Journal of Organometallic Chemistry, 235 (1982) 151—159
Elsevier Sequoia S.A., Lausanne — Printed in The Netherlands

THE CRYSTAL AND MOLECULAR STRUCTURE OF THE COMPLEX OF YTTRIUM BISCYCLOPENTADIENYL CHLORIDE WITH ALUMINIUM HYDRIDE MONOETHERATE $[(\eta^5\text{-}C_5H_5)_2YCl]_2AlH_3 \cdot OEt_2$

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(Received March 30th, 1982)

Summary

The structure of the bimetallic complex of composition $(Cp_2YCl)_2AlH_3 \cdot Et_2O$ has been determined by X-ray diffraction. The complex forms rhombic crystals, $a = 13.521(5)$, $b = 12.877(5)$, $c = 30.73(1)$ Å, *Pcab*, $Z = 8$, $d = 1.52$ g/cm^3. The structure was refined to an R factor of 4.8%. The molecule is a polymeric chain made up of dimeric $Cp_2Y(\mu\text{-}Cl)_2YCp_2$ fragments and aluminium hydride monoetherate units linked with each other through Y—H—Al bridges, r(X—H) = 2.35 Å, r(Al—H) = 1.5 Å, and secondary Al···Cl bonds, r = 3.007 Å. The aluminium coordination polyhedron is a trigonal bipyramid.

Introduction

The coordination chemistry of lanthanoid biscyclopentadienyl compounds and their bimetallic derivatives has not been studied thoroughly. At present, the compounds of composition $(Cp_2MX)_2$ and $Cp_2MR_2AlR_2$, where M stands for Sc, Y, Gd, Dy, Ho, Er, Tm, Yb, X is Cl or R, and R is Me or Et [1—3], have been isolated. Bimetallic complexes involving alkyl bridges have been shown to be effective catalysts of ethylene polymerization [4]. In view of this observation, the synthesis and study of bimetallic hydride complexes as possible catalysts for hydrogenation and isomerization of olefins (cf. titanium and

0022-328X/82/0000—0000/$02.75

aluminium hydride complexes [5]) seems of interest. We have chosen yttrium as the transition metal that forms one of the most stable ternary hydrides with aluminium, $Y(AlH_4)_3$ [6]. The reaction of Cp_2MCl with aluminium hydride which yields stable aluminium hydride complexes even with titanium [7], has been used as synthetic procedure.

Results and discussion

The asymmetrical part of the unit cell (Fig. 1) contains a molecule of complex I comprising two distinct fragments, aluminium hydride monoetherate, and dimeric bis-η^5-cyclopentadienylyttrium chloride. Each of the two moieties may in principle exist as independent molecules. However, at present neither of them has been studied as individual species by direct methods.

The IR spectral data suggest [8] that aluminium is four-coordinate in ether solutions of AlH_3 and that the structure of aluminium hydride monoetherate resembles those of tetrahedral trialkylaminealanes, AlH_3NR_3 [9], having C_{3v} skeleton symmetry. Because of the attack of the electronegative hydrogen atom at the electron-deficient aluminium atom, attempts at the isolation of $AlH_3 \cdot Et_2O$ from its solutions lead to a partial dissociation of the Al—O bond, which is the weakest of all Al—O bonds known for complexes of this type ($\Delta H(\mathrm{Al} \leftarrow \mathrm{O}) \sim$ 50—70 kJ/mol [10]). As a result, an insoluble polymer of the composition $AlH_3 \cdot 0.3 Et_2O$, probably involving Al—H—Al bridges [11], precipitates. The aluminium coordination number may also increase to 5, e.g. in reactions with stronger and/or less bulky Lewis bases such as NMe_3, diamines, THF and dioxane [12]. Five-coordinate aluminium has a trigonal bipyramidal environment (D_{3h} symmetry).

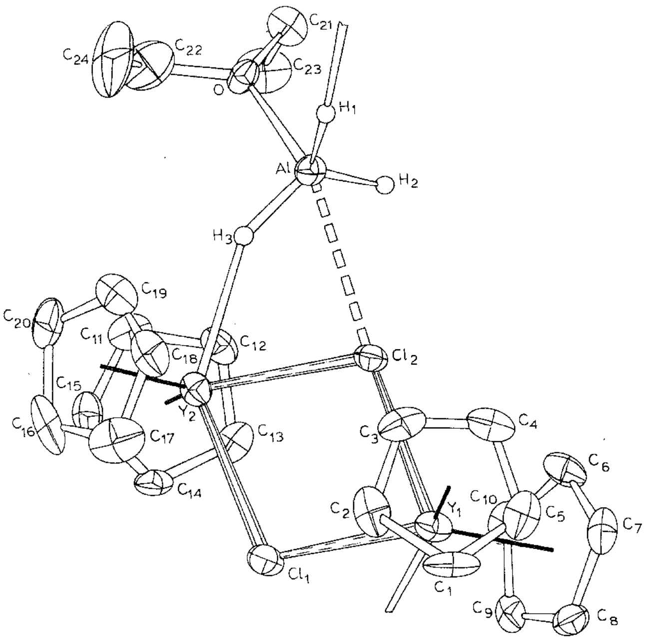

Fig. 1. The asymmetric part of the crystal structure of I.

Biscyclopentadienylmetal chlorides, Cp_2MCl, where M is a Group III metal or a lanthanoid have been prepared for scandium and yttrium and high-atomic weight lanthanoids with $r_{cov.} \leqslant 1.62$ Å [13]. Increasing $r_{cov.}$ results in the disproportionation of Cp_2MCl to Cp_3M and MCl_3 during their syntheses [13]. The yttrium derivative ($r_{cov.} = 1.62$ Å) is just the limiting case. One may suggest that coordinative unsaturation of yttrium should result in certain structural differences between derivatives of Cp_2YCl and dimeric $(Cp_2ScCl)_2$ ($r_{cov.} = 1.44$ Å). The presence of two vacant metal orbitals in Group III metal derivatives Cp_2MX [14] provides the possibility of the formation of 18-electronic complexes of the types $(Cp_2MCl \cdot L)_2$ or $Cp_2MCl \cdot 2L$, where L is a two-electron ligand. There is, however, no data on such compounds containing yttrium or even elements with smaller covalent radii, because of strict limitations imposed on the size of the ligands. Electronegative hydrogen atoms in molecular hydrides, e.g. in aluminium hydride etherate, are the least restrictive from the point of view of steric hindrances. Indeed, the molecules $AlH_3 \cdot OEt_2$ and Cp_2YCl react fairly readily to give a comparatively stable adduct I ($T_{decomp.} >$ 80° C) where the structures of the fragments change from those expected for the free individual molecules.

Structure of the $AlH_3 \cdot OEt_2$ fragment

The geometrical parameters of the ether molecule, $(C_2H_5)_2O$, remain practically unaffected by the complex formation (Tables 2 and 3) which is typical

TABLE 1

ATOMIC COORDINATES (× 10^4 for Y, Cl, Al, O, C; × 10^3 for H)

Atom	*X*	*Y*	*Z*	Atom	*X*	*Y*	*Z*
Y(1)	1196(1)	1578(1)	3243(1)	C(12)	1324(9)	5846(9)	3823(4)
Y(2)	1805(1)	4037(1)	4172(1)	C(13)	475(9)	5237(8)	3797(4)
Cl(1)	448(2)	2525(2)	3957(1)	C(14)	143(8)	5072(9)	4223(5)
Cl(2)	2410(2)	3307(2)	3388(1)	C(15)	758(11)	5597(10)	4506(4)
Al	3845(2)	5058(2)	3530(1)	C(16)	1881(23)	3558(34)	5002(7)
O	4387(5)	6335(4)	3718(2)	C(17)	1977(28)	2624(24)	4789(10)
C(1)	1750(9)	−353(9)	3370(6)	C(18)	2862(27)	2598(27)	4577(8)
C(2)	2170(12)	207(12)	3713(4)	C(19)	3323(19)	3456(34)	4666(9)
C(3)	2916(10)	843(11)	3523(7)	C(20)	2752(32)	4123(17)	4903(9)
C(4)	2891(10)	612(11)	3080(6)	C(21)	4466(8)	7211(8)	3428(4)
C(5)	2189(12)	−82(11)	2994(5)	C(22)	4515(18)	6650(12)	4173(6)
C(6)	1249(10)	2471(13)	2470(4)	C(23)	3593(11)	7870(10)	3424(5)
C(7)	998(11)	1489(13)	2386(4)	C(24)	5111(16)	6012(21)	4427(6)
C(8)	79(11)	1267(9)	2551(4)	H(1)	486(10)	447(11)	354(5)
C(9)	−293(9)	2198(12)	2748(4)	H(2)	342(10)	543(11)	311(11)
C(10)	454(11)	2923(9)	2689(4)	H(3)	321(11)	484(11)	387(5)
C(11)	1489(8)	6053(9)	4264(4)	H′(1) [a]	−14	53	354
				Al′ [a]	−1155	−58	3530

[a] Atoms related to the initial ones by the glide mirror plane *a* normal to the *Y* axis. The coordinate transformation matrix is $\begin{pmatrix} 1 & 0 & 0 \\ 0 & \bar{1} & 0 \\ 0 & 0 & 1 \\ -\frac{1}{2} & \frac{1}{2} & 0 \end{pmatrix}$.

TABLE 2

BOND LENGTHS, *d* (Å)

Bond	*d*	Bond	*d*	Bond	*d*
Y(1)—Cl(1)	2.705(5)	Y(2)—Cl(1)	2.756(5)	C(1)—C(2)	1.40(4)
Y(1)—Cl(2)	2.802(3)	Y(2)—Cl(2)	2.711(4)	C(2)—C(3)	1.43(4)
Y(1)—CpI [a]	2.366	Y(2)—CpIII [a]	2.364	C(3)—C(4)	1.39(4)
Y(1)—CpII [a]	2.364	Y(2)—CpIV [a]	2.363	C(4)—C(5)	1.33(4)
Y(1)—H′(1)	2.4(1)	Y(2)—H(3)	2.3(1)	C(5)—C(1)	1.35(3)
Y(1)—C(1)	2.63(2)	Y(2)—C(11)	2.65(2)	C(6)—C(7)	1.34(4)
Y(1)—C(2)	2.63(3)	Y(2)—C(12)	2.65(2)	C(7)—C(8)	1.37(3)
Y(1)—C(3)	2.65(2)	Y(2)—C(13)	2.64(2)	C(8)—C(9)	1.44(4)
Y(1)—C(4)	2.66(2)	Y(2)—C(14)	2.62(1)	C(9)—C(10)	1.39(3)
Y(1)—C(5)	2.64(2)	Y(2)—C(15)	2.66(2)	C(10)—C(6)	1.40(3)
Y(1)—C(6)	2.64(2)	Y(2)—C(16)	2.63(2)	C(11)—C(12)	1.40(3)
Y(1)—C(7)	2.65(2)	Y(2)—C(17)	2.64(3)	C(12)—C(13)	1.39(3)
Y(1)—C(8)	2.64(2)	Y(2)—C(18)	2.65(3)	C(13)—C(14)	1.40(3)
Y(1)—C(9)	2.65(2)	Y(2)—C(19)	2.66(3)	C(14)—C(15)	1.38(2)
Y(1)—C(10)	2.63(2)	Y(2)—C(20)	2.59(3)	C(15)—C(11)	1.37(3)
Al—O	1.890(9)	O—C(21)	1.44(2)	C(16)—C(17)	1.38(3)
Al—H(1)	1.6(1)	O—C(22)	1.47(3)	C(17)—C(18)	1.36(5)
Al—H(2)	1.5(1)	C(21)—C(23)	1.45(2)	C(18)—C(19)	1.30(5)
Al—H(3)	1.4(1)	C(22)—C(24)	1.39(3)	C(19)—C(20)	1.37(5)
Al···Cl(2)	3.007(7)			C(20)—C(16)	1.42(5)

[a] CpI is the mean square plane I (C(1)C(2)C(3)C(4)C(5)).

for compounds of this type. On the other hand, the structure of the almost planar AlH_3 group differs considerably from that observed in tributylamine-alane, $AlH_3 \cdot NBu_3$, where all the valence angles at aluminium are nearly tetra-

TABLE 3

VALENCE ANGLES, *ω* (degrees)

Angle	*ω*	Angle	*ω*	Angle	*ω*
Cl(1)—Y(1)—Cl(2)	74.4(1)	Cl(1)—Y(2)—Cl(2)	75.1(1)	C(1)—C(2)—C(3)	106(2)
c(1)—Y(1)—Cl(1) [a]	118.6	c(3)—Y(2)—Cl(1) [a]	101.3	C(2)—C(3)—C(4)	105(2)
c(1)—Y(1)—Cl(2) [a]	99.3	c(3)—Y(2)—Cl(2) [a]	113.6	C(3)—C(4)—C(5)	111(2)
c(2)—Y(1)—Cl(1) [a]	116.1	c(4)—Y(2)—Cl(1) [a]	101.1	C(4)—C(5)—C(1)	108(2)
c(2)—Y(1)—Cl(2) [a]	99.3	c(4)—Y(2)—Cl(2) [a]	115.6	C(5)—C(1)—C(2)	109(2)
c(1)—Y(1)—H′(1) [a]	93	c(3)—Y(2)—H(3) [a]	94	C(6)—C(7)—C(8)	111(2)
c(2)—Y(1)—H′(1) [a]	100	c(4)—Y(2)—H(3) [a]	98	C(7)—C(8)—C(9)	107(2)
Cl(1)—Y(1)—H′(1)	71	Cl(2)—Y(2)—H(3)	64	C(8)—C(9)—C(10)	105(2)
Y(1)—Cl(1)—Y(2)	105.3(2)	Y(1)—Cl(2)—Y(2)	103.9(1)	C(9)—C(10)—C(6)	110(2)
O—Al—H(1)	95(5)	H(1)—Al—H(2)	118(7)	C(10)—C(6)—C(7)	107(2)
O—Al—H(2)	102(6)	H(1)—Al—H(3)	116(8)	C(11)—C(12)—C(13)	107(2)
O—Al—H(3)	101(6)	H(2)—Al—H(3)	118(8)	C(12)—C(13)—C(14)	107(1)
Al—O—C(21)	121(1)	Al—O—C(22)	125(1)	C(13)—C(14)—C(15)	109(1)
O—C(21)—C(23)	114(1)	O—C(22)—C(24)	116(2)	C(14)—C(15)—C(11)	108(2)
C(21)—O—C(22)	111(1)	O—Al···Cl(2)	161.2(6)	C(15)—C(11)—C(12)	109(2)
Y(1)—H′(1)—Al′	156(9)	Y(2)—H(3)—Al	152(8)	C(16)—C(17)—C(18)	109(3)
				C(17)—C(18)—C(19)	107(3)
				C(18)—C(19)—C(20)	112(3)
				C(19)—C(20)—C(16)	105(3)
				C(20)—C(16)—C(17)	106(3)

[a] c(1) is the centre of the ring I (C(1)C(2)C(3)C(4)C(5)).

hedral [9], and resembles that found for bipyramidal $AlH_3 \cdot 2NMe_3$ [11].

The Al—O coordination bond length in I, equal to 1.890(1) Å, falls within the range of reasonable values (r(Al—O) varies from 1.87 Å in the trimer $(Me_2AlONMe_2)_3$ [15] to 2.004(6) Å in the complex $Me_3Al(\overline{ONNMeO)Al}Me_2$ [16]). The average Al—H bond length in I is 1.5(1) Å, i.e. this bond is shorter than it is in polymeric AlH_3 (1.71(1) Å) [17] and somewhat longer than in $AlH_3 \cdot NBu_3$ (1.4(1) Å) [9].

Structure of the $(Cp_2YCl)_2$ fragment

Two wedge-like sandwiches Cp_2Y are linked with each other by the $(\mu_2\text{-}Cl)_2$ bridges (Fig. 1). The near-rhombic $(YCl)_2$ heterocycle is slightly nonplanar, the dihedral angle about the Cl—Cl axis being equal to 10.5°. The obvious non-equivalence of the Y—Cl bonds (Table 2) is noteworthy: the Y(1)—Cl(1) and Y(1)—Cl(2) bond lengths differ by more than 10σ.

The Y—C bond lengths of 2.59—2.66 Å (2.64 Å on average) are typical for yttrium η-cyclopentadienyl derivatives. Thus, the Y—C bond length is equal to 2.655(18) Å in the $(Cp_2YMe)_2$ molecule. The cyclopentadienyl rings are planar (Table 4). The structure of the $(Cp_2YCl)_2$ fragment differs significantly from the structure of $(Cp_2ScCl)_2$ [1] and from those of binuclear cyclopentadienylmetal complexes of the type $Cp_2MX_2M'X'_2$ (M' = M, B, Al; X' = X, Cp) in general, in that the cyclopentadienyl rings are arranged asymmetrically with respect to the M—M' axis in the former compound. The centra of the (C_5H_5) moieties and the Y and Y' atoms do not lie in the same plane: the angle made by the c(1)Y(1)c(2) plane (c(1) and c(2) are the centra of the two rings) and the Y(1)—Y(2) direction is equal to 32.3°, and the angle between that direction and the c(3)Y(2)c(4) plane is equal to 26.1°.

Bond system between $AlH_3 \cdot OEt_2$ and $(Cp_2YCl)_2$ fragments

Because of the specific features of the yttrium fragment described above, there are free coordination sites in the yttrium environment. These sites are involved in bonding with a hydride atom of the $H_3Al \cdot OEt_2$ molecule. As a result, a single Y—H—Al bridge is formed (Fig. 1). With transition metals, structures containing single hydride bridges between different metal atoms are only known for titanium, e.g. $[Cp(C_5H_4)TiHAlEt_2]_2$ [18], $(C_{10}H_8)[(C_5H_4)TiHAlEt_2]_2$ [19], and niobium, $Cp_2Nb(CO)HZn(BH_4)_2$ [20]. Y—H—Al bridges link alternate Al and Y fragments to produce an infinite chain directed along the crystal X axis (Fig. 2). The H'(1) and H(3) atoms lie in the bisector planes of the wedge-shaped sandwiches at the Y(1) and Y(2) atoms, respectively (Table 4). The fragment sequence in the chain is as follows: —Al—H(3)—Y(2)—$(Cl)_2$—Y(1)—H'(1)—Al'— (primed atoms are generated by the glide mirror plane "a" from non-primed ones). Therefore, two of the AlH_3 hydrogen atoms participate in bridge bonding while the third one (H(2)) remains terminal. This is probably the reason for the increased stability of the coordinated aluminium hydride: it is known that the formation of bridge structures increases the stability of electron-deficient hydrides. Thus, non-solvated beryllium and aluminium hydrides where all the M—H bonds form bridges have unusually high stability [17].

The average valence angle at bridge H atoms in I, 154(8)°, differs only insig-

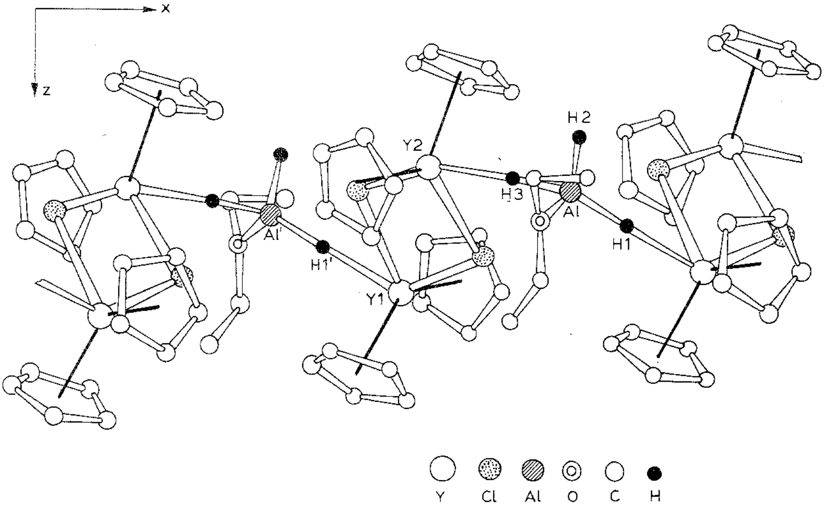

Fig. 2. A projection of a polymer chain section onto the *XZ* plane.

nificantly from the Al—H—Al angle in $(AlH_3)_x$, (141(2)°) [17], but it is considerably broadened from the M—H—M' angles in $[Cp(C_5H_4)TiHAlEt_2]_2$ (101°, [18]) and $Cp_2Nb(CO)HZn(BH_4)_2$ (107°, [20]). The Y—H bond length, 2.35 Å, coincides with the average Y—H distance in the Y—H—B bridges of the complex $Y(BH_4)_3 \cdot 3THF$ [21]. Like the Y(1)—Cl(1) and Y(1)—Cl(2) bonds, the Y—H bond lies in the bisector plane of the wedge-like sandwich. The central Y(1)—Cl(1)/Y(2)—Cl(2) bond direction almost coincides with the fictitious symmetry axis of the Cp_2Y fragment. This is in agreement with the results of the quantum chemical calculations of the Cp_2ML_3 molecules [14] and with the structure determination of Cp_2TaH_3 [22]. The nonequivalence of the chlorine bridges in the yttrium fragment is probably caused by the formation of the so-called secondary Al···Cl(2) bond shown by the dashed line in Fig. 1. In fact, although the Al···Cl distance, 3.007 Å, far exceeds an ordinary Al—Cl bond length (2.10 to 2.20 Å), it is shortened considerably from the normal Van der Waals contact, 3.8 Å. It is important that Al and Cl do interact with each other, and the short distance between them is not a mere consequence of steric effects. The Al···Cl(2) separation might easily be increased by rotating one of the fragments about the Y—H(bridge) bond (Fig. 1). The formation of the secondary Al···Cl bond thus leads firstly to additional stabilization of the complex molecule, secondly, to a change of the aluminium coordination polyhedron, which becomes a trigonal bipyraminid (this explains the planarity of the AlH_3 unit and the increase of the O—Al···Cl(2) angle to 161.2(6)°), and thirdly, to an elongation and weakening of the Y(1)—Cl(2) bond, which is a continuation of the bipyramid axis (the Al···Cl(2)—Y(1) angle is equal to 175.7°).

The yttrium atom attains an 18-electron configuration in complex I, and all its valence orbitals are involved in bonding. Both Y and Al fragments thus become electronically and coordinatively saturated. The above discussion of the structural features of I leads to the conclusion that stabilities of binuclear

TABLE 4

THE COEFFICIENTS OF THE $Ax + By + Bz + D = 0$ EQUATIONS FOR THE PLANAR FRAGMENTS AND THE DISPLACEMENT OF ATOMS FROM THOSE PLANES (Å)

Coefficient	Plane											
	CpI		CpII		CpIII		CpIV		B1 [a]		B2 [b]	
	Atom	Displ.	Atom	Displ.	Atom	Displ.	Atom	Displ.	Atom	Displ.	Atom	Displ.
	C(1)	−0.005	C(6)	−0.008	C(11)	0.007	C(16)	−0.016	Y(1)	−0.002	Y(2)	−0.026
	C(2)	0.000	C(7)	0.009	C(12)	0.000	C(17)	−0.002	Cl(1)	−0.064	Cl(1)	−0.021
	C(3)	0.005	C(8)	−0.006	C(13)	−0.008	C(18)	0.021	Cl(2)	−0.017	Cl(2)	−0.155
	C(4)	−0.008	C(10)	−0.004	C(14)	0.012	C(19)	0.032	H′(1)	0.175	H(3)	−0.173
	C(5)	0.008	C(9)	−0.004	C(15)	−0.009	C(20)	0.028	Al′	−0.236	Al	−0.424
A	9.038		5.512		−7.618		5.363		8.160		7.263	
B	−9.420		−3.536		10.505		−5.031		−7.264		−8.740	
C	4.125		26.761		−2.282		25.221		17.319		15.386	
D	3.299		6.417		4.313		11.870		5.449		4.228	

Dihedral angles: CpI—CpII, 126.18°; CpIII—CpIV, 128.12°; B1—B2, 8.41°.

[a] Bisector plane between the planes CpI and CpII. [b] Bisector plane between the planes CpIII and CpIV.

complexes of the type we are considering depends on the radius of the Group III metal involved, i.e. on the free volume of its coordination sphere, and on the basicity of the "molecular ligand", i.e. on the hydride atom negative charge which in turn depends, in particular, on the strength of the Lewis base.

Experimental

All the operations involved in the synthesis and study of the compounds prepared in this work were carried out under dry argon. Solvents (ether, benzene) were distilled over $LiAlH_4$ prior to use. The analyses for Y and Al were carried out as recommended in ref. [23]. Chlorine was determined by titration with silver nitrate, with potentiometric indication of the end point. Cp_2YCl was prepared as described in ref. 2 and purified by vacuum sublimation.

Reaction of Cp_2YCl with aluminium hydride in ether

Aluminium hydride, AlH_3, (10.14 mmol) in 60 ml Et_2O was added to biscyclopentadienylyttrium chloride (2.46 g, 9.66 mmol) suspended in 70 ml Et_2O. During the reaction Cp_2YCl dissolved almost fully. The unreacted colloidal residue was filtered off, and a transparent colourless filtrate was evaporated under vacuum to one third of its initial volume. The crystalline precipitate was isolated from the mother liquor and dried under vacuum. The product (0.5 g, 20%) was a white crystalline powder sensitive to air oxygen and moisture. According to the analytical data, the ratio of the elements in the product was Y : Al : Cl = 2 : 1 : 2.

Determination of the crystal and molecular structure

Colourless crystals of $(Cp_2YCl)_2AlH_3 \cdot OEt_2$ (I) are rhombic, $a = 13.521(5)$, $b = 12.877(5)$, $c = 30.73(1)$ Å, $V = 5350(3)$ Å^3, space group *Pcab*, $Z = 8$, $d_{calcd.} = 1.52$ g/cm^3. 1620 reflections with $I > 2\sigma$ were collected with a "Syntex P$\bar{1}$" diffractometer (λMo-K_α, graphite monochromator, $\theta/2\theta$ scan, $2\theta \leqslant 48°$, without corrections for absorption, $\mu = 47.0$ cm^{-1}).

The structure was solved by the direct method. The Y and Cl atoms were located from the Fourier synthesis. The other non-hydrogen atom positions were determined by sequential Fourier syntheses. The model was refined first in isotropic and then in anisotropic approximations to an R factor of 7.6%. The cyclopentadienyl ring hydrogen atom positions were calculated from geometrical considerations; the hydride and ether hydrogen atoms were located from the difference synthesis. The final refinement gave R = 4.8%. The coordinates of the major atoms are listed in Table 1 and the bond lengths and valence angles in Tables 2 and 3.

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