

## Structure of $\{(1\alpha,3\alpha,5\beta)\text{-(NSOPh)}_2\text{NPF}\}_2[(\eta^5\text{-C}_5\text{H}_4)\text{Fe}(\eta^5\text{-C}_5\text{H}_4)]$

BY A. MEETSMA

Department of Chemistry, University of Groningen, Nijenborgh 16, 9747 AG Groningen, The Netherlands

H. F. M. SCHOON AND J. C. VAN DE GRAMPPEL\*

Department of Inorganic Chemistry, University of Groningen, Nijenborgh 16, 9747 AG Groningen, The Netherlands

AND W. P. BOSMAN

Crystallography Laboratory, University of Nijmegen, Toernooiveld, 6525 ED Nijmegen, The Netherlands

(Received 1 July 1987; accepted 28 July 1987)

**Abstract.** 5,5'-Ferrocenediyl-di(5-fluoro-1,3-diphenyl-1 $\lambda^6$ ,3 $\lambda^6$ ,2,4,6,5 $\lambda^5$ -dithiatriazaphosphorine 1,3-dioxide),  $\text{C}_{34}\text{H}_{28}\text{F}_2\text{FeN}_6\text{O}_4\text{P}_2\text{S}_4$ ,  $M_r=868.66$ , monoclinic,  $P2_1/a$ ,  $a=24.356$  (5),  $b=10.018$  (9),  $c=24.402$  (7) Å,  $\beta=116.99$  (2)°,  $V=5306$  (5) Å<sup>3</sup>,  $Z=6$ ,  $D_x=1.631$  g cm<sup>-3</sup>,  $\lambda(\text{Mo } K\alpha)=0.71073$  Å,  $\mu=8.0$  cm<sup>-1</sup>,  $F(000)=2664$ ,  $T=130$  K,  $R=0.040$  for 6292 observed reflections with  $I > 2.5\sigma(I)$ . Two independent molecules are present in the unit cell, one obeying symmetry  $\bar{1}$ . The title compound consists of two *S*-phenyl-substituted SNP ring systems connected by a bridging ferrocenyl unit. The pentadienyl groups exhibit a staggered conformation. The mutually *cis*-located phenyl groups are in *trans* position towards the  $\text{C}_5\text{H}_4\text{FeC}_5\text{H}_4$  part. Two sets of S–N bond lengths can be discerned with mean values 1.558 (2) (in a P–N–S segment) and 1.586 (2) Å (in an S–N–S segment). Mean P–N is 1.580 (2) Å. Exocyclic bond lengths (mean values) are S–O = 1.427 (2), S–C = 1.757 (3), P–F = 1.553 (2) and P–C = 1.741 (3) Å. The ferrocenyl groups display normal geometry.

**Introduction.** Under well chosen reaction conditions the application of the organolithium reagent  $(\eta^5\text{-C}_5\text{H}_4\text{Li})_2\text{Fe.TMEDA}$  towards the new ring system *cis*-(NSOPh)<sub>2</sub>NPF<sub>2</sub> leads to the formation of the title compound. From an NMR analysis only one of the possible isomers appeared to be formed. This finding prompted us to carry out an X-ray diffraction study in order to establish the mutual positions of the exocyclic ligands and in this way to gather some insight into the mode of attack of the organometallic reagent. Moreover, it seemed interesting to compare the conformation of the ferrocenyl unit with that found in related structures.

**Experimental.** Suitable orange-coloured block-shaped crystals obtained by recrystallization from benzene. Crystal (0.45 × 0.38 × 0.05 mm) was glued on top of a glass fibre and transferred into the cold nitrogen stream of the low-temperature unit on an Enraf–Nonius CAD-4F diffractometer interfaced to a PDP-11/23 computer. Graphite-monochromated Mo  $K\alpha$  radiation. Cell dimensions from setting angles of 22 reflections ( $6.16 < \theta < 11.91^\circ$ ). The cell was checked for the presence of higher lattice symmetry (Le Page, 1982); the observed pseudo *oS* (de Wolff *et al.*, 1985) Bravais lattice type was found to be inconsistent with the symmetry of the reflection data. Intensity data of 16 482 reflections were collected within one half of the reflection sphere;  $h -28 \rightarrow 28$ ,  $k 0 \rightarrow 11$ ,  $l -28 \rightarrow 23$ ;  $1.6 < \theta < 25.0^\circ$ ;  $\omega/2\theta$  scan mode with  $\Delta\omega = (0.85 + 0.35 \tan\theta)^\circ$ . Two reference reflections measured every 100 min (031: r.m.s.d. 2.1%; 031: r.m.s.d. 1.8%) showed no indication of crystal decomposition during the 311 h of X-ray exposure time. Intensities corrected for scale variation (min. and max. 0.95 and 1.04, respectively) and for Lorentz and polarization effects. No absorption correction applied. Variance  $\sigma^2(I)$  calculated on the basis of counting statistics plus the term  $(PI)^2$ , where  $P (=0.026)$  is the instability constant as derived from the excess variance in the reference reflections (McCandlish, Stout & Andrews, 1975). Data set averaged in a set of 9316 reflections. The Fe atoms were located by Patterson techniques. Extension of the model was accomplished by using direct methods applied to difference structure factors (DIRDIF; Beurskens *et al.*, 1982) and refined on  $F$  by block-diagonal least-squares techniques with anisotropic thermal parameters for non-hydrogen atoms. H atoms located on a difference Fourier map and included in the final refinement with isotropic temperature factors. Convergence with 888 parameters was reached at  $R=0.040$ ,  $wR=0.038$ ,  $w=1/\sigma^2(F)$ ,  $S=1.981$ ;

\* Author to whom correspondence should be addressed.

6293 observed reflections with  $I > 2.5\sigma(I)$ ; average  $\Delta/\sigma = 0.025$ , maximum  $\Delta/\sigma = 0.964$ . Minimum and maximum residual densities in final Fourier map  $-0.60$  and  $0.40 \text{ e } \text{Å}^{-3}$ . Correction for isotropic secondary extinction applied (Zachariasen, 1967),  $g = 3.4(3) \times 10^{-4}$ . The  $\bar{1}3,3,17$  reflection with a  $\Delta F/\sigma(F)$  ratio of 45.6 was excluded from the final refinement. Final fractional atomic coordinates and equivalent isotropic thermal parameters for the non-hydrogen atoms are given in Table 1.\* Scattering factors from Cromer & Mann (1968). Anomalous-dispersion factors were taken from Cromer & Liberman (1970). All calculations carried out on the CDC-Cyber 170/760 computer of the University of Groningen with the program packages *XTAL* (Stewart & Hall, 1983), *EUCLID* (Spek, 1982; calculation of geometric data) and a locally modified version of the program *PLUTO* (Meetsma, 1986; Motherwell & Clegg, 1978; preparation of illustrations).

\* Lists of structure factors, anisotropic thermal parameters, H-atom parameters and bond lengths and angles involving H atoms and an *ORTEP* plot (Johnson, 1965) have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 44290 (52 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. Final fractional atomic coordinates and equivalent isotropic thermal parameters for non-H atoms with e.s.d.'s in parentheses

$$U_{eq} = \frac{1}{3} \sum_i \sum_j U_{ij} a_i^* a_j^* a_i a_j$$

	x	y	z	$U_{eq}(\text{Å}^2)$
<b>Molecule A</b>				
Fe(1)	0.50000	0.00000	0.50000	0.0280 (3)
S(11)	0.52878 (4)	0.4072 (1)	0.60392 (5)	0.0244 (3)
S(12)	0.55516 (4)	0.2018 (1)	0.68941 (4)	0.0228 (3)
P(11)	0.44977 (5)	0.1938 (1)	0.57957 (5)	0.0266 (4)
F(11)	0.3854 (1)	0.2101 (3)	0.5773 (1)	0.040 (1)
O(11)	0.5588 (1)	0.4578 (3)	0.5700 (1)	0.031 (1)
O(12)	0.6041 (1)	0.1066 (3)	0.7165 (1)	0.029 (1)
N(11)	0.4666 (1)	0.3356 (3)	0.5636 (1)	0.026 (1)
N(12)	0.5758 (1)	0.3141 (3)	0.6572 (1)	0.023 (1)
N(13)	0.4928 (2)	0.1355 (3)	0.6454 (1)	0.027 (1)
C(11)	0.5100 (2)	0.5465 (4)	0.6367 (2)	0.025 (1)
C(12)	0.5474 (2)	0.6566 (4)	0.6521 (2)	0.035 (2)
C(13)	0.5343 (2)	0.7662 (5)	0.6778 (2)	0.048 (2)
C(14)	0.4832 (2)	0.7633 (5)	0.6888 (2)	0.047 (2)
C(15)	0.4462 (2)	0.6533 (5)	0.6738 (2)	0.043 (2)
C(16)	0.4593 (2)	0.5428 (5)	0.6478 (2)	0.033 (2)
C(17)	0.5445 (2)	0.2795 (4)	0.7484 (2)	0.020 (1)
C(18)	0.5878 (2)	0.3711 (5)	0.7851 (2)	0.034 (2)
C(19)	0.5800 (2)	0.4324 (5)	0.8308 (2)	0.043 (2)
C(110)	0.5306 (2)	0.4015 (4)	0.8415 (2)	0.033 (2)
C(111)	0.4890 (2)	0.3074 (4)	0.8058 (2)	0.033 (2)
C(112)	0.4955 (2)	0.2451 (4)	0.7591 (2)	0.024 (1)
C(113)	0.4349 (2)	0.0807 (4)	0.5204 (2)	0.024 (1)
C(114)	0.4326 (2)	-0.0606 (4)	0.5221 (2)	0.029 (1)
C(115)	0.4197 (2)	-0.1076 (5)	0.4629 (2)	0.037 (2)
C(116)	0.4146 (2)	0.0010 (5)	0.4254 (2)	0.035 (1)
C(117)	0.4240 (2)	0.1185 (5)	0.4595 (2)	0.031 (1)
<b>Molecule B</b>				
Fe(2)	0.18363 (2)	0.09443 (6)	0.17247 (3)	0.0186 (2)
S(21)	0.16439 (4)	0.5088 (1)	0.25401 (4)	0.0193 (3)
S(22)	0.22425 (5)	0.3298 (1)	0.34875 (4)	0.0214 (3)
S(31)	0.20146 (4)	-0.3232 (1)	0.09409 (4)	0.0205 (3)
S(32)	0.14744 (5)	-0.1446 (1)	-0.00268 (4)	0.0208 (3)
P(21)	0.25812 (5)	0.3580 (1)	0.25569 (5)	0.0187 (3)
P(31)	0.10730 (5)	-0.1629 (1)	0.08602 (5)	0.0208 (3)

Table 1 (cont.)

	x	y	z	$U_{eq}(\text{Å}^2)$
F(21)	0.31713 (9)	0.4332 (2)	0.2642 (1)	0.0264 (6)
F(31)	0.0444 (1)	-0.2288 (2)	0.0717 (1)	0.0310 (6)
O(21)	0.1022 (1)	0.5408 (3)	0.2118 (1)	0.024 (1)
O(22)	0.2074 (1)	0.2078 (3)	0.3674 (1)	0.029 (1)
O(31)	0.2621 (1)	-0.3518 (3)	0.1401 (1)	0.028 (1)
O(32)	0.1677 (1)	-0.0297 (3)	-0.0229 (1)	0.027 (1)
N(21)	0.2018 (1)	0.4564 (3)	0.2207 (1)	0.018 (1)
N(22)	0.1631 (1)	0.4041 (3)	0.3019 (1)	0.020 (1)
N(23)	0.2714 (1)	0.3119 (3)	0.3224 (1)	0.022 (1)
N(31)	0.1589 (1)	-0.2705 (3)	0.1213 (1)	0.021 (1)
N(32)	0.2063 (1)	-0.2213 (3)	0.0468 (1)	0.020 (1)
N(33)	0.0995 (1)	-0.1124 (3)	0.0215 (1)	0.025 (1)
C(21)	0.2014 (2)	0.6532 (4)	0.2952 (2)	0.018 (1)
C(22)	0.2524 (2)	0.7049 (4)	0.2919 (2)	0.021 (1)
C(23)	0.2804 (2)	0.8160 (4)	0.3259 (2)	0.022 (1)
C(24)	0.2581 (2)	0.8772 (4)	0.3618 (2)	0.025 (1)
C(25)	0.2075 (2)	0.8239 (4)	0.3654 (2)	0.027 (1)
C(26)	0.1794 (2)	0.7123 (4)	0.3326 (2)	0.022 (1)
C(27)	0.2594 (2)	0.4320 (4)	0.4142 (2)	0.021 (1)
C(28)	0.3175 (2)	0.4814 (4)	0.4335 (2)	0.025 (1)
C(29)	0.3421 (2)	0.5612 (4)	0.4852 (2)	0.032 (2)
C(31)	0.1674 (2)	-0.4696 (4)	0.0531 (2)	0.022 (1)
C(32)	0.1143 (2)	-0.5205 (4)	0.0520 (2)	0.023 (1)
C(33)	0.0874 (2)	-0.6319 (4)	0.0167 (2)	0.029 (1)
C(34)	0.1135 (2)	-0.6917 (4)	-0.0160 (2)	0.036 (2)
C(35)	0.1664 (2)	-0.6405 (4)	-0.0150 (2)	0.036 (2)
C(36)	0.1936 (2)	-0.5291 (4)	0.0193 (2)	0.026 (1)
C(37)	0.1108 (2)	-0.2499 (4)	-0.0660 (2)	0.020 (1)
C(38)	0.0570 (2)	-0.3176 (4)	-0.0773 (2)	0.028 (1)
C(39)	0.0327 (2)	-0.4070 (4)	-0.1249 (2)	0.034 (2)
C(210)	0.3090 (2)	0.5936 (4)	0.5161 (2)	0.030 (2)
C(211)	0.2504 (2)	0.5425 (4)	0.4960 (2)	0.029 (1)
C(212)	0.2253 (2)	0.4616 (4)	0.4456 (2)	0.028 (1)
C(213)	0.2549 (2)	0.2239 (4)	0.2087 (2)	0.016 (1)
C(214)	0.2757 (2)	0.0918 (4)	0.2290 (2)	0.020 (1)
C(215)	0.2634 (2)	0.0140 (4)	0.1774 (2)	0.024 (1)
C(216)	0.2347 (2)	0.0946 (4)	0.1243 (2)	0.024 (1)
C(217)	0.2296 (2)	0.2247 (4)	0.1437 (2)	0.020 (1)
C(310)	0.0607 (2)	-0.4281 (4)	-0.1618 (2)	0.040 (2)
C(311)	0.1137 (2)	-0.3599 (5)	-0.1510 (2)	0.039 (2)
C(312)	0.1395 (2)	-0.2716 (4)	-0.1032 (2)	0.030 (1)
C(313)	0.1119 (2)	-0.0327 (4)	0.1350 (2)	0.019 (1)
C(314)	0.1387 (2)	-0.0384 (4)	0.2002 (2)	0.023 (1)
C(315)	0.1342 (2)	0.0874 (5)	0.2220 (2)	0.030 (2)
C(316)	0.1050 (2)	0.1739 (4)	0.1710 (2)	0.033 (2)
C(317)	0.0909 (2)	0.1003 (4)	0.1168 (2)	0.023 (1)

**Discussion.** Relevant data on the geometry are given in Table 2. The molecular structure (molecules *A* and *B*) is depicted in Fig. 1, which also explains the atom-numbering scheme.

The monoclinic unit cell contains two sets of crystallographically independent molecules with two *A* molecules and four *B* molecules, respectively. Molecule *A* has a centre of inversion imposed by the crystal symmetry. A projection of the unit-cell contents down [010] is shown in Fig. 2.

In each molecule the arrangement of the ligands with respect to the SNP ring systems appears to be equal, *viz.* phenyl, *cis*-phenyl, *trans*- $\text{C}_5\text{H}_4$ . This means that the nucleophilic attack of  $(\text{LiC}_5\text{H}_4)_2\text{Fe}$  towards *cis*-(NSOPH)<sub>2</sub>NPF<sub>2</sub> takes place exclusively at the side of the oxygen atoms, thus avoiding the steric hindrance of the large phenyl groups.

The observed conformations of the SNP rings approach that of an envelope for [S(11)–N(12)] (Boeyens, 1978) with torsion angles ranging from  $-31.0(2)$  to  $32.6(3)^\circ$ , lowest asymmetry parameter value  $\Delta C_s[\text{N}(12)] = 2.3(3)^\circ$  (Duax, Weeks & Rohrer, 1976); a skew boat for [S(21)–N(22)] with torsion angles ranging from  $-30.8(2)$  to  $46.0(2)^\circ$ , lowest

Table 2. Selected bond distances (Å), angles (°) and torsion angles (°)

Molecule A			
S(11)–N(11)	1.557 (3)	S(11)–O(11)	1.423 (3)
S(11)–N(12)	1.588 (3)	S(12)–O(12)	1.433 (3)
S(12)–N(12)	1.579 (3)	S(11)–C(11)	1.769 (5)
S(12)–N(13)	1.556 (4)	S(12)–C(17)	1.756 (5)
P(11)–N(11)	1.576 (3)	P(11)–F(11)	1.552 (3)
P(11)–N(13)	1.579 (3)	P(11)–C(113)	1.739 (5)
N(11)–S(11)–N(12)	112.3 (2)	S(12)–N(13)–P(11)	122.3 (2)
N(12)–S(12)–N(13)	113.1 (1)	O(11)–S(11)–C(11)	106.8 (2)
N(11)–P(11)–N(13)	117.3 (2)	O(12)–S(12)–C(17)	108.6 (2)
P(11)–N(11)–S(11)	123.1 (2)	C(113)–P(11)–F(11)	102.3 (2)
S(11)–N(12)–S(12)	123.5 (2)		
N(12)–S(11)–N(11)–P(11)	7.3 (3)	N(12)–S(12)–N(13)–P(11)	–10.5 (4)
N(11)–S(11)–N(12)–S(12)	–31.0 (2)	N(13)–P(11)–N(11)–S(11)	11.5 (3)
N(13)–S(12)–N(12)–S(11)	32.6 (3)	N(11)–P(11)–N(13)–S(12)	–9.8 (4)
Molecule B			
S(21)–N(21)	1.562 (3)	S(31)–N(31)	1.555 (3)
S(21)–N(22)	1.581 (3)	S(31)–N(32)	1.585 (3)
S(22)–N(22)	1.592 (3)	S(32)–N(32)	1.591 (3)
S(22)–N(23)	1.560 (3)	S(32)–N(33)	1.562 (3)
P(21)–N(21)	1.587 (3)	P(31)–N(31)	1.581 (3)
P(21)–N(23)	1.578 (3)	P(31)–N(33)	1.581 (3)
S(21)–O(21)	1.429 (3)	S(31)–O(31)	1.421 (3)
S(22)–O(22)	1.428 (3)	S(32)–O(32)	1.426 (3)
S(21)–C(21)	1.759 (5)	S(31)–C(31)	1.757 (5)
S(22)–C(27)	1.758 (5)	S(32)–C(37)	1.745 (5)
P(21)–F(21)	1.551 (3)	P(31)–F(31)	1.555 (3)
P(21)–C(213)	1.745 (5)	P(31)–C(313)	1.738 (5)
N(21)–S(21)–N(22)	111.9 (2)	N(31)–S(31)–N(32)	112.1 (2)
N(22)–S(22)–N(23)	112.4 (1)	N(32)–S(32)–N(33)	112.5 (1)
N(21)–P(21)–N(23)	117.3 (2)	N(31)–P(31)–N(33)	117.8 (2)
P(21)–N(21)–S(21)	119.7 (2)	P(31)–N(31)–S(31)	121.2 (2)
S(21)–N(22)–S(22)	121.0 (2)	S(31)–N(32)–S(32)	121.6 (2)
S(22)–N(23)–P(21)	123.2 (2)	S(32)–N(33)–P(31)	122.5 (2)
O(21)–S(21)–C(21)	109.2 (2)	O(31)–S(31)–C(31)	109.5 (2)
O(22)–S(22)–C(27)	107.5 (2)	O(32)–S(32)–C(37)	107.4 (2)
C(213)–P(21)–F(21)	103.0 (2)	C(313)–P(31)–F(31)	102.9 (2)
N(22)–S(21)–N(21)–P(21)	–30.8 (2)	N(32)–S(31)–N(31)–P(31)	23.4 (2)
N(21)–S(21)–N(22)–S(22)	46.0 (2)	N(31)–S(31)–N(32)–S(32)	–42.4 (2)
N(23)–S(22)–N(22)–S(21)	–29.4 (3)	N(33)–S(32)–N(32)–S(31)	33.1 (3)
N(22)–S(22)–N(23)–P(21)	–2.2 (3)	N(32)–S(32)–N(33)–P(31)	–5.0 (3)
N(23)–P(21)–N(21)–S(21)	3.0 (3)	N(33)–P(31)–N(31)–S(31)	1.1 (3)
N(21)–P(21)–N(23)–S(22)	14.9 (3)	N(31)–P(31)–N(33)–S(32)	–11.2 (3)

The iron atoms are equidistant between the planes [in *A* 1.65 (4) and in *B* 1.66 (2) and 1.65 (1) Å, respectively]. Mean C–C bond lengths: [C(113)–C(117)] = 1.411 (3), [C(213)–C(217)] = 1.411 (3) and [C(313)–C(317)] = 1.413 (3) Å. For symmetry reasons the C<sub>5</sub>H<sub>4</sub> groups in molecule *A* have a staggered conformation. In molecule *B* the ferrocenyl moiety exhibits an almost staggered conformation [torsion angle C(213)–ct(2)–ct(3)–C(313) = 177.5 (3)° (ct is the centroid of the ring)]. This arrangement differs from that observed in related molecules (NPCl<sub>2</sub>)<sub>2</sub>–NP<sup>i</sup>PrCH(OH)C<sub>5</sub>H<sub>4</sub>FeC<sub>5</sub>H<sub>5</sub> (Meetsma, Buwalda & van de Grampel, 1987), N<sub>3</sub>P<sub>3</sub>F<sub>5</sub>C<sub>5</sub>H<sub>4</sub>FeC<sub>5</sub>H<sub>5</sub> (Allcock, Lavin, Riding, Suszko & Whittle, 1984), ferrocene

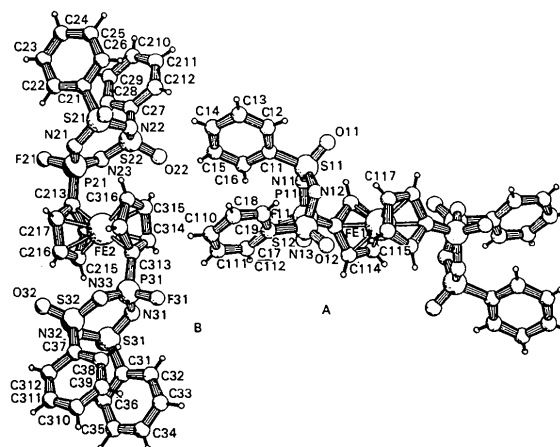


Fig. 1. PLUTO drawing of the two independent molecules (*A* and *B*) of the title compound with the adopted atom-numbering scheme; some carbon atoms are not labelled for the sake of clarity.

asymmetry parameter value  $\Delta C_2[\text{P}(21)\text{--N}(23)] = 3.8 (3)^\circ$ ; a skew boat for [S(31)–N(32)] with torsion angles ranging from  $-42.4 (2)$  to  $33.1 (3)^\circ$ , lowest asymmetry parameter value  $\Delta C_2[\text{P}(31)\text{--N}(33)] = 8.1 (3)^\circ$ . Puckering parameters are:  $Q = 0.305 (2) \text{ \AA}$ ,  $\theta = 108.8 (4)^\circ$ ,  $\phi = 116.4 (5)^\circ$  (Cremer & Pople, 1975; e.s.d. following Norrestam, 1981),  $Q = 0.393 (2) \text{ \AA}$ ,  $\theta = 66.9 (3)^\circ$ ,  $\phi = 334.1 (3)^\circ$  and  $Q = 0.356 (2) \text{ \AA}$ ,  $\theta = 115.2 (3)^\circ$ ,  $\phi = 140.6 (4)^\circ$ , respectively.

The S–N and P–N bond lengths are not equal. Their sequence can be explained from the different electronegativities of the SOPh and PFC<sub>5</sub>H<sub>4</sub>R centres (Winter, van de Grampel, de Boer, Meetsma & Spek, 1987). The phenyl rings display normal geometry with mean C–C bond lengths: [C(11)–C(16)] = 1.376 (3), [C(17)–C(112)] = 1.372 (3), [C(21)–C(26)] = 1.375 (3), [C(27)–C(212)] = 1.376 (3), [C(31)–C(36)] = 1.376 (3) and [C(37)–C(312)] = 1.377 (3) Å.

In the ferrocenyl moieties the planar C<sub>5</sub>H<sub>4</sub> groups are in parallel position (for molecule *A* imposed by symmetry) and separated by 3.30 (*A*) and 3.31 Å (*B*).

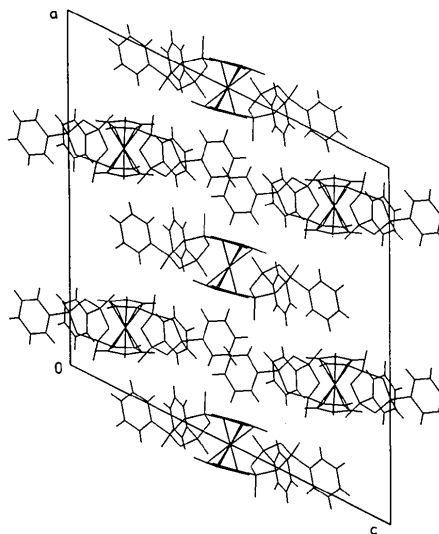


Fig. 2. Molecular packing viewed down [010]; hydrogen bonds omitted.

(Seiler & Dunitz, 1979) and ferrocene derivatives (Krukoniš, Silverman & Yannoni, 1972; Hall & Brown, 1971; Palenik, 1969; Palenik, 1970).

Between the molecules weak hydrogen bonding (Berkovitch-Yellin & Leiserowitz, 1984) is observed: H(28)···O(11) ( $1-x, 1-y, 1-z$ ) = 2.41 (3) Å [sum of the van der Waals radii is 2.72 Å (Bondi, 1964)], the angle C(28)—H(28)···O(11) = 127 (2)°, H(33)···F(31) ( $-x, -1-y, -z$ ) = 2.47 (5) Å [sum of the van der Waals radii is 2.67 Å], C(33)—H(33)···F(31) = 131 (3)°, and H(210)···O(22) ( $\frac{1}{2}-x, \frac{1}{2}+y, 1-z$ ) = 2.49 (3) Å, C(210)—H(210)···O(22) = 133 (2)°.

#### References

- ALLCOCK, H. R., LAVIN, K. D., RIDING, G. H., SUSZKO, P. R. & WHITTLE, R. R. (1984). *J. Am. Chem. Soc.* **106**, 2337–2347.  
BERKOVITCH-YELLIN, Z. & LEISEROWITZ, L. (1984). *Acta Cryst.* **B40**, 159–165.  
BEURSKENS, P. T., BOSMAN, W. P., DOESBURG, H. M., VAN DEN HARK, TH. E. M., PRICK, P. A. J., NOORDIK, J. H., BEURSKENS, G., GOULD, R. O. & PARTHASARATHI, V. (1982). *Conformation in Biology*, edited by R. SRINIVASAN & R. H. SARMA, pp. 389–406. New York: Adenine Press.  
BOEYENS, J. C. A. (1978). *J. Cryst. Mol. Struct.* **8**, 317–320.  
BONDI, A. (1964). *J. Phys. Chem.* **68**, 441–451.  
CREMER, D. & POPLE, J. A. (1975). *J. Am. Chem. Soc.* **97**, 1354–1358.  
CROMER, D. T. & LIBERMAN, D. (1970). *J. Chem. Phys.* **53**, 1891–1898.  
CROMER, D. T. & MANN, J. B. (1968). *Acta Cryst.* **A24**, 321–324.

- DUAX, W. L., WEEKS, C. M. & ROHRER, D. C. (1976). *Topics in Stereochemistry*, Vol. 9, edited by E. L. ELIEL & N. ALLINGER, pp. 271–383. New York: John Wiley.  
HALL, L. H. & BROWN, G. M. (1971). *Acta Cryst.* **B27**, 81–90.  
JOHNSON, C. K. (1965). *ORTEP*. Report ORNL-3794. Oak Ridge National Laboratory, Tennessee, USA.  
KRUKONIS, A. P., SILVERMAN, J. & YANNONI, N. F. (1972). *Acta Cryst.* **B28**, 987–990.  
LE PAGE, Y. (1982). *J. Appl. Cryst.* **15**, 255–259.  
MCCANDLISH, L. E., STOUT, G. H. & ANDREWS, L. C. (1975). *Acta Cryst.* **A31**, 245–249.  
MEETSMA, A. (1986). Extended version of the program *PLUTO*. Univ. of Groningen, The Netherlands.  
MEETSMA, A., BUWALDA, P. L. & VAN DE GRAMPPEL, J. C. (1987). *Acta Cryst.* Submitted.  
MOTHERWELL, W. D. S. & CLEGG, W. (1978). *PLUTO*. Program for plotting molecular and crystal structures. Univ. of Cambridge, England.  
NORRESTAM, R. (1981). *Acta Cryst.* **A37**, 764–765.  
PALENIK, G. J. (1969). *Inorg. Chem.* **8**, 2744–2749.  
PALENIK, G. J. (1970). *Inorg. Chem.* **9**, 2424–2430.  
SEILER, P. & DUNITZ, J. D. (1979). *Acta Cryst.* **B35**, 2020–2032.  
SPEK, A. L. (1982). *The EUCLID Package*. In *Computational Crystallography*, edited by D. SAYRE, p. 528. Oxford: Clarendon Press.  
STEWART, J. H. & HALL, S. R. (1983). *The XTAL System*. Tech. Rep. TR-1364. Computer Science Center, Univ. of Maryland, College Park, Maryland, USA.  
WINTER, H., VAN DE GRAMPPEL, J. C., DE BOER, J. L., MEETSMA, A. & SPEK, A. L. (1987). *Phosphorus Sulfur*, **32**, 145–151.  
WOLFF, P. M. DE, BELOV, N. V., BERTAUT, E. F., BUERGER, M. J., DONNAY, J. D. H., FISCHER, W., HAHN, TH., KOPSTIK, V. A., MACKAY, A. L., WONDRA TSCHKE, H., WILSON, A. J. C. & ABRAHAMS, S. C. (1985). *Acta Cryst.* **A41**, 278–280.  
ZACHARIASEN, W. H. (1967). *Acta Cryst.* **23**, 558–564.

*Acta Cryst.* (1987). **C43**, 2317–2319

## Structure of Di(*tert*-butyl)bis(*N,N*-dimethyldithiocarbamate)tin(IV)

BY KIMOON KIM AND JAMES A. IBERS

*Department of Chemistry, Northwestern University, Evanston, IL 60208, USA*

AND OK-SANG JUNG AND YOUN SOO SOHN

*Inorganic Chemistry Laboratory, Korean Advanced Institute of Science and Technology, Seoul 131, Korea*

(Received 11 June 1987; accepted 10 July 1987)

**Abstract.** [Sn(C<sub>4</sub>H<sub>9</sub>)<sub>2</sub>(C<sub>3</sub>H<sub>6</sub>NS<sub>2</sub>)<sub>2</sub>], *M<sub>r</sub>* = 473.35, monoclinic, *P*2<sub>1</sub>/*c*, *a* = 8.405 (2), *b* = 15.020 (4), *c* = 16.377 (5) Å, β = 96.01 (1)°, *V* = 2056 (1) Å<sup>3</sup>, *Z* = 4, *D<sub>x</sub>* = 1.529 g cm<sup>-3</sup>, graphite-monochromated Mo *K*α, λ(*K*α<sub>1</sub>) = 0.7093 Å, μ = 16.34 cm<sup>-1</sup>, *F*(000) = 966.30 (including anomalous dispersion), *T* = 113 K, *R*(*F*<sup>2</sup>) = 0.033 for 4291 unique reflections. The Sn atom is five-coordinate: one of the *N,N*-dimethyldithiocarbamate (dtc) ligands acts as a bidentate ligand while the other is monodentate. The coordination geometry around the Sn atom is a distorted trigonal bipyramid:

two C atoms of the *tert*-butyl groups and one S atom of the bidentate dtc ligand are equatorial while the other two S atoms are axial.

**Introduction.** Organotin(IV) dithiocarbamate complexes have demonstrated a variety of coordination geometries around the Sn atom, with coordination numbers ranging from 4 to 7 (Zubieta & Zuckerman, 1978). The coordination geometry depends upon the bonding mode of the dithiocarbamate moiety, which acts either as a monodentate or as a bidentate ligand. In