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Structure of Bis(triphenylphosphine)iminium Tetracarbonyl(α-ethoxycarbonylethyl)-ferrate: $[\{(C_6H_5)_3P\}_2N][Fe(CO)_4\{CH(CH_3)COOC_2H_5\}]$

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Abstract. $(C_{36}H_{30}NP_2)[FeC_9H_9O_6]$, $M_r = 807.6$, triclinic, $P\bar{1}$, $a = 16.908$ (2), $b = 27.038$ (3), $c = 9.531$ (1) Å, $\alpha = 93.88$ (1), $\beta = 95.71$ (1), $\gamma = 106.86$ (1)°, $V = 4128$ (2) Å^3, $Z = 4$, $D_x = 1.30$ Mg m^{-3}, λ(Mo $K\alpha$) = 0.71073 Å, $\mu = 0.49$ mm^{-1}, $F(000) = 1680$, $T = 293$ K. Full-matrix least-squares refinement based on 7968 reflections led to R and wR values of 0.025 and 0.027, respectively. In the anionic moiety of the molecule, the geometry around Fe consists of a trigonal bipyramid with the α-ethoxycarbonylethyl group in an axial position.

Experimental. Ethanolic solution of $KHFe(CO)_4$ prepared from 1 equivalent of $Fe(CO)_5$ with 2 equivalents of potassium hydroxide in ethanol and further elimination of the coproduced $KHCO_3$ (filtration under argon at 253 K) reacted with 1.1 equivalent of ethyl acrylate at 308 K for a 6.5 h period. Bis(triphenylphosphine)iminium chloride was then added. The reaction medium was stirred for 2 h at 308 K and then for a further 20 h at room temperature. The reaction medium was then placed in the refrigerator and yellow plate crystals of the title compound appeared within two days. IR and NMR data confirmed the structure (Brunet & Passelaigue, 1989). Crystals suitable for X-ray diffraction were obtained by slow evaporation of a two-phase dichloromethane/diisopropyl ether (1/1) solution. Analysis: calculated C 66.93, H 4.87, N 1.73; found C 66.76, H 5.07, N 1.73.

Data-collection crystal of dimensions 0.50 × 0.40 × 0.10 mm. Enraf–Nonius CAD-4 diffractometer, graphite-monochromated Mo $K\alpha$ radiation. Cell dimensions from setting angles of 25 reflections having $10.5 < \theta < 12.5°$, 11484 reflections measured, using $\omega/2\theta$ scan for 2θ from 3 to 46°, $-18 \le h \le 18$, $-29 \le k \le 29$, $0 \le l \le 10$, scan range $(0.80 + \tan\theta)°$. Intensities of three reflections $(76\bar{3}, 10,0,\bar{12}, 24,\bar{8},10)$ measured every 2 h showed a linear decay of 7%; linear decay corrections. Corrections for Lp and absorption by ψ scans (North, Phillips & Mathews, 1968), minimum relative transmission 0.91. Direct methods followed by Fourier and least-squares techniques using 7968 reflections having $F_o^2 > 3\sigma(F_o^2)$ based on counting statistics. Full-matrix least-squares refinement minimizing $\sum w(|F_o| - |F_c|)^2$ with anisotropic thermal parameters for non-H atoms, but phenyl rings refined as isotropic rigid groups (C—C = 1.395, C—H = 0.97 Å). All H atoms located by ΔF, H(C5*a*) and H(C5*b*) atoms refined, other H atoms in constrained geometry (C—H = 0.97 Å). Isotropic U_H allowed to vary, then kept fixed at 0.06 Å^2 for H(C5*a*) and H(C5*b*), 0.07 Å^2 for methyl and phenyl H atoms and 0.08 Å^2 for ethyl H atoms. $R = 0.025$, $wR = 0.027$, 493 variables, unit weights. Mean and maximum parameter shifts 0.0004σ and 0.004σ, respectively. Maximum and minimum heights in final ΔF map 0.18 and -0.19 e Å^{-3}. Scattering factors, including real and imaginary parts of anomalous dispersion, from *International Tables for X-ray Crystallography* (1974, Vol. IV, pp. 99–101, 149) and from Stewart, Davidson & Simpson (1965) for H atoms. MicroVAX 3400 DEC computer. *SDP* (B. A. Frenz & Associates, Inc., 1985), *SHELXS*86 (Sheldrick, 1986), *SHELX*76 (Sheldrick, 1976), *ORFFE* (Busing, Martin & Levy, 1964) and *NRC* (Ahmed, Hall, Pippy & Huber, 1966) programs.

The final positional and equivalent isotropic thermal parameters are listed in Table 1, bond lengths and angles in Table 2.† The thermal ellipsoid plot of the $[(CO)_4Fe\{CH(Me)CO_2Et\}]$ entities is shown in Fig. 1 with the atomic numbering. A view of the asymmetric unit is given in Fig. 2.

Related literature. Structure of $[\{(C_6H_5)_3P\}_2N][Fe(CO)_4C_3H_7]$ (Huttner & Gartzke, 1975); structure of $[\{(C_6H_5)_3P\}_2N][Fe(CO)_4(CH_2CO_2Me)]$ (Keim, Röper, Strutz & Krüger, 1984).

† Lists of structure factors, anisotropic thermal parameters, least-squares-planes equations and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54818 (43 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: PA0260]

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Table 1. *Fractional atomic coordinates and isotropic or equivalent isotropic temperature factors* ($Å^2 \times 10^2$) *with e.s.d.'s in parentheses*

U_{eq} is defined as one third of the trace of the orthogonalized U_{ij} tensor.

	x	y	z	U_{eq}/U_{iso}
Fe*a*	0.20862 (2)	0.20392 (1)	0.60309 (4)	3.79 (7)
C(1*a*)	0.2747 (2)	0.2306 (1)	0.7622 (3)	5.8 (6)
O(1*a*)	0.3172 (2)	0.2487 (1)	0.8653 (3)	10.6 (7)
C(2*a*)	0.1956 (2)	0.1389 (1)	0.5458 (3)	5.2 (6)
O(2*a*)	0.1875 (1)	0.09522 (9)	0.5121 (3)	8.2 (5)
C(3*a*)	0.1768 (2)	0.2501 (1)	0.5064 (3)	6.1 (6)
O(3*a*)	0.1530 (2)	0.2806 (1)	0.4625 (3)	10.6 (6)
C(4*a*)	0.1148 (2)	0.1897 (1)	0.6798 (3)	4.9 (6)
O(4*a*)	0.0539 (1)	0.18174 (9)	0.7287 (3)	7.5 (5)
C(5*a*)	0.3217 (2)	0.2221 (1)	0.5031 (3)	4.7 (5)
H(C5*a*)	0.342 (2)	0.2598 (2)	0.518 (3)	6.0
C(6*a*)	0.3861 (2)	0.1967 (1)	0.5661 (4)	7.2 (7)
C(7*a*)	0.2936 (2)	0.2014 (1)	0.3545 (3)	5.4 (6)
O(5*a*)	0.2924 (2)	0.16052 (8)	0.2949 (2)	7.7 (5)
O(6*a*)	0.2664 (2)	0.23605 (8)	0.2799 (2)	7.2 (5)
C(8*a*)	0.2277 (2)	0.2181 (2)	0.1359 (4)	8.6 (9)
C(9*a*)	0.1422 (3)	0.2196 (2)	0.1171 (4)	10.0 (1)
Fe*b*	0.31290 (2)	0.68762 (1)	0.54799 (4)	4.10 (7)
C(1*b*)	0.2874 (2)	0.7189 (1)	0.6994 (3)	5.2 (6)
O(1*b*)	0.2781 (1)	0.7408 (1)	0.8018 (3)	7.8 (5)
C(2*b*)	0.3295 (2)	0.7222 (1)	0.3962 (3)	5.1 (6)
O(2*b*)	0.3431 (2)	0.74550 (9)	0.2993 (2)	8.0 (5)
C(3*b*)	0.3104 (2)	0.6216 (1)	0.5497 (3)	4.1 (5)
O(3*b*)	0.3113 (1)	0.57900 (8)	0.5516 (2)	6.4 (4)
C(4*b*)	0.4188 (2)	0.7096 (1)	0.6189 (3)	5.1 (6)
O(4*b*)	0.4880 (1)	0.72437 (9)	0.6654 (3)	7.1 (5)
C(5*b*)	0.1830 (2)	0.6545 (1)	0.4540 (3)	4.8 (5)
H(C5*b*)	0.160 (2)	0.6284 (8)	0.516 (2)	6.0
C(6*b*)	0.1735 (2)	0.6318 (1)	0.3023 (3)	6.9 (7)
C(7*b*)	0.1449 (2)	0.6967 (1)	0.4690 (4)	5.5 (6)
O(5*b*)	0.1383 (1)	0.72665 (9)	0.3830 (3)	8.4 (5)
O(6*b*)	0.1141 (1)	0.69895 (7)	0.5941 (2)	6.0 (4)
C(8*b*)	0.0844 (2)	0.7422 (1)	0.6299 (4)	8.2 (8)
C(9*b*)	0.0556 (2)	0.7372 (2)	0.7709 (4)	9.2 (9)
N(1)	0.7406 (1)	0.50464 (7)	0.8623 (2)	3.1 (3)
P(1)	0.71409 (4)	0.47503 (2)	0.99553 (6)	2.8 (1)
C(1*c*)	0.67813 (9)	0.51257 (6)	1.1235 (1)	2.74 (5)
C(2*c*)	0.60994 (9)	0.52907 (6)	1.0773 (1)	3.30 (6)
C(3*c*)	0.58429 (9)	0.56281 (6)	1.1669 (1)	4.02 (6)
C(4*c*)	0.62683 (9)	0.58005 (6)	1.3025 (1)	4.70 (7)
C(5*c*)	0.69503 (9)	0.56355 (6)	1.3487 (1)	5.40 (8)
C(6*c*)	0.72068 (9)	0.52981 (6)	1.2592 (1)	4.28 (7)
C(7*c*)	0.79911 (9)	0.45562 (5)	1.0772 (2)	3.02 (5)
C(8*c*)	0.87863 (9)	0.49123 (5)	1.0938 (2)	4.26 (7)
C(9*c*)	0.94646 (9)	0.47766 (5)	1.1567 (2)	4.65 (7)
C(10*c*)	0.93476 (9)	0.42847 (5)	1.2029 (2)	4.40 (7)
C(11*c*)	0.85524 (9)	0.39286 (5)	1.1863 (2)	5.26 (7)
C(12*c*)	0.78742 (9)	0.40643 (5)	1.1234 (2)	4.74 (7)
C(13*c*)	0.62946 (9)	0.41780 (6)	0.9411 (1)	2.91 (5)
C(14*c*)	0.61447 (9)	0.39616 (6)	0.8004 (1)	3.91 (6)
C(15*c*)	0.55204 (9)	0.34928 (6)	0.7599 (1)	4.79 (7)
C(16*c*)	0.50459 (9)	0.32405 (6)	0.8602 (1)	5.78 (8)
C(17*c*)	0.51958 (9)	0.34570 (6)	1.0009 (1)	5.24 (7)
C(18*c*)	0.58201 (9)	0.39257 (6)	1.0414 (1)	4.03 (6)
P(2)	0.76712 (4)	0.56141 (2)	0.81760 (6)	2.7 (1)
C(19*c*)	0.83247 (9)	0.56249 (4)	0.6800 (2)	2.60 (5)
C(20*c*)	0.86981 (9)	0.60901 (4)	0.6253 (2)	3.27 (6)
C(21*c*)	0.91924 (9)	0.60941 (4)	0.5164 (2)	4.15 (6)
C(22*c*)	0.93132 (9)	0.56328 (4)	0.4621 (2)	4.49 (7)
C(23*c*)	0.89397 (9)	0.51676 (4)	0.5168 (2)	4.15 (6)
C(24*c*)	0.84455 (9)	0.51637 (4)	0.6257 (2)	3.29 (6)
C(25*c*)	0.67756 (9)	0.57910 (4)	0.7458 (2)	2.84 (5)
C(26*c*)	0.60275 (9)	0.53972 (4)	0.7061 (2)	3.51 (6)
C(27*c*)	0.53184 (9)	0.55194 (4)	0.6515 (2)	4.02 (6)
C(28*c*)	0.53574 (9)	0.60353 (4)	0.6366 (2)	4.31 (7)
C(29*c*)	0.61056 (9)	0.64291 (4)	0.6763 (2)	4.46 (7)
C(30*c*)	0.68147 (9)	0.63070 (4)	0.7309 (2)	3.72 (6)
C(31*c*)	0.82580 (9)	0.61116 (6)	0.9548 (2)	3.06 (5)
C(32*c*)	0.91225 (9)	0.62233 (6)	0.9765 (2)	4.27 (7)
C(33*c*)	0.95851 (9)	0.65779 (6)	1.0894 (2)	5.39 (8)
C(34*c*)	0.91833 (9)	0.68208 (6)	1.1805 (2)	5.86 (8)
C(35*c*)	0.83188 (9)	0.67091 (6)	1.1587 (2)	5.61 (8)
C(36*c*)	0.78561 (9)	0.63545 (6)	1.0459 (2)	4.32 (7)
N(2)	0.6884 (1)	0.01404 (8)	0.0918 (2)	3.7 (4)
P(3)	0.74683 (4)	0.06952 (2)	0.15549 (7)	3.2 (1)
C(1*d*)	0.68901 (9)	0.09834 (6)	0.2642 (1)	3.33 (6)
C(2*d*)	0.60395 (9)	0.08896 (6)	0.2239 (1)	4.23 (6)
C(3*d*)	0.55846 (9)	0.11318 (6)	0.3041 (1)	5.10 (7)
C(4*d*)	0.59804 (9)	0.14677 (6)	0.4247 (1)	4.97 (7)

Table 1 (*cont.*)

	x	y	z	U_{eq}/U_{iso}
C(5*d*)	0.68311 (9)	0.15614 (6)	0.4650 (1)	4.87 (7)
C(6*d*)	0.72859 (9)	0.13193 (6)	0.3848 (1)	4.13 (6)
C(7*d*)	0.83709 (8)	0.06747 (6)	0.2686 (2)	3.33 (6)
C(8*d*)	0.82701 (8)	0.02869 (6)	0.3605 (2)	4.39 (7)
C(9*d*)	0.89313 (8)	0.02853 (6)	0.4603 (2)	5.30 (7)
C(10*d*)	0.96934 (8)	0.06715 (6)	0.4681 (2)	5.35 (8)
C(11*d*)	0.97943 (8)	0.10593 (6)	0.3763 (2)	5.40 (8)
C(12*d*)	0.91330 (8)	0.10609 (6)	0.2765 (2)	4.55 (7)
C(13*d*)	0.7829 (1)	0.11228 (7)	0.0246 (2)	3.66 (6)
C(14*d*)	0.7564 (1)	0.15601 (7)	0.0072 (2)	4.79 (7)
C(15*d*)	0.7835 (1)	0.18743 (7)	−0.0993 (2)	6.38 (9)
C(16*d*)	0.8372 (1)	0.17512 (7)	−0.1885 (2)	6.33 (9)
C(17*d*)	0.8637 (1)	0.13139 (7)	−0.1711 (2)	6.08 (8)
C(18*d*)	0.8366 (1)	0.09996 (7)	−0.0646 (2)	4.90 (7)
P(4)	0.68562 (4)	−0.03248 (2)	−0.01943 (7)	3.1 (1)
C(19*d*)	0.6567 (1)	−0.02043 (5)	−0.1978 (2)	3.07 (6)
C(20*d*)	0.6617 (1)	−0.05328 (5)	−0.3137 (2)	3.99 (6)
C(21*d*)	0.6384 (1)	−0.04332 (5)	−0.4509 (2)	4.78 (7)
C(22*d*)	0.6100 (1)	−0.00050 (5)	−0.4722 (2)	5.09 (7)
C(23*d*)	0.6050 (1)	0.03235 (5)	−0.3563 (2)	5.12 (7)
C(24*d*)	0.6284 (1)	0.02239 (5)	−0.2191 (2)	4.13 (6)
C(25*d*)	0.60770 (9)	−0.08848 (6)	0.0217 (2)	3.11 (6)
C(26*d*)	0.60007 (9)	−0.13733 (6)	−0.0459 (2)	4.33 (7)
C(27*d*)	0.53720 (9)	−0.18068 (6)	−0.0185 (2)	5.31 (8)
C(28*d*)	0.48196 (9)	−0.17517 (6)	0.0766 (2)	5.41 (8)
C(29*d*)	0.48959 (9)	−0.12632 (6)	0.1442 (2)	5.39 (8)
C(30*d*)	0.55246 (9)	−0.08297 (6)	0.1168 (2)	4.29 (7)
C(31*d*)	0.7807 (1)	−0.04908 (7)	−0.0163 (2)	3.76 (6)
C(32*d*)	0.8341 (1)	−0.03556 (7)	−0.1194 (2)	4.72 (7)
C(33*d*)	0.9097 (1)	−0.04699 (7)	−0.1084 (2)	6.11 (8)
C(34*d*)	0.9319 (1)	−0.07195 (7)	0.0058 (2)	6.73 (9)
C(35*d*)	0.8786 (1)	−0.08547 (7)	0.1089 (2)	6.38 (9)
C(36*d*)	0.8030 (1)	−0.07404 (7)	0.0979 (2)	4.81 (7)

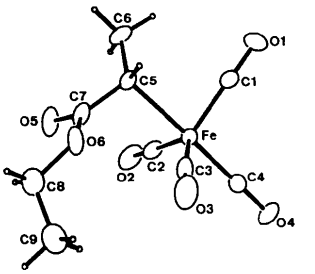

Fig. 1. An *ORTEP* plot (Johnson, 1965) of the $[(CO)_4Fe\{CH(Me_3)CO_2Et\}]$ entity (*i.e.* molecule *a*) showing 35% probability thermal ellipsoids. H atoms are drawn at an arbitrary scale.

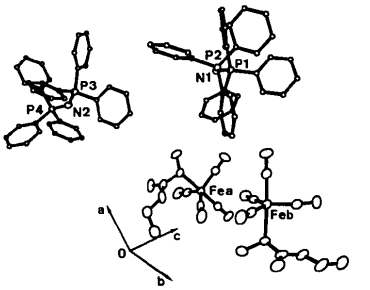

Fig. 2. An *ORTEP* plot (Johnson, 1965) of the asymmetric unit showing 35% probability thermal ellipsoids. Phenyl rings are drawn at an arbitrary scale. H atoms are omitted.

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Table 2. *Interatomic bond lengths* (Å) *and angles* (°) *with e.s.d.'s in parentheses*

Fe*a*—C(1*a*)	1.765 (3)	Fe*b*—C(1*b*)	1.776 (3)
Fe*a*—C(2*a*)	1.749 (3)	Fe*b*—C(2*b*)	1.777 (3)
Fe*a*—C(3*a*)	1.773 (4)	Fe*b*—C(3*b*)	1.773 (3)
Fe*a*—C(4*a*)	1.764 (3)	Fe*b*—C(4*b*)	1.762 (3)
Fe*a*—C(5*a*)	2.164 (3)	Fe*b*—C(5*b*)	2.188 (3)
C(1*a*)—O(1*a*)	1.146 (4)	C(1*b*)—O(1*b*)	1.156 (4)
C(2*a*)—O(2*a*)	1.167 (4)	C(2*b*)—O(2*b*)	1.158 (4)
C(3*a*)—O(3*a*)	1.147 (5)	C(3*b*)—O(3*b*)	1.159 (4)
C(4*a*)—O(4*a*)	1.143 (4)	C(4*b*)—O(4*b*)	1.151 (4)
C(5*a*)—C(6*a*)	1.542 (5)	C(5*b*)—C(6*b*)	1.507 (4)
C(5*a*)—C(7*a*)	1.466 (4)	C(5*b*)—C(7*b*)	1.469 (5)
C(5*a*)—H(C5*a*)	0.974 (6)	C(5*b*)—H(C5*b*)	0.97 (2)
C(7*a*)—O(5*a*)	1.202 (4)	C(7*b*)—O(5*b*)	1.211 (4)
C(7*a*)—O(6*a*)	1.367 (4)	C(7*b*)—O(6*b*)	1.353 (4)
O(6*a*)—C(8*a*)	1.447 (4)	O(6*b*)—C(8*b*)	1.433 (5)
C(8*a*)—C(9*a*)	1.451 (6)	C(8*b*)—C(9*b*)	1.476 (6)
N(1)—P(1)	1.581 (2)	N(2)—P(3)	1.579 (2)
N(1)—P(2)	1.570 (2)	N(2)—P(4)	1.576 (2)
P(1)—C(1*c*)	1.796 (2)	P(3)—C(1*d*)	1.781 (2)
P(1)—C(7*c*)	1.794 (2)	P(3)—C(7*d*)	1.796 (2)
P(1)—C(13*c*)	1.781 (1)	P(3)—C(13*d*)	1.790 (2)
P(2)—C(19*c*)	1.793 (2)	P(4)—C(19*d*)	1.799 (2)
P(2)—C(25*c*)	1.802 (2)	P(4)—C(25*d*)	1.791 (2)
P(2)—C(31*c*)	1.798 (1)	P(4)—C(31*d*)	1.789 (2)
C(1*a*)—Fe*a*—C(2*a*)	117.5 (2)	C(1*b*)—Fe*b*—C(2*b*)	117.9 (1)
C(1*a*)—Fe*a*—C(3*a*)	114.3 (1)	C(1*b*)—Fe*b*—C(3*b*)	118.5 (1)
C(1*a*)—Fe*a*—C(4*a*)	95.9 (1)	C(1*b*)—Fe*b*—C(4*b*)	89.7 (1)
C(1*a*)—Fe*a*—C(5*a*)	85.5 (1)	C(1*b*)—Fe*b*—C(5*b*)	93.8 (1)
C(2*a*)—Fe*a*—C(3*a*)	127.3 (1)	C(2*b*)—Fe*b*—C(3*b*)	123.3 (1)
C(2*a*)—Fe*a*—C(4*a*)	93.8 (1)	C(2*b*)—Fe*b*—C(4*b*)	93.6 (1)
C(2*a*)—Fe*a*—C(5*a*)	86.5 (1)	C(2*b*)—Fe*b*—C(5*b*)	87.2 (1)
C(3*a*)—Fe*a*—C(4*a*)	89.6 (2)	C(3*b*)—Fe*b*—C(4*b*)	92.0 (1)
C(3*a*)—Fe*a*—C(5*a*)	88.8 (1)	C(3*b*)—Fe*b*—C(5*b*)	83.8 (1)
C(4*a*)—Fe*a*—C(5*a*)	178.2 (1)	C(4*b*)—Fe*b*—C(5*b*)	175.4 (1)
Fe*a*—C(1*a*)—O(1*a*)	178.9 (4)	Fe*b*—C(1*b*)—O(1*b*)	174.1 (2)
Fe*a*—C(2*a*)—O(2*a*)	177.7 (3)	Fe*b*—C(2*b*)—O(2*b*)	177.7 (2)
Fe*a*—C(3*a*)—O(3*a*)	174.7 (3)	Fe*b*—C(3*b*)—O(3*b*)	178.0 (2)
Fe*a*—C(4*a*)—O(4*a*)	178.2 (3)	Fe*b*—C(4*b*)—O(4*b*)	179.4 (3)
Fe*a*—C(5*a*)—C(6*a*)	113.4 (2)	Fe*b*—C(5*b*)—C(6*b*)	113.5 (2)
Fe*a*—C(5*a*)—C(7*a*)	104.3 (2)	Fe*b*—C(5*b*)—C(7*b*)	105.9 (2)
Fe*a*—C(5*a*)—H(C5*a*)	103. (2)	Fe*b*—C(5*b*)—H(C5*b*)	103. (1)
C(6*a*)—C(5*a*)—C(7*a*)	109.7 (3)	C(6*b*)—C(5*b*)—C(7*b*)	112.2 (3)
C(6*a*)—C(5*a*)—H(C5*a*)	112. (2)	C(6*b*)—C(5*b*)—H(C5*b*)	112. (1)
C(7*a*)—C(5*a*)—H(C5*a*)	114. (2)	C(7*b*)—C(5*b*)—H(C5*b*)	109. (2)
C(5*a*)—C(7*a*)—O(5*a*)	129.4 (3)	C(5*b*)—C(7*b*)—O(5*b*)	127.1 (3)
C(5*a*)—C(7*a*)—O(6*a*)	111.1 (3)	C(5*b*)—C(7*b*)—O(6*b*)	112.2 (3)
O(5*a*)—C(7*a*)—O(6*a*)	119.4 (3)	O(5*b*)—C(7*b*)—O(6*b*)	120.7 (3)
C(7*a*)—O(6*a*)—C(8*a*)	116.8 (3)	C(7*b*)—O(6*b*)—C(8*b*)	117.9 (3)

Table 2 (*cont.*)

O(6*a*)—C(8*a*)—C(9*a*)	111.4 (3)	O(6*b*)—C(8*b*)—C(9*b*)	108.5 (3)
P(1)—N(1)—P(2)	139.9 (1)	P(3)—N(2)—P(4)	140.8 (1)
N(1)—P(1)—C(1*c*)	113.03 (9)	N(2)—P(3)—C(1*d*)	107.7 (1)
N(1)—P(1)—C(7*c*)	109.99 (9)	N(2)—P(3)—C(7*d*)	113.4 (1)
N(1)—P(1)—C(13*c*)	109.75 (8)	N(2)—P(3)—C(13*d*)	114.0 (1)
C(1*c*)—P(1)—C(7*c*)	110.16 (7)	C(1*d*)—P(3)—C(7*d*)	106.04 (8)
C(1*c*)—P(1)—C(13*c*)	105.96 (7)	C(1*d*)—P(3)—C(13*d*)	108.26 (9)
C(7*c*)—P(1)—C(13*c*)	107.74 (7)	C(7*d*)—P(3)—C(13*d*)	107.11 (8)
N(1)—P(2)—C(19*c*)	107.45 (9)	N(2)—P(4)—C(19*d*)	112.0 (1)
N(1)—P(2)—C(25*c*)	110.93 (8)	N(2)—P(4)—C(25*d*)	107.2 (1)
N(1)—P(2)—C(31*c*)	115.66 (9)	N(2)—P(4)—C(31*d*)	114.72 (9)
C(19)—P(2)—C(25*c*)	107.65 (8)	C(19*d*)—P(4)—C(25*d*)	107.95 (7)
C(19*c*)—P(2)—C(31*c*)	106.50 (7)	C(19*d*)—P(4)—C(31*d*)	107.67 (8)
C(25*c*)—P(2)—C(31*c*)	108.28 (8)	C(25*d*)—P(4)—C(31*d*)	107.00 (8)

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Structure of Diaquadichlorobis(hydrazinium)iron(II) Dichloride

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Abstract. $[Fe(N_2H_5)_2(H_2O)_2Cl_2].Cl_2$, $M_r = 299.65$, monoclinic, $P2_1/c$, $a = 8.027$ (1), $b = 5.725$ (2), $c = 11.430$ (2) Å, $\beta = 97.08$ (1)°, $V = 521.3$ (2) Å^3, $Z = 2$, $D_m = 1.92$, $D_x = 1.910$ g cm^{-3}, λ(Mo $K\alpha$) = 0.71069 Å, $\mu = 24.5$ cm^{-1}, $F(000) = 304$, $T = 295$ K, final $R = 0.0242$ and $wR = 0.0292$ for 1411 significant $[F_o > 5.0\sigma(F_o)]$ reflections. The crystal contains discrete Cl^- ions and complex $[Fe(N_2H_5)_2(H_2O)_2Cl_2]^{2+}$ cations. In the complex cation, the Fe atom is bonded to two hydrazinium cations, two Cl atoms and two water molecules. The coordinated atoms are *trans* to each other. The ions are connected by both N—H···Cl and O—H···Cl type hydrogen bonds.

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Experimental. The title compound was prepared by refluxing an aqueous solution of $FeCl_3.6H_2O$ and