

1984) where the edda coordination to the cobalt is *trans*. The three chelate rings of edda have a $\delta\lambda\delta$ conformation and the nitrogen configurations are *R* and *S*, the same as in [Co(edda)(*R*-1,2-diaminopropane)]⁺ (Halloran *et al.*, 1975), but differing from the $\delta\lambda\lambda$ conformation and *SS* or *RR* nitrogen configuration in the complex [Co(edda)(CO₃)] (Egan, 1988). The picolinate ligand binds the Co atom with bond lengths and angles similar to those in [Co(pipecolinato)(en)₂]²⁺ (Brown, Majeste, Chung & Trefonos, 1977), where pipecolinate is the unsaturated analogue of picolinate. One other cobalt complex of picolinate has been reported, [Co(pic)₃].H₂O (Pellizzi & Pellizzi, 1981).

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Structure of 1,1'-Bis(diphenylphosphinetetramethyl)ferrocene

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Abstract. [Fe(C₂₁H₂₂P)₂], *M_r* = 666.61, triclinic, *P* $\bar{1}$, *a* = 7.698 (3), *b* = 8.663 (2), *c* = 13.724 (4) Å, α = 80.25 (2), β = 83.58 (3), γ = 75.69 (3)°, *V* = 871.6 Å³, *Z* = 1, *D_x* = 1.270 g cm⁻³, λ (Mo *K*α) = 0.71073 Å, μ = 5.490 cm⁻¹, *F*(000) = 352, *T* = 296 (1) K, *R* = 0.047 for 2712 reflections with *I* > 3.5σ(*I*). The title complex, a precursor for bi- or polymetallic compounds, bears a new bifunctional permethylated ligand which is more electron rich than the known diphenylphosphinecyclopentadienyl moiety. The geometry of the complex is affected little by the substitution of methyl groups for H atoms in the rings. The molecule is centrosymmetric with the Fe atom at the crystallographic inversion centre.

Experimental. The compound was synthesized by reaction of FeCl₂ with C₅Me₄PPh₂Li in THF. Recrystallization from CHCl₃/pentane afforded yellow single crystals. A specimen (0.08 × 0.08 × 0.15 mm) was mounted on an Enraf-Nonius CAD-4 diffractometer. The unit cell was determined and refined from angle data of 25 randomly selected reflections in the range 11 < θ < 19° (CAD-4 rou-

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tines). Intensities measured with filtered Mo *K*α radiation, ω–2θ scan, 2 < θ < 28°. Data (4506 reflections) collected in the range *h* 0 → 10, *k* –11 → 11, *l* –18 → 18 corresponding to 2712 unique reflections with *I* > 3.5σ(*I*) used in structure determination and refinement. Intensities of three standard reflections (108, 136 and 008) measured at 60 min intervals showed a positive (0.6%) linear decay which was corrected. Lorentz–polarization correction. Empirical absorption corrections [*ψ* scan; transmission factors: min. 54.00, max. 99.94%, and *DIFABS* (Walker & Stuart, 1983) absorption: min. 0.7017, max. 1.2214]. Structure solved by heavy-atom, least-squares and difference Fourier techniques and refined (on *F*) by full-matrix least squares with unit weights and anisotropic thermal parameters for non-H atoms. All H atoms of the methyl groups located from difference Fourier map and refined with *B*_{iso} fixed at 5.0 Å², those of the phenyl groups placed in calculated positions, riding on the C atoms bearing them and included in the final refinements with *B*_{iso} = 5.0 Å². Secondary-extinction coefficient refined to *g* = 2.7636 × 10⁻⁷ {*F_c* = *F_c*/[1 + *g*(*F_c*)²L*p*]}]. Δ/σ_{max} = 0.02 for non-H atoms, Δρ max. and min. +0.5 and –0.6 e Å⁻³. Final *R* factor:

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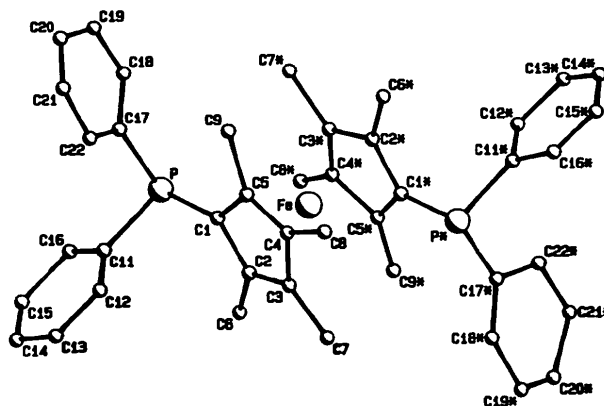
Table 1. Fractional atomic coordinates and equivalent isotropic temperature factors

$$B_{eq} = (4/3)[a^2B(1,1) + b^2B(2,2) + c^2B(3,3) + ab(\cos\gamma)B(1,2) + ac(\cos\beta)B(1,3) + bc(\cos\alpha)B(2,3)].$$

	x	y	z	$B_{eq}(\text{\AA}^2)$
Fe	0.500	0.500	0.500	2.00 (1)
P	0.3867 (1)	0.6449 (1)	0.72632 (8)	3.07 (2)
C(1)	0.3315 (5)	0.5593 (4)	0.6237 (3)	2.42 (7)
C(2)	0.2697 (5)	0.6621 (4)	0.5355 (3)	2.60 (7)
C(3)	0.2352 (5)	0.5631 (4)	0.4688 (3)	2.69 (7)
C(4)	0.2813 (5)	0.3998 (4)	0.5144 (3)	2.56 (7)
C(5)	0.3430 (5)	0.3964 (4)	0.6089 (3)	2.60 (7)
C(6)	0.2385 (6)	0.8428 (5)	0.5176 (3)	3.68 (9)
C(7)	0.1574 (5)	0.6217 (6)	0.3695 (3)	3.9 (1)
C(8)	0.2582 (6)	0.2603 (5)	0.4712 (3)	4.07 (9)
C(9)	0.3981 (6)	0.2425 (5)	0.6801 (3)	3.78 (9)
C(11)	0.1677 (5)	0.7737 (5)	0.7603 (3)	3.16 (8)
C(12)	0.0032 (6)	0.7508 (5)	0.7395 (3)	4.0 (1)
C(13)	-0.1576 (6)	0.8540 (6)	0.7683 (3)	4.6 (1)
C(14)	-0.1533 (7)	0.9780 (6)	0.8168 (4)	5.0 (1)
C(15)	0.0069 (8)	1.0032 (6)	0.8374 (4)	5.2 (1)
C(16)	0.1681 (6)	0.9015 (5)	0.8084 (3)	4.0 (1)
C(17)	0.4027 (6)	0.4845 (5)	0.8335 (3)	3.58 (9)
C(18)	0.5681 (7)	0.4252 (6)	0.8737 (3)	4.4 (1)
C(19)	0.5885 (8)	0.3028 (6)	0.9556 (3)	5.5 (1)
C(20)	0.4454 (9)	0.2446 (7)	0.9976 (4)	6.3 (2)
C(21)	0.2790 (9)	0.3031 (7)	0.9621 (4)	6.5 (2)
C(22)	0.2542 (7)	0.4274 (6)	0.8791 (4)	5.0 (1)

Table 2. Selected interatomic distances (\AA) and bond angles ($^\circ$)

Fe—C(1)	2.074 (3)	Fe—C(5)	2.044 (3)
Fe—C(2)	2.040 (3)	P—C(1)	1.832 (4)
Fe—C(3)	2.050 (4)	P—C(11)	1.837 (4)
Fe—C(4)	2.053 (4)	P—C(17)	1.837 (4)
C(1)—P—C(11)	101.3 (2)	P—C(1)—C(2)	120.1 (3)
C(1)—P—C(17)	105.7 (2)	P—C(1)—C(5)	132.6 (3)
C(11)—P—C(17)	99.6 (2)	C(2)—C(1)—C(5)	107.3 (3)

Fig. 1. Molecular structure of $[\text{Fe}(\text{C}_{21}\text{H}_{22}\text{P})_2]_2$.

0.047, S 0.896. Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV). Calculations performed on a VAX730 computer with the *SDP* program package (Frenz, 1978). Final positional parameters are in Table 1, and selected bond lengths and bond angles are in Table 2.* The atomic labelling scheme and molecular structure are shown in Fig. 1.

Related literature. Crystal structures of related metallocenes have been reported: $\text{Fe}(\text{C}_5\text{H}_4\text{PPh}_2)_2$ (Casellato, Ajo, Valle, Corain, Longato & Graziani, 1988), $\text{Co}(\text{C}_5\text{H}_4\text{PPh}_2)_2$ (Dubois, Eigenbrot, Miedaner, Smart & Haltiwanger, 1986), $\text{Fe}(\text{C}_5\text{Me}_4\text{H})_2$ and $\text{Fe}(\text{C}_5\text{Me}_5)_2$ (Struchkov, Andrianov, Sal'nikova, Lyatitov & Materikova, 1978; Freyberg, Robbins, Raymond & Smart, 1979). The structures of some homo- and heterobimetallic ferrocenes with a bridging diphenylphosphinocyclopentadienylidene ligand have been studied [$\text{dppf} = \text{Fe}(\text{C}_5\text{H}_4\text{PPh}_2)_2$]: $\text{PdCl}_2(\text{dppf})$ (Hayashi, Konishi, Kobori, Kumada, Higuchi & Hirotsu, 1984), $[\text{Rh}(\text{NBD})(\text{dppf})]^+$ (Cullen, Kim, Einstein & Jones, 1985), $\text{NiBr}_2(\text{dppf})$, $\text{Mo}(\text{CO})_4(\text{dppf})$ (Butler, Cullen, Kim, Rettig & Trotter, 1985), $\text{PtCl}_2(\text{dppf})$ (Clemente, Pilloni, Corain, Longato & Tiripicchio-Camellini, 1986), $\text{Fe}(\text{CO})_3(\text{dppf})$ (Kim, Kwon, Kwon, Baeg, Shim &

* Lists of structure factors, anisotropic thermal parameters, H-atom parameters, least-squares-planes details and a full list of bond lengths and angles have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54049 (21 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Lee, 1990), $[\text{Ir}(\text{dppf})_2]\text{B}(\text{C}_6\text{H}_5)_4$ (Casellato, Corain, Graziani, Longato & Pilloni, 1990) and $\text{Pd}(\text{B}_3\text{H}_7)(\text{dppf})$ (Housecroft, Owen, Raithby & Shaykh, 1990).

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