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Structure of [μ-Hexamethylenebis(diphenylphosphine)-*P*,*P'*]bis[di-μ-carbonyl-nonacarbonyl-*triangulo*-triiron(3 *Fe–Fe*)] $\{[Fe_3(CO)_{11}]Ph_2P(CH_2)_3\}_2$

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Abstract. (I), $[Fe_6(C_{52}H_{32}O_{22}P_2)]$, $M_r = 1405{\cdot}9$, triclinic, $P\bar{1}$, $a = 12{\cdot}190$ (15), $b = 14{\cdot}908$ (8), $c = 9{\cdot}209$ (6) Å, $\alpha = 95{\cdot}56$ (5), $\beta = 112{\cdot}29$ (7), $\gamma = 108{\cdot}37$ (8)°, $V = 1424$ (5) Å^3, $Z = 1$, $D_x = 1{\cdot}65$ g cm^{-3}, λ(Mo $K\alpha$) = 0·71073 Å, $\mu = 16{\cdot}3$ cm^{-1}, $F(000) = 706$, $T = 294$ K, $R = 0{\cdot}042$ for 2219 observed reflections. The midpoint of the C_6 chain in the bis(diphenylphosphino)hexane ligand lies on a crystallographic inversion centre and each of the P atoms of bis-1,6-(diphenylphosphino)hexane is coordinated to one Fe atom of a $Fe_3(CO)_{11}$ cluster. In each cluster one Fe—Fe bond is bridged by two CO ligands; the remaining CO ligands are all terminal. The P atoms are each bonded to one of the bridged Fe atoms. Both bridging carbonyl ligands are asymmetrically bonded but the asymmetry in one bridge is more marked than in the other. The less symmetric system has one remarkably short Fe—C distance, 1·856 (7) Å, and one remarkably long one, 2·348 (6) Å. The unbridged Fe—Fe bond *trans* to the P atom is significantly longer, 2·702 (1) Å, than the other unbridged Fe—Fe bond, 2·681 (2) Å.

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Introduction. Whereas the substitution of carbonyl ligands on $[Fe_3(CO)_{12}]$ by monodentate phosphines has afforded many $[Fe_3(CO)_{12-n}(PR)_n]$ derivatives (n = 1, 2 or 3) (see *e.g.* Grant & Manning, 1978), reaction with $(Ph_2PCH_2)_2$ (diphos) disrupts the Fe_3-core and produces Fe- and Fe_2-based compounds. We have observed that the reaction between $[Fe_3(CO)_{12}]$ and bis-1,6-(diphenylphosphino)hexane (dpph) in tetrahydrofuran at 298 K for 4 h afforded three products which contained the Fe_3-core intact. One of these products analyzed as $[Fe_3(CO)_{11}]_2$-(dpph) (I) and could be recrystallized from CH_2Cl_2 solution to give dark-green crystals. The only $[Fe_3(CO)_{11}PR_3]$ compound to have been characterised previously (R = Ph) had a crystal structure in which the asymmetric unit contained two molecules which were structural isomers (Dahm & Jacobson, 1968). It was decided to characterize (I) in the anticipation that it would have a less complicated structure than $[Fe_3(CO)_{11}PPh_3]$.

Experimental. Dark-green crystal, 0·35 × 0·41 × 0·48 mm, mounted on glass fibre in random orientation. Accurate cell dimensions and crystal orientation matrix determined on CAD-4 diffractometer

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from a least-squares refinement of data collected using setting angles of 19 reflections, $6 < \theta < 10°$. Intensities of reflections with indices h 0 to 13, k −16 to 15, l −9 to 8, with $2 < 2\theta < 45{\cdot}2°$ measured; ω–2θ scans; ω-scan width $(1{\cdot}0 + 0{\cdot}35\tan\theta)°$; graphite-monochromated Mo $K\alpha$ radiation. Intensities of three reflections measured at 2 h intervals showed some evidence of slight crystal decay (intensities of standard reflections dropped by 3·6% during data collection), anisotropic decay correction applied. 3856 reflections measured, 2219 with $I > 3\sigma(I)$ labelled observed and used in structure solution and refinement. Data corrected for Lorentz, polarization and absorption effects, Gaussian integration. Space group $P\bar{1}$ assumed and confirmed by the successful analysis. Coordinates of the Fe atoms were obtained by analysis of the three-dimensional Patterson map; remaining non-H atoms were located in successive difference Fourier syntheses. H atoms were included in the refinement but restrained to ride on the C atom to which they were bonded (C—H 0·95 Å). Refinement was by full-matrix least-squares calculations on F, initially with isotropic and later with anisotropic thermal parameters for non-H atoms. The final refinement cycle included 370 variable parameters, $R = 0{\cdot}042$, $wR = 0{\cdot}053$, goodness-of-fit 1·37, $w = 1/[\sigma^2(F_o) + 0{\cdot}060(F_o)^2]$. Max shift/e.s.d. < 0·005; maximum density in final difference map 0·37 e Å^{-3}, no chemically significant features. Scattering factors and anomalous-dispersion corrections from *International Tables for X-ray Crystallography* (1974, Vol. IV). All calculations were performed on a PDP-11/73 computer using *SDP-Plus* (B. A. Frenz & Associates, Inc., 1984). Atomic coordinates and selected bond lengths and angles are given in Tables 1 and 2 respectively.* Fig. 1 is a view of the molecule prepared using *ORTEP*II (Johnson, 1976).

Discussion. The general view of (I) (Fig. 1) shows the centrosymmetric molecule to contain two (necessarily) identical Fe_3-triangles linked by the $Ph_2P(CH_2)_6PPh_2$ ligand. There are two CO ligands bridging one Fe—Fe bond and nine terminal CO groups. Each P atom of the dpph ligand is attached to one of the bridged Fe atoms and is *trans* to an unbridged Fe—Fe bond. The arrangement of carbonyl and phosphine ligands around the Fe_3 triangle is the same as that found in one of the isomers of $[Fe_3(CO)_{11}PPh_3]$ (Dahm & Jacobson, 1968). (I)

* Full details of molecular dimensions, calculated hydrogen coordinates, anisotropic thermal parameters, mean-plane data, selected torsion angles and a list of structure factors have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 53516 (33 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. *Positional and thermal parameters and their e.s.d.'s*

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

	x	y	z	B_{eq}(Å^2)
Fe(1)	−0·12079 (8)	0·22461 (6)	0·0833 (1)	3·12 (2)
Fe(2)	−0·17533 (9)	0·24499 (8)	−0·2093 (1)	4·87 (3)
Fe(3)	−0·36877 (8)	0·17419 (7)	−0·1271 (1)	4·17 (3)
P(1)	0·0880 (1)	0·2450 (1)	0·2249 (2)	2·80 (4)
O(1)	−0·0299 (5)	0·4319 (3)	0·0877 (6)	6·3 (2)
O(2)	−0·1756 (4)	0·2892 (4)	0·3492 (5)	6·0 (2)
O(3)	−0·2183 (5)	0·0232 (3)	0·1121 (6)	6·5 (2)
O(4)	−0·0857 (4)	0·0848 (4)	−0·1445 (5)	5·8 (1)
O(5)	0·0391 (6)	0·3408 (6)	−0·2830 (6)	11·6 (3)
O(6)	−0·3118 (6)	0·1092 (5)	−0·5289 (6)	9·7 (3)
O(7)	−0·2814 (6)	0·3944 (4)	−0·3015 (7)	9·7 (2)
O(8)	−0·3869 (5)	−0·0158 (4)	−0·2837 (6)	6·5 (2)
O(9)	−0·5979 (6)	0·1622 (5)	−0·4021 (7)	10·1 (2)
O(10)	−0·4830 (5)	0·1008 (4)	0·0874 (6)	7·9 (2)
O(11)	−0·3272 (5)	0·3721 (4)	0·0201 (7)	7·2 (2)
C(1)	−0·0752 (6)	0·3495 (5)	0·0509 (7)	4·6 (2)
C(2)	−0·1516 (6)	0·2667 (4)	0·2467 (7)	3·7 (2)
C(3)	−0·1819 (6)	0·1000 (5)	0·0956 (8)	4·3 (2)
C(4)	−0·1150 (6)	0·1501 (5)	−0·1243 (7)	4·0 (2)
C(5)	−0·0377 (7)	0·3067 (7)	−0·2467 (8)	7·2 (3)
C(6)	−0·2618 (7)	0·1624 (6)	−0·4045 (9)	6·1 (2)
C(7)	−0·2425 (7)	0·3369 (6)	−0·2610 (9)	6·6 (3)
C(8)	−0·3735 (6)	0·0582 (5)	−0·2206 (8)	5·0 (2)
C(9)	−0·5072 (7)	0·1660 (6)	−0·2999 (8)	6·0 (2)
C(10)	−0·4397 (6)	0·1283 (5)	0·0035 (8)	5·4 (2)
C(11)	−0·3374 (6)	0·2967 (5)	−0·0367 (8)	4·9 (2)
C(21)	0·1849 (5)	0·2666 (4)	0·1084 (6)	2·7 (1)
C(22)	0·2102 (6)	0·1949 (4)	0·0385 (7)	3·9 (2)
C(23)	0·2791 (6)	0·2152 (5)	−0·0544 (8)	5·3 (2)
C(24)	0·3247 (6)	0·3063 (5)	−0·0728 (8)	5·0 (2)
C(25)	0·3003 (6)	0·3775 (5)	−0·0045 (8)	4·9 (2)
C(26)	0·2311 (6)	0·3589 (5)	0·0879 (8)	4·3 (2)
C(31)	0·1930 (6)	0·3467 (4)	0·4046 (6)	3·2 (2)
C(32)	0·1512 (6)	0·4134 (5)	0·4598 (8)	4·6 (2)
C(33)	0·2380 (7)	0·4901 (5)	0·5913 (9)	5·9 (2)
C(34)	0·3621 (7)	0·5034 (6)	0·6656 (8)	6·0 (2)
C(35)	0·4048 (7)	0·4375 (6)	0·6128 (8)	5·8 (2)
C(36)	0·3212 (6)	0·3601 (5)	0·4826 (8)	5·0 (2)
C(41)	0·1044 (5)	0·1372 (4)	0·2960 (7)	3·3 (2)
C(42)	0·0570 (6)	0·1182 (4)	0·4275 (7)	3·6 (2)
C(43)	0·0305 (6)	0·0124 (4)	0·4428 (6)	3·7 (2)

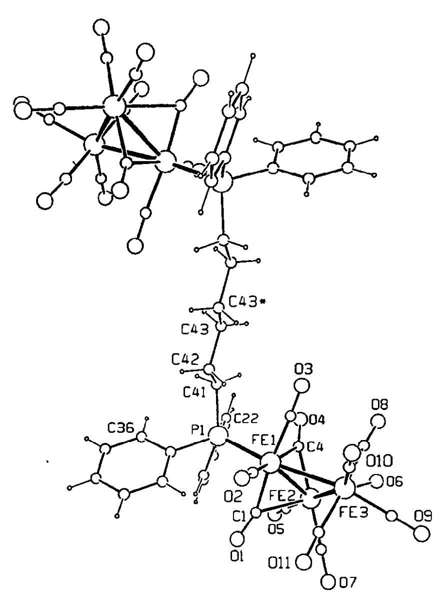

Fig. 1. View of the $\{[Fe_3(CO)_{11}]Ph_2P(CH_2)_3\}_2$ molecule with atom-numbering scheme. For clarity atoms are shown by spheres of arbitrary size.

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

Fe(1)—Fe(2)	2·593 (1)	O(4)—C(4)	1·159 (10)
Fe(1)—Fe(3)	2·702 (1)	O(5)—C(5)	1·103 (11)
Fe(1)—P(1)	2·278 (2)	O(6)—C(6)	1·141 (9)
Fe(1)—C(1)	1·856 (7)	O(7)—C(7)	1·133 (12)
Fe(1)—C(2)	1·787 (8)	O(8)—C(8)	1·128 (10)
Fe(1)—C(3)	1·803 (7)	O(9)—C(9)	1·128 (9)
Fe(1)—C(4)	2·153 (8)	O(10)—C(10)	1·131 (11)
Fe(2)—Fe(3)	2·681 (2)	O(11)—C(11)	1·140 (10)
Fe(2)—C(1)	2·348 (6)	C(21)—C(22)	1·369 (10)
Fe(2)—C(4)	1·895 (7)	C(21)—C(26)	1·374 (9)
Fe(2)—C(5)	1·819 (9)	C(22)—C(23)	1·402 (12)
Fe(2)—C(6)	1·782 (7)	C(23)—C(24)	1·349 (11)
Fe(2)—C(7)	1·821 (10)	C(24)—C(25)	1·350 (12)
Fe(3)—C(8)	1·829 (8)	C(25)—C(26)	1·399 (12)
Fe(3)—C(9)	1·786 (7)	C(31)—C(32)	1·388 (11)
Fe(3)—C(10)	1·799 (9)	C(31)—C(36)	1·387 (9)
Fe(3)—C(11)	1·789 (8)	C(32)—C(33)	1·374 (8)
P(1)—C(21)	1·854 (7)	C(33)—C(34)	1·340 (11)
P(1)—C(31)	1·833 (5)	C(34)—C(35)	1·375 (14)
P(1)—C(41)	1·833 (7)	C(35)—C(36)	1·368 (8)
O(1)—C(1)	1·131 (8)	C(41)—C(42)	1·540 (10)
O(2)—C(2)	1·140 (10)	C(42)—C(43)	1·538 (9)
O(3)—C(3)	1·139 (8)	C(43)—C(43)*	1·514 (10)

Fe(2)—Fe(1)—Fe(3)	60·80 (4)	Fe(3)—Fe(2)—C(1)	82·9 (2)
Fe(2)—Fe(1)—P(1)	113·16 (7)	Fe(3)—Fe(2)—C(4)	87·6 (2)
Fe(2)—Fe(1)—C(1)	61·1 (2)	Fe(3)—Fe(2)—C(5)	173·0 (3)
Fe(2)—Fe(1)—C(2)	136·0 (2)	Fe(3)—Fe(2)—C(6)	92·9 (3)
Fe(2)—Fe(1)—C(3)	114·2 (2)	Fe(3)—Fe(2)—C(7)	83·8 (3)
Fe(2)—Fe(1)—C(4)	45·9 (2)	C(1)—Fe(2)—C(4)	90·9 (3)
Fe(3)—Fe(1)—P(1)	168·87 (7)	C(1)—Fe(2)—C(5)	90·6 (3)
Fe(3)—Fe(1)—C(1)	92·3 (2)	C(1)—Fe(2)—C(6)	175·8 (4)
Fe(3)—Fe(1)—C(2)	90·8 (2)	C(1)—Fe(2)—C(7)	82·1 (3)
Fe(3)—Fe(1)—C(3)	81·4 (2)	C(4)—Fe(2)—C(5)	95·4 (4)
Fe(3)—Fe(1)—C(4)	82·1 (2)	C(4)—Fe(2)—C(6)	89·4 (4)
P(1)—Fe(1)—C(1)	92·3 (2)	C(4)—Fe(2)—C(7)	169·4 (4)
P(1)—Fe(1)—C(2)	99·4 (2)	C(5)—Fe(2)—C(6)	93·5 (4)
P(1)—Fe(1)—C(3)	93·6 (2)	C(5)—Fe(2)—C(7)	92·6 (4)
P(1)—Fe(1)—C(4)	87·2 (2)	C(6)—Fe(2)—C(7)	97·0 (4)
C(1)—Fe(1)—C(2)	89·9 (3)	Fe(1)—Fe(3)—Fe(2)	57·59 (4)
C(1)—Fe(1)—C(3)	173·6 (3)	Fe(1)—Fe(3)—C(8)	90·8 (2)
C(1)—Fe(1)—C(4)	98·3 (3)	Fe(1)—Fe(3)—C(9)	160·8 (3)
C(2)—Fe(1)—C(3)	91·5 (3)	Fe(1)—Fe(3)—C(10)	97·6 (2)
C(2)—Fe(1)—C(4)	169·3 (2)	Fe(1)—Fe(3)—C(11)	82·0 (2)
C(3)—Fe(1)—C(4)	79·6 (3)	Fe(2)—Fe(3)—C(8)	81·7 (3)
Fe(1)—Fe(2)—Fe(3)	61·61 (4)	Fe(2)—Fe(3)—C(9)	104·0 (3)
Fe(1)—Fe(2)—C(1)	43·8 (2)	Fe(2)—Fe(3)—C(10)	155·0 (2)
Fe(1)—Fe(2)—C(4)	54·7 (2)	Fe(2)—Fe(3)—C(11)	88·5 (3)
Fe(1)—Fe(2)—C(5)	115·1 (2)	C(8)—Fe(3)—C(9)	91·8 (3)
Fe(1)—Fe(2)—C(6)	134·1 (3)	C(8)—Fe(3)—C(10)	96·9 (4)
Fe(1)—Fe(2)—C(7)	115·4 (3)	C(8)—Fe(3)—C(11)	169·9 (4)
C(9)—Fe(3)—C(10)	101·0 (4)	Fe(2)—C(4)—O(4)	148·5 (6)
C(9)—Fe(3)—C(11)	92·7 (3)	Fe(2)—C(5)—O(5)	174·0 (6)
C(10)—Fe(3)—C(11)	91·0 (4)	Fe(2)—C(6)—O(6)	176·7 (9)
Fe(1)—P(1)—C(21)	114·9 (2)	Fe(2)—C(7)—O(7)	175·1 (9)
Fe(1)—P(1)—C(31)	119·7 (3)	Fe(3)—C(8)—O(8)	174·4 (6)
Fe(1)—P(1)—C(41)	112·5 (2)	Fe(3)—C(9)—O(9)	175·4 (9)
C(21)—P(1)—C(31)	99·9 (3)	Fe(3)—C(10)—O(10)	178·9 (5)
C(21)—P(1)—C(41)	103·7 (3)	Fe(3)—C(11)—O(11)	174·4 (7)
C(31)—P(1)—C(41)	104·2 (3)	P(1)—C(31)—C(32)	122·8 (4)
Fe(1)—C(1)—Fe(2)	75·1 (2)	P(1)—C(31)—C(36)	118·1 (6)
Fe(1)—C(1)—O(1)	156·1 (6)	P(1)—C(21)—C(22)	123·5 (5)
Fe(2)—C(1)—O(1)	128·7 (6)	P(1)—C(21)—C(26)	118·2 (6)
Fe(1)—C(2)—O(2)	176·7 (5)	P(1)—C(41)—C(42)	113·2 (5)
Fe(1)—C(3)—O(3)	176·3 (6)	C(41)—C(42)—C(43)	111·2 (6)
Fe(1)—C(4)—Fe(2)	79·4 (3)	C(42)—C(43)—C(43)*	112·8 (6)
Fe(1)—C(4)—O(4)	132·1 (5)		

* Refers to equivalent position $-x$, $-y$, $1-z$.

has the P atom approximately in the plane containing the three Fe atoms. This is a feature found in both isomers of $[Fe_3(CO)_{11}PPh_3]$, in $[Fe_3(CO)_{10}\{P(OMe)_3\}_2]$ (Adams, Bailey, Bentley & Mann, 1989) and $[Fe_3(CO)_9(PMe_2Ph)_3]$ (Raper & McDonald, 1971).

The asymmetry of both the bridging carbonyl ligands is a notable feature of (I), with the bonding to the carbonyl C(1)—O(1) more asymmetric than to C(4)—O(4) (Table 2). The Fe(1)—C(1) and Fe(2)—C(1) bond lengths are 1·856 (7) and 2·348 (6) Å respectively, with angles Fe(1)—C(1)—O(1) 156·1 (6) and Fe(2)—C(1)—O(1) 128·7 (6)°. The corresponding Fe—C distances in the Fe(1)—C(4)—Fe(2) bridge are 2·153 (8) and 1·895 (7) Å respectively and the angles are 132·1 (5) and 148·5 (6)°. Asymmetric bonding to bridging CO groups was noted previously in $[Fe_3(CO)_{12}]$ (Cotton & Troup, 1974) but it is much more marked in (I). The isomer of $[Fe_3(CO)_{11}PPh_3]$ which is similar to (I) has an even less asymmetric bridged section than $[Fe_3(CO)_{12}]$. In (I), the Fe(1)—C(1) distance is remarkably short and is only 0·054 Å longer than the mean value of all terminal Fe—C distances (1·802 Å). Hence the Fe(2)—C(1) bond length is remarkably long.

Another noteworthy feature in (I) is the difference in the lengths of the bonds between the unbridged Fe and the bridged Fe atoms. That between Fe(1), the phosphine substituted site, and Fe(3) is 2·702 (1) Å which is significantly longer than Fe(2)—Fe(3), 2·681 (2) Å, even at the 3σ level. A similar situation occurred in the related $[Fe_3(CO)_{11}PPh_3]$ isomer but the difference was not discernable at the 3σ level. Since the equivalent Fe—Fe distances in $[Fe_3(CO)_{12}]$ were 2·677 (2) and 2·681 (3) Å, it is clear that the phosphine substitution has weakened the Fe—Fe bond *trans* to the phosphine ligand. With respect to the disintegration of the Fe_3 core in the reaction of $[Fe_3(CO)_{12}]$ with diphos, it seems possible that this is initiated by the double substitution by the phosphine leading to weakening of both Fe—Fe bonds. This would occur most probably at the unbridged iron site for steric reasons. The bridged Fe—Fe iron bond in (I) is also affected by the phosphine substitution. The distance in (I) [2·593 (1) Å] is much longer than that in $[Fe_3(CO)_{12}]$ [2·558 (1) Å].

The Fe—P distance, 2·278 (2) Å, is similar to those in the $[Fe_3(CO)_{11}PPh_3]$ isomers [2·24 (1) and 2·25 (1) Å] and the mean value of 2·237 Å in $[Fe_3(CO)_9(PMe_2Ph)_3]$.

The mean terminal Fe—C and C—O distances in (I), 1·802 and 1·131 Å respectively, compare with values of 1·82 and 1·13 Å in $[Fe_3(CO)_{12}]$. All bond distances and angles in the dpph ligand are normal.

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Structure of Imidazolium Hexachlorotantalate(V)

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Abstract. $C_3H_5N_2^+.[TaCl_6]^-$, $M_r = 462{\cdot}75$, orthorhombic, *Pnma*, $a = 14{\cdot}796\,(8)$, $b = 6{\cdot}985\,(3)$, $c = 11{\cdot}011\,(6)$ Å, $V = 1138{\cdot}0$ Å^3, $D_x = 2{\cdot}701$ Mg m^{-3}, $Z = 4$, λ(Mo $K\overline{\alpha}$) = 0·71069 Å, $\mu = 10{\cdot}92$ mm^{-1}, $F(000) = 848$, $T = 185$ K, $R = 0{\cdot}042$ for 847 observed reflections. The structure consists of roughly octahedral $[TaCl_6]^-$ anions [Ta—Cl = 2·317 (3)–2·362 (3) Å] and imidazolium cations interacting *via* N—H···Cl hydrogen bonds. There is also cohesion between the layers due to normal van der Waals contacts.

Introduction. The reactions of Nb and Ta halides with various nitrogen-containing aromatic ligands are currently being investigated in this laboratory. When imidazole ligands are reacted with $TaCl_5$ in 1:1 ratio in benzene or toluene, the main product is the sparingly soluble $TaCl_5$(imidazole) monoadduct (Levasseur & Beauchamp, 1990). The filtrate from one such preparation was kept under argon in a Schlenk tube at room temperature. Three months later, a few red crystals had appeared. Under similar conditions, $NbCl_5$ and 7-azaindole had produced a condensed [(7-azaindolyl)-7-azaindolium]$^+$ cation (Poitras & Beauchamp, 1990). The few crystals available of the red imidazole material were used to identify the compound by X-ray diffraction. Our results show that the imidazole unit remains monomeric in the present case.

Experimental. Red crystals very sensitive to moisture. Specimen transferred under dry argon and sealed in a Lindemann capillary. Bounded by the following pairs of faces (separation in mm): (010)/(0$\bar{1}$0),0·10; (001)/(00$\bar{1}$), 0·20; ($\bar{1}$01)/(10$\bar{1}$), 0·17; (100)/($\bar{1}$00), 0·24. Nonius CAD-4 diffractometer, equipped with an N_2 cooling system, graphite-monochromatized Mo $K\overline{\alpha}$ radiation. Unit-cell dimensions from 25 centered reflections in the range $20 \leq 2\theta \leq 25°$. Laue symmetry and cell dimensions checked with long-exposure axial photographs along the three axes. ω–2θ scan, $\omega = (1{\cdot}00 + 0{\cdot}35\tan\theta)°$, $2\theta_{max} = 50{\cdot}0°$. Orientation monitored every 200 measurements, intensity checked every hour by using seven standard reflections, intensity fluctuation within ±2·5%. 3393 hkl, $\bar{h}\bar{k}l$, $\bar{h}kl$ reflections measured (h: 0–17, k: 0–8, l: 0–12). Absorption correction based on crystal geometry applied (Gaussian integration, grid 10 × 10 × 10, transmission range 0·05–0·14). 1093 independent hkl reflections after octant averaging ($R_{av} = 0{\cdot}042$), 847 with $I \geq 3{\cdot}0\sigma(I)$ retained for structure determination and refinement. Data corrected for Lp.

Orthorhombic Laue symmetry and systematic absences ($0kl$ $k + l \neq 2n$, $hk0$ $h \neq 2n$) consistent with space groups *Pnma* and $Pn2_1a$ (alternate setting for $Pna2_1$). Structure solved in the centrosymmetric space group *Pnma* by the heavy-atom method. Ta found to lie on mirror plane [equipoint 4(c)] from a Patterson map. Four Cl [two in general position, two on 4(c)] and the five non-H atoms [all on 4(c)] of the imidazolium cation located from difference Fourier (ΔF) maps.

Special care was taken to identify the N and C atoms in the imidazole unit. The five ring atoms were first defined as carbons C*‡ and refined isotropically, whereas the Ta and Cl atoms were refined anisotropically. All H atoms were visible on the ΔF synthesis. They were fixed at idealized positions [C(N)—H = 0·95 Å, $U = 0{\cdot}10$ Å^2] and repositioned after each

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‡ Asterisked symbols represent the ring atoms defined as carbon at this early stage.

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