

and C—N = 1.496 (6) Å] are within the range normally observed for the aet metal complexes (Konno, Nagashio, Okamoto & Hidaka, 1992, and references therein).

The single crystal used in this work consists of the $\Delta\Delta$ and $\Lambda\Lambda$ isomers, which combine to form the racemic compound. All the aet chelate rings possess a distinct *gauche* form with the λ conformation for the $\Delta\Delta$ isomer and the δ conformation for the $\Lambda\Lambda$ isomer, and, furthermore, all the bridging S atoms are fixed to the *S* configuration for the $\Delta\Delta$ isomer and the *R* configuration for the $\Lambda\Lambda$ isomer.

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Structure of *trans*-Bis[1,2-bis(dimethylphosphino)ethane]dibromoiron(II)

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Abstract. [FeBr₂(C₆H₁₆P₂)₂], $M_r = 515.94$, monoclinic, $P2_1/n$, $a = 8.739$ (2), $b = 12.743$ (2), $c = 9.533$ (1) Å, $\beta = 91.32$ (1)°, $V = 1061.2$ Å³, $Z = 2$, $D_x = 1.615$ g cm⁻³, $\lambda(\text{Mo K}\alpha) = 0.71069$ Å, $\mu = 45.56$ cm⁻¹, $F(000) = 520$, $T = 293$ K, final $R = 0.043$ for 1178 observed reflections. The structure consists of a neutral *trans*-[FeBr₂{1,2-bis(dimethylphosphino)ethane}₂] molecule with the Fe atom located at a centre of symmetry. The Fe—P bond lengths [2.230 (2) and 2.236 (1) Å] are the same as those in the analogous chloro complex and are indicative of low-spin Fe^{II}. The Fe—Br distance is 2.492 (1) Å.

Introduction. As part of a project involving a mechanistic study of C—H bond activation by iron bis(diphosphine) dihydride complexes, a series of dihalide complexes [FeX₂(pp)₂] [pp = 1,2-bis(dimethylphosphino)ethane (dmpe), 1,2-bis(dipropylphosphino)ethane (dprpe) or 1,2-bis(diphenylphosphino)ethane (dppe); X = Cl, Br] were synthesized and their properties examined (Baker, Field & Hambley, 1988). These dihalide complexes can easily be converted to the corresponding dihydrides by reduction with lithium aluminium hydride in THF

(Baker, Field & Young, 1990). In this paper, the structure of [FeBr₂(dmpe)₂] (1) is reported.

Experimental. [FeBr₂(dmpe)₂] was synthesized by stirring a THF solution of anhydrous iron(II) bromide in the presence of dmpe at room temperature for 6 h. The reaction was accompanied by a colour change from yellow–brown to pale green. Removal of the solvent produced a green solid which was characterized as a symmetrical *trans* complex by ³¹P and ¹H NMR spectroscopy. ³¹P NMR (C₆D₆): δ 58.10 (s, 4P). ¹H{³¹P} NMR (C₆D₆): δ 1.482 (s, 8 × CH₃); 2.204 (s, 2 × PCH₂CH₂P). Details of the synthesis have been published elsewhere (Baker, Field & Hambley, 1988). Pale green crystals of *trans*-[FeBr₂(dmpe)₂] were obtained by slow crystallization from benzene/light petroleum.

Cell constants were determined by a least-squares fit to 25 independent reflections, measured and refined on an Enraf–Nonius CAD-4F diffractometer with a graphite monochromator. The crystallographic data are summarized in Table 1. Data were reduced and Lorentz, polarization and absorption corrections were applied using the Enraf–Nonius SDP system (Frenz, 1985). The structure was solved by heavy-atom methods and refined by full-matrix

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Table 1. Summary of data-collection and processing parameters

Crystal dimensions (mm)	0.075 × 0.125 × 0.175
Crystal faces	(110), (011), (111)
Data collection range (°)	2 < 2θ < 50
Scan width (°)	0.80 + 0.34tanθ
Horizontal counter aperture (mm)	2.70 + 1.05tanθ
Scan type	ω-θ
Absorption correction*	
Number of sampling points	8 × 12 × 12
Max., min. corrections	2.164, 1.460
Range of hkl	h -10 → 10, k 0 → 15, l 0 → 11
R _{int}	0.027
Total data collected	2089
Number of unique reflections	1598
Data with I > 2.5σ(I)	1178
Total variables	95
Function minimized	Σw(F _o - F _c) ²
R	0.043
wR	0.048
w	2.24/[σ ² (F _o) + 0.00038F _o ²]
Max. Δ/σ in final cycle	0.05
Max., min. Δρ (e Å ⁻³)	0.6, -0.5

* Coppens, Leiserowitz & Rabinovich (1965).

least-squares analysis with *SHELX76* (Sheldrick, 1976). H atoms were refined with individual refined isotropic thermal parameters, all other atoms were refined anisotropically. Scattering factors used were the values supplied in *SHELX76*. Non-H-atom coordinates are listed in Table 2* and bond lengths and angles are given in Table 3. The atomic nomenclature is shown on the *ORTEP* plot (Johnson, 1965) given in Fig. 1.

Discussion. The structure of [FeBr₂(dmpe)₂] is formed by isolated molecules in which the metal atom has a P₄Br₂ environment. The arrangement of the donor atoms is *trans* octahedral, with the two bidentate phosphine ligands occupying the equatorial plane and the two Br atoms defining the vertical axis. The symmetry of the coordination polyhedron is close to *D*_{4h} and is crystallographically *C*₂. The only other published structure of an iron complex with a P₄Br₂ donor set and a *trans* octahedral geometry is the high-spin compound [FeBr₂(dppen)₂] (2) [dppen = (*Z*)-1,2-bis(diphenylphosphino)ethylene] (Cecconi, Di Vaira & Sacconi, 1981).

A perspective view of the [FeBr₂(dmpe)₂] molecule is shown in Fig. 1. The Fe—P distances are 2.230 (2) and 2.236 (1) Å in [FeBr₂(dmpe)₂] and these are similar to the 2.230 (2) and 2.236 (2) Å Fe—P distances in the analogous compound [FeCl₂(dmpe)₂] (3) (Di Vaira, Midollini & Sacconi, 1981). However, the Fe—P distances in both (1) and (3) are significantly shorter than those in (2) [2.640 (2) and 2.674 (2) Å] and this strongly confirms the low-spin nature of the complex. The Fe—Br bond length of 2.492 (1) Å

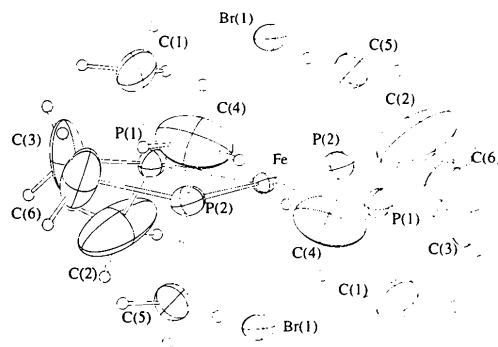
* Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 55431 (10 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: HL1006]

Table 2. Positional parameters for *trans*-[FeBr₂(dmpe)₂] (1) and equivalent isotropic thermal parameters
$$U_{eq} = \frac{1}{3} \sum_i \sum_j U_{ij} a_i^* a_j^* \mathbf{a}_i \cdot \mathbf{a}_j$$

	x	y	z	U _{eq} (Å ²)
Fe(1)	0.5000	0.5000	0.5000	0.035
Br(1)	0.3012 (1)	0.3692 (1)	0.5635 (1)	0.063
P(1)	0.6188 (2)	0.3763 (2)	0.3767 (2)	0.059
P(2)	0.3635 (3)	0.5339 (2)	0.3047 (2)	0.070
C(1)	0.6137 (19)	0.2384 (8)	0.4323 (14)	0.152
C(2)	0.8067 (18)	0.3863 (14)	0.3297 (29)	0.346
C(3)	0.5307 (25)	0.3591 (19)	0.2058 (14)	0.256
C(4)	0.1636 (15)	0.5233 (14)	0.2911 (20)	0.238
C(5)	0.3952 (13)	0.6516 (8)	0.2035 (9)	0.089
C(6)	0.4178 (25)	0.4313 (11)	0.1696 (16)	0.192

Table 3. Bond lengths (Å) and bond angles (°) for the title compound (1)

Br(1)—Fe(1)	2.492 (1)	P(1)—Fe(1)	2.236 (2)
P(2)—Fe(1)	2.230 (2)	C(1)—P(1)	1.836 (11)
C(2)—P(1)	1.717 (13)	C(3)—P(1)	1.799 (14)
C(4)—P(2)	1.754 (12)	C(5)—P(2)	1.808 (9)
C(6)—P(2)	1.903 (14)	C(6)—C(3)	1.387 (19)
P(1)—Fe(1)—Br(1)	89.5 (1)	P(2)—Fe(1)—Br(1)	88.3 (1)
P(2)—Fe(1)—P(1)	86.8 (1)	C(2)—P(1)—Fe(1)	122.9 (5)
C(1)—P(1)—Fe(1)	120.6 (4)	C(3)—P(1)—Fe(1)	111.6 (6)
C(2)—P(1)—C(1)	100.2 (9)	C(3)—P(1)—C(2)	99.5 (12)
C(3)—P(1)—C(1)	97.6 (9)	C(4)—P(2)—Fe(1)	124.0 (6)
C(5)—P(2)—Fe(1)	121.4 (3)	C(5)—P(2)—C(4)	100.9 (6)
C(6)—P(2)—Fe(1)	107.2 (5)	C(6)—P(2)—C(4)	99.2 (10)
C(6)—P(2)—C(5)	99.5 (6)	C(3)—C(6)—P(2)	118.3 (11)
C(6)—C(3)—P(1)	115.6 (12)		

Fig. 1. *ORTEP* plot of *trans*-bis[1,2-bis(dimethylphosphino)ethane]dibromoiron(II).

is longer than the Fe—Cl bond length in (3) [2.352 (1) Å], as expected owing to the difference in steric bulk and electronic properties of the halide ligands. The lengths of the P—CH₃ bonds in the ligand range from 1.717 (13) to 1.836 (11) Å and the two P—C bond lengths to the ethane bridge are 1.799 (14) and 1.903 (14) Å. The abnormally short P—C and C—C distances and the large P—C—C angles in the ligand molecule (Table 3) are a consequence of thermal motion. The Br—Fe—P angles in (1) [86.8 (1) and 89.5 (1)°] are closer to 90° than both the corresponding Cl—Fe—P angles in (3) [85.8 (1)

and 88.9 (1)° and Br—Fe—P angles in (2) [84.8 (1) and 86.5 (1)°].

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Structure of Bis[ferrocenyl(*p*-tolyl)methyl] Ether

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Abstract. C₃₆H₃₄Fe₂O, *M_r* = 594.36, monoclinic, *C*2/*c*, *a* = 17.316 (5), *b* = 11.328 (2), *c* = 14.837 (4) Å, β = 109.18 (3)°, *V* = 2749 (1) Å³, *Z* = 4, *D_x* = 1.436 g cm⁻³, λ(Mo *K*α) = 0.71069 Å, μ = 10.8 cm⁻¹, *F*(000) = 1240, room temperature, *R* = 0.038 for 1795 unique reflections with *I* > 2σ(*I*). The molecule lies on a crystallographic twofold axis which passes through the O atom. The cyclopentadienyl rings are twisted 7.6 (1)° from the eclipsed conformation. In the ferrocene moiety, the Fe—C distances are in the range 2.030 (5)–2.074 (4) Å and C—C distances in the range 1.402 (6)–1.428 (4) Å. The substitution of one cyclopentadienyl ring causes some distortion within the ferrocene moiety.

Introduction. The title compound is the major product from the reaction of ferrocene with tolu-aldehyde, whereas the corresponding carbinol (α-*p*-tolyl)ferrocenylmethanol was obtained as the by-product. We decided to carry out a single-crystal X-ray study of several related ferrocenyl ethers and carbinols of this type in order to investigate the influence of different substituents on the ferrocene geometry. In this work we describe the structure of bis[ferrocenyl(*p*-tolyl)methyl] ether. As far as we know, the X-ray diffraction studies of ethers of this type have not been described so far.

Experimental. Condensation of *p*-tolualdehyde with ferrocene in conc. H₂SO₄ at 265 K gave a 42% yield of the title compound (Kovač & Rapić, 1990). Orange crystals (0.06 × 0.07 × 0.20 mm) were obtained by recrystallization from ethanol. Data were collected on a Philips PW1100 diffractometer with graphite-monochromated Mo *K*α radiation. Intensities were measured using the θ–2θ scan mode, scan width of 1.4° and scan speed of 0.04° s⁻¹. Unit-cell parameters were determined by the least-squares procedure from 18 reflections, 8.5 < θ < 14.1°. Three standard reflections (400, $\bar{4}2\bar{3}$, $\bar{6}\bar{4}\bar{3}$) measured every 2 h showed no significant intensity variations. 1884 reflections were measured within the range 2 ≤ θ ≤ 30° (–24 ≤ *h* ≤ 22, 0 ≤ *k* ≤ 15, 0 ≤ *l* ≤ 18), 1825 unique (*R_{int}* = 0.029), 1795 with *I* > 2σ(*I*) were used in the refinement. Intensities were corrected for Lorentz and polarization effects. No absorption or extinction corrections were applied. The structure was solved by direct methods using *MULTAN*80 (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1980). H-atom positions were determined from a difference Fourier map. The structure was refined by the full-matrix least-squares procedure based on *F* with anisotropic temperature factors for all non-H atoms and isotropic for H atoms. Refinement (245 parameters)