three adjacent coordination sites, completing the octahedral coordination about each Mo. In the η^7-cycloheptatrienyl rings the C—C bonds are in the range 1·38 (2)–1·46 (2) [mean 1·41 (2) Å], while the C—C—C angles vary from 126 (1) to 131 (1) [mean 128 (1)°], with no systematic variation. They can be described as essentially regular heptagons and are planar within experimental error (planes 1 to 4 in Table 3). The Mo—C distances are in the range 2·23 (1)–2·30 (1) [mean 2·27 (2) Å]; the perpendicular distances from the Mo atoms to the rings vary from 1·573–1·598 [mean 1·583 (11) Å], and the perpendiculars to the η^7-cycloheptatrienyl rings at the Mo atoms meet the rings close (0·02 Å mean) to their centroids. The difference in formal valences of the Mo atoms in the tetramer, Mo^{II}, and the dimer, Mo^{IV}, is reflected in these ring-normal distances, those in the tetramer [1·583 (11) Å, mean] being significantly longer than those in the dimer (1·567 Å, mean).

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The Crystal and Molecular Structures of Dichloro(triphenylphosphine)-(η^5-tetramethylethylcyclopentadienyl)cobalt Toluene Solvate and Tri-μ-chloro-bis(η^5-tetramethylethylcyclopentadienyl)dicobalt Tetrachloroferrate

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The crystal structures of dichloro(triphenylphosphine)(η^5-tetramethylethylcyclopentadienyl)cobalt toluene solvate, $C_{36}H_{40}Cl_2CoP$, compound I, monoclinic, a = 8·560 (6), b = 17·791 (5), c = 21·065 (5) Å, β = 98·87 (6)°, Z = 4, space group $P2_1/n$, 2679 reflexions, R = 0·061 and tri-μ-chloro-bis(η^5-tetramethylethylcyclopentadienyl)dicobalt tetrachloroferrate, $C_{22}H_{34}Cl_7Co_2Fe$, compound II, orthorhombic, a = 15·940 (4), b = 25·819 (3), c = 15·067 (1) Å, Z = 8, space group $Pbca$, 2108 reflexions, R = 0·086 have been determined from diffractometer data. The coordination around the Co atoms in both compounds may be described as distorted octahedral; in compound II the two Co octahedra share one face, with a Co···Co distance of 2·887 (2) Å.

Introduction

The chemistry of pentaalkylcyclopentadienyl transition metal complexes has been comparatively unexplored. The work of Maitlis and coworkers (Lee, Moseley, White & Maitlis, 1975) has shown that certain η^5-pentamethylcyclopentadienyl complexes of rhodium and iridium possess properties markedly different from their unsubstituted analogues. Therefore, in the development of the chemistry of η^5-tetramethylethylcyclopentadienylcobalt complexes (Pardy, 1977) (η^5-tetramethylethylcyclopentadienyl)tin tri-*n*-butyl was found to react with cobalt(II) chloride in tetrahydrofuran to give a red-brown oil. Treatment of this oil with chlorine gave the complexes $[(\eta^5$-tetramethylethylcyclopentadienyl)$CoCl_2]_2$ and $[(\eta^5$-tetramethylethylcyclopentadienyl$)_2Co_3Cl_6]$. Both complexes dissolved in water giving blue solutions believed to contain the $[(\eta^5$-tetramethylethylcyclopentadienyl$)_2$-$Co_2(\mu$-$Cl)_3]^+$ ion, which was precipitated from concentrated HCl using $FeCl_4^-$, and the crystal structure of $[(\eta^5$-tetramethylethylcyclopentadienyl$)_2Co_2(\mu$-$Cl)_3]$-$[FeCl_4]$ is reported here together with that of the toluene solvate of the reaction product of $[(\eta^5$-tetramethylethylcyclopentadienyl)$CoCl_2]_2$ with triphenylphosphine.

Experimental

Samples of both compounds (I, blue needles; II, dark blue needles) were provided by Drs R. B. A. Pardy and M. L. H. Green. They are sensitive to air and moisture and were sealed in glass capillaries.

After survey photography by Weissenberg and precession techniques, the selected crystals were mounted on a Nonius CAD-4F, PDP-8 controlled, κ-geometry diffractometer and accurate cell dimensions and orientation matrix obtained by a least-squares fit to the setting angles of 25 reflexions.

Crystal data

Compound I: $C_{36}H_{40}Cl_2CoP$, $M_r = 633\cdot5$. Monoclinic, $a = 8\cdot560\,(6)$, $b = 17\cdot791\,(5)$, $c = 21\cdot065\,(5)$ Å, $\beta = 98\cdot87\,(6)°$, $U = 3169\cdot6$ Å^3. Systematic extinctions: $h0l$, $h + l = 2n + 1$; $0k0$, $k = 2n + 1$. Space group $P2_1/n$ $(\frac{1}{2} + x, \frac{1}{2} - y, \frac{1}{2} + z)$. $D_c = 1\cdot33$ g cm^{-3} for $Z = 4$. $D_m = 1\cdot34$ g cm^{-3}. Mo $K\alpha$ radiation, $\lambda = 0\cdot71069$ Å, $\mu = 7\cdot8$ cm^{-1}.

Compound II: $C_{22}H_{34}Cl_7Co_2Fe$, $M_r = 720\cdot4$. Orthorhombic, $a = 15\cdot940\,(4)$, $b = 25\cdot819\,(3)$, $c = 15\cdot067\,(1)$ Å, $U = 6201\cdot1$ Å^3. Systematic extinctions: $0kl$, $k = 2n + 1$; $h0l$, $l = 2n + 1$; $hk0$, $h = 2n + 1$. Space group $Pbca$ (No. 61, D_{2h}^{15}). $D_c = 1\cdot54$ g cm^{-3} for $Z = 8$. $D_m = 1\cdot52$ g cm^{-3}. Mo $K\alpha$ radiation, $\lambda = 0\cdot71069$ Å, $\mu = 21\cdot34$ cm^{-1}.

For each crystal, the intensities of every independent reflexion with $\sin\theta/\lambda < 0\cdot53$ Å^{-1} were measured with an $\omega/2\theta$ scan, a variable scan rate and an ω scan angle of $1\cdot20 + 0\cdot35\tan\theta$. Mo $K\alpha$ radiation was used with a graphite monochromator. Reflexions with $I < 3\sigma(I)$, where $\sigma(I)$ is the standard deviation based on simple counting statistics, were not included in subsequent calculations. I and II gave sets of 2679 and 2108 independent structure amplitudes corrected for Lorentz and polarization effects but not absorption.

Structure solution and refinement

The structure of I was solved by Patterson and Fourier techniques, and refined by a blocked-matrix least-squares method [block 1: scale, dummy overall isotropic temperature factor (Rollett, 1965); block 2: x,y,z for the Co, Cl and P atoms; block 3: Co, Cl and P anisotropic temperature factors; block 4: η^5-tetramethylethylcyclopentadienyl x,y,z; block 5: η^5-tetramethylethylcyclopentadienyl temperature factors; blocks 6, 7 and 8: x,y,z for each C_6H_5 ring; blocks 9, 10, 11: temperature factors for each C_6H_5 ring; block 12: toluene x,y,z; block 13: toluene temperature factors]. All non-hydrogen atoms had anisotropic temperature factors. The H atoms were located from difference syntheses and were included in the refinement with isotropic temperature factors. The C—H bond lengths were constrained (Waser, 1963; Rollett, 1969) to be $1\cdot00$ Å with an estimated standard deviation of $0\cdot03$ Å; C—C—H angles in the six-membered rings were constrained to be 120° with an e.s.d. of 1°; H—C—H and C—C—H angles in the η^5-tetramethylethylcyclopentadienyl ring and toluene methyl group were constrained to be 109° with an e.s.d. of 1°. In addition, the difference in mean square displacement along the bond direction of the two atoms (C and H) forming each bond was constrained to be zero with an e.s.d. of $0\cdot002$ Å^2. In the final stages of refinement each reflexion was assigned a weight: $w^{1/2} = |F_o|/5\cdot5$ if $|F_o| \le 5\cdot5$, otherwise $w^{1/2} = 5\cdot5/|F_o|$, and the final R_w was $0\cdot061$ for 2679 reflexions.

For compound II, the Co, Fe and Cl atoms in the molecule were located from an E map based on the best phase set after weighted, multi-solution tangent refinement, with three origin-defining phases (102, 6,16,11, 112) and two multi-solution phases (7,4,14, 13,10,8); 417 reflexions with $|E|$ greater than $1\cdot5$ were used in this refinement.

The light atoms were located from subsequent difference syntheses. Refinement was by a least-squares method with a large block approximation to the normal matrix: a 2×2 block was calculated from the derivatives of the scale and dummy overall isotropic temperature factor, two blocks from those of the positional parameters of the cation and anion respectively, and a further two from those of the corresponding temperature factors. All non-hydrogen atoms had anisotropic temperature factors. Difference syntheses showed some evidence for H atoms at their expected positions. However, they were positioned geometrically (C—H = $1\cdot0$ Å, $U_{\rm iso} = 0\cdot05$ Å^2) and included in the structure factor calculations, their locations being readjusted after each cycle. In the final stages an overall isotropic extinction parameter ($363\cdot2$) was introduced into the refinement (Larson, 1967). Each reflexion was assigned a weight

$$w = 1/\sum_{r=1}^{n} A_r T_r^*(X)$$

where n is the number of coefficients A_r, for a Chebyshev series, T_r^* is the polynomial function, and X is $|F_o|/|F_o(\max)|$. Three coefficients, A_r, were used with values 16·94, 23·25 and 7·38 (Rollett, 1965) and the final weighted R_w was 0·086 for 2108 reflexions.

All calculations for both structures were performed on the Oxford University ICL 1906A computer with the Oxford *CRYSTALS* package (Carruthers, 1975). Complex neutral-atom scattering factors were taken from *International Tables for X-ray Crystallography* (1974).

Results and discussion

The final atomic positional parameters are given in Tables 1 and 2,† and bond lengths and angles, with e.s.d.'s calculated from the variance–covariance matrix, in Tables 3 and 4. Projections of the molecules are shown in Figs. 1 and 2. Details of important molecular planes are given in Table 5.

For compound I, the asymmetric unit contains one $C_{29}H_{32}Cl_2CoP$ molecule and a molecule of toluene solvate. The shortest intermolecular distances (not involving H atoms) are C(3)···C(11) 3·560 Å, C(2)···C(11) 3·642 Å, C(6)···C(34) 3·648 Å, with C(11) and C(34) related by $-x,-y,-z$ to the parameters in Table 1. The toluene solvent molecule is planar [plane (6) in Table 5] and interatomic distances and angles are normal but with somewhat high standard deviations, probably a consequence of high thermal motion.

Table 1. *Fractional atomic coordinates and isotropic temperature factors, with estimated standard deviations in parentheses, for compound* I ($C_{36}H_{40}Cl_2CoP$)

	x	y	z	U_{iso} (Å^2)
Co(1)	0·33042 (9)	0·03796 (5)	0·18677 (4)	
P(1)	0·4709 (2)	−0·0430 (1)	0·25845 (8)	
Cl(1)	0·3341 (2)	0·1191 (1)	0·26969 (8)	
Cl(2)	0·5497 (2)	0·0883 (1)	0·15325 (9)	
C(1)	0·2412 (7)	0·0042 (4)	0·0927 (3)	
C(2)	0·1881 (7)	0·0795 (4)	0·1021 (3)	
C(3)	0·0964 (7)	0·0768 (4)	0·1524 (3)	
C(4)	0·0953 (7)	0·0013 (4)	0·1758 (3)	
C(5)	0·1798 (7)	−0·0448 (4)	0·1366 (3)	
C(6)	0·1841 (9)	−0·1280 (4)	0·1352 (4)	
C(7)	0·0006 (8)	−0·0242 (4)	0·2254 (3)	
C(8)	0·0069 (8)	0·1399 (4)	0·1757 (4)	
C(9)	0·2159 (9)	0·1459 (4)	0·0627 (4)	
C(10)	0·3233 (8)	−0·0197 (4)	0·0393 (3)	
C(11)	0·2071 (9)	−0·0292 (5)	−0·0233 (3)	
C(12)	0·3394 (7)	−0·0934 (4)	0·3046 (3)	
C(13)	0·2913 (7)	−0·1667 (4)	0·2912 (3)	
C(14)	0·1770 (9)	−0·1990 (4)	0·3236 (4)	
C(15)	0·1161 (9)	−0·1600 (5)	0·3690 (4)	
C(16)	0·1663 (9)	−0·0869 (5)	0·3838 (4)	
C(17)	0·2766 (8)	−0·0529 (4)	0·3518 (3)	
C(18)	0·5888 (7)	−0·1155 (4)	0·2266 (3)	
C(19)	0·6592 (8)	−0·1745 (4)	0·2653 (3)	
C(20)	0·7578 (8)	−0·2255 (4)	0·2415 (4)	
C(21)	0·7909 (9)	−0·2178 (5)	0·1800 (4)	
C(22)	0·7218 (9)	−0·1613 (5)	0·1412 (4)	
C(23)	0·6227 (8)	−0·1100 (4)	0·1647 (3)	
C(24)	−0·1200 (7)	0·4972 (4)	0·1779 (3)	
C(25)	−0·2091 (8)	0·5590 (4)	0·1900 (3)	
C(26)	−0·3292 (8)	0·5847 (4)	0·1421 (4)	
C(27)	−0·3597 (8)	0·5484 (5)	0·0839 (4)	
C(28)	−0·2708 (9)	0·4880 (5)	0·0720 (4)	
C(29)	−0·1526 (8)	0·4610 (5)	0·1189 (3)	
C(30)	−0·139 (1)	0·2812 (5)	0·0354 (4)	
C(31)	−0·272 (1)	0·2452 (6)	0·0442 (5)	
C(32)	−0·399 (1)	0·2427 (6)	−0·0043 (5)	
C(33)	−0·389 (1)	0·2756 (6)	−0·0601 (5)	
C(34)	−0·254 (1)	0·3130 (5)	−0·0703 (4)	
C(35)	−0·126 (1)	0·3153 (5)	−0·0219 (4)	
C(36)	0·019 (1)	0·3565 (8)	−0·0312 (7)	
H(61)	0·116 (4)	−0·144 (2)	0·095 (1)	0·049 (5)
H(62)	0·145 (4)	−0·151 (3)	0·172 (2)	0·063 (6)
H(63)	0·295 (4)	−0·146 (3)	0·134 (2)	0·050 (5)
H(71)	0·020 (5)	−0·080 (2)	0·234 (2)	0·057 (6)
H(72)	−0·109 (4)	−0·017 (2)	0·205 (2)	0·041 (5)
H(73)	0·019 (5)	0·004 (2)	0·267 (2)	0·056 (5)
H(81)	0·044 (5)	0·189 (2)	0·163 (2)	0·074 (7)
H(82)	−0·107 (3)	0·135 (3)	0·159 (2)	0·037 (5)
H(83)	0·022 (5)	0·137 (2)	0·223 (1)	0·058 (6)
H(91)	0·130 (4)	0·150 (3)	0·026 (2)	0·043 (5)
H(92)	0·216 (5)	0·192 (2)	0·089 (2)	0·064 (6)
H(93)	0·318 (4)	0·142 (3)	0·047 (2)	0·063 (6)
H(101)	0·378 (5)	−0·069 (2)	0·051 (1)	0·069 (6)
H(102)	0·402 (4)	0·018 (2)	0·031 (3)	0·047 (5)
H(111)	0·137 (3)	0·014 (2)	−0·037 (2)	0·087 (7)
H(112)	0·141 (3)	−0·073 (2)	−0·013 (3)	0·083 (7)
H(113)	0·264 (5)	−0·043 (2)	−0·059 (2)	0·034 (5)
H(131)	0·346 (5)	−0·198 (1)	0·263 (2)	0·054 (5)
H(141)	0·140 (6)	−0·252 (2)	0·313 (2)	0·056 (6)
H(151)	0·037 (5)	−0·183 (1)	0·392 (2)	0·034 (5)
H(161)	0·126 (5)	−0·060 (2)	0·418 (2)	0·051 (6)
H(171)	0·307 (6)	0·000 (2)	0·361 (2)	0·050 (6)
H(191)	0·642 (6)	−0·178 (2)	0·311 (2)	0·057 (5)
H(201)	0·810 (6)	−0·265 (2)	0·270 (1)	0·060 (6)
H(211)	0·855 (5)	−0·256 (2)	0·162 (1)	0·038 (5)
H(221)	0·756 (5)	−0·152 (2)	0·100 (2)	0·057 (6)
H(231)	0·578 (6)	−0·068 (2)	0·137 (1)	0·059 (6)
H(251)	−0·191 (5)	0·583 (2)	0·233 (1)	0·049 (5)
H(261)	−0·395 (5)	0·629 (2)	0·151 (1)	0·049 (5)
H(271)	−0·435 (4)	0·570 (2)	0·049 (1)	0·029 (5)
H(281)	−0·295 (6)	0·461 (3)	0·030 (2)	0·08 (2)
H(291)	−0·097 (5)	0·413 (2)	0·111 (1)	0·046 (5)
H(301)	−0·046 (4)	0·280 (3)	0·070 (2)	0·062 (6)
H(311)	−0·275 (3)	0·215 (3)	0·084 (2)	0·058 (7)
H(321)	−0·495 (4)	0·214 (3)	0·001 (1)	0·080 (9)
H(331)	−0·480 (4)	0·272 (3)	−0·096 (1)	0·072 (8)
H(341)	−0·243 (3)	0·330 (3)	−0·114 (1)	0·058 (6)
H(361)	0·088 (5)	0·312 (2)	−0·027 (2)	0·17 (1)
H(362)	0·082 (4)	0·401 (2)	−0·014 (2)	0·09 (1)
H(363)	−0·021 (6)	0·365 (2)	−0·078 (2)	0·18 (1)

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† Lists of structure factors and anisotropic thermal parameters for the non-hydrogen atoms have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 33529 (50 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 2. *Fractional atomic coordinates, with estimated standard deviations in parentheses, for compound* II ($C_{22}H_{34}Cl_7Co_2Fe$)

	x	*y*	*z*
Co(1)	0·2569 (1)	0·49013 (7)	0·3400 (1)
Co(2)	0·1767 (1)	0·38987 (7)	0·3403 (1)
Cl(1)	0·2179 (5)	0·4399 (2)	0·4550 (3)
Cl(2)	0·1317 (4)	0·4662 (2)	0·2800 (5)
Cl(3)	0·3010 (4)	0·4136 (2)	0·2780 (5)
C(1)	0·2455 (9)	0·5645 (6)	0·380 (1)
C(2)	0·3184 (8)	0·5411 (6)	0·4208 (9)
C(3)	0·3720 (8)	0·5250 (5)	0·3549 (9)
C(4)	0·3353 (8)	0·5386 (5)	0·269 (1)
C(5)	0·2575 (9)	0·5629 (6)	0·284 (1)
C(6)	0·170 (1)	0·5893 (9)	0·427 (1)
C(7)	0·334 (1)	0·5374 (8)	0·517 (1)
C(8)	0·4537 (9)	0·4972 (7)	0·367 (1)
C(9)	0·375 (1)	0·5274 (7)	0·180 (1)
C(10)	0·200 (1)	0·5862 (7)	0·218 (1)
C(11)	0·218 (1)	0·641 (1)	0·212 (2)
C(12)	0·1767 (9)	0·3181 (7)	0·285 (1)
C(13)	0·1922 (9)	0·3157 (6)	0·377 (1)
C(14)	0·122 (1)	0·3351 (6)	0·423 (1)
C(15)	0·0644 (9)	0·3515 (6)	0·355 (1)
C(16)	0·0986 (9)	0·3405 (6)	0·2724 (9)
C(17)	0·231 (1)	0·2957 (9)	0·212 (2)
C(18)	0·272 (1)	0·2911 (8)	0·418 (2)
C(19)	0·110 (2)	0·3378 (9)	0·521 (1)
C(20)	−0·018 (1)	0·3788 (8)	0·376 (2)
C(21)	0·056 (1)	0·3477 (7)	0·185 (1)
C(22)	0·003 (2)	0·302 (1)	0·163 (1)
Fe(1)	0·0116 (1)	0·17368 (9)	0·3882 (2)
Cl(4)	−0·0509 (3)	0·1131 (2)	0·4648 (3)
Cl(5)	−0·0612 (3)	0·2443 (2)	0·4020 (4)
Cl(6)	0·1376 (3)	0·1841 (2)	0·4415 (4)
Cl(7)	0·0215 (4)	0·1503 (2)	0·2495 (3)
H(61)	0·1288	0·6022	0·3819
H(62)	0·1890	0·6189	0·4648
H(63)	0·1415	0·5628	0·4657
H(71)	0·2849	0·5521	0·5503
H(72)	0·3857	0·5574	0·5329
H(73)	0·3416	0·5003	0·5342
H(81)	0·4652	0·4925	0·4316
H(82)	0·4999	0·5182	0·3395
H(83)	0·4509	0·4626	0·3372
H(91)	0·4300	0·5098	0·1887
H(92)	0·3836	0·5606	0·1470
H(93)	0·3369	0·5044	0·1445
H(101)	0·1404	0·5811	0·2377
H(102)	0·2084	0·5693	0·1592
H(111)	0·1801	0·6571	0·1678
H(112)	0·2099	0·6572	0·2715
H(113)	0·2779	0·6454	0·1930
H(171)	0·2049	0·3031	0·1530
H(172)	0·2880	0·3117	0·2147
H(173)	0·2358	0·2574	0·2204
H(181)	0·3118	0·2810	0·3695
H(182)	0·3006	0·3170	0·4578
H(183)	0·2568	0·2599	0·4535
H(191)	0·1604	0·3234	0·5517
H(192)	0·1013	0·3746	0·5395
H(193)	0·0593	0·3169	0·5381
H(201)	−0·0482	0·3867	0·3194
H(202)	−0·0066	0·4116	0·4088
H(203)	−0·0538	0·3555	0·4134
H(211)	0·0194	0·3792	0·1884
H(212)	0·0989	0·3524	0·1379
H(221)	−0·0255	0·3075	0·1049
H(222)	−0·0408	0·2970	0·2105
H(223)	0·0388	0·2702	0·1600

Table 3. *Compound* I ($C_{36}H_{40}Cl_2CoP$): *interatomic distances* (Å) *and bond angles* (°)

Co(1)—P(1)	2·289 (2)	C(12)—C(13)	1·384 (9)
Co(1)—Cl(1)	2·263 (2)	C(13)—C(14)	1·401 (9)
Co(1)—Cl(2)	2·288 (2)	C(14)—C(15)	1·349 (11)
		C(15)—C(16)	1·389 (11)
Co(1)—C(1)	2·099 (6)	C(16)—C(17)	1·382 (9)
Co(1)—C(2)	2·132 (6)	C(17)—C(12)	1·399 (9)
Co(1)—C(3)	2·137 (6)	C(18)—C(19)	1·407 (9)
Co(1)—C(4)	2·094 (6)	C(19)—C(20)	1·385 (10)
Co(1)—C(5)	2·128 (6)	C(20)—C(21)	1·376 (11)
		C(21)—C(22)	1·371 (11)
C(1)—C(2)	1·439 (9)	C(22)—C(23)	1·388 (10)
C(2)—C(3)	1·413 (9)	C(23)—C(18)	1·382 (9)
C(3)—C(4)	1·431 (9)	C(24)—C(25)	1·384 (9)
C(4)—C(5)	1·437 (9)	C(25)—C(26)	1·403 (9)
C(5)—C(1)	1·428 (9)	C(26)—C(27)	1·374 (10)
C(1)—C(10)	1·477 (9)	C(27)—C(28)	1·362 (11)
C(2)—C(9)	1·483 (10)	C(28)—C(29)	1·387 (10)
C(3)—C(8)	1·485 (9)	C(29)—C(24)	1·388 (9)
C(4)—C(7)	1·488 (9)	C(30)—C(31)	1·346 (13)
C(5)—C(6)	1·481 (9)	C(31)—C(32)	1·371 (13)
C(10)—C(11)	1·534 (9)	C(32)—C(33)	1·327 (13)
		C(33)—C(34)	1·377 (12)
P(1)—C(12)	1·831 (6)	C(34)—C(35)	1·375 (12)
P(1)—C(18)	1·827 (6)	C(35)—C(30)	1·371 (11)
P(1)—C(24)	1·847 (6)	C(35)—C(36)	1·482 (13)
P(1)—Co(1)—Cl(1)	87·08 (7)	P(1)—C(12)—C(13)	122·7 (5)
P(1)—Co(1)—Cl(2)	94·43 (7)	P(1)—C(12)—C(17)	117·4 (5)
Cl(1)—Co(1)—Cl(2)	94·25 (7)	C(17)—C(12)—C(13)	119·7 (6)
		C(12)—C(13)—C(14)	119·7 (6)
Co(1)—P(1)—C(12)	110·8 (2)	C(13)—C(14)—C(15)	120·7 (6)
Co(1)—P(1)—C(18)	117·8 (2)	C(14)—C(15)—C(16)	120·0 (6)
Co(1)—P(1)—C(24)	118·0 (2)	C(15)—C(16)—C(17)	120·8 (6)
C(12)—P(1)—C(18)	105·6 (3)	C(16)—C(17)—C(12)	119·2 (6)
C(12)—P(1)—C(24)	102·5 (3)	P(1)—C(18)—C(19)	121·7 (5)
C(18)—P(1)—C(24)	100·4 (3)	P(1)—C(18)—C(23)	120·3 (5)
		C(23)—C(18)—C(19)	117·7 (6)
C(5)—C(1)—C(2)	108·9 (6)	C(18)—C(19)—C(20)	120·6 (6)
C(1)—C(2)—C(3)	107·1 (6)	C(19)—C(20)—C(21)	120·2 (6)
C(2)—C(3)—C(4)	109·0 (6)	C(20)—C(21)—C(22)	120·1 (6)
C(3)—C(4)—C(5)	107·8 (6)	C(21)—C(22)—C(23)	120·0 (6)
C(4)—C(5)—C(1)	107·0 (6)	C(22)—C(23)—C(18)	121·4 (6)
C(5)—C(1)—C(10)	125·7 (7)	P(1)—C(24)—C(25)	120·8 (5)
C(2)—C(1)—C(10)	124·8 (6)	P(1)—C(24)—C(29)	119·4 (5)
C(1)—C(2)—C(9)	125·7 (6)	C(29)—C(24)—C(25)	119·5 (6)
C(3)—C(2)—C(9)	127·1 (6)	C(24)—C(25)—C(26)	119·3 (6)
C(2)—C(3)—C(8)	126·6 (7)	C(25)—C(26)—C(27)	120·4 (6)
C(4)—C(3)—C(8)	124·3 (6)	C(26)—C(27)—C(28)	120·0 (6)
C(3)—C(4)—C(7)	124·2 (6)	C(27)—C(28)—C(29)	120·6 (6)
C(5)—C(4)—C(7)	127·4 (7)	C(28)—C(29)—C(24)	120·0 (6)
C(4)—C(5)—C(6)	126·8 (6)	C(35)—C(30)—C(31)	121·1 (7)
C(1)—C(5)—C(6)	125·7 (6)	C(30)—C(31)—C(32)	120·1 (7)
C(1)—C(10)—C(11)	111·4 (6)	C(31)—C(32)—C(33)	119·6 (8)
C(34)—C(35)—C(30)	118·5 (8)	C(32)—C(33)—C(34)	121·5 (7)
C(30)—C(35)—C(36)	121·2 (9)	C(33)—C(34)—C(35)	119·1 (7)
C(34)—C(35)—C(36)	120·2 (9)		

For compound II the asymmetric unit contains one discrete dimeric cation and one anion, both in general positions. The shortest interionic distances (not involving H atoms) are C(18)···Cl(6) 3·517 Å, C(20)···Cl(5) 3·560 Å, and C(13)···Cl(6) 3·637 Å. The tetrachloroferrate anion is an almost regular tetrahedron. The bond lengths, in the range 2·171 (5)–2·185 (5) Å, and bond angles, in the range 107·9 (2)–111·3 (2)°, compare well with values for this anion found in other structures (Sheldrick & Stelzer, 1977; Constant, Daran & Jeannin, 1973).

If the η^5-tetramethylethylcyclopentadienyl ring is considered to occupy three coordination sites, the bonding to cobalt in both compounds may be described as six coordination.

Table 4. *Compound* II ($C_{22}H_{34}Cl_7Co_2Fe$): *interatomic distances* (Å) *and bond angles* (°)

Co(1)–Co(2)	2·887 (2)		
Co(1)–Cl(1)	2·251 (5)	Co(2)–Cl(1)	2·255 (5)
Co(1)–Cl(2)	2·277 (5)	Co(2)–Cl(2)	2·285 (5)
Co(1)–Cl(3)	2·297 (5)	Co(2)–Cl(3)	2·276 (5)
Co(1)–C(1)	2·02 (2)	Co(2)–C(12)	2·03 (2)
Co(1)–C(2)	2·04 (1)	Co(2)–C(13)	2·01 (2)
Co(1)–C(3)	2·06 (1)	Co(2)–C(14)	2·08 (1)
Co(1)–C(4)	2·07 (1)	Co(2)–C(15)	2·06 (1)
Co(1)–C(5)	2·06 (2)	Co(2)–C(16)	2·06 (2)
C(1)–C(2)	1·45 (2)	C(12)–C(13)	1·42 (2)
C(2)–C(3)	1·37 (2)	C(13)–C(14)	1·41 (2)
C(3)–C(4)	1·47 (2)	C(14)–C(15)	1·44 (2)
C(4)–C(5)	1·41 (2)	C(15)–C(16)	1·39 (2)
C(5)–C(1)	1·46 (2)	C(16)–C(12)	1·38 (2)
C(1)–C(6)	1·54 (2)	C(12)–C(17)	1·51 (2)
C(2)–C(7)	1·48 (2)	C(13)–C(18)	1·56 (2)
C(3)–C(8)	1·50 (2)	C(14)–C(19)	1·49 (2)
C(4)–C(9)	1·51 (2)	C(15)–C(20)	1·53 (2)
C(5)–C(10)	1·48 (2)	C(16)–C(21)	1·49 (2)
C(10)–C(11)	1·44 (3)	C(21)–C(22)	1·49 (3)
Fe(1)–Cl(4)	2·185 (5)	Fe(1)–Cl(6)	2·181 (5)
Fe(1)–Cl(5)	2·171 (5)	Fe(1)–Cl(7)	2·181 (6)
Cl(1)–Co(1)–Cl(2)	84·7 (2)	Cl(1)–Co(2)–Cl(2)	84·4 (3)
Cl(1)–Co(1)–Cl(3)	84·4 (3)	Cl(1)–Co(2)–Cl(3)	84·7 (2)
Cl(2)–Co(1)–Cl(3)	82·7 (3)	Cl(2)–Co(2)–Cl(3)	83·0 (3)
Co(1)–Cl(1)–Co(2)	79·7 (2)	Co(1)–Cl(2)–Co(2)	78·5 (2)
Co(1)–Cl(3)–Co(2)	78·3 (2)		
C(5)–C(1)–C(2)	107 (1)	C(16)–C(12)–C(13)	108 (1)
C(1)–C(2)–C(3)	109 (1)	C(12)–C(13)–C(14)	109 (1)
C(2)–C(3)–C(4)	109 (1)	C(13)–C(14)–C(15)	105 (1)
C(3)–C(4)–C(5)	108 (1)	C(14)–C(15)–C(16)	109 (1)
C(4)–C(5)–C(1)	107 (1)	C(15)–C(16)–C(12)	109 (1)
C(2)–C(1)–C(6)	128 (2)	C(13)–C(12)–C(17)	127 (2)
C(5)–C(1)–C(6)	125 (2)	C(16)–C(12)–C(17)	125 (2)
C(3)–C(2)–C(7)	126 (1)	C(14)–C(13)–C(18)	127 (2)
C(1)–C(2)–C(7)	125 (1)	C(12)–C(13)–C(18)	123 (2)
C(4)–C(3)–C(8)	125 (1)	C(15)–C(14)–C(19)	128 (2)
C(2)–C(3)–C(8)	127 (1)	C(13)–C(14)–C(19)	127 (2)
C(5)–C(4)–C(9)	127 (1)	C(16)–C(15)–C(20)	128 (2)
C(3)–C(4)–C(9)	125 (1)	C(14)–C(15)–C(20)	122 (2)
C(1)–C(5)–C(10)	125 (2)	C(12)–C(16)–C(21)	126 (1)
C(4)–C(5)–C(10)	128 (2)	C(15)–C(16)–C(21)	126 (1)
C(5)–C(10)–C(11)	108 (2)	C(16)–C(21)–C(22)	111 (2)
Cl(4)–Fe(1)–Cl(5)	107·9 (2)	Cl(5)–Fe(1)–Cl(6)	110·7 (2)
Cl(4)–Fe(1)–Cl(6)	108·3 (2)	Cl(5)–Fe(1)–Cl(7)	111·3 (2)
Cl(4)–Fe(1)–Cl(7)	109·9 (2)	Cl(6)–Fe(1)–Cl(7)	108·7 (3)

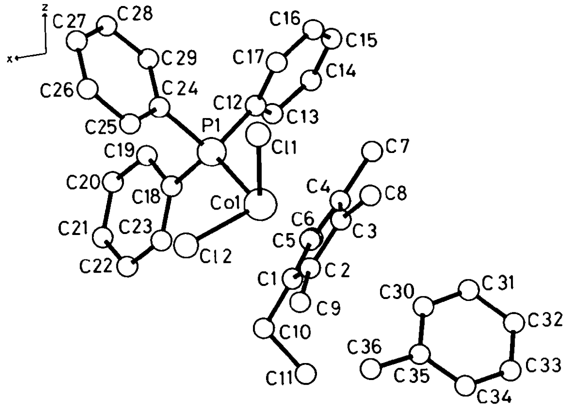

Fig. 1. The molecular structure of dichloro(triphenylphosphine)-(η^5-tetramethylethylcyclopentadienyl)cobalt toluene solvate projected down *b*.

Table 5. *Equations of least-squares best planes and, in square brackets, distances* (Å) *of atoms from the planes*

The equations are referred to the crystal axes and *x*,*y*,*z* are fractional coordinates.

Compound I

Plane (1): ring C(1) to C(5)

$$6\cdot299x + 2\cdot902y + 11\cdot287z = 2\cdot565$$

[C(1) 0·012, C(2) 0·002, C(3) –0·016, C(4) 0·023, C(5) –0·022, C(6) –0·252, C(7) –0·088, C(8) –0·133, C(9) –0·075, C(10) –0·142, Co(1) 1·734]

Normal to Co(1) meets plane 0·02 Å from centroid.

Plane (2): C(6) C(7) C(8) C(9) C(10)

$$-6\cdot340x - 2\cdot492y - 11\cdot271z = -2\cdot422$$

[C(6) 0·051, C(7) –0·062, C(8) 0·049, C(9) –0·017, C(10) –0·022, Co(1) –1·872]

Plane (3): ring C(12) to C(17)

$$-5\cdot392x + 6\cdot063y - 12\cdot481z = -6\cdot205$$

[C(12) 0·007, C(13) –0·011, C(14) 0·005, C(15) 0·004, C(16) 0·003, C(17) –0·008]

Plane (4): ring C(18) to C(23)

$$6\cdot345x + 10\cdot431y + 4\cdot397z = 3\cdot526$$

[C(18) 0·002, C(19) 0·002, C(20) –0·009, C(21) 0·012, C(22) –0·008, C(23) 0·001]

Plane (5): ring C(24) to C(29)

$$-6\cdot140x - 10\cdot670y + 9\cdot714z = -2\cdot835$$

[C(24) –0·004, C(25) 0·001, C(26) –0·002, C(27) 0·007, C(28) –0·010, C(29) 0·009]

Plane (6): ring C(30) to C(36)

$$3\cdot547x - 15\cdot043y - 8\cdot353z = -5\cdot023$$

[C(30) 0·003, C(31) –0·001, C(32) –0·008, C(33) 0·001, C(34) 0·002, C(35) 0·015, C(36) –0·012]

Compound II

Plane (7): ring C(1) to C(5)

$$7\cdot270x + 22\cdot965y + 0\cdot447z = 14\cdot924$$

[C(1) –0·005, C(2) 0·006, C(3) –0·005, C(4) 0·002, C(5) 0·002, C(6) 0·034, C(7) 0·076, C(8) –0·043, C(9) –0·009, C(10) 0·090, Co(1) –1·649]

Normal to Co(1) meets plane 0·04 Å from centroid.

Plane (8): C(6) C(7) C(8) C(9) C(10)

$$-7\cdot532x - 22\cdot746y - 0\cdot379z = -14\cdot891$$

[C(6) 0·046, C(7) –0·044, C(8) 0·025, C(9) 0·005, C(10) –0·032, Co(1) 1·679]

Plane (9): ring C(12) to C(16)

$$-6\cdot665x - 23\cdot449y + 0\cdot276z = -8\cdot566$$

[C(12) 0·008, C(13) –0·013, C(14) 0·013, C(15) –0·008, C(16) 0·000, C(17) 0·152, C(18) 0·040, C(19) 0·059, C(20) –0·090, C(21) 0·094, Co(2) –1·659]

Normal to Co(2) meets plane 0·04 Å from centroid.

Plane (10): C(17) C(18) C(19) C(20) C(21)

$$7\cdot032x + 23\cdot150y - 0\cdot574z = 8\cdot363$$

[C(17) –0·015, C(18) 0·053, C(19) –0·071, C(20) 0·062, C(21) –0·029, Co(2) 1·710]

For I, the monodentate ligands are disposed about the Co atom with a very regular geometry [P(1)–Co(1)–Cl(1) 87·08 (7), P(1)–Co(1)–Cl(2) 94·43 (7), Cl(1)—Co(1)—Cl(2) 94·25 (7)°]. They also have pseudo threefold symmetry relative to the η^5-tetramethylethylcyclopentadienyl ligand, where the angles are Cp—Co(1)—P(1) 129·6, Cp—Co(1)—Cl(1) 121·9, Cp—Co(1)—Cl(2) 120·2° (Cp is the centroid of the five-membered ring). The Co(1)—P(1) distance of 2·289 (2) Å is not much shorter than that calculated for a single bond length from the sum of the covalent radii (2·32 Å) (Brückner & Randaccio, 1974). The two Co—Cl bond distances of 2·263 (2) and 2·288 (2) Å differ by over ten e.s.d.'s, although they are both in the range [2·247 (3)–2·286 (2) Å] observed for other Co^{III}—Cl bond distances in six-coordinate complexes: 2·247 (3) and 2·263 (3) Å in $\{Co[NH_2(CH_2)_3NH(CH_2)_2NH(CH_2)_3NH_2]Cl_2\}^+$ (Payne, 1973), 2·286 (2) Å in $Co(NH_3)_5Cl^{2+}$ (Messmer & Amma, 1968), and 2·27 (1) Å in $[Co(1,9\text{-diamino-}3,7\text{-dithianonane})NO_2Cl]^+$ (Murray-Rust & Murray-Rust, 1973). Departure from ideal tetrahedral geometry is marked about the phosphorus: Co—P—C 110·8 (2)–118·0 (2)°, C—P—C 100·4 (3)–105·6 (3)°. In systems where there is a significant contraction of the metal—phosphorus bond, such effects have been attributed to an increase in the *p*-orbital character in the P—C bonds (Mason & Towl, 1970). In this molecule, however, steric interactions are a more likely cause of the observed distortions, although there are no remarkable non-bonded intramolecular contacts, with Cl(1)···C(3) = 3·044, C(4)···C(12) = 3·581 Å. The P—C bond lengths do not differ significantly, and the mean [1·835 (11) Å] is not appreciably greater than that [1·828 (3) Å] for free triphenylphosphine (Daly, 1964). Each of the three phenyl rings is planar within experimental error (Table 5), the mean C—C distance is 1·384 (14) Å, and the mean C—C—C angle is 120 (1)°, similar to values [1·400 (2) Å, 120·0 (1)°] found for the free ligand.

For II the mean Co—Cl distance [2·274 (18) Å] compares well with the values found for I. However, both this distance and the Co—Co distance of 2·887 (2) Å are shorter than the corresponding distances for other chloro-bridged cobalt species: 2·316 (4), 2·334 (4) and 3·151 (1) Å respectively for $(NH_3)_4Co(\mu\text{-Cl})(\mu\text{-NH}_2)Co(NH_3)_4^{4+}$ (Barro, Marsh & Schaefer, 1970), and 2·417 (1) and 3·470 (1) Å respectively for $[Co_2Cl_2(C_2H_6O_2)_4]^{2+}$ (Antti, 1975). The mean Cl—Co—Cl angle of 84·0 (9)° indicates that there is considerable deviation from octahedral bonding about the Co atoms, and, although the Co—Co distance is relatively short, it is believed that there is no direct bonding interaction. The angles about Co [mean 84·0 (9)°] and the Co—Cl—Co bridging angles [mean 78·8 (8)°] compare well with those found for the related $(NH_3)_3Co(OH)_3Co(NH_3)_3^{3+}$ cation [80·6 (3) and 83·3 (3)° respectively] (Mandel, Mandel, Marsh & Schaefer, 1977), where the Co—Co distance is 2·565 (1) Å.

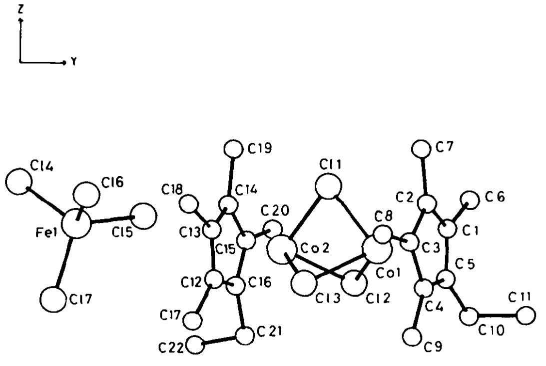

Fig. 2. The molecular structure of tri-μ-chloro-bis(η^5-tetramethylethylcyclopentadienyl)dicobalt tetrachloroferrate projected down *a*.

For I, the Co—C(ring) distances are in the range 2·094 (6)–2·137 (6) Å, and those for II are in the range 2·01 (2)–2·08 (1) Å, indicating that the bulky η^5-tetramethylethylcyclopentadienyl ring only deviates slightly from a symmetric position with respect to the Co atom. In unsubstituted η^5-cyclopentadienyl cobalt complexes Co—C(ring) distances found are 2·03 (1) Å in $[Co(\eta^5\text{-}C_5H_5)(C_4F_6)_2PO(OH)]$ (Barrow, Freer, Harrison, Sim, Taylor & Wilson, 1975), 2·05 (3) Å in $[(\eta^5\text{-}C_5H_5)(1\text{-benzoylcyclopentadienyl})Co]$ (Churchill, 1965) and 2·05 (2) Å in $[(C_6H_5)_2PCo(\eta^5\text{-}C_5H_5)]_2$ (Coleman & Dahl, 1967). The slightly longer values for I could be the result of steric crowding. Similar small deviations from a symmetric position with respect to the metal atom are observed in structures containing η^5-pentamethylcyclopentadienyl rings; for example, Rh—C 2·196 (6)–2·237 (6) Å in $Rh(Me_5C_5)(C_6H_5CH{=}CHCOCH{=}CHC_6H_5)$ (Ibers, 1974); Fe—C 2·090 (8)–2·137 (8) Å in $[(Me_5C_5)Fe(CO)_2SO_2(CH_2CH{=}CHC_6H_5)]$ (Churchill & Wormald, 1971); Rh—C 2·109 (6)–2·178 (6) Å in $[(Me_5C_5)RhCl]_2HCl$ (Churchill & Ni, 1973); and Cr—C 2·197 (6)–2·236 (6) Å in $[(Me_5C_5)(CO)_2Cr]_2$ (Potenza, Giordano, Mastropaolo & Efraty, 1974).

In both I and II the C_5 ring atoms are coplanar [planes (1), (7) and (9) in Table 5], as are the $(CH_3)_4(CH_2)$ groups [planes (2), (8) and (10)]. The perpendicular distances from the Co atom to the ring planes are 1·734 Å in I, and 1·649 and 1·659 Å in II. In I all the ring substituents are bent away from the Co atom. This, coupled with the longer perpendicular distance of the ring plane from the Co atom, indicates substantial steric crowding in the molecule (Churchill & Ni, 1973). For II, with shorter perpendicular distances, not all the substituents in both rings are bent back from the cyclopentadienyl ring planes, possibly due to a combination of steric and electronic factors (Rees & Coppens, 1973). The cyclopentadienyl ring planes for II are very nearly parallel and are inclined at an angle of 3·7° to each other, and 2·0 and 2·1° respectively to the plane formed by the three bridging Cl atoms.

The dimensions of the η^5-tetramethylethylcyclopentadienyl rings are consistent with η^5-pentamethylcyclopentadienyl–metal structures. The ring C—C distances are in the ranges 1·413 (9)–1·439 (9) Å and 1·37 (2)–1·47 (2) Å, the C—CH_3(or CH_2) distances are in the ranges 1·477 (9)–1·488 (9) Å and 1·48 (2)–1·56 (2) Å, while the ring angles vary from 107·0 (6)–109·0 (6)° and 105 (1)–109 (1)° and the CH_3(or CH_2)—C—C angles vary from 124·2 (6)–127·4 (7)° and 122 (2)–128 (2)°, with no systematic variation, for compounds I and II respectively.

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