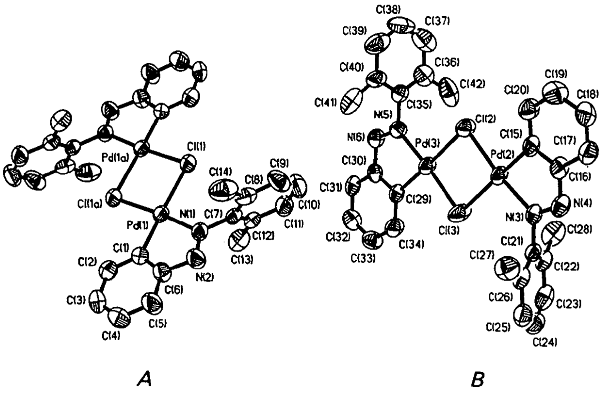

Fig. 1. *ORTEP* (Johnson, 1965) view of the complex $C_{28}H_{26}Cl_2N_4Pd_2$ with molecules *A* and *B*. H atoms are omitted for clarity.

inversion centre. The length of the Pd—Cl bond *trans* to C, and the Pd—Cl bond *trans* to N are in agreement with the stronger *trans* effect exerted by a σ-bonded phenyl ring (Barr, Dyke, Smith, Kennard & McKee, 1985) and within the range expected.

The few X-ray structural analyses carried out on similar cyclopalladated compounds show that in the solid state the Pd_2Cl_2 fragment can be either planar (Barr, Dyke, Smith, Kennard & McKee, 1985) or bent, with a dihedral angle between $PdCl_2$ planes of 58° (Constable, McDonald, Sawkins & Shaw, 1980) or 39° (Ghedini, Armentano, De Munno, Crispini & Neve, 1990). In the present case, the Pd_2Cl_2 fragment is strictly planar in molecule *A* and nearly planar in molecule *B*.

Some distortions can be observed in one molecule with respect to the other. In fact, with respect to the Pd_2Cl_2 plane the five-membered metallocycles form a dihedral angle of 3·1 (2)° in *A* and of 7·3 (2), 0·7 (2)° in *B*. The planes of the dimethyl-substituted phenyl groups form, with the metallocycle planes, dihedral angles of 72·9 (3), 72·9 (3), 75·3 (2) and 100·1 (3)°. Therefore the methyl groups are on opposite sides of the Pd_2Cl_2 planes. The shortest Pd—C distances, namely Pd(1)—C(13), Pd(1*a*)—C(13*a*), Pd(2)—C(28) and Pd(3)—C(42), are 3·52 (1), 3·52 (1), 3·66 (1) and 3·76 (1) Å, respectively. Therefore the methyl groups, through an H atom, might be thought as interacting with the metal in a pseudo-axial position.

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Structure of Tris(η^5-cyclopentadienyl)yttrium(III)*

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Abstract. $[Y(C_5H_5)_3]$, $M_r = 284{\cdot}2$, orthorhombic, $P2_1cn$ (non-standard setting of $Pna2_1$), $a = 8{\cdot}631$ (3), $b = 13{\cdot}910$ (4), $c = 19{\cdot}662$ (4) Å, $V = 2360$ (1) Å^3, $Z = 8$, $D_x = 1{\cdot}599$ g cm^{-3}, λ(Mo $K\alpha$) = 0·709261 Å, $\mu = 48{\cdot}3$ cm^{-1}, $F(000) = 1152$, $T = 295$ K, $R = 0{\cdot}059$ for 1558 reflections $[I > 1{\cdot}5\sigma(I)]$. Each Y atom is η^5-bonded to three cyclopentadienyl rings, individual $[Y(C_5H_5)_3]$ molecules being loosely aligned to form zigzag chains as in the lanthanoid homologues $[Ln(C_5H_5)_3]$ with Ln = Er and Tm. The structure of

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* Coordination Behaviour in Base-Free Tris(cyclopentadienyl) Complexes of Rare Earth Elements. VII. Part VI: Paolucci, Fischer, Breitbach, Pelli & Traldi (1988).

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[$Y(C_5H_5)_3$] differs, however, markedly from those of the congeners containing the other genuine Group 3 rare-earth elements Sc and La.

Introduction. Considerable interest has been focused on the structures of base-free rare-earth tris(cyclopentadienyl) complexes, Cp_3M^{III} ($Cp = C_5H_5$, C_5H_4R or $C_5H_3R_2$; R = alkyl group; M = Sc, Y, La and Ce–Lu), a unique class of homoleptic MX_3 compounds which displays an unusual wealth of structural variations in particular for $Cp = C_5H_5$ (Hammel, Schwarz & Weidlein, 1989; Stults, Andersen & Zalkin, 1990). While the nature of the central metal atom contributes primarily to this interesting tuning of the crystal and molecular structures, more recent findings indicate that the structure may vary with the specific technique of crystal preparation (Rebizant, Apostolidis, Spirlet & Kanellakopulos, 1988; Eggers, Kopf & Fischer, 1987). The completely unexpected isomorphism of [$Lu(C_5H_5)_3$] and [$Sc(C_5H_5)_3$] has raised the question whether their polymeric [$(\eta^5\text{-}C_5H_5)_2M(\mu\text{-}\eta^1{:}\eta^1\text{-}C_5H_5)]_\infty$ arrangement might be particularly attractive for early non-*f* transition metals (Eggers, Schultze, Kopf & Fischer, 1986). While the large ionic radius (Shannon, 1976) of La^{3+} (1·216 Å) may be responsible for the so far unique structure of [$(\eta^5\text{-}C_5H_5)_2La(\mu\text{-}\eta^5{:}\eta^2\text{-}C_5H_5)]_\infty$ (Eggers, Kopf & Fischer, 1986), the ionic radius of Y (1·075 Å) is intermediate between the radii of Lu (1·032 Å) and Eu (1·120 Å) making predictions for [$Y(C_5H_5)_3$] quite difficult. We report here a single-crystal X-ray diffraction study of [$Y(C_5H_5)_3$].

Experimental. Tris(cyclopentadienyl)yttrium was prepared under N_2 atmosphere from anhydrous YCl_3 and NaC_5H_5 in tetrahydrofuran (THF) (Birmingham & Wilkinson, 1956); the crude product resulting after solvent removal *in vacuo* (*ca* 400 K) was sublimed slowly at 400 K and *ca* 10^{-2} Pa. From the colourless crystalline sublimate one specimen of 0·25 × 0·25 × 0·4 mm was selected for X-ray analysis and sealed under argon in a Lindemann capillary.

Intensity data recorded on a Syntex $P2_1$ automatic diffractometer, graphite-monochromated Mo $K\alpha$ radiation; $\theta/2\theta$ scan mode. Cell parameters refined by least squares from angle data of 18 selected reflections, $12 \leq 2\theta \leq 32°$. Space group determined from systematic absences (refinement in centrosymmetric space group *Pnam* was unsuccessful). 2422 reflections measured in range $4·5 \leq 2\theta \leq 58·0°$ (*hkl* range: 0→10, 0→16, 0→21), three standard reflections (218, 242, 244) displaying insignificant deviation from average; 2247 unique reflections, 1558 of which [with $I \geq 1·5\sigma(I)$] were used for refinement. Numeric absorption correction with program *SHELX*76 (Sheldrick, 1976), transmission factors 0·4699 (min.) and 0·5829 (max.). Structure solved by direct methods using program *SHELXS*84 (Sheldrick, 1984) and Fourier techniques (*SHELX*76), scattering factors for all atoms except Y included in the *SHELX* program; for Y from *International Tables for X-ray Crystallography* (1974, Vol. IV). Anisotropic thermal parameters for non-H atoms, common temperature factor for H atoms $U = 0·0745$ Å^2. Refinement of 235 parameters keeping the C_5H_5 rings as rigid groups (with C—C distance 1·40, C—H distance 0·96 Å). $R = 0·059$, $wR = 0·051$ for $w = [\sigma^2(F) + 0·0004(F)^2]^{-1}$. Final difference maps showed no peaks higher than 0·8 or less than $-0·6$ e Å^{-3}, with next-following peaks very close to the Y center. All Δ/σ values < 0·26. When the two final cycles of refinement were repeated with the enantiomorphic data set in which the signs of all coordinates were reversed, the final R values increased by *ca* 0·2%.

Table 1. *Atomic coordinates and temperature factors, U_{eq}, with e.s.d.'s*

$$U_{eq} = (U_{11} + U_{22} + U_{33})/3.$$

	x	y	z	U_{eq}(Å^2)
Y1	0·0000	0·2793 (1)	0·2405 (1)	0·032 (1)
Y2	0·3666 (2)	0·2472 (1)	0·4873 (1)	0·032 (1)
C111	−0·1071 (17)	0·1216 (9)	0·3033 (5)	0·05 (2)
C112	−0·2380 (17)	0·1704 (9)	0·2793 (5)	0·05 (2)
C113	−0·2351 (17)	0·1676 (9)	0·2081 (5)	0·07 (3)
C114	−0·1024 (17)	0·1171 (9)	0·1881 (5)	0·08 (3)
C115	−0·0233 (17)	0·0886 (9)	0·2469 (5)	0·07 (2)
C121	0·1799 (18)	0·2426 (8)	0·1253 (5)	0·05 (2)
C122	0·2697 (18)	0·2011 (8)	0·1768 (5)	0·06 (2)
C123	0·3214 (18)	0·2748 (8)	0·2196 (5)	0·05 (2)
C124	0·2635 (18)	0·3619 (8)	0·1946 (5)	0·05 (2)
C125	0·1761 (18)	0·3420 (8)	0·1363 (5)	0·05 (2)
C131	−0·1650 (14)	0·4344 (8)	0·2019 (4)	0·04 (2)
C132	−0·0311 (14)	0·4749 (8)	0·2301 (4)	0·05 (2)
C133	−0·0290 (14)	0·4531 (8)	0·2996 (4)	0·06 (2)
C134	−0·1617 (14)	0·3991 (8)	0·3144 (4)	0·07 (3)
C135	−0·2457 (14)	0·3875 (8)	0·2540 (4)	0·06 (2)
C211	0·0816 (16)	0·2802 (7)	0·4326 (6)	0·06 (2)
C212	0·1819 (16)	0·2731 (7)	0·3769 (6)	0·04 (2)
C213	0·2333 (16)	0·1778 (7)	0·3727 (6)	0·04 (2)
C214	0·1649 (16)	0·1261 (7)	0·4259 (6)	0·05 (2)
C215	0·0711 (16)	0·1894 (7)	0·4629 (6)	0·05 (2)
C221	0·5144 (18)	0·3975 (7)	0·5431 (4)	0·05 (2)
C222	0·5891 (18)	0·3830 (7)	0·4807 (4)	0·07 (3)
C223	0·4855 (18)	0·4080 (7)	0·4289 (4)	0·05 (2)
C224	0·3469 (18)	0·4379 (7)	0·4592 (4)	0·05 (2)
C225	0·3648 (18)	0·4315 (7)	0·5298 (4)	0·05 (2)
C231	0·5309 (15)	0·1278 (9)	0·5705 (4)	0·05 (2)
C232	0·4505 (15)	0·0701 (9)	0·5240 (4)	0·06 (2)
C233	0·5178 (15)	0·0830 (9)	0·4598 (4)	0·06 (2)
C234	0·6398 (15)	0·1486 (9)	0·4667 (4)	0·06 (2)
C235	0·6479 (15)	0·1763 (9)	0·5351 (4)	0·06 (2)

Discussion. Atomic parameters are listed in Table 1,* selected interatomic distances and angles in Table 2. An *ORTEP*II (Johnson, 1976) drawing of [$Y(C_5H_5)_3$] is given in Fig. 1. Unlike [$Sc(C_5H_5)_3$] and [$La(C_5H_5)_3$], just three η^5-coordinated C_5H_5 ligands

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* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 53707 (11 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 2. *Selected distances* (Å) *and angles* (°); *Cpnm is the center of the mth cyclopentadienyl ligand of the nth* Cp_3Y *molecule* (*see also Fig.* 1)

Y1—C111	2·682 (12)	Y2—C211	2·723 (14)
Y1—C112	2·664 (14)	Y2—C212	2·717 (13)
Y1—C113	2·634 (14)	Y2—C213	2·707 (12)
Y1—C114	2·633 (13)	Y2—C214	2·706 (12)
Y1—C115	2·663 (13)	Y2—C215	2·717 (14)
Y1—C121	2·794 (12)	Y2—C221	2·684 (11)
Y1—C122	2·859 (14)	Y2—C222	2·697 (13)
Y1—C123	2·805 (15)	Y2—C223	2·715 (11)
Y1—C124	2·704 (14)	Y2—C224	2·715 (10)
Y1—C125	2·696 (12)	Y2—C225	2·697 (10)
Y1—C131	2·694 (11)	Y2—C231	2·729 (11)
Y1—C132	2·741 (11)	Y2—C232	2·667 (12)
Y1—C133	2·693 (11)	Y2—C233	2·685 (13)
Y1—C134	2·614 (11)	Y2—C234	2·758 (13)
Y1—C135	2·614 (12)	Y2—C235	2·784 (13)
Y1—Cp11	2·371 (6)	Y2—Cp21	2·439 (6)
Y1—Cp12	2·503 (6)	Y2—Cp22	2·425 (5)
Y1—Cp13	2·392 (5)	Y2—Cp23	2·450 (6)
Y1—C212	3·108 (13)	Y2—C121	3·159 (12)
Y1—C213	3·578 (12)	Y2—C125	3·581 (12)
Cp11—Y1—Cp12	118·0 (4)	Cp21—Y2—Cp22	117·9 (3)
Cp11—Y1—Cp13	120·8 (4)	Cp21—Y2—Cp23	118·8 (4)
Cp12—Y1—Cp13	116·4 (4)	Cp22—Y2—Cp23	116·7 (4)

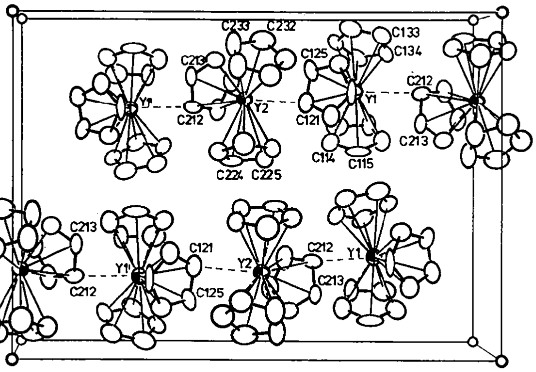

Fig. 1. *ORTEP*II drawing of $[(C_5H_5)_3Y^{III}]$. Dashed line: trace of closest-packed zigzag chain in the lattice.

Table 3. *Some properties of the polymeric nature and molecular packing of* $[M(C_5H_5)_3]$ *complexes investigated so far*

The two lines refer to two nonequivalent molecules.

M	$r(M^{III})$ (Å)*	Space group	Δ (Å)†	Δ (%)‡	$M\cdots M'$ (Å)	$10^3 \times D_x/M_r$ (moles cm^{-3})
La^a	1·216	$P2_1/c$	0·127/0·484	4·5/17·0	5·814	5·338
			0·154/0·559	5·4/19·6	5·814	
La^b	1·216	$P2_1$	0·228^m	8·13§	5·849	5·257
Pr^c	1·179	$P2_1$	0·126/0·316	4·5/11·2	5·760	5·467
Y^d	1·075	$Pna2_1$	0·409	15·2	5·810	5·626
			0·446	16·4	5·910	
Er^e	1·062	$Pna2_1$	0·444	16·5	5·806	5·621
			0·520	19·5	5·934	
Tm^e	1·052	$Pna2_1$	0·568	21·2	5·891	5·615
			0·670	25·2	6·040	
Yb^f	1·042	$P2_12_12_1$	1·500	56·8	6·739	5·503
Lu^g	1·032	$Pbc2_1$	0·060/0·449	2·3/17·3	6·163	5·672
Sc^h	0·926¶	$Pbc2_1$	0·137/0·528	5·5/21·2	6·245	5·869

References: (*a*) Rebizant *et al.* (1988) (non-sublimed sample); (*b*) Eggers, Kopf & Fischer (1986); (*c*) Hinrichs, Melzer, Rehwoldt, Jahn & Fischer (1983); (*d*) present study; (*e*) Eggers, Hinrichs, Kopf, Jahn & Fischer (1986); (*f*) Eggers *et al.* (1987) (non-sublimed sample); (*g*) Eggers, Schultze, Kopf & Fischer (1986); (*h*) Atwood & Smith (1973).

* From Shannon (1976) for coordination number 9.

† Difference between shortest intermolecular $M\cdots C'$ distance(s) [two different ones only for M = La, Pr, Sc and Lu; see §] and average intramolecular M—C distance.

‡ Referred to average of all intramolecular M—C distances of the sample in question.

§ Average for two almost equally short La···C′ distances.

¶ Extrapolated value.

surround each Y^{III} ion in crystalline $Y(C_5H_5)_3$. The average Y—C distance of 2·706 Å compares well with that of $[Y(\eta^5\text{-}C_5H_5)_3.THF]$ (2·707 Å; Rogers, Atwood, Emad, Sikora & Rausch, 1981). The crystal structure of $[Y(C_5H_5)_3]$ also differs from that of $[Yb(C_5H_5)_3]$ (Eggers *et al.*, 1987) but compares well (Table 3) with those of $[Tm(C_5H_5)_3]$ and $[Er(C_5H_5)_3]$ (Eggers, Hinrichs, Kopf, Jahn & Fischer, 1986). The shortest intermolecular C···Y′ distance within a $[Y(C_5H_5)_3]_n$ chain exceeds the average intramolecular C—Y bond distance by 15–16% (Table 3); this percentage is intermediate between the corresponding data for $[Lu(C_5H_5)_3]$ and $[La(C_5H_5)_3]$ on the one hand and $[Tm(C_5H_5)_3]$ and $[Yb(C_5H_5)_3]$ on the other (see Table 3). The isomorphism of $[Y(C_5H_5)_3]$ with two of its *lanthanoid* congeners (*i.e.* Ln = Er and Tm) appears to be dictated by the very similar ionic radii of Y^{III}, Er^{III} and Tm^{III} and not by any elusive property of the non-*f* element yttrium. Yet powder diffraction studies of $[Ho(C_5H_5)_3]$ (radius 1·072 Å) and a few complexes of somewhat larger *f*-element ions (Haug, 1971; Laubereau & Burns, 1970) suggest successful competition of at least one further modification for Ln = Ho to Pr (possible space groups *Pbcm* and $Pbc2_1$). Interestingly, the reciprocal molar density, D_x/M_r (Table 3), of all $[M(C_5H_5)_3]$ structures turns out to lie within the narrow range $5{\cdot}55 \pm 0{\cdot}30 \times 10^{-3}$ mol cm^{-3}, exceeding the average D_x/M_r value of the related $[M(CH_3C_5H_4)_3]$ systems (Stults *et al.*, 1990; Hammel *et al.*, 1989) by more than 20%. Optimal molecular packing appears thus to be reached for Cp = C_5H_5 equally well by a variety of zigzag chains whose energies may, nevertheless, differ only by minute amounts.

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Structure of *cis*-Dichloro[methylphosphinylidenebis(methylamine-*N*,*N*′)]platinum(II) Hemihydrate

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Abstract. $[PtCl_2(C_3H_{11}N_2OP)].\frac{1}{2}H_2O$, $M_r = 397{\cdot}11$, monoclinic, $C2/c$, $a = 18{\cdot}177$ (3), $b = 7{\cdot}175$ (4), $c = 17{\cdot}155$ (1) Å, $\beta = 119{\cdot}97$ (1)°, $V = 1938$ (1) Å^3, $Z = 8$, $D_x = 2{\cdot}722$ g cm^{-3}, λ(Mo $K\alpha$) = 0·71073 Å, $\mu = 76{\cdot}525$ cm^{-1}, $F(000) = 1464$, $T = 292$ K, $R = 0{\cdot}019$ for 1514 observed reflections with $I > 3\sigma(I)$. Molecules of the complex placed in general positions are linked *via* hydrogen bonds to water molecules on twofold axes. The Pt atom is planar coordinated by two *cis* Cl atoms [Pt—Cl 2·311 (2), 2·300 (2) Å, Cl—Pt—Cl 91·53 (7)°] and two amino N atoms from the chelating ligand [Pt—N 2·048 (6), 2·038 (6) Å, N—Pt—N 93·9 (2)°]. The conformation of the six-membered chelate ring is a slightly distorted chair; Pt and P atoms deviate from the least-squares plane of the N_2C_2 fragment by −0·915 and 0·922 Å respectively. The phosphonyl O atom takes an axial position with respect to the ring.

Introduction. Since the establishment of the antitumor activity of *cis*-$[PtCl_2(NH_3)_2]$ by Rosenberg, van Camp, Trosko & Mansour (1969), various analogues of the parent compound have been synthesized and tested in order to find alternative drugs with lower toxicity and better solubility (Hacker, Douple & Krakoff, 1984). In this respect, special attention has been drawn to the platinum complexes of functionalized amines (Hydes & Russell, 1988). Recently Dodoff, Varbanov, Borisov & Spassovska (1990) reported the preparation, characterization and *in vivo* antitumor activity of a series of Pt^{II}, Pt^{IV} and Pd^{II} complexes of amine-containing phosphine oxide ligands, $(CH_2)_2P(O)CH_2NH_2$ and $CH_3P(O)(CH_2NH_2)_2$ (L). One of the compounds previously formulated as $[PtCl_2L].H_2O$ on the basis of elemental analysis and spectral data has been re-examined in the present X-ray structure analysis, which shows that the correct formula is $[PtCl_2L].\frac{1}{2}H_2O$.

Experimental. The title complex was prepared as described by Dodoff *et al.* (1990). Yellow crystals, suitable for X-ray analysis, were grown from a saturated aqueous solution. An irregularly shaped crystal with approximate dimensions 0·15 × 0·15 × 0·1 mm was examined. D_m was not determined. Intensity data were collected with an Enraf–Nonius CAD-4 diffractometer (graphite monochromator, Mo $K\alpha$ radiation); ω–2θ scan, scan speed 1–7° min^{-1}, $\Delta\omega = (0{\cdot}8 + 0{\cdot}4\tan\theta)$°. Cell constants were determined

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