Table 9. *Intermolecular distances less than* 3·5 Å

Hydrogen atoms are not included. Symmetry operations are listed in Table 8.

OXOFMB		FMB	
$C(2)\cdots N(3^{ii})$	3·317 (7) Å	$C(2)\cdots O(6^{ii})$	3·421 (5) Å
$C(2)\cdots O(1'^{ii})$	3·383 (6)	$C(5)\cdots C(2^{vi})$	3·243 (7)
$O(2)\cdots N(3^{ii})$	3·413 (6)	$C(5)\cdots N(3^{vi})$	3·446 (6)
$O(2)\cdots C(4^{ii})$	3·288 (7)	$C(6)\cdots N(2^{vi})$	3·248 (6)
$O(2)\cdots N(8^{vi})$	3·486 (5)	$O(6)\cdots C(2^{iii})$	3·035 (7)
$O(2)\cdots C(9^{ii})$	3·323 (6)	$O(6)\cdots N(3^{vi})$	3·476 (6)
$O(2)\cdots O(1'^{ii})$	3·108 (5)	$N(7)\cdots N(1^{vi})$	3·488 (6)
$O(1')\cdots O(2'^{vii})$	3·481 (5)	$N(7)\cdots C(2^{vi})$	3·363 (6)
$C(2')\cdots O(3'^{iv})$	3·363 (6)	$N(7)\cdots O(2'^{iii})$	3·088 (5)
$O(2')\cdots O(6^{v})$	3·378 (5)	$N(8)\cdots O(2'^{iii})$	3·083 (6)
$C(4')\cdots O(2'^{vii})$	3·201 (6)	$N(8)\cdots O(3'^{iii})$	3·007 (5)
$C(5')\cdots O(2'^{vii})$	3·254 (7)	$O(1')\cdots N(1^{vii})$	3·067 (5)
$O(5')\cdots N(1^{ii})$	3·265 (6)	$O(1')\cdots C(2^{vii})$	3·288 (6)
$O(5')\cdots C(6^{ii})$	3·250 (7)	$O(1')\cdots C(5^{vii})$	3·367 (6)
		$O(1')\cdots C(6^{vii})$	3·104 (6)
		$O(1')\cdots O(3'^{iii})$	3·425 (6)
		$C(2')\cdots O(5'^{v})$	3·250 (6)
		$C(4')\cdots O(2'^{vii})$	3·449 (5)
		$C(5')\cdots O(2'^{vii})$	3·408 (6)

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The Crystal and Molecular Structure of (Pentahaptofluorenyl)(trihaptofluorenyl)dichlorozirconium(IV)

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(Pentahaptofluorenyl)(trihaptofluorenyl)dichlorozirconium(IV), fl_2ZrCl_2, (fl = fluorenyl) is monoclinic, space group $P2_1/n$, with $a = 12·347$, $b = 13·771$, $c = 11·922$ Å, $\beta = 98·2°$, $Z = 4$. The structure was solved by the heavy-atom technique and refined by least-squares methods with 2697 independent reflexions to give $R = 0·053$. The normally tetrahedral coordination of two h^5-bonded ligands and two Cl atoms is distorted to the extent that the bonding of one fluorenyl group is reduced to an h^3 system, that the fl–Zr–fl angle is opened out to 125·0°, and that the Cl–Zr–Cl angle is closed down to 93·6°.

Introduction

The reaction of $ZrCl_4$ and sodium fluorenide in diethoxyethane gave orange prisms on sublimation of the evaporated reaction mixture. The complex is insoluble in most organic solvents and shows reactivity towards others (*e.g.* THF). It is an efficient catalyst (in combination with aluminum alkyls) for olefin polymerization (Wailes & Weigold, 1974). These unusual properties prompted an X-ray investigation to obtain information about its geometry and type of bonding. A preliminary report of its preparation and structure has been published (Kowala, Wailes, Weigold & Wunderlich, 1974).

Experimental

The space group and preliminary cell dimensions were determined from Weissenberg photographs. Accurate cell parameters were based on high-order reflexions,

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scanned with a diffractometer, in which Cu $K\alpha_1$ and Cu $K\alpha_2$ peaks were resolved.

Crystal data

$C_{26}H_{18}Cl_2Zr$, M.W. 492·56, monoclinic, a = 12·347, b = 13·771, c = 11·922 Å, β = 98·2°, U = 2006·2 Å^3, D_c = 1·63 g cm^{-3}, μ(Cu $K\alpha$) = 87·13 cm^{-1}, F(000) = 992, space group $P2_1/n$.

The crystal, 0·3 × 0·1 × 0·1 mm, was coated with Araldite and mounted with **b** parallel to the φ axis of the diffractometer. Intensities for 2697 independent reflexions were measured (up to 2θ = 130°) with Ni-filtered Cu $K\alpha$ radiation on a Canberra Industries automated four-circle Siemens diffractometer. The θ–2θ scan technique was employed with the 200, 020, and 002 reflexions as reference.

The positions of Zr and the two Cl atoms were determined from a Patterson function. The resultant electron density map revealed the locations of all C atoms. Several cycles of refinement by least-squares procedures of positional and at first isotropic, and in the final two cycles anisotropic, thermal parameters, reduced R to 0·063. A difference synthesis revealed the positions of the H atoms. A further cycle of refinement including all H atoms gave a final value of R = 0·053.

The scattering factors were those of Cromer & Waber (1965). Copies of observed and calculated structure factors are available from the authors on request.* The final positional and thermal parameters are in Table 1.

Bond lengths and angles are in Tables 2 and 3 respectively with the following e.s.d.'s: Zr–Cl = 0·002 Å, Zr–C = 0·01 Å, C–C = 0·015 Å, C–H = 0·1 Å, Cl–Zr–Cl = 0·1° and C–C–C = 1·0°.

Discussion

As can be seen from Fig. 1 and Table 2, the Zr–C distances in the two fluorenyl groups are different.

The average Zr–C distance in the five-membered ring of one fluorenyl group is 2·56 (7) Å which is comparable to values published for a number of (π-C_5H_5)$_2$ZrX$_2$ (X = halogen) complexes (Bush & Sim, 1971; Saldarragia-Molina, Clearfield & Bernal, 1974). The Zr–C bonding for this fluorenyl group can be described as 22,23,24,25,26-pentahapto (1-5-η).

In the other fluorenyl group, the Zr–C(9), Zr–C(10) and Zr–C(13) lengths are 2·40, 2·59 and 2·56 Å respectively (mean 2·52 (8) Å). The Zr–C(11) and Zr–C(12) distances are much longer (mean 2·80 (1) Å).

Table 1. *Final atomic fractional coordinates and thermal parameters with their e.s.d.'s in parentheses*

The anisotropic temperature factors are of the form $T = \exp[-(\beta_{11}h^2 + \beta_{22}k^2 + \beta_{33}l^2 + 2\beta_{12}hk + 2\beta_{13}hl + 2\beta_{23}kl)]$. Values are × 10^4.

	x	y	z	β_{11}	β_{22}	β_{33}	β_{12}	β_{13}	β_{23}
Zr	2572 (1)	350 (0)	2554 (1)	23 (2)	17 (2)	30 (2)	1 (0)	4 (0)	1 (0)
Cl(1)	3920 (0)	655 (0)	1830 (1)	37 (2)	31 (2)	58 (3)	4 (1)	11 (1)	−11 (0)
Cl(2)	2530 (0)	−653 (0)	4219 (0)	43 (3)	32 (2)	37 (3)	−6 (1)	3 (1)	6 (1)
C(1)	2130 (6)	1952 (0)	4695 (0)	32 (6)	34 (6)	66 (8)	2 (4)	21 (5)	−13 (5)
C(2)	2471 (7)	1670 (7)	5775 (8)	50 (7)	44 (6)	63 (8)	−4 (5)	30 (6)	−3 (5)
C(3)	3512 (5)	1262 (2)	6109 (8)	50 (7)	40 (6)	48 (7)	−12 (5)	12 (5)	−12 (5)
C(4)	4241 (7)	1150 (2)	5340 (5)	44 (6)	35 (5)	45 (6)	−11 (5)	−14 (5)	−10 (5)
C(5)	5540 (7)	1151 (1)	3065 (8)	33 (6)	30 (6)	81 (9)	−3 (4)	22 (6)	2 (5)
C(6)	5857 (8)	1245 (1)	2004 (1)	47 (7)	45 (7)	98 (10)	−10 (5)	24 (7)	8 (6)
C(7)	5141 (17)	1615 (3)	1115 (9)	89 (9)	41 (6)	82 (10)	−19 (6)	55 (7)	6 (6)
C(8)	4087 (7)	1944 (1)	1199 (8)	69 (8)	24 (6)	72 (9)	−14 (5)	41 (6)	2 (5)
C(9)	2744 (5)	2081 (7)	2637 (3)	24 (6)	16 (4)	75 (8)	3 (4)	−8 (5)	−3 (5)
C(10)	2855 (1)	1847 (3)	3876 (1)	46 (6)	17 (5)	30 (6)	−3 (4)	7 (4)	2 (4)
C(11)	3898 (4)	1433 (0)	4202 (1)	35 (6)	20 (5)	42 (7)	−3 (4)	8 (5)	−9 (4)
C(12)	4470 (4)	1453 (1)	3206 (2)	46 (6)	25 (5)	38 (6)	−11 (4)	14 (5)	−1 (4)
C(13)	3735 (1)	1848 (1)	2282 (1)	43 (6)	17 (5)	29 (6)	−10 (4)	12 (4)	−3 (4)
C(14)	1990 (7)	−190 (7)	−343 (7)	51 (7)	60 (7)	27 (6)	10 (5)	4 (5)	−18 (5)
C(15)	2111 (1)	569 (2)	−1030 (4)	71 (8)	54 (7)	35 (7)	10 (6)	15 (5)	5 (5)
C(16)	1751 (8)	1512 (8)	−828 (1)	59 (8)	69 (8)	35 (7)	−12 (6)	8 (6)	20 (6)
C(17)	1260 (5)	1718 (7)	104 (1)	46 (7)	31 (5)	71 (9)	11 (5)	−10 (6)	13 (5)
C(18)	−53 (5)	1623 (7)	2395 (8)	44 (7)	42 (6)	71 (8)	22 (5)	8 (6)	−12 (6)
C(19)	−609 (2)	1341 (8)	3237 (5)	44 (7)	62 (8)	74 (9)	5 (6)	11 (6)	−4 (7)
C(20)	−599 (7)	371 (8)	3582 (8)	44 (7)	98 (9)	54 (6)	−9 (7)	26 (5)	8 (8)
C(21)	−44 (7)	−350 (8)	3137 (2)	34 (5)	71 (7)	44 (7)	−19 (6)	3 (5)	7 (6)
C(22)	1170 (7)	−637 (6)	1524 (7)	46 (6)	22 (5)	38 (7)	−5 (4)	4 (5)	0 (4)
C(23)	1435 (6)	−17 (6)	637 (7)	30 (5)	30 (5)	34 (6)	3 (4)	−4 (4)	−2 (4)
C(24)	1097 (7)	951 (6)	881 (7)	49 (6)	27 (6)	30 (6)	3 (4)	5 (5)	4 (4)
C(25)	536 (6)	905 (7)	1859 (6)	15 (5)	43 (6)	27 (6)	4 (4)	−2 (4)	−4 (4)
C(26)	533 (7)	−80 (6)	2211 (7)	41 (5)	36 (6)	32 (6)	1 (4)	−5 (4)	0 (4)

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* These tables have also been deposited with the British Library Lending Division as Supplementary Publication No. SUP 31303 (15 pp., 1 microfiche). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 13 White Friars, Chester CH1 1NZ, England.

Table 1 (*cont.*)

	x	y	z	B
H(1)	1464 (83)	2284 (80)	4577 (88)	31600
H(2)	1994 (84)	1859 (76)	6329 (84)	35100
H(3)	3801 (79)	1269 (74)	7191 (85)	31900
H(4)	4886 (77)	1073 (70)	5694 (78)	28400
H(5)	6100 (81)	851 (77)	3457 (85)	33700
H(6)	6631 (87)	1141 (79)	1868 (93)	43000
H(7)	5291 (92)	1698 (80)	579 (96)	47100
H(8)	3648 (88)	2199 (85)	349 (95)	39100
H(9)	1919 (73)	2216 (72)	2507 (83)	26300
H(14)	2271 (76)	−908 (77)	−631 (81)	29800
H(15)	2567 (77)	466 (79)	−1736 (81)	34700
H(16)	1737 (82)	2110 (80)	−1523 (89)	35100
H(17)	1060 (81)	2348 (77)	224 (81)	32000
H(18)	−184 (79)	2190 (81)	2384 (82)	33400
H(19)	−1303 (85)	2067 (79)	3488 (88)	40300
H(20)	−1022 (84)	176 (82)	3899 (89)	39500
H(21)	−88 (78)	1158 (74)	3500 (84)	31000
H(22)	1458 (76)	1295 (70)	1531 (78)	25000

The Zr–C bonding for this ring is therefore 10,9,13-trihapto (π-allylic or 1-3-η). Although there are known examples of mono-, di- and pentahapto bonding modes in cyclopentadienyl–metal complexes (Calderon, Cotton, De Boer & Takats, 1971; Lucas, Green, Forder & Prout, 1973), the present complex is the first with trihapto bonding in a planar π-bonded system and therefore requires reassessment of the comment by Cotton (1969) that trihapto bonding in such systems is 'mythical'.

The two Zr–Cl distances (Table 2) have a mean of 2·420 (4) Å which is similar to the 2·44 (1) in $(\pi\text{-}C_5H_5)_2$-$ZrCl_2$ (Prout, Cameron, Forder, Critchley, Denton & Rees, 1974), 2·44 in $(CH_2)_3(C_5H_4)_2$ $ZrCl_2$ (Saldarragia-Molina *et al.*, 1974) and 2·43 Å in $(\pi\text{-}C_5H_5)_2$ $ZrCl\text{-}Si(C_6H_5)_3$ (Muir, 1971).

The Cl(1)–Zr–Cl(2) angle of 93·6° (Table 3) is somewhat less than the 97·1 for $(\pi\text{-}C_5H_5)_2ZrCl_2$ (Prout *et al.*, 1974) and 93·9° for $(CH_2)_3(C_5H_4)_2HfCl_2$ (Saldarragia-Molina *et al.*, 1974). However, it is of the right order to fit well into the recently proposed unified M.O. bonding model of bis-π-cyclopentadienyl metal complexes with d^0 electron configuration (Green, Green & Prout, 1972; Prout *et al.*, 1974).

The C–C bonds (Table 2) range from 1·35 to 1·50 Å (mean 1·41 (3) Å) and are consistent with those of similar aromatic systems (Sutton, 1965).

Because of the poor least-squares refinement of the positional parameters of the H atoms, the C–H bond

Table 2. *Interatomic distances* (Å) (*numbering of* H *atoms same as attached* C *atoms*)

Zr–Cl(1)	2·416	Zr–Cl(2)	2·425
Zr–C(9)	2·395	Zr–C(22)	2·397
Zr–C(10)	2·589	Zr–C(23)	2·555
Zr–C(11)	2·801	Zr–C(24)	2·635
Zr–C(12)	2·807	Zr–C(25)	2·645
Zr–C(13)	2·561	Zr–C(26)	2·562
C(1)—C(2)	1·35	C(14)–C(15)	1·35
C(2)—C(3)	1·41	C(15)–C(16)	1·41
C(3)—C(4)	1·38	C(16)–C(17)	1·37
C(4)—C(11)	1·42	C(17)–C(24)	1·44
C(11)–C(10)	1·41	C(24)–C(23)	1·44
C(10)–C(1)	1·42	C(23)–C(14)	1·46
C(11)–C(12)	1·47	C(24)–C(25)	1·44
C(12)–C(13)	1·43	C(25)–C(26)	1·42
C(13)–C(9)	1·39	C(26)–C(22)	1·44
C(9)—C(10)	1·50	C(22)–C(23)	1·43
C(12)–C(5)	1·42	C(25)–C(18)	1·43
C(5)—C(6)	1·38	C(18)–C(19)	1·35
C(6)—C(7)	1·38	C(19)–C(20)	1·40
C(7)—C(8)	1·40	C(20)–C(21)	1·36
C(8)—C(13)	1·43	C(21)–C(26)	1·45
C(1)—H(1)	0·93	C(14)–H(14)	1·12
C(2)—H(2)	0·98	C(15)–H(15)	1·09
C(3)—H(3)	1·29	C(16)–H(16)	1·17
C(4)—H(4)	0·85	C(17)–H(17)	0·92
C(5)—H(5)	0·88	C(18)–H(18)	0·80
C(6)—H(6)	1·00	C(19)–H(19)	1·38
C(7)—H(7)	0·70	C(20)–H(20)	0·74
C(8)—H(8)	1·13	C(21)–H(21)	1·20
C(9)—H(9)	1·03	C(22)–H(22)	0·97

Table 3. *Bond angles* (°)

Cl(1) -Zr——Cl(2)	93·6	C(14)–C(15)–C(16)	123·2
C(1)—C(2)—C(3)	122·2	C(15)–C(16)–C(17)	121·2
C(2)—C(3)—C(4)	120·8	C(17)–C(24)–C(23)	118·6
C(4)—C(11)–C(10)	120·7	C(24)–C(23)–C(14)	119·8
C(11)–C(10)–C(1)	119·4	C(23)–C(14)–C(15)	117·7
C(10)–C(1)—C(2)	118·8	C(16)–C(17)–C(24)	119·4
C(3)—C(4)—C(11)	118·1	C(23)–C(22)–C(26)	107·7
C(10)–C(9)—C(13)	106·9	C(22)–C(26)–C(25)	108·5
C(9)—C(13)–C(12)	110·0	C(24)–C(25)–C(26)	107·6
C(11)–C(12)–C(13)	107·7	C(23)–C(24)–C(25)	108·0
C(10)–C(11)–C(12)	107·2	C(22)–C(23)–C(24)	107·5
C(9)—C(10)–C(11)	108·0	C(20)–C(21)–C(26)	116·3
C(7)—C(8)—C(13)	116·3	C(19)–C(20)–C(21)	124·6
C(6)—C(7)—C(8)	124·6	C(18)–C(19)–C(20)	120·6
C(5)—C(6)—C(7)	120·2	C(19)–C(18)–C(25)	118·5
C(6)—C(5)—C(12)	118·2	C(18)–C(25)–C(26)	120·4
C(5)—C(12)–C(13)	121·3	C(21)–C(26)–C(25)	119·5
C(8)—C(13)–C(12)	119·3		

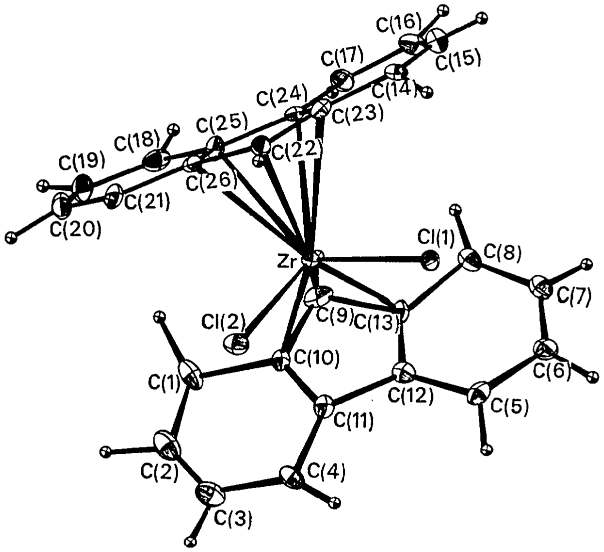

Fig. 1. An *ORTEP* plot (Johnson, 1965) of fl_2ZrCl_2 looking down **b**. The numbering scheme is the same as that used in the tables.

lengths vary appreciably about a mean value of 1·01 Å in the two fluorenyl rings (*cf.* Table 2).

The least-squares plane equations for the two fluorenyl ligands are

$$4{\cdot}012x+12{\cdot}778y+2{\cdot}204z+4{\cdot}370=0 \qquad (h^3\text{-system})$$

and

$$9{\cdot}692x+2{\cdot}815y+5{\cdot}784z+1{\cdot}728=0 \qquad (h^5\text{-system})$$

where the constants are in Å and refer to the monoclinic axes.

The maximum deviation of atoms from each plane is 0·039 and 0·140 Å respectively and the standard deviations, $[\sum_i^n (d-d_i)^2/(n-3)]^{1/2}$, are 0·023 and 0·096 Å respectively, thus showing that the two fluorenyl ligands are essentially planar.

The angle formed by the normals to the least-squares planes intersecting at the Zr atom is 125° which is somewhat smaller than values reported for other cyclopentadienylzirconium complexes (Wailes, Coutts & Weigold, 1974). However, it is significant that both Zr–fl normals are of length 2·33 Å, that is, about 0·1 Å longer than found in the above complexes. This finding adds substance to the suggestion by Prout *et al.* (1974) that the size of the angle between the normals bears an inverse relationship to the length of the normals.

The dihedral angle is approximately bisected by the Cl(1)–Zr–Cl(2) plane.

To conclude, it is seen that the type of Zr–C bonding and the general geometry of the molecule is determined by the steric requirements imposed by the bulky fluorenyl groups. Not only are the fl ligands staggered, but the angle between the normals from the Zr atom to the ligand planes is opened out by about 17° from the normal tetrahedral angle. More importantly still, one fl ligand is displaced laterally so that only three of the five central C atoms are at normal π-bond distances from the metal atom, implying a change from h^5 to h^3(π-allylic) bonding.

Despite these extensive readjustments, this molecule must still be under some internal strain as evidenced by the ten Cl···C intramolecular contacts listed in Table 4 which are all less than the sum of van der Waals radii of 3·52 Å (Bondi, 1964).

Because of the very low solubility of the complex in most organic solvents, it was anticipated that a number of short intermolecular contacts would exist. However only that between H(6) at (x,y,z) and H(14) at $(1-x, -y, -z)$, 2·17 Å, is less than the sum of van der Waals radii (2·40 Å) but little significance should be attached to this because of the low accuracy of the H atom coordinates. Therefore no plausible explanation of the low solubility can be advanced at this stage.

Table 4. *Most relevant intramolecular contacts* (Å)

Cl(1)···C(5)	3·393	Cl(2)···C(4)	3·406
Cl(1)···C(12)	3·355	Cl(2)···C(10)	3·498
Cl(1)···C(14)	3·321	Cl(2)···C(11)	3·334
Cl(1)···C(22)	3·363	Cl(2)···C(21)	3·282
Cl(1)···C(23)	3·311	Cl(2)···C(26)	3·278

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