Table 3. *Bond angles* (°)

P(1)—Pt—P(2)	106·6 (1)	Pt—C(41)—C(46)	120·5 (5)
P(1)—Pt—C(41)	141·9 (2)	C(42)—C(41)—C(46)	122·1 (7)
P(2)—Pt—C(41)	111·1 (2)	C(45)—C(41)—C(46)	122·4 (7)
P(1)—Pt—C(42)	103·4 (2)	Pt—C(42)—C(41)	72·1 (5)
P(2)—Pt—C(42)	150·0 (2)	Pt—C(42)—C(43)	101·2 (5)
C(41)—Pt—C(42)	39·5 (3)	C(41)—C(42)—C(43)	110·4 (7)
Pt—P(1)—C(6)	114·1 (2)	O(2)—C(43)—C(42)	127·2 (8)
Pt—P(1)—C(12)	113·9 (2)	O(2)—C(43)—C(44)	122·9 (7)
C(6)—P(1)—C(12)	103·0 (3)	C(42)—C(43)—C(44)	109·8 (8)
Pt—P(1)—C(18)	117·6 (2)	C(43)—C(44)—C(45)	101·3 (6)
C(6)—P(1)—C(18)	105·0 (2)	C(43)—C(44)—C(50)	112·6 (7)
C(12)—P(1)—C(18)	101·4 (3)	C(45)—C(44)—C(50)	112·6 (6)
Pt—P(2)—C(24)	115·2 (2)	C(43)—C(44)—C(54)	111·7 (7)
Pt—P(2)—C(30)	114·3 (2)	C(45)—C(44)—C(54)	110·7 (7)
C(24)—P(2)—C(30)	104·0 (2)	C(50)—C(44)—C(54)	107·9 (6)
Pt—P(2)—C(36)	114·7 (2)	O(1)—C(45)—C(41)	127·6 (8)
P(24)—P(2)—C(36)	101·9 (3)	O(1)—C(45)—C(44)	122·3 (7)
C(30)—OP(2)—C(36)	105·3 (3)	C(41)—C(45)—C(44)	110·0 (7)
C(56)—O(3)—C(57)	115·9 (17)	C(41)—C(46)—C(47)	111·0 (8)
P(1)—C(6)—C(1)	124·4 (2)	C(41)—C(46)—C(48)	111·5 (7)
P(1)—C(6)—C(5)	115·6 (2)	C(47)—C(46)—C(48)	107·4 (8)
P(1)—C(12)—C(7)	121·4 (2)	C(41)—C(46)—C(49)	108·5 (7)
P(1)—C(12)—C(11)	118·6 (2)	C(47)—C(46)—C(49)	109·9 (8)
P(1)—C(18)—C(13)	119·6 (2)	C(48)—C(46)—C(49)	108·5 (8)
P(1)—C(18)—C(17)	120·4 (2)	C(44)—C(50)—C(51)	111·2 (8)
P(2)—C(24)—C(19)	117·9 (2)	C(44)—C(50)—C(52)	111·5 (8)
P(2)—C(24)—C(23)	122·0 (2)	C(51)—C(50)—C(52)	106·8 (8)
P(2)—C(30)—C(25)	116·9 (2)	C(44)—C(50)—C(53)	108·6 (7)
P(2)—C(30)—C(29)	123·1 (2)	C(51)—C(50)—C(53)	109·6 (9)
P(2)—C(36)—C(31)	122·2 (2)	C(52)—C(50)—C(53)	109·0 (8)
P(2)—C(36)—C(35)	117·8 (2)	N(1)—C(54)—C(44)	179·2 (10)
Pt—C(41)—C(42)	68·4 (4)	O(3)—C(56)—C(55)	114·9 (20)
Pt—C(41)—C(45)	101·6 (5)	O(3)—C(57)—C(58)	112·3 (17)
C(42)—C(41)—C(45)	108·5 (7)		

least-squares refinement on 479 parameters; all non-H atoms anisotropic, H atoms idealized and updated (C—H = 0·96 Å, $U = 1{\cdot}2U$ of attached C), phenyl rings constrained to rigid hexagons (C—C = 1·395 Å). $R_F = 4{\cdot}11\%$, $wR_F = 5{\cdot}00\%$, $S = 1{\cdot}072$, $w^{-1} = \sigma^2(F_o) + gF_o^2$, $g = 0{\cdot}001$; $(\Delta/\sigma)_{max} = 0{\cdot}18$; $\Delta\rho_{max} = 1{\cdot}075$, $\Delta\rho_{min} = -0{\cdot}407$ e Å^{-3}; atomic scattering factors from *International Tables for X-ray Crystallography* (1974); *SHELXTL* computer program (Sheldrick, 1983).

Atomic coordinates and equivalent isotropic thermal parameters are given in Table 1. Bond lengths are given in Table 2 and angles are given in Table 3. Fig. 1 shows the labeled molecular structure of the compound and Fig. 2 shows the unit-cell packing diagram.*

Related literature. To our knowledge, no other structures of cyclopentenedione platinum diphosphine complexes have been published.

* Lists of structure factors, anisotropic thermal parameters and full lists of bond lengths have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52368 (35 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Carbonyl(2-cyano-3,3-dimethylbutanoic acid-*N*)(η^5-cyclopentadienyl)-(triphenylphosphine)iron(I) Tetrafluoroborate

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Abstract. $[Fe(C_5H_5)(CO)(C_7H_{11}NO_2)\{P(C_6H_5)_3\}][BF_4]$, $M_r = 627{\cdot}2$, triclinic, $P\bar{1}$, $a = 11{\cdot}123$ (5), $b = 12{\cdot}085$ (6), $c = 12{\cdot}855$ (6) Å, $\alpha = 101{\cdot}91$ (4), $\beta = 93{\cdot}67$ (4), $\gamma = 108{\cdot}43$ (3)°, $V = 1588{\cdot}4$ (12) Å^3, $Z = 2$, $D_x = 1{\cdot}311$ g cm^{-3}, λ(Mo $K\alpha$) = 0·71073 Å, $\mu = 5{\cdot}71$ cm^{-1}, $F(000) = 648$, $T = 296$ K, $R_F = 5{\cdot}29\%$ for 5411 observed reflections and 343 parameters. The coordination geometry at iron in the cation is the expected three-legged piano-stool type; if the cyclopentadienyl ring is considered to occupy three coordination sites, the overall geometry is approximately octahedral. Hydrogen bonding occurs between the acidic hydrogen of the carboxy group and one of the F atoms of the counterion: H*x*···F(1)

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Table 1. *Atomic coordinates* ($\times 10^4$) *and isotropic thermal parameters* ($Å^2 \times 10^3$)

	x	y	z	U*
Fe	7280·6 (8)	585·9 (7)	3595·9 (7)	36·1 (3)
P	8087 (1)	323 (1)	2051 (1)	37·4 (6)
B	3208 (9)	6171 (9)	3100 (10)	78 (5)
F(1)	2079 (5)	6282 (7)	2818 (7)	174 (5)
F(2)	2996 (8)	5225 (7)	3485 (7)	204 (6)
F(3)	3889 (7)	6085 (8)	2332 (5)	190 (6)
F(4)	3888 (7)	7104 (7)	3866 (8)	218 (5)
O(1)	4635 (4)	−241 (5)	2601 (4)	81 (3)
O(2)	9816 (5)	4724 (5)	3174 (5)	95 (3)
O(3)	9799 (6)	6255 (5)	4434 (6)	124 (3)
N(1)	7590 (4)	2246 (4)	3648 (4)	46 (2)
C(1)	5681 (6)	92 (6)	2953 (5)	51 (3)
C(2)	7763 (6)	3228 (5)	3765 (5)	49 (3)
C(3)	7926 (6)	4523 (6)	4006 (6)	60 (3)
C(4)	9291 (7)	5264 (7)	3898 (7)	76 (4)
C(5)	6856 (7)	4742 (6)	3335 (6)	68 (3)
C(6)	6950 (10)	6040 (7)	3727 (8)	134 (6)
C(7)	5579 (7)	3906 (6)	3449 (9)	126 (6)
C(8)	7073 (10)	4521 (8)	2159 (7)	126 (6)
C(11)	8815 (6)	640 (6)	4663 (5)	52 (3)
C(12)	8096 (7)	−548 (6)	4138 (5)	55 (3)
C(13)	6852 (7)	−788 (6)	4394 (5)	61 (3)
C(14)	6814 (6)	271 (7)	5102 (5)	65 (4)
C(15)	8036 (7)	1140 (6)	5258 (5)	58 (3)
C(21)	10551 (4)	1837 (3)	2859 (4)	56 (3)
C(22)	11858	2116	3171	68 (3)
C(23)	12419	1228	2936	70 (4)
C(24)	11674	62	2388	68 (4)
C(25)	10368	−217	2076	50 (3)
C(26)	9806	671	2311	40 (2)
C(31)	7879 (4)	−1389 (4)	158 (3)	62 (3)
C(32)	7424	−2538	−521	73 (4)
C(33)	6549	−3476	−203	70 (4)
C(34)	6129	−3265	795	63 (3)
C(35)	6584	−2116	1475	50 (6)
C(36)	7459	−1178	1156	41 (2)
C(41)	6597 (4)	1143 (4)	802 (4)	66 (3)
C(42)	6354	1775	66	87 (4)
C(43)	7367	2506	−334	95 (5)
C(44)	8622	2605	2	106 (6)
C(45)	8865	1973	737	80 (4)
C(46)	7853	1242	1137	49 (3)

*Equivalent isotropic U defined as one third of the trace of the orthogonalized U_{ij} tensor.

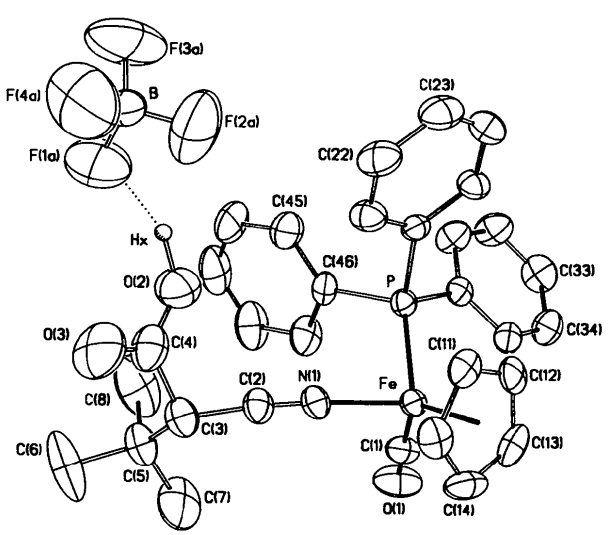

Fig 1. Molecular structure and labeling scheme for $\{[(C_5H_5)FeP(C_6H_5)_3(CO)NCC_5H_{11}CO_2][BF_4]\}_2$.

= 1·71 (1) Å and O(2)—Hx···F(1) = 2·756 (8) Å. Both C(3) and Fe are chiral centers; the crystal is composed of a racemic mixture of [*R*,*S*] and [*S*,*R*] enantiomers.

Table 2. *Bond lengths* (Å) *and angles* (°)

Fe—P	2·236 (2)	O(1)—C(1)	1·132 (7)
Fe—N(1)	1·911 (5)	O(2)—C(4)	1·301 (11)
Fe—C(1)	1·772 (6)	O(3)—C(4)	1·187 (9)
Fe—C(11)	2·096 (7)	N(1)—C(2)	1·116 (8)
Fe—C(12)	2·070 (8)	C(2)—C(3)	1·480 (9)
Fe—C(13)	2·074 (8)	C(3)—C(4)	1·532 (10)
Fe—C(14)	2·114 (7)	C(3)—C(5)	1·547 (11)
Fe—C(15)	2·132 (6)	C(5)—C(6)	1·513 (12)
P—C(26)	1·817 (4)	C(5)—C(7)	1·497 (10)
P—C(36)	1·826 (4)	C(5)—C(8)	1·527 (12)
P—C(46)	1·834 (6)	C(11)—C(12)	1·401 (8)
B—F(1)	1·340 (13)	C(11)—C(15)	1·388 (11)
B—F(2)	1·297 (15)	C(12)—C(13)	1·396 (10)
B—F(3)	1·289 (14)	C(13)—C(14)	1·424 (11)
B—F(4)	1·312 (12)	C(14)—C(15)	1·402 (9)
		Cp*—Fe	1·725 (7)
P—Fe—N(1)	91·7 (2)	Fe—N(1)—C(2)	174·3 (5)
P—Fe—C(1)	93·6 (2)	Fe—C(1)—O(1)	175·7 (6)
N(1)—Fe—C(1)	95·0 (3)	N(1)—C(2)—C(3)	174·7 (7)
P—Fe—C(11)	99·9 (2)	C(2)—C(3)—C(4)	110·4 (6)
N(1)—Fe—C(11)	102·8 (2)	C(2)—C(3)—C(5)	110·5 (5)
C(1)—Fe—C(11)	157·2 (3)	C(4)—C(3)—C(5)	115·0 (7)
P—Fe—C(12)	91·2 (2)	O(2)—C(4)—O(3)	124·1 (7)
N(1)—Fe—C(12)	141·7 (2)	O(2)—C(4)—C(3)	114·6 (6)
C(1)—Fe—C(12)	122·9 (3)	O(3)—C(4)—C(3)	121·3 (8)
C(11)—Fe—C(12)	39·3 (2)	C(3)—C(5)—C(6)	108·3 (6)
P—Fe—C(13)	119·0 (2)	C(3)—C(5)—C(7)	109·7 (7)
N(1)—Fe—C(13)	148·1 (2)	C(6)—C(5)—C(7)	111·2 (8)
C(1)—Fe—C(13)	91·5 (3)	C(3)—C(5)—C(8)	108·7 (7)
C(11)—Fe—C(13)	65·9 (3)	C(6)—C(5)—C(8)	108·9 (8)
C(12)—Fe—C(13)	39·4 (3)	C(7)—C(5)—C(8)	110·1 (6)
P—Fe—C(14)	156·7 (3)	Fe—C(11)—C(12)	69·4 (4)
N(1)—Fe—C(14)	108·4 (3)	Fe—C(11)—C(15)	72·2 (4)
C(1)—Fe—C(14)	96·1 (3)	C(12)—C(11)—C(15)	108·5 (6)
C(11)—Fe—C(14)	64·9 (3)	Fe—C(12)—C(11)	71·4 (4)
C(12)—Fe—C(14)	65·8 (3)	Fe—C(12)—C(13)	70·4 (5)
C(13)—Fe—C(14)	39·7 (3)	C(11)—C(12)—C(13)	108·3 (6)
P—Fe—C(15)	135·7 (2)	Fe—C(13)—C(12)	70·2 (4)
N(1)—Fe—C(15)	86·8 (3)	Fe—C(13)—C(14)	71·7 (5)
C(1)—Fe—C(15)	130·6 (3)	C(12)—C(13)—C(14)	107·3 (5)
C(11)—Fe—C(15)	38·3 (3)	Fe—C(14)—C(13)	68·6 (4)
C(12)—Fe—C(15)	65·2 (3)	Fe—C(14)—C(15)	71·4 (4)
C(13)—Fe—C(15)	65·7 (3)	C(13)—C(14)—C(15)	107·6 (6)
C(14)—Fe—C(15)	38·5 (2)	Fe—C(15)—C(11)	69·4 (4)
Fe—P—C(26)	110·3 (2)	Fe—C(15)—C(14)	70·0 (4)
Fe—P—C(36)	116·8 (2)	C(11)—C(15)—C(14)	108·2 (6)
C(26)—P—C(36)	106·0 (2)	P—C(26)—C(21)	118·0 (1)
Fe—P—C(46)	116.3 (2)	P—C(26)—C(25)	121·7 (1)
C(26)—P—C(46)	105·4 (2)	P—C(36)—C(31)	119·7 (1)
C(36)—P—C(46)	100·9 (2)	P—C(36)—C(35)	120·3 (1)
F(1)—B—F(2)	108·3 (8)	P—C(46)—C(41)	117·3 (1)
F(1)—B—F(3)	114·7 (10)	P—C(46)—C(45)	122·6 (1)
F(2)—B—F(3)	109·0 (11)	Cp—Fe—P	124·4 (2)
F(1)—B—F(4)	109·5 (10)	Cp—Fe—C(1)	123·2 (3)
F(2)—B—F(4)	107·6 (10)	Cp—Fe—N(1)	120·7 (3)
F(3)—B—F(4)	107·6 (8)		

*Cp represents the center of the cyclopentadienyl ring.

Experimental. The title compound was obtained from the reaction of *tert*-butylcyanoketene [*t*-Bu(CN)C=C=O] with $[C_5H_5(CO)(PPh_3)Fe(CH_2Cl_2)]BF_4$ followed by hydrolytic workup (Macklin, 1988). Orange-brown crystals from CH_2Cl_2 and hexane (0·30 × 0·35 × 0·35 mm); Nicolet *R*3*m* diffractometer with graphite monochromator; ω scans; lattice parameters were from the least-squares fit of 25 reflections ($22 \le 2\theta \le 26°$); empirical absorption correction ($T_{max}/T_{min} = 1·04$); $2\theta_{max} = 48°$ ($h = \pm 13$, $k = \pm 14$, $l = +15$); 115, 250, 602 standard reflections, 5411 reflections collected, 4943 unique ($R_{int} = 2·46\%$), 2906 were observed with $F_o > 5\sigma(F_o)$, 1827 unobserved reflections. Direct methods (*SOLV*) structure solution; least-squares refinement on 343 parameters; all non-H atoms were

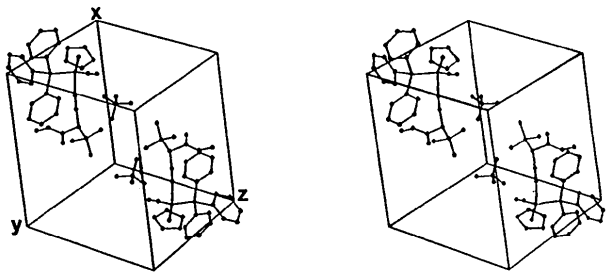

Fig. 2. Unit-cell packing diagram for $\{[(C_5H_5)FeP(C_6H_5)_3(CO)NCC_5H_{11}CO_2][BF_4]\}_2$.

anisotropic, H atoms idealized (except for H*x* which was found and refined) and updated (C—H = 0·96 Å, $U = 1{\cdot}2U$ of attached C); phenyl rings constrained to rigid hexagons, C—C = 1·395 Å. $R_F = 5{\cdot}29\%$, $wR_F = 6{\cdot}54\%$, $S = 0{\cdot}35$, $w^{-1} = \sigma^2(F) + gF_o^2$, $g = 0{\cdot}0002$; $(\Delta/\sigma)_{max} = 0{\cdot}461$, $\Delta\rho_{max} = 0{\cdot}644$, $\Delta\rho_{min} = -0{\cdot}391$ e Å^{-3}; atomic scattering factors were from *International Tables for X-ray Crystallography* (1974); *SHELXTL* computer program (Sheldrick, 1984). Atomic and equivalent isotropic thermal parameters are given in Table 1. Bond lengths and angles are given in Table 2.* Fig. 1 shows the molecular structure and Fig. 2 the unit-cell packing.

Related literature. To our knowledge no other structures of a cationic cyclopentadienyl iron complex with similar substitution have been previously reported.

* 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 52365 (22 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Structure of Bis(imidazole)(5,10,15,20-tetraphenylporphinato)iron(III) Bis(*cis*-1,2-dicyanoethylenedithiolato)cuprate(III) Tetrakis(tetrahydrofuran) Solvate

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Abstract. $[Fe(C_{44}H_{28}N_4)(C_3H_4N_2)_2][Cu(C_4N_2S_2)_2]{\cdot}4C_4H_8O$, $M_r = 1437{\cdot}1$, triclinic, $P\bar{1}$, $a = 10{\cdot}012$ (4), $b = 11{\cdot}604$ (6), $c = 15{\cdot}802$ (7) Å, $\alpha = 71{\cdot}42$ (4), $\beta = 87{\cdot}12$ (3), $\gamma = 78{\cdot}45$ (4)°, $V = 1704$ (1) Å^3, $Z = 1$, $D_x = 1{\cdot}40$ g cm^{-3}, λ(Mo $K\alpha$) = 0·7107 Å, $\mu = 6{\cdot}9$ cm^{-1}, $F(000) = 747$, $T = 143$ K, $R = 0{\cdot}063$ for 4935 unique observed reflections. Fe is six-coordinate, Fe—N(porphinato) (av.) = 1·99 (2), Fe—N(imidazole) = 1·981 (3) Å; Cu—S (av.) = 2·172 (8) Å.

Experimental. Fe(tpp) (Collman *et al.*, 1980) (0·025 g, 0·037 mmol) (tpp^{2-} = 5,10,15,20-tetraphenylporphinate) dissolved in 35 mL tetrahydrofuran. Solution of (tba)[Cu(mnt)$_2$] (Muetterties, 1961) (0·022 g, 0·037 mmol) (mnt^{2-} = *cis*-1,2-dicyanoethylenedithiolate, tba^+ = tetra-*n*-butylammonium) in 5 mL tetrahydrofuran added; reaction mixture stirred for 3 h. Solution of imidazole (0·0025 g, 0·37 mmol) in 5 mL tetrahydrofuran added; vapor diffusion of *n*-hexane into the reaction solution yielded dark rectangular crystals.

Crystal size 0·32 × 0·32 × 0·18 mm. Nicolet *R3m* diffractometer, unit-cell constants from least-squares fit of setting angles for 25 reflections ($2\theta_{av} = 22{\cdot}59°$). Data collected (Wyckoff ω scans) to $(\sin\theta)/\lambda$ of 0·595 Å^{-1}, $-12 < h < 12$, $-14 < k < 14$, $0 < l < 19$. Three standard reflections (400, 070, 008) every 100, no significant variation; data corrected for Lorentz and polarization factors; no absorption correction applied due to low absorption coefficient; 6229 unique reflections, 4935 reflections with $F > 2{\cdot}5\sigma(F)$ observed, $R_{int} = 0{\cdot}0325$.

Structure solved by direct methods (*RANT*); block-diagonal (max. 103 parameters/block, 436 parameters total, data/parameters = 11·3) weighted $\{w = [\sigma^2(F) + gF^2]^{-1}$, $g = 0{\cdot}0106\}$ least-squares refinement on *F*. H atoms in idealized positions

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