metal-organic papers

A solvated ytterbium diaryl: bis(1,1′:3′,1″-terphenyl-$C^{2'}$)bis(tetrahydrofuran-O)ytterbium(II)

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Key indicators

Single-crystal X-ray study
$T = 173$ K
Mean σ(C–C) = 0.006 Å
R factor = 0.032
wR factor = 0.078
Data-to-parameter ratio = 17.2

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

The title compound, [Yb(Dpp)$_2$(thf)$_2$] [Dpp = 2,6-Ph$_2$C$_6$H$_3$ (*m*-terphenyl), C$_{18}$H$_{13}$, and thf = tetrahydrofuran, C$_4$H$_8$O], is the first example of a structurally characterized Yb^{II} diaryl. The Yb atom shows a strongly distorted tetrahedral environment formed by the C atoms of two aryl groups (average Yb—C = 2.520 Å) and the O atoms of two tetrahydrofuran ligands (average Yb—O = 2.412 Å). In addition, there are two weak η^1–π-arene interactions (average Yb···C = 3.138 Å) involving *ortho*-C atoms of the m-terphenyl phenyl groups.

Comment

σ-Bonded ytterbium^{II} organyls may be conveniently prepared by a direct synthesis route from organyl iodides and Yb metal (Evans *et al.*, 1971). Using *m*-terphenyl groups as supporting ligands we recently reported the synthesis and structural characterization of europium^{II} and ytterbium^{II} aryls (Heckmann & Niemeyer, 2000). For the Grignard-analogous ytterbium^{II} aryl [Yb(Dpp)I(thf)$_3$], it was possible to detect a Schlenk-like equilibrium between [Yb(Dpp)I(thf)$_3$], [Yb(Dpp)$_2$(thf)$_2$] and [YbI$_2$(thf)$_4$] species in THF solution, using ^{171}Yb NMR spectroscopy as a probe. Depending on the nature of the aryl substituent and the solvent used, either the diaryl compound or the mixed aryl ytterbium iodide species may be obtained from solution. Thus, recrystallization of [Yb(Dpp)I(thf)$_3$] from aromatic solvents yielded dark-red prisms of the title compound, (I).
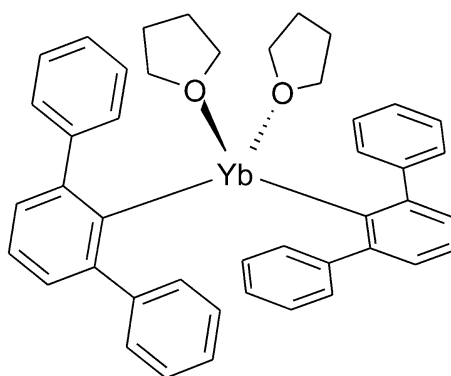
(I)

The molecular structure of (I) shows monomeric units (Fig. 1) wherein two aryl ligands and two thf molecules form a strongly distorted tetrahedral environment of the Yb atom. The different size of the ligands is clearly reflected in the huge difference between the C1—Yb—C19 [129.79 (11)°] and
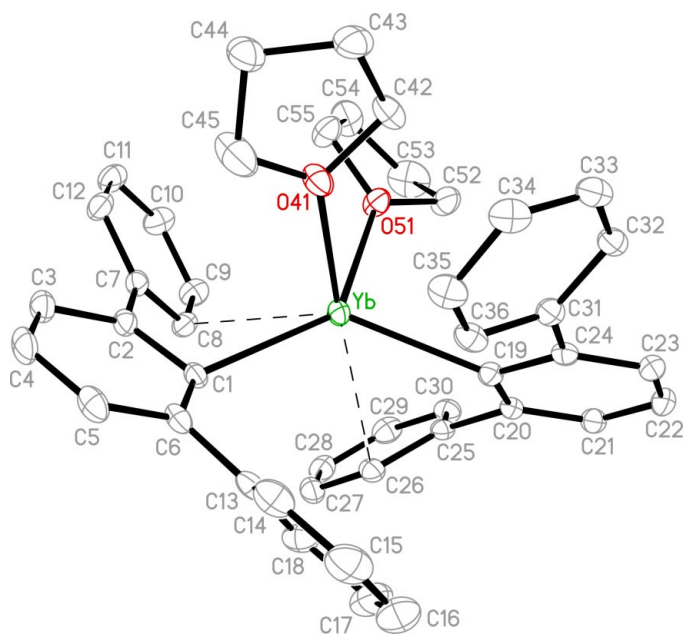
Figure 1
Displacement ellipsoid plot (at the 30% probability level) for (I). H atoms have been omitted for clarity.

O41—Yb—O51 [78.34 (9)°] bond angles. With almost identical values of 2.521 (3) (Yb—C1) and 2.518 (3) Å (Yb—C19), the Yb—C distances in (I) are close to the bond lengths in [Yb(Dpp)I(thf)₃] [2.529 (4) Å; Heckmann & Niemeyer, 2000] and [Yb{C(SiMe₃)₃}₂] (average 2.496 Å; Eaborn *et al.*, 1994), which contain five- and two-coordinate metal centers, respectively. The distorted tetrahedral coordination of the Yb atom in (I) leaves room for two additional weak η^1-π-arene interactions [Yb···C8 = 3.127 (3) Å and Yb···C26 = 3.148 (3) Å] involving the *ortho*-C atoms of phenyl groups of *m*-terphenyl ligands. As a consequence, considerable differences in the Yb—C(*ipso*)—C(*ortho*) angles [Yb—C1—C2/ Yb—C1—C6 = 109.2 (2)/126.3(2)° and Yb—C19—C20/Yb— C19—C24 = 114.8 (2)/127.0(2)°] are observed. Another notable feature is the significant displacement of the Yb atom from the C1—C6 and C19—C24 aromatic ring planes [1.341 (5) and 0.745 (5) Å, respectively]. These unusually large displacements may be explained by steric overcrowding inside the molecule and the highly ionic character of the metal– carbon bonds. In addition, a favourable arrangement regarding the secondary η^1–π-arene interactions is provided; the angles between the Yb···C vectors and the normals of the aromatic ring planes are 32.2 (2) [Yb···C8/C7—C12] and 30.1 (2)° [Yb···C26/C25—C30].

Finally, taking into account the difference in the ionic radii for Yb^{II} and Eu^{II} (0.15 Å for coordination number 6), it is interesting to compare the pertinent bond distances in (I) with the corresponding values in the isostructural compound [Eu(Dpp)₂(thf)₂] (Heckmann & Niemeyer, 2000). It turns out that the difference in the ionic radii has the most significant effect on the M—C bonds [2.615 Å (average Eu—C) *versus* 2.520 Å (average Yb—C)], shows somewhat smaller influence

on the M—O coordination [2.498 Å (average Eu—O) *versus* 2.412 Å (average Yb—O)] and has least effect on the secondary interactions [3.186 Å (average Eu···C) *versus* 3.138 Å (average Yb···C)].

Experimental

The title compound was synthesized and characterized as described earlier (Heckmann & Niemeyer, 2000). Dark-red crystals were grown from C_6D_6 at 278 K.

Crystal data

[Yb(C₁₈H₁₃)₂(C₄H₈O)₂]	$Z = 2$
$M_r = 775.82$	$D_x = 1.487$ Mg m^{-3}
Triclinic, $P\bar{1}$	Mo $K\alpha$ radiation
$a = 9.754$ (3) Å	Cell parameters from 18
$b = 11.9028$ (18) Å	reflections
$c = 17.093$ (3) Å	$\theta = 8.1$–12.2°
$\alpha = 75.923$ (14)°	$\mu = 2.74$ mm^{-1}
$\beta = 74.367$ (18)°	$T = 173$ (2) K
$\gamma = 66.560$ (16)°	Prism, dark red
$V = 1732.5$ (6) Å^3	$0.30 \times 0.20 \times 0.15$ mm

Data collection

Rebuilt Syntex $P2_1$/Siemens $P3$ four-circle diffractometer	$R_{int} = 0.032$
	$\theta_{max} = 27.5°$
Wyckoff scans	$h = -12 \rightarrow 12$
Absorption correction: ψ scan (North *et al.*, 1968)	$k = -14 \rightarrow 15$
	$l = -21 \rightarrow 22$
$T_{min} = 0.494$, $T_{max} = 0.684$	2 standard reflections
8446 measured reflections	every 198 reflections
7975 independent reflections	intensity decay: none
7095 reflections with $I > 2\sigma(I)$	

Refinement

Refinement on F^2	Only H-atom U's refined
$R[F^2 > 2\sigma(F^2)] = 0.032$	$w = 1/[\sigma^2(F_o^2) + (0.0409P)^2]$
$wR(F^2) = 0.078$	where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.19$	$(\Delta/\sigma)_{max} = 0.001$
7975 reflections	$\Delta\rho_{max} = 0.86$ e Å^{-3}
463 parameters	$\Delta\rho_{min} = -0.73$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Yb—C1	2.521 (3)	Yb···C26	3.148 (3)
Yb—C19	2.518 (3)	C1—C2	1.417 (5)
Yb—O41	2.396 (3)	C1—C6	1.420 (5)
Yb—O51	2.427 (2)	C19—C20	1.413 (5)
Yb···C8	3.127 (3)	C19—C24	1.422 (5)
C1—Yb—C19	129.79 (11)	Yb—C1—C2	109.2 (2)
O41—Yb—O51	78.34 (9)	Yb—C1—C6	126.3 (2)
C1—Yb—O41	88.62 (10)	C2—C1—C6	115.0 (3)
C1—Yb—O51	136.61 (10)	Yb—C19—C20	114.8 (2)
C19—Yb—O41	114.73 (10)	Yb—C19—C24	127.0 (2)
C19—Yb—O51	92.82 (10)	C20—C19—C24	115.2 (3)

The H atoms were positioned with idealized geometry and refined in a riding-model approximation. For most H atoms, the assigned U_{iso} was allowed to refine freely. Isotropic displacement parameters larger than 0.15 Å^2 were constrained to 1.2U_{eq} of the parent atom.

Data collection: *P3* (Siemens, 1989); cell refinement: *P3*; data reduction: *XDISK* (Siemens, 1989); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *SHELXTL* (Sheldrick, 1998); software used to prepare material for publication: *SHELXTL*.

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