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Reduction of vanadium(II) to vanadium(0) by naphthaleneytterbium: synthesis and X-ray crystal structure of the two-dimensional multidecker complex $[(\eta^5\text{-}C_5H_5)V(\mu\text{-}\eta^6{:}\eta^2\text{-}C_{10}H_8)Yb(THF)(\eta^5\text{-}C_5H_5)]_n$*

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Abstract

The reaction of $(C_5H_5)_2V$ with an excess of $(C_{10}H_8)Yb(THF)_2$ in tetrahydrofuran at room temperature generated the polymeric two-dimensional multidecker complex $[(\eta^5\text{-}C_5H_5)V(\mu\text{-}\eta^6{:}\eta^2\text{-}C_{10}H_8)Yb(THF)(\eta^5\text{-}C_5H_5)]_n$ (**1**), which is also obtained from the reaction of $YbI_2(THF)_2$ with an equimolar mixture of KC_5H_5 and $K[(C_5H_5)V(C_{10}H_8)]$ in tetrahydrofuran. The new compound was characterized by elemental analyses, magnetic susceptibility, IR and ESR spectra. The X-ray structural analysis of **1** showed the compound to be monoclinic, space group *C2/c*, $Z=8$, with $a=19.07(5)$, $b=13.61(3)$, $c=17.40(3)$ Å, $\beta=91.9(2)°$ and $D_{calc}=1.84$ g cm^{-3}. The structure was solved from 2482 observed reflections with $F_o>4\sigma(F_o)$ and refined to a final *R* value of 0.047. The molecular structure consists of infinite zig-zag chains formed by $(\eta^5\text{-}C_5H_5)Yb$ moieties and with one $(\eta^5\text{-}C_5H_5)V(\eta^6\text{-}C_{10}H_8)$ unit coordinated η^2 via the naphthalene to each Yb atom.

Introduction

Up to now only a few complexes are known in which rare earth elements are coordinated by alkenes, alkines or aromatic hydrocarbons. The butadiene complexes of La, Nd, Sm and Er were synthesized in 1978 [2] but it was not before 1986 that the first X-ray crystal structure analyses of lanthanide complexes with $\eta^2\text{-}C_2H_4$ [3], $\eta^2\text{-}C_2Me_2$ [4], η^6-arene derivatives [5] or $\eta^3\text{-}\eta^1\text{-}C_4H_6$ [6] were reported. Recently we found that SmI_2, EuI_2 and YbI_2 react with lithium naphthalide in tetrahydrofuran to give pyrophoric products which, according to their reactivity and their ESR spectroscopic data [7], seem to be π-complexes of zerovalent lanthanoides. On the other hand, the reaction of $(C_5H_5)_2V$ with $(C_{10}H_8)Yb(THF)_2$, yielding $(C_5H_5)_2Yb$ and $(C_5H_5)V(C_{10}H_8)V(C_5H_5)$, indicates the presence of Yb(II) [8]. Thus, initiated by a metal naphthalide, a reductive transfer of a cyclopentadienyl ligand from vanadium to the more electropositive ytterbium will occur, as previously described for the formation of $(C_5H_5)V(C_{10}H_8)$ [9] and $K[(C_5H_5)V(C_{10}H_8)]$ [9]. With the use of an excess of $(C_{10}H_8)Yb(THF)_2$, we have now succeeded in the isolation of a polymeric cyclopentadienyl ytterbium complex with naphthalene bridges between each ytterbium and cyclopentadienyl vanadium unit.

Experimental

All manipulations were carried out *in vacuo* using standard Schlenk techniques. The solvents were dried and freed of oxygen by refluxing and keeping them over sodium ketyl. $(C_{10}H_8)Yb(THF)_2$† and $(C_5H_5)_2V$ were prepared by literature procedures [7, 10],

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†The number of coordinated THF molecules in $(C_{10}H_8)Yb(THF)_n$ depends on the method of its isolation and drying. In contrast to previous studies [7, 8] here we used the complex $(C_{10}H_8)Yb(THF)_2$.

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$K[(C_5H_5)V(C_{10}H_8)]$ and KC_5H_5 *in situ* from $(C_5H_5)_2V$ and $KC_{10}H_8$ [9]. The ESR spectra were obtained using a Bruker ER 200D-SCR spectrometer, the IR spectra using a Perkin-Elmer 577 infrared spectrophotometer with the samples as nujol mulls between KBr discs. The magnetic susceptibility was estimated by the Faraday method [11].

Reaction of $(C_5H_5)_2V$ with excess $(C_{10}H_8)Yb(THF)_2$

(a) A solution of $(C_5H_5)_2V$ (0.73 g, 4.03 mmol) in 15 ml of THF was added at room temperature to a suspension of $(C_{10}H_8)Yb(THF)_2$ (3.5 g, 7.86 mmol) in 15 ml of THF. During four hours stirring, the color of the solution turned from black to dark red–brown. Centrifugation, decantation of the red–brown solution and removal of the solvent from the organic layer *in vacuo* left a black solid which was purified by washing with 20 ml of hexane and recrystallized from THF. 1.07 g of **1** (48%), m.p. 180–190 °C (decomp.) could be isolated as black crystals. IR (cm^{-1}): 1360(m), 1320(m), 1095(w), 1060(w), 1020(m), 1005(m), 995(w), 940(w), 810(m), 780(s), 715(m), 660(w), 610(w), 470(m). Magnetic susceptibility: $\chi_M = 824 \times 10^{-6}$ cgsu, $\mu_{eff}^{(293)} = 1.4$ μB. ESR: 8 lines, $g_i = 1.982$, $A_i = 4.47$ mT; anisotropic: $g_{\parallel} = 2.000$ mT, $g_{\perp} = 1.973$, $A_{\parallel} \cong 0$ mT, $A_{\perp} = 7.46$ mT. *Anal.* Calc. for $C_{24}H_{26}OVYb$ (one THF was removed during the purification of the product): C, 51.99; H, 4.73. Found: C, 51.69; H, 4.87%.

(b) Neat crystalline $(C_5H_5)_2V$ (0.77 g, 4.25 mmol) and black powdery $(C_{10}H_8)Yb(THF)_2$ (3.27 g, 7.34 mmol) were mixed at room temperature. After 15 h, 20 ml of THF were added and the suspension formed was stirred for a few min. Filtration afforded a black residue and a red–brown solution. After slow removal of the THF from the filtrate, 1.36 g of black crystals of **1** (57%) remained.

Reaction of $YbI_2(THF)_2$ with an equimolar mixture of $K[(C_5H_5)V(C_{10}H_8)]$ and KC_5H_5

$YbI_2(THF)_2$ (4.1 g, 7.18 mmol) was added to an equimolar mixture of KC_5H_5 and $K[(C_5H_5)V(C_{10}H_8)]$, obtained *in situ* from $(C_5H_5)_2V$ (1.2 g, 7.18 mmol) and $KC_{10}H_8$ (2.4 g, 14.3 mmol) in 50 ml of THF. The suspension was stirred for 10 min and then centrifugated. After decantation from a white precipitate, the solvent was removed slowly from the clear red–brown solution to give 3.9 g of black crystalline **1** (97%).

X-ray data collection, structure determination and refinement of 1

Suitable crystals were selected using a special device [12], set in a Lindemann capillary and placed in the diffractometer. Determination of lattice parameters and intensity measurements were carried out on a Syntex $P2_1$ instrument, using Mo Kα radiation (graphite monochromator, $\lambda = 0.71073$ Å).

Cell dimensions were obtained from a least-squares fit of 15 reflections in the range $4.22 \leqslant 2\theta \leqslant 19.8°$. **1** crystallizes monoclinic; from the systematical absences (hkl: $h+k=2n$; $h0l$: h, $l=2n$; $0kl$: $k+l=2n$) the space group was determined to be $C2/c$ (No. 15) or Cc (No. 9). One fourth of the reflections in the range $1 \leqslant 2\theta \leqslant 45°$ was collected. Two attempts to take the usual θ–2θ scans led over a period of 4 days to a total decomposition of the crystal. Therefore Wyckoff scans were taken. Two standard reflections were measured every 100 reflections (only little changes observed). From a total of 2564 obtained reflections, 2482 with $F_o > 4\sigma(F_o)$ were used for subsequent calculations. The raw data were corrected for Lorentz and polarization effects.

The heavy atoms were found by Patterson methods and all non-hydrogen atoms were located by successive difference Fourier syntheses. The carbon atom C22 lies on a two-fold axis (Wyckoff position e) that generated the second part of the cyclopentadienyl ring. The centroid of the ring formed by C23 up to C27 (occupation factor 0.5) represents a center of symmetry (Wyckoff position a), thus causing a disorder in the cyclopentadienyl system. Therefore in the asymmetric unit there are two half cyclopentadienyl rings. The carbon atoms of the disordered ring were put at calculated positions and refined as a rigid group (d(C–C) = 1.43 Å).

TABLE 1. Crystal and data collection parameters and details for the structure refinement of $[(\eta^5\text{-}C_5H_5)V(\mu\text{-}\eta^6{:}\eta^2\text{-}C_{10}H_8)Yb(THF)(\eta^5\text{-}C_5H_5)]_n \cdot (THF)$

Formula	$C_{24}H_{26}OVYb(C_4H_8O)$
Formula weight (g mol^{-1})	626.57
Space group	$C2/c$
a (Å)	19.07(5)
b (Å)	13.61(3)
c (Å)	17.40(3)
β (°)	91.9(2)
V ($Å^3$)	4514(12)
Z	8
D_{calc} (g cm^{-3})	1.84
μ (cm^{-1})	45.6
$F(000)$	2488
Diffractometer	Siemens-Nicolet Syntex $P2_1$
Radiation, λ (Å)	Mo Kα, 0.71073
Monochromator	graphite crystal
Temperature (K)	293(5)
Scan technique	Wyckoff scans
2θ Limits (°)	$1 \leqslant 2\theta \leqslant 45$
No. unique data	2564 ($R_{int} = 0.074$)
No. observed data	2482, $F_o > 4\sigma F_o$
No. refined parameters	240
$R = \Sigma\|\|F_o\| - \|F_c\|\|/\Sigma\|F_o\|$	0.047
$R_w = [\Sigma w(\|F_o\| - \|F_c\|)^2/\Sigma\|F_o\|^2]^{1/2}$	0.063
w	$1/[\sigma^2(F_o) + 0.001390(F_o)^2]$

There was one THF molecule in the asymmetric unit located near the two-fold axis. The resulting disorder could not be refined. It was possible to resolve the structure in *Cc* but refinement led to unreasonable bond distances. Refinement in *C*2/*c* gave satisfactory bond lengths and has therefore been chosen as the correct space group. At this stage an empirical absorption correction was applied using the method of Walker and Stuart [13]; minimum absorption correction coefficient 0.872, maximum 1.178. Subsequently all non-hydrogen atoms were refined anisotropically and the hydrogen atoms were added at calculated positions (d(C–H) = 0.95 Å [14], $U_{iso} = 0.08$ Å^2). The maximum ratio of shift to error in the final refinement cycle was 0.003. The final difference Fourier map showed a maximum electron density of 2.63 e Å^{-3} close to the disordered THF molecule in the crystal lattice. Scattering factors for non-hydrogen atoms and the anomalous dispersion terms for Yb, V, C and O were taken from refs. 15 and 16 and scattering factors for H from ref. 17. All calculations were done with SHELX 76 [18]. Crystal and data collection parameters as well as details on the refinement of the structure are summarized in Table 1. The final atomic parameters are listed in Table 2, bond distances and angles in Tables 3 and 4. See also 'Supplementary material'.

Results and discussion

The reaction proceeding after the addition of an excess of $(C_{10}H_8)Yb(THF)_2$ to a solution of $(C_5H_5)_2V$ in THF was monitored by ESR spectroscopy in a THF matrix at 130 K [19]. In the course of the reaction, the intensity of the $(C_5H_5)_2V$ signal quickly decreased and disappeared completely within 30–40 min. Simultaneously a new signal appeared in the expected field range showing 8 lines with $g_i = 1.982$ and $A_i = 4.47$ mT, thus indicating paramagnetic V(0). The anisotropic spectrum of **1** has an axial symmetry of the g and $\boldsymbol{T}$ tensors ($g_{\parallel} = 2.000$, $g_{\perp} = 1.973$, $A_{\parallel} \cong 0$ mT, $A_{\perp} = 7.46$ mT) which is typical for a 17-electron d^5-complex of V(0). An identical signal was observed for the anion $[(C_5H_5)V(C_{10}H_8)]^-$ formed in the reaction of $(C_5H_5)_2V$ with $KC_{10}H_8$ [9]. ESR investigations on $(\eta^6\text{-arene})_2V$ [20, 21] and $(C_5H_5)V(\eta^7\text{-}C_7H_7)$ [22] led to the same result. The product, catena(cyclopentadienyl)(tetrahydrofuran)ytterbium(II)[μ-η^6:η^2-napthalene(cyclopentadienyl)vanadium(0)] (**1**), was isolated from the reaction mixture as black crystals, which do not change their crystal habit up to 180 °C.

$$(C_{10}H_8)Yb(THF)_2 + (C_5H_5)_2V \xrightarrow{THF}$$

$$[(\eta^5\text{-}C_5H_5)V(\mu\text{-}\eta^6\text{:}\eta^2\text{-}C_{10}H_8)Yb(THF)(\eta^5\text{-}C_5H_5)]_n$$

1

TABLE 2. Positional parameters of **1** with e.s.d.s of the last significant digit in parentheses and equivalent isotropic thermal parameters (Å^2)

Atom	*x*/*a*	*y*/*b*	*z*/*c*	B_{eq} [a]
Yb	0.56544(2)	0.46574(3)	0.87826(2)	1.94
V	0.7745(1)	0.5950(2)	0.8584(1)	2.77
O1	0.5935(4)	0.2922(5)	0.9028(4)	2.80
C1	0.8568(7)	0.706(1)	0.824(1)	4.75
C2	0.8406(7)	0.722(1)	0.902(1)	5.05
C3	0.8617(8)	0.636(2)	0.945(1)	6.11
C4	0.8897(7)	0.567(1)	0.8919(9)	4.29
C5	0.8853(6)	0.612(1)	0.8209(8)	4.08
C6	0.7242(5)	0.4906(9)	0.7787(7)	3.07
C7	0.7212(7)	0.456(1)	0.8561(9)	4.69
C8	0.6946(7)	0.521(1)	0.9146(7)	5.17
C9	0.6697(7)	0.617(1)	0.8979(8)	4.26
C10	0.6743(6)	0.649(1)	0.8204(8)	3.82
C11	0.7004(5)	0.5900(9)	0.7583(7)	2.88
C12	0.7081(7)	0.624(1)	0.6823(8)	4.57
C13	0.7344(9)	0.566(2)	0.6259(9)	6.31
C14	0.7550(8)	0.470(2)	0.6439(11)	6.08
C15	0.7497(7)	0.432(1)	0.717(1)	4.92
C16	0.5465(7)	0.2084(9)	0.8897(8)	3.78
C17	0.575(1)	0.127(1)	0.9400(12)	7.27
C18	0.6217(8)	0.175(1)	0.9994(9)	5.06
C19	0.6482(6)	0.263(1)	0.9557(8)	3.95
C20	0.4696(6)	0.537(1)	0.7674(6)	2.97
C21	0.4504(7)	0.436(1)	0.7790(7)	4.07
C22 [b]	0.5000	0.378(1)	0.7500	3.68
C23 [b]	0.5258	0.5817	0.9910	3.08
C24 [b]	0.4679	0.5411	0.9486	6.73
C25 [b]	0.4557	0.4450	0.9772	2.52
C26 [b]	0.5062	0.4262	1.0373	5.34
C27 [b]	0.5495	0.5107	1.0458	2.79

[a] $B_{eq} = (8\pi^2/3)\Sigma_i\Sigma_j U_{ij} a^*_i a^*_j a_i \cdot a_j$. [b] Calculated carbon position.

TABLE 3. Selected bond distances (Å) for **1** with e.s.d.s of the last significant digit in parentheses

V–C1	2.27(1)	V–C2	2.26(1)
V–C3	2.27(1)	V–C4	2.28(1)
V–C5	2.24(1)	V–C6	2.19(1)
V–C7	2.15(1)	V–C8	2.10(1)
V–C9	2.16(1)	V–C10	2.13(1)
V–C11	2.21(1)	Yb···C7	3.01(1)
Yb–C8	2.63(1)	Yb–C9	2.87(1)
Yb–C20	2.79(1)	Yb–C21	2.78(1)
Yb–C22	2.788(7)	Yb–C23	2.645(7)
Yb–C24	2.482(5)	Yb–C25	2.767(1)
Yb–C26	3.068(2)	Yb–C27	3.001(2)
Yb–O1	2.454(7)		

1 was also obtained from the neat reaction between the solids $(C_5H_5)_2V$ and $(C_{10}H_8)Yb(THF)_2$ for 12 to 15 h. Independent of the molar ratio, the yields of **1** were moderate.

The formation of the anion $[(C_5H_5)V(C_{10}H_8)]^-$ with V(0) demonstrates that $(C_{10}H_8)Yb(THF)_2$ acts as a reducing agent, comparable to $KC_{10}H_8$ [9]. At room

TABLE 4. Selected bond angles (°) for **1** with e.s.d.s of the last significant digit in parentheses

CT1–V–CT2	175.0(4)
CT2–Yb–CT3	125.2(7)
CT2–Yb–CT4	101.3(7)
CT3–Yb–CT4	119.7(7)
CT2–Yb–O1	105.5(3)
CT3–Yb–O1	98.2(2)
CT4–Yb–O1	104.4(3)
Yb–Cp–Yb′	180.0

CT1 defines the centroid of the ring formed by C1–C5; CT2 defines the centroid of the ring formed by C6–C11; CT3 defines the centroid of the ring formed by C23–C27; CT4 defines the centroid of the ring formed by C20, C21, C22, C20′, C21′. The symmetry equivalent positions (′) can be achieved using the transformation: $1-x$, y, $1.5-z$.

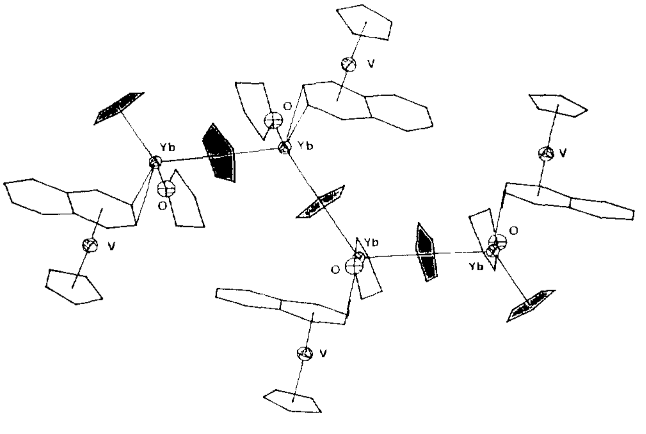

Fig. 1. ORTEP plot of **1** showing the infinite zig-zag chain formed by cyclopentadienylytterbium moieties. Thermal ellipsoids for the non-carbon atoms are drawn at the 50% probability level. The thermal ellipsoids for the carbon atoms are reduced variably. Hydrogen atoms are omitted for clarity.

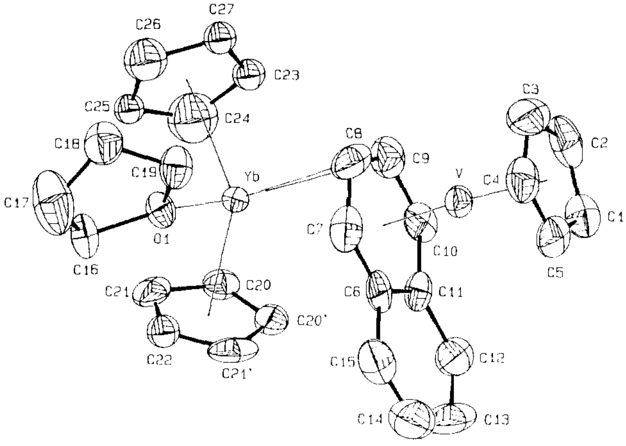

Fig. 2. ORTEP drawing of **1** with the atomic numbering scheme at the 50% probability level. The second part of a cyclopentadienyl system (C20–C22), generated by the two-fold axis, is added for clarity. Hydrogen atoms are omitted.

temperature, **1** possesses a magnetic moment $\mu_{\text{eff}}=1.4$ BM. It is soluble in THF, but insoluble in ether, toluene or hexane.

Finally, we found that an equimolar mixture of $K[(C_5H_5)V(C_{10}H_8)]$ and KC_5H_5, prepared *in situ* from $(C_5H_5)_2V$ and $KC_{10}H_8$, reacts with $YbI_2(THF)_2$ in tetrahydrofuran to form **1** in a yield of 97%.

$$YbI_2(THF)_2+K[(C_5H_5)V(C_{10}H_8)]+KC_5H_5\xrightarrow{\text{THF}}$$

$$\underset{\mathbf{1}}{[(\eta^5\text{-}C_5H_5)V(\mu\text{-}\eta^6{:}\eta^2\text{-}C_{10}H_8)Yb(THF)(\eta^5\text{-}C_5H_5)]_n}+2KI$$

Until now, all our attempts to synthesize disubstituted ytterbium complexes like $[(C_5H_5)V(C_{10}H_8)]_2Yb(THF)_x$ or corresponding anthracene derivatives failed.

Molecular structure of $[(\eta^5\text{-}C_5H_5)V(\mu\text{-}\eta^6{:}\eta^2\text{-}C_{10}H_8)Yb(THF)(\eta^5\text{-}C_5H_5)]_n$ *(1)*

The X-ray crystal structure analysis proves **1** is a polymer built up from infinite zig-zag chains formed by cyclopentadienylytterbium moieties, $(C_5H_5Yb)_n$. Each ytterbium atom of the chain is further η^2 coordinated by the naphthalene of one $(C_5H_5)V(C_{10}H_8)$ unit and by one THF molecule (Fig. 1). The ytterbium–cyclopentadienyl–ytterbium chain is bent at ytterbium, but linear at the cyclopentadienyl centroids with η^5 bonds of the cyclopentadienyl rings to both neighboring ytterbium atoms. This kind of cyclopentadienyl bridging is also found in $[(MeC_5H_4)_2Yb(THF)]_n$ [23], but not in most of the other compounds of this type investigated crystallographically until now. Thus, in the Cp_3Ln derivatives of lanthanum and lutetium the bridging C_5H_5 unit is $\eta^5{:}\eta^2$ or even $\eta^1{:}\eta^1$ bonded to the neighbouring metal atoms, $(\eta^5\text{-}C_5H_5)_2La(\mu\text{-}\eta^5{:}\eta^2\text{-}C_5H_5)$ [24] or $(\eta^5\text{-}C_5H_5)_2Lu(\mu\text{-}\eta^1{:}\eta^1\text{-}C_5H_5)$ [25].

Figure 2 shows an ORTEP [26] drawing of **1** with the atomic numbering scheme in the asymmetric unit and the second part of the cyclopentadienyl system (C20–C22) which is generated by the two-fold axis. The Yb–C_5H_5 centroid distances (2.534(7) and 2.520(7) Å), which are equal in the margin of the standard deviation, are long compared with that in monomeric cyclopentadienylytterbium(II) compounds, e.g. in $(C_5H_4(CH_2)_3\text{-}C_5H_4)Yb(THF)_2$ (2.42 Å [27], $(C_5Me_5)_2Yb(THF)$ (2.37 Å) [28], or $(t\text{-}C_4H_9C_5H_4)_2Yb(THF)_2$ (2.438–2.445 Å) [29], but in the same range as in $[(MeC_5H_4)_2Yb(THF)]_n$ (mean C–Yb distances 2.76, 2.87 and 2.91 Å) [23]. The same effect is found for cyclopentadienylindium and thallium derivatives with longer C–In or C–Tl distances in the polymeric chains than in the monomeric units in the gas phase (e.g. 2.621 Å for monomeric [30] and 2.85–3.09 Å for polymeric $(C_5H_5In)_n$ [31]).

The ytterbium atom is coordinated by two η^5-cyclopentadienyl rings belonging to the chain, the oxygen atom of one THF ligand and two carbon atoms of the naphthalene. The Yb–$O_{(THF)}$ distance (2.454(7) Å) is

TABLE 5. Comparison of the carbon–carbon distances (Å) in different kinds of naphthalene compounds

Bond	1	$[(C_5H_5)V]_2(C_{10}H_8)$	$[(C_6H_6)Cr]_2C_{10}H_8$	$C_{10}H_8$
C6–C7	1.43(2)	1.455	1.443	1.426
C7–C8	1.45(2)	1.418	1.388	1.378
C8–C9	1.43(2)	1.397	1.410	1.415
C9–C10	1.43(2)	1.420	1.392	1.378
C10–C11	1.44(2)	1.448	1.438	1.426
C6–C11	1.44(2)	1.468	1.466	1.426

in the same range as in the cyclopentadienylytterbium complexes containing coordinated THF (2.41 Å [27], 2.412 Å [28], 2.426–2.435 Å [29]). There are only two short contacts between Yb and the aromatic hydrocarbon, 2.63(1) Å to C8 and 2.87(1) Å to C9, which are still longer as common ytterbium–carbon σ bonds. The naphthalene system is influenced by both the η^2-coordination to ytterbium and the η^6-coordination to vanadium. All C–C bonds are longer than in solid naphthalene [32], but consistent with the C–C bond lengths in $(C_5H_5)V(C_{10}H_8)V(C_5H_5)$ [8] and $(C_6H_6)Cr(C_{10}H_8)Cr(C_6H_6)$ [33] (Table 5). The expected shortening of the C8–C9 bond (1.415 Å in $C_{10}H_8$ [32], 1.410 Å in $(C_6H_6)Cr(C_{10}H_8)Cr(C_6H_6)$ [33], 1.397 Å in $(C_5H_5)V(C_{10}H_8)V(C_5H_5)$ [8], but 1.43 Å in **1**) was not confirmed. It may be considered as the engagement of the π-electron density between C8 and C9 into the coordinative bond to ytterbium.

The vanadium atoms are η^5-bonded to the cyclopentadienyl ring and η^6 to that six-membered ring of the naphthalene system which is also the bridging part to ytterbium, an arrangement resulting in an almost coplanar sandwich system (angle centroid (C_5H_5)–V–ring centroid $(C_{10}H_8) = 175.0(4)°$). The bonding distances metal–centroid (C_5H_5) (1.92(4) Å) and metal–centroid $(C_{10}H_8)$ (1.589(7) Å) are similar to those found in $(C_5H_5)V(C_{10}H_8)V(C_5H_5)$ (1.918 and 1.652 Å) [8].

Supplementary material

Further details of the structure investigation are available on request from the Fachinformationszentrum Karlsruhe, Gesellschaft für wissenschaftlich-technische Information mbH, W-7514 Eggenstein-Leopoldshafen 2, Germany, on quoting the depository number CSD 56607, the authors, and the full citation of the journal.

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