1*

AND ORLA NI DHUBHGHAILL AND TREVOR R. SPALDING

*Chemistry Department, University College, Cork, Ireland*

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**Abstract.**  $[\text{Et}_4\text{N}]_2[\text{Fe}(\text{SeB}_{10}\text{H}_{10})_2]$ ,  $2\text{C}_8\text{H}_{20}\text{N}^+\cdot\text{B}_{20}\text{H}_{20}^-\text{FeSe}_2^-$ ,  $M_r = 710.7$ , monoclinic,  $P2_1/c$ ,  $a = 8.306$  (2),  $b = 16.999$  (5),  $c = 14.589$  (3) Å,  $\beta = 116.91$  (2)°,  $V = 1837$  (2) Å<sup>3</sup>,  $Z = 2$ ,  $D_x = 1.28$  g cm<sup>-3</sup>,  $\lambda(\text{Mo K}\alpha) = 0.71073$  Å,  $\mu = 23.9$  cm<sup>-1</sup>,  $F(000) = 728$ , room temperature. Final  $R = 0.037$  for 1260 observed reflections. The anion has a commo-bisicosahedral structure with two (SeB<sub>10</sub>H<sub>10</sub>) cages sharing a common Fe apex and the Se atoms bonded to Fe which lies on an inversion centre; the Fe—Se distance is 2.280 (1) Å. The cation is disordered. The <sup>57</sup>Fe Mössbauer spectrum of the ferraselenaborane shows a simple doublet [ $\sigma = 0.53$  (1),  $\Delta = 2.11$  (1) mm s<sup>-1</sup>] consistent with the Fe<sup>II</sup> situation.

**Introduction.** The ferraselenaborane anion was first prepared as the  $[\text{Me}_4\text{N}]^+$  salt (Little, Friesen & Todd, 1977). We have prepared the  $[\text{Et}_4\text{N}]^+$  salt in the same manner to establish its solid-state structure and Mössbauer spectrum. A previous X-ray diffraction study of the sulfur analogue  $[\text{Me}_4\text{N}]_2[\text{Fe}(\text{SB}_{10}\text{H}_{10})_2]$  (Davis & Bernal, 1972) showed the complex to crystallize in space group *Ibam* and have a molecular structure based on commo-FeSB<sub>10</sub> icosahedra. However, the S atom and one B atom were disordered which precluded the determination of accurate Fe—S distances. Very few structural studies of metallaheteroboranes have been reported except derivatives of carbaboranes.

**Experimental.** Maroon needle crystals grown from aqueous ethanol. Accurate cell dimensions and crystal-orientation matrix determined on a CAD-4 diffractometer by a least-squares treatment of the setting angles of 25 reflections in the range  $8 < \theta < 13^\circ$ . Crystal dimensions 0.10 × 0.15 × 0.40 mm; intensities of reflections with indices  $h$  0 to 10,  $k$  0 to 21,  $l$  -18 to 18, with  $2 < 2\theta < 54^\circ$  measured;  $\omega$ - $2\theta$  scans;  $\omega$ -scan width  $(0.60 + 0.35 \tan \theta)^\circ$ ; graphite-monochromatized MoK $\alpha$  radiation; intensities of three reflections measured every 2 h showed no evidence of crystal decay. 4551 reflections measured, 4006 unique, 1260 with  $I > 3\sigma(I)$  labelled observed and used in structure solution and refinement;  $R_{\text{int}} = 0.020$ . Data corrected for Lorentz, polarization and absorption effects (max. and min. transmission factors 0.804, 0.694). Gaussian integration, grid  $8 \times 8 \times 16$ . Space group  $P2_1/c$  uniquely from systematic absences  $h0l$   $l = 2n + 1$ ,  $0k0$   $k = 2n + 1$ . With  $Z = 2$ , the Fe atom lies at an inversion centre; the coordinates of the Se atom were determined from analysis of the three-dimensional Patterson function and those of the remaining non-H atoms were found *via* the heavy-atom method. The Et<sub>4</sub>N cation is disordered; the CH<sub>2</sub> groups of each ethyl moiety occupy two sites with occupancy factors of 0.65 and 0.35 (from isotropic refinement). Refinement was by full-matrix least-squares calculations on  $F$ , initially with isotropic and then with anisotropic thermal parameters. At an intermediate stage in the refinement, difference

Table 1. *Positional parameters and equivalent isotropic temperature factors with their e.s.d.'s*

	<i>x</i>	<i>y</i>	<i>z</i>	<i>B</i> <sub>eq</sub> <sup>*</sup> (Å <sup>2</sup> )
Se	0.26276 (7)	0.00370 (5)	−0.01352 (5)	5.33 (2)
Fe	0.0	0.0	0.0	2.98 (2)
B1	0.1017 (9)	−0.0972 (4)	−0.0584 (5)	5.0 (2)
B2	−0.1327 (10)	−0.0683 (5)	−0.1356 (5)	6.0 (2)
B3	−0.1573 (10)	0.0328 (5)	−0.1547 (6)	5.9 (2)
B4	0.0483 (10)	0.0830 (5)	−0.1001 (5)	5.0 (2)
B5	−0.0044 (12)	−0.1027 (5)	−0.1971 (7)	8.4 (3)
B6	−0.1608 (11)	−0.0266 (7)	−0.2535 (6)	9.5 (4)
B7	−0.0439 (11)	0.0623 (6)	−0.2313 (6)	7.5 (3)
B8	0.1881 (11)	0.0419 (5)	−0.1650 (5)	6.1 (2)
B9	0.2087 (10)	−0.0647 (5)	−0.1442 (6)	5.8 (2)
B10	0.0439 (11)	−0.0236 (6)	−0.2579 (6)	6.9 (3)
N	0.3383 (6)	0.3313 (3)	0.0747 (3)	4.2 (1)
C11A	0.4756 (14)	0.3328 (6)	0.0333 (8)	6.6 (3)
C21A	0.1538 (12)	0.3256 (6)	−0.0164 (7)	6.0 (4)
C31A	0.3693 (15)	0.2640 (7)	0.1410 (8)	7.1 (4)
C41A	0.3581 (13)	0.4068 (6)	0.1311 (7)	5.8 (3)
C12	0.4937 (11)	0.2629 (6)	−0.0181 (6)	12.9 (3)
C22	0.0003 (10)	0.3262 (5)	0.0072 (7)	10.3 (3)
C32	0.5517 (11)	0.2550 (5)	0.2329 (6)	10.3 (4)
C42	0.3061 (10)	0.4797 (4)	0.0719 (6)	7.2 (3)
C11B	0.3547 (22)	0.2634 (14)	0.0106 (14)	6.9 (7)
C21B	0.1655 (24)	0.3135 (11)	0.0878 (17)	6.8 (7)
C31B	0.4987 (29)	0.3273 (14)	0.1816 (15)	8.3 (8)
C41B	0.3325 (26)	0.4074 (11)	0.0240 (15)	6.8 (6)

\* Anisotropically refined atoms are given in the form of the isotropic equivalent thermal parameter defined as:

$$\frac{4}{3}[a^2B_{11} + b^2B_{22} + c^2B_{33} + abc\cos\gamma B_{12} + accos\beta B_{13} + bccos\alpha B_{23}].$$

Table 2. *Interatomic distances (Å) and selected angles (°)*

Se—Fe	2.280 (1)	B6—B7	1.745 (15)
Se—B1	2.089 (7)	B6—B10	1.731 (14)
Se—B4	2.133 (7)	B7—B8	1.757 (12)
Se—B8	2.109 (8)	B7—B10	1.753 (15)
Se—B9	2.100 (9)	B8—B9	1.832 (12)
Fe—B1	2.195 (8)	B8—B10	1.744 (11)
Fe—B2	2.122 (7)	B9—B10	1.749 (10)
Fe—B3	2.108 (7)	N—C11A	1.513 (14)
Fe—B4	2.194 (9)	N—C21A	1.510 (9)
B1—B2	1.821 (10)	N—C31A	1.444 (12)
B1—B5	1.807 (11)	N—C41A	1.493 (11)
B1—B9	1.916 (14)	C11A—C12	1.45 (2)
B2—B3	1.737 (12)	C21A—C22	1.46 (2)
B2—B5	1.774 (15)	C31A—C32	1.509 (12)
B2—B6	1.775 (13)	C41A—C42	1.460 (12)
B3—B4	1.745 (11)	N—C11B	1.53 (2)
B3—B6	1.750 (14)	N—C21B	1.56 (3)
B3—B7	1.826 (15)	N—C31B	1.53 (2)
B4—B7	1.745 (11)	N—C41B	1.48 (2)
B4—B8	1.931 (14)	C12—C11B	1.39 (3)
B5—B6	1.752 (13)	C22—C21B	1.36 (2)
B5—B9	1.706 (12)	C32—C31B	1.40 (2)
B5—B10	1.755 (14)	C42—C41B	1.48 (2)
Angles around Fe			
Se—Fe—B1	55.6 (2)	B3—Fe—B4	47.8 (3)
B1—Fe—B2	49.9 (3)	B4—Fe—Se	56.9 (2)
B2—Fe—B3	48.5 (3)		
Angles around Se			
Fe—Se—B1	60.1 (2)	B8—Se—B4	54.1 (4)
B1—Se—B9	54.4 (4)	B4—Se—Fe	59.5 (3)
B9—Se—B8	51.6 (3)		
Angles in SeB <sub>4</sub> ring			
B1—Se—B4	95.0 (3)	B2—B3—B4	113.0 (5)
Se—B1—B2	109.0 (4)	B3—B4—Se	110.1 (5)
B1—B2—B3	112.4 (5)		

maps showed maxima in positions consistent with the expected locations of most of the H atoms; in the final rounds of calculations the H atoms were positioned on geometrical grounds (C—H 0.95, B—H 1.10 Å) and included (as riding atoms) in the structure-factor calculations with an overall  $B_{iso}$  of 7.0 Å<sup>2</sup>. The final cycle of refinement included 223 variable parameters,  $R = 0.037$ ,  $wR = 0.044$ , goodness-of-fit 1.49,  $w = 1/[\sigma^2(F_o) + 0.040(F_o)^2]$ . Max. shift/e.s.d. was less than 0.01; density in final difference map  $\pm 0.50$  e Å<sup>−3</sup>, no chemically significant features. Scattering factors and anomalous-dispersion corrections from *International Tables for X-ray Crystallography* (1974). All calculations were performed on a PDP11/73 computer using *SDP-Plus* (B. A. Frenz & Associates Inc., 1983). Atomic coordinates and details of molecular geometry are given in Tables 1 and 2.\* Figs. 1 and 2 are views of the ions prepared using *ORTEP* (Johnson, 1976).

The <sup>57</sup>Fe Mössbauer spectrum (Fig. 3) of a polycrystalline sample of [Et<sub>4</sub>N]<sub>2</sub>[Fe(SeB<sub>10</sub>H<sub>10</sub>)<sub>2</sub>] was recorded at 78 K on an apparatus which has been described previously (Brint, O'Cuill, Spalding & Deeney, 1983). Isomer shift and quadrupole splitting parameters are recorded with respect to sodium nitroprusside as standard in Table 3.

\* Lists of structure factors, thermal parameters, calculated H-atom coordinates, bond angles and mean-planes data have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 43775 (21 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

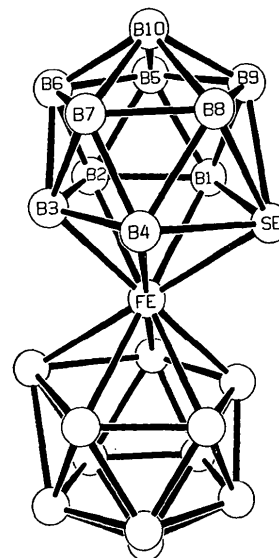


Fig. 1. A view of the Fe(SeB<sub>10</sub>H<sub>10</sub>)<sub>2</sub> anion with our numbering scheme; H atoms are omitted for clarity and the atoms are shown as spheres of arbitrary size.

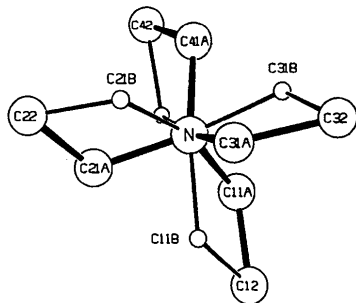


Fig. 2. A view of the disordered  $Et_4N$  cation; the atoms are shown as spheres of arbitrary size.

Table 3. Mössbauer parameters ( $mm\ s^{-1}$ ) for  $[Et_4N]_2[Fe(SeB_{10}H_{10})_2]$  and related compounds with reference to sodium nitroprusside, data recorded at 78 or 80 K

	Isomer shift	Quadrupole splitting
$[Et_4N]_2[Fe(SeB_{10}H_{10})_2]^a$	0.53	2.11
$Cs_2[Fe(SB_{10}H_{10})_2]^b$	0.41	2.14
$Cs_2[Fe(1,2-C_2B_9H_{11})_2]^b$	0.48	2.38
$[Me_4N]_2[Fe(1,2-C_2B_9H_{11})_2]^c$	0.56	2.80
$[Fe(C_2H_5)_2]^c$	0.79	2.42
$[Me_4N][Fe(SB_{10}H_{10})_2]^b$	0.91	0.79
$[Me_4N][Fe(1,2-C_2B_9H_{11})_2]$	0.50 <sup>c</sup>	0.64 <sup>c</sup>
	0.56 <sup>b</sup>	0.58 <sup>b</sup>
$[Fe(C_2H_5)(1,2-C_2B_9H_{11})]^b$	0.70	0.43
$[Fe(C_2H_5)_2][BF_4]^c$	0.48	*

References: (a) present work; (b) Good, Buttone & Foyt (1974); (c) Birchall & Drummond (1971).

\* Unresolved, quadrupole splitting  $\approx 0$ .

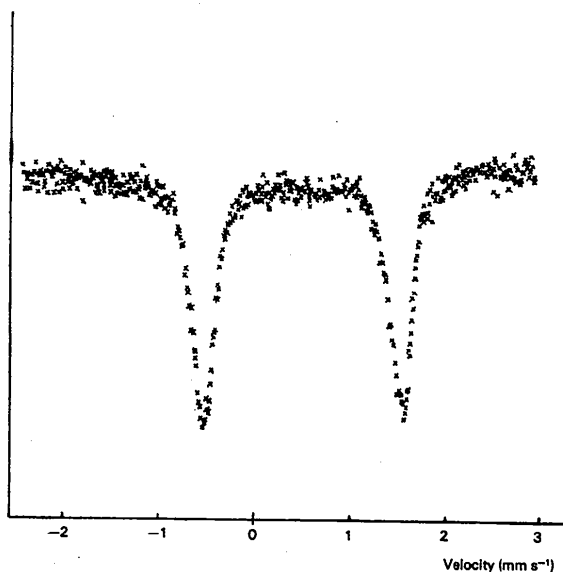


Fig. 3.  $^{57}Fe$  Mössbauer spectrum of  $[Et_4N]_2[Fe(SeB_{10}H_{10})_2]$  at 78 K.

**Discussion.** In the anion (Fig. 1, Table 2), the B—B distances cover a wide range, 1.706 (12) to 1.931 (14) Å, typical of borane cluster compounds (Kennedy, 1984). The particular values reflect the positions of the B atoms *vis à vis* the heteroatoms. For B atoms (B1, B4, B8, B9) adjacent to Se, the range of B—B distances is 1.832 (12) to 1.931 (14) Å whereas for B atoms (B2, B3, B5, B6, B7, B10) not adjacent to Se the range is 1.706 (12) to 1.826 (15) Å. These latter values are similar to those reported for analogous data in *closo*-[1-( $\eta^5$ -C<sub>5</sub>H<sub>5</sub>)-1,2,3-FeC<sub>2</sub>B<sub>9</sub>H<sub>11</sub>] (Zalkin, Templeton & Hopkins, 1965), *closo*-[1-( $\eta^6$ -C<sub>6</sub>H<sub>5</sub>Me)-2,4-Me<sub>2</sub>-1,2,4-FeC<sub>2</sub>B<sub>9</sub>H<sub>9</sub>] (Garcia, Green, Stone, Somerville, Welch, Briant, Cox & Mingos, 1985) and *closo*-[1-( $\eta^6$ -C<sub>6</sub>H<sub>3</sub>Me<sub>3</sub>)-1,2,3-FeC<sub>2</sub>B<sub>9</sub>H<sub>11</sub>] (Hanusa, Huffman & Todd, 1982). Distances between B atoms and the heteroatoms show much less variation. The

B—Se values are between 2.089 (7) and 2.133 (7) Å and those of B—Fe are between 2.108 (7) and 2.195 (8) Å. Again, the latter values are similar to the Fe—B data in the ferracarboranes mentioned above [2.09, 2.069 (7) to 2.008 (12), and 2.083 (13) to 2.147 (11) Å, respectively].

The Fe—Se distance, 2.280 (1) Å, is notably short compared to the Fe—Se distances in other clusters such as *nido*-[Fe<sub>2</sub>(CO)<sub>6</sub>Se<sub>2</sub>] (Campana, Lo & Dahl, 1979), which are in the range 2.354 (2) to 2.378 (2) Å. With the Fe atom occupying an inversion centre, the planes containing the SeB<sub>4</sub> rings attached to the Fe atom are of necessity parallel. The cations (Fig. 2) are disordered with CH<sub>2</sub> groups in two sets with anticipated dimensions. There are no untoward inter-ion contacts.

Mössbauer spectra have previously been recorded for a number of compounds related to the title complex. Generally, the values of isomer shift do not vary greatly, but quadrupole splittings clearly show two distinct ranges of values depending on the oxidation state of the Fe atom, Table 3. For compounds with Fe<sup>II</sup> the quadrupole splitting values lie between 2.11 and 2.80  $mm\ s^{-1}$ , whereas for Fe<sup>III</sup> compounds the values are much closer to zero ( $\sim 0$  to 0.79  $mm\ s^{-1}$ ). For the complexes with 2,3-C<sub>2</sub>B<sub>9</sub>H<sub>11</sub> or SB<sub>10</sub>H<sub>10</sub> ligands, both Fe<sup>II</sup> and Fe<sup>III</sup> data have been obtained. The spectrum of  $[Et_4N]_2[Fe(SeB_{10}H_{10})_2]$  (Fig. 3) is a simple doublet whose parameters are close to those for the sulfur analogue, suggesting a very similar electronic environment at Fe (Table 3).

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### *cis*-(*E*)-Bis(7-methyl-1-naphthyl)bis(triphenylphosphine)platinum(II)

BY T. DEBAERDEMAEKER AND K. BERHALTER

*Sektion Röntgen- und Elektronenbeugung, Universität Ulm, D-7900 Ulm, Federal Republic of Germany*

AND C. WEISEMANN AND H. A. BRUNE

*Lehrstuhl Organische Chemie I, Universität Ulm, D-7900 Ulm, Federal Republic of Germany*

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**Abstract.**  $[\text{Pt}(\text{C}_{11}\text{H}_9)_2(\text{C}_{18}\text{H}_{15}\text{P}_2)] \cdot \text{CH}_2\text{Cl}_2$ ,  $M_r = 1087.08$ , monoclinic, space group  $P2_1/a$ ,  $a = 18.104$  (4),  $b = 20.872$  (4),  $c = 13.680$  (3) Å,  $\beta = 106.27$  (4)°,  $V = 4962.1$  Å<sup>3</sup>,  $Z = 4$ ,  $D_x = 1.455$ ,  $D_m = 1.468$  g cm<sup>-3</sup> (flotation method in  $\text{CCl}_4$ /hexane), graphite-monochromated Mo  $K\alpha$ ,  $\lambda = 0.71069$  Å,  $\mu = 30.65$  cm<sup>-1</sup>,  $F(000) = 2184$ ,  $T = 295$  K, 6082 reflections measured, 3133 of which were taken as observed; final  $R$  value 0.049. Each molecule of the title compound crystallizes with one molecule of  $\text{CH}_2\text{Cl}_2$ . The carbon atoms C(1) and C(12) of the naphthyl ligands and the P atoms of the phosphine ligands lie in a plane containing nearly  $dsp^2$ -hybridized Pt. The two naphthyl ligands are approximately perpendicular (88.8 and 87.2°) to this plane and *anti*-orientated to one another (*E* conformation).

**Introduction.** In the course of experiments on the stereochemistry of platinum–organic compounds we were interested in the mutual orientation of large planar aryl ligands directly bonded to platinum in *cis*- and *trans*-bis(aryl)bis(ligand)platinum(II) compounds (Brune, Wiege & Debaerdemaeker, 1984; Debaerdemaeker, Stapp & Brune, 1987; Debaerdemaeker, Weisemann & Brune, 1987). As a contribution to the stereochemistry of these compounds the structure of *cis*-(*E*)-bis(7-methyl-1-naphthyl)bis(triphenylphosphine)platinum(II) (Fig. 1) is reported here.

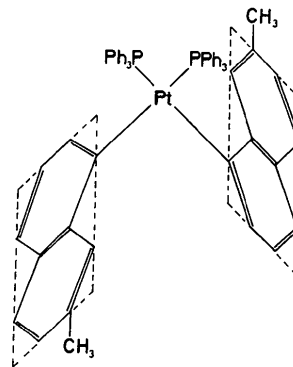


Fig. 1. Schematic illustration of *cis*-(*E*)-bis(7-methyl-1-naphthyl)-bis(triphenylphosphine)platinum(II).

**Experimental.** The synthesis of the title compound was previously reported (Brune, Schäfer, Spohn & Weisemann, 1986). Crystals (0.3 × 0.1 × 0.1 mm approximately) were obtained by the very slow cooling of solutions of the title compound in  $\text{CH}_2\text{Cl}_2$ /methanol (1/1). Philips PW-1100 four-circle diffractometer; Mo  $K\alpha$  radiation, graphite monochromator,  $\lambda = 0.71069$  Å.  $\theta$ – $2\theta$  scan; 6082 unique reflections measured, 3133 observed reflections [ $F > 2.0\sigma(F)$ ,  $-18 \leq h \leq 18$ ,  $0 \leq k \leq 20$ ,  $0 \leq l \leq 13$ ,  $\theta_{\max} = 22^\circ$ ]. The intensities of three monitor reflections did not vary