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Cubic di- μ -amido-bis[bis(η^5 -cyclopentadienyl)ytterbium(III)]

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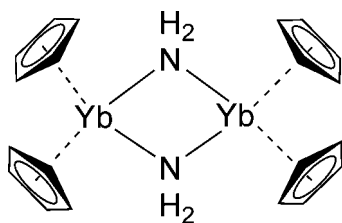
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 Key indicators: single-crystal X-ray study; $T = 293$ K; mean $\sigma(\text{C}-\text{C}) = 0.008$ Å; R factor = 0.024; wR factor = 0.051; data-to-parameter ratio = 23.4.

The title compound, $[\text{Yb}_2(\text{C}_5\text{H}_5)_4(\text{NH}_2)_2]$, was synthesized in a Parr high-pressure vessel starting from monoclinic $[\text{Cp}_2\text{YbNH}_2]_2$ (where Cp = cyclopentadienyl). The complex is located on a special position of site symmetry mmm with just an eighth of the complex in the asymmetric unit. The Yb atom is in a distorted tetrahedral coordination environment, coordinated by two cyclopentadienyl rings and two amino groups forming a dimeric complex bridged through the amino groups. This compound represents the high-pressure cubic modification of monoclinic $[\text{Cp}_2\text{YbNH}_2]_2$ obtained at 250 bar (1 bar = 10^5 Pa) under an atmosphere of argon.

Related literature

The monoclinic $[\text{Cp}_2\text{YbNH}_2]_2$ complex was first described by Fischer & Fischer (1966). The first single X-ray diffraction data of this compound were presented by Baisch, Pagano, Zeuner, Barros *et al.* (2006). For related literature, see Schumann *et al.* (1995); Hayes & Thomas (1989); Hammel & Weidlein (1990); Baisch, Pagano, Zeuner & Schnick (2006).



Experimental

Crystal data

$[\text{Yb}_2(\text{C}_5\text{H}_5)_4(\text{NH}_2)_2]$
 $M_r = 638.5$
 Cubic, $Im\bar{3}$
 $a = 14.4104$ (17) Å
 $V = 2992.5$ (6) Å³

$Z = 6$
 Mo $K\alpha$ radiation
 $\mu = 9.32$ mm⁻¹
 $T = 293$ (2) K
 $0.22 \times 0.17 \times 0.12$ mm

Data collection

Stoe IPDS diffractometer
 Absorption correction: multi-scan (*XPREP* in *SHELXTL-Plus*; Sheldrick, 1996)
 $T_{\min} = 0.160$, $T_{\max} = 0.390$
 (expected range = 0.134–0.327)

16043 measured reflections
 843 independent reflections
 578 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.080$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.025$
 $wR(F^2) = 0.051$
 $S = 0.87$
 843 reflections
 36 parameters
 1 restraint

H atoms treated by a mixture of independent and constrained refinement
 $\Delta\rho_{\max} = 0.58$ e Å⁻³
 $\Delta\rho_{\min} = -0.50$ e Å⁻³

Data collection: *X-AREA* (Stoe & Cie, 2002); cell refinement: *X-AREA*; data reduction: *X-AREA*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1990); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *DIAMOND* (Brandenburg, 1999); software used to prepare material for publication: *SHELXL97*.

The authors are indebted to Thomas Miller for performing the single-crystal X-ray diffractometry. Financial support by the Deutsche Forschungsgemeinschaft (DFG) (Schwerpunktprogramm SPP 1166, Lanthanoidspezifische Funktionalitäten in Molekül und Material, project SCHN377/10) and the Fonds der Chemischen Industrie is also gratefully acknowledged.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BT2511).

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supplementary materials

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Cubic di- μ -amido-bis[bis(η^5 -cyclopentadienyl)ytterbium(III)]

M. Zeuner and W. Schnick

Comment

The structure of the title compound, $[\text{Cp}_2\text{YbNH}_2]_2$, shows the high-pressure cubic modification of the monoclinic compound $[\text{Cp}_2\text{YbNH}_2]_2$. The asymmetric unit consists of three C atoms, one N atom and one Yb atom (figure 1). Geometric parameters of the title compound are in the usual ranges. It crystallizes isotyp with cubic $[\text{Cp}_2\text{ErNH}_2]_2$, which was the only cubic compound of the system $[\text{Cp}_2\text{LnNH}_2]_2$ so far.

Experimental

Anhydrous ammonia was condensed at 195 K (dry ice, *i*PrOH) onto YpCp_3 (4–10 mmol). The resulting colourless mixture was stirred rapidly for 5 h and then further stirred for additional 6 – 24 h without further cooling. After complete evaporation of residual ammonia the pale-coloured solid was dried under vacuum and sublimed under reduced pressure yielding green Cp_3YbNH_3 . Heating of Cp_3YbNH_3 to 523 K yielded yellow monoclinic $[\text{Cp}_2\text{YbNH}_2]_2$. Subsequent treatment of this compound to 250 bar yielded yellow cubic $[\text{Cp}_2\text{YbNH}_2]_2$.

Refinement

The H atoms bonded to C were positioned geometrically and refined using a riding model, C—H = 0.93 Å and $U_{\text{iso}}(\text{H}) = 1.2 U_{\text{iso}}(\text{C})$. The amino H atom was freely refined.

Figures

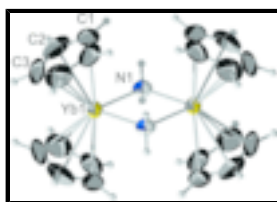


Fig. 1. A view of the title compound showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level.

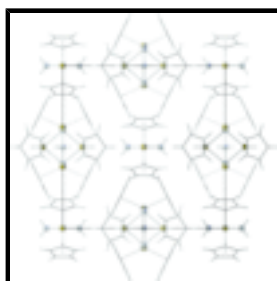


Fig. 2. shows the crystal packing diagram of the title compound viewed along the *c* axis.

di- μ -amido-bis[bis(η^5 -cyclopentadienyl)ytterbium(III)]

Crystal data

[Yb ₂ (C ₅ H ₅) ₄ (NH ₂) ₂]	$Z = 6$
$M_r = 638.5$	$F_{000} = 1788$
Cubic, $Im\bar{3}$	$D_x = 2.126 \text{ Mg m}^{-3}$
Hall symbol: -I223	Melting point: not measured K
$a = 14.4104 (17) \text{ \AA}$	Mo $K\alpha$ radiation
$b = 14.4104 (17) \text{ \AA}$	$\lambda = 0.71073 \text{ \AA}$
$c = 14.4104 (17) \text{ \AA}$	Cell parameters from 6512 reflections
$\alpha = 90^\circ$	$\theta = 4.6\text{--}60.9^\circ$
$\beta = 90^\circ$	$\mu = 9.32 \text{ mm}^{-1}$
$\gamma = 90^\circ$	$T = 293 (2) \text{ K}$
$V = 2992.5 (6) \text{ \AA}^3$	Platelet, yellow
	$0.22 \times 0.17 \times 0.12 \text{ mm}$

Data collection

Stoe IPDS diffractometer	843 independent reflections
Radiation source: fine-focus sealed tube	578 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{\text{int}} = 0.080$
$T = 293(2) \text{ K}$	$\theta_{\text{max}} = 30.4^\circ$
oscillation scans	$\theta_{\text{min}} = 2.8^\circ$
Absorption correction: multi-scan correction based on equivalents (XPREP in SHELXTL-PLUS; Sheldrick, 1996)	$h = -20 \rightarrow 20$
$T_{\text{min}} = 0.160$, $T_{\text{max}} = 0.390$	$k = -20 \rightarrow 20$
16043 measured reflections	$l = -20 \rightarrow 20$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.025$	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2) = 0.051$	$w = 1/[\sigma^2(F_o^2) + (0.0285P)^2]$
$S = 0.87$	where $P = (F_o^2 + 2F_c^2)/3$
843 reflections	$(\Delta/\sigma)_{\text{max}} < 0.001$
36 parameters	$\Delta\rho_{\text{max}} = 0.58 \text{ e \AA}^{-3}$
1 restraint	$\Delta\rho_{\text{min}} = -0.50 \text{ e \AA}^{-3}$
Primary atom site location: structure-invariant direct methods	Extinction correction: none

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted R -factor wR and goodness of fit S are based on F^2 , conventional R -factors R are based on F , with F set to zero for negative F^2 . The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating R -factors(gt) *etc.* and is not relevant to the choice of reflections for refinement. R -factors based on F^2 are statistically about twice as large as those based on F , and R -factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (\AA^2)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
Yb1	0.5000	0.12174 (2)	0.5000	0.03854 (11)
N1	0.5000	0.0000	0.6022 (4)	0.0400 (14)
C1	0.3244 (3)	0.1320 (4)	0.4523 (4)	0.0776 (17)
H1	0.3009	0.0854	0.4143	0.093*
C2	0.3644 (4)	0.2112 (5)	0.4235 (5)	0.090 (2)
H2	0.3739	0.2286	0.3621	0.108*
C3	0.3888 (6)	0.2621 (5)	0.5000	0.105 (4)
H3	0.4167	0.3202	0.5000	0.126*
H11	0.551 (3)	0.0000	0.638 (4)	0.07 (2)*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Yb1	0.03668 (19)	0.03207 (18)	0.0469 (2)	0.000	0.000	0.000
N1	0.044 (4)	0.049 (4)	0.026 (3)	0.000	0.000	0.000
C1	0.040 (2)	0.071 (3)	0.122 (4)	0.015 (2)	-0.018 (2)	-0.026 (3)
C2	0.068 (4)	0.103 (5)	0.100 (5)	0.034 (4)	-0.004 (3)	0.036 (4)
C3	0.057 (5)	0.036 (4)	0.224 (14)	0.018 (3)	0.000	0.000

Geometric parameters (\AA , $^\circ$)

Yb1—N1 ⁱ	2.291 (4)	Yb1—C1 ^{iv}	2.626 (5)
Yb1—N1	2.291 (4)	Yb1—H11	2.75 (3)
Yb1—C3 ⁱⁱ	2.580 (6)	N1—Yb1 ⁱ	2.291 (4)
Yb1—C3	2.580 (6)	N1—H11	0.894 (10)
Yb1—C2 ⁱⁱⁱ	2.588 (5)	C1—C2	1.344 (8)
Yb1—C2 ⁱⁱ	2.588 (5)	C1—C1 ⁱⁱⁱ	1.376 (12)
Yb1—C2	2.588 (5)	C1—H1	0.9300
Yb1—C2 ^{iv}	2.588 (5)	C2—C3	1.369 (8)
Yb1—C1 ⁱⁱⁱ	2.626 (5)	C2—H2	0.9300
Yb1—C1	2.626 (5)	C3—C2 ⁱⁱⁱ	1.369 (8)

supplementary materials

Yb1—C1 ⁱⁱ	2.626 (5)	C3—H3	0.9300
N1 ⁱ —Yb1—N1	80.1 (2)	C2 ⁱⁱ —Yb1—C1 ⁱⁱ	29.87 (19)
N1 ⁱ —Yb1—C3 ⁱⁱ	126.87 (15)	C2—Yb1—C1 ⁱⁱ	144.2 (2)
N1—Yb1—C3 ⁱⁱ	126.87 (15)	C2 ^{iv} —Yb1—C1 ⁱⁱ	49.90 (19)
N1 ⁱ —Yb1—C3	126.87 (15)	C1 ⁱⁱⁱ —Yb1—C1 ⁱⁱ	148.9 (3)
N1—Yb1—C3	126.87 (15)	C1—Yb1—C1 ⁱⁱ	173.6 (2)
C3 ⁱⁱ —Yb1—C3	76.8 (4)	N1 ⁱ —Yb1—C1 ^{iv}	82.80 (12)
N1 ⁱ —Yb1—C2 ⁱⁱⁱ	130.95 (16)	N1—Yb1—C1 ^{iv}	102.21 (14)
N1—Yb1—C2 ⁱⁱⁱ	96.19 (19)	C3 ⁱⁱ —Yb1—C1 ^{iv}	50.0 (2)
C3 ⁱⁱ —Yb1—C2 ⁱⁱⁱ	94.5 (3)	C3—Yb1—C1 ^{iv}	123.7 (3)
C3—Yb1—C2 ⁱⁱⁱ	30.72 (18)	C2 ⁱⁱⁱ —Yb1—C1 ^{iv}	144.2 (2)
N1 ⁱ —Yb1—C2 ⁱⁱ	130.95 (16)	C2 ⁱⁱ —Yb1—C1 ^{iv}	49.90 (19)
N1—Yb1—C2 ⁱⁱ	96.19 (19)	C2—Yb1—C1 ^{iv}	126.0 (2)
C3 ⁱⁱ —Yb1—C2 ⁱⁱ	30.72 (18)	C2 ^{iv} —Yb1—C1 ^{iv}	29.87 (19)
C3—Yb1—C2 ⁱⁱ	94.5 (3)	C1 ⁱⁱⁱ —Yb1—C1 ^{iv}	173.6 (2)
C2 ⁱⁱⁱ —Yb1—C2 ⁱⁱ	98.1 (3)	C1—Yb1—C1 ^{iv}	148.9 (3)
N1 ⁱ —Yb1—C2	96.19 (19)	C1 ⁱⁱ —Yb1—C1 ^{iv}	30.4 (3)
N1—Yb1—C2	130.95 (16)	N1 ⁱ —Yb1—H11	88.7 (8)
C3 ⁱⁱ —Yb1—C2	94.5 (3)	N1—Yb1—H11	17.5 (5)
C3—Yb1—C2	30.72 (18)	C3 ⁱⁱ —Yb1—H11	109.6 (4)
C2 ⁱⁱⁱ —Yb1—C2	50.4 (3)	C3—Yb1—H11	131.6 (12)
C2 ⁱⁱ —Yb1—C2	120.2 (3)	C2 ⁱⁱⁱ —Yb1—H11	102.1 (13)
N1 ⁱ —Yb1—C2 ^{iv}	96.19 (19)	C2 ⁱⁱ —Yb1—H11	79.0 (4)
N1—Yb1—C2 ^{iv}	130.95 (16)	C2—Yb1—H11	145.7 (12)
C3 ⁱⁱ —Yb1—C2 ^{iv}	30.72 (18)	C2 ^{iv} —Yb1—H11	115.2 (10)
C3—Yb1—C2 ^{iv}	94.5 (3)	C1 ⁱⁱⁱ —Yb1—H11	95.8 (12)
C2 ⁱⁱⁱ —Yb1—C2 ^{iv}	120.2 (3)	C1—Yb1—H11	118.7 (9)
C2 ⁱⁱ —Yb1—C2 ^{iv}	50.4 (3)	C1 ⁱⁱ —Yb1—H11	65.9 (8)
C2—Yb1—C2 ^{iv}	98.1 (3)	C1 ^{iv} —Yb1—H11	88.3 (12)
N1 ⁱ —Yb1—C1 ⁱⁱⁱ	102.21 (14)	Yb1 ⁱ —N1—Yb1	99.9 (2)
N1—Yb1—C1 ⁱⁱⁱ	82.80 (12)	Yb1 ⁱ —N1—H11	112 (3)
C3 ⁱⁱ —Yb1—C1 ⁱⁱⁱ	123.7 (3)	Yb1—N1—H11	112 (3)
C3—Yb1—C1 ⁱⁱⁱ	50.0 (2)	C2—C1—C1 ⁱⁱⁱ	107.9 (4)
C2 ⁱⁱⁱ —Yb1—C1 ⁱⁱⁱ	29.87 (19)	C2—C1—Yb1	73.5 (3)
C2 ⁱⁱ —Yb1—C1 ⁱⁱⁱ	126.0 (2)	C1 ⁱⁱⁱ —C1—Yb1	74.82 (13)
C2—Yb1—C1 ⁱⁱⁱ	49.90 (19)	C2—C1—H1	126.0
C2 ^{iv} —Yb1—C1 ⁱⁱⁱ	144.2 (2)	C1 ⁱⁱⁱ —C1—H1	126.0
N1 ⁱ —Yb1—C1	82.80 (12)	Yb1—C1—H1	117.7
N1—Yb1—C1	102.21 (14)	C1—C2—C3	108.5 (7)
C3 ⁱⁱ —Yb1—C1	123.7 (3)	C1—C2—Yb1	76.6 (3)
C3—Yb1—C1	50.0 (2)	C3—C2—Yb1	74.3 (4)

C2 ⁱⁱⁱ —Yb1—C1	49.90 (19)	C1—C2—H2	125.8
C2 ⁱⁱ —Yb1—C1	144.2 (2)	C3—C2—H2	125.8
C2—Yb1—C1	29.87 (19)	Yb1—C2—H2	115.4
C2 ^{iv} —Yb1—C1	126.0 (2)	C2 ⁱⁱⁱ —C3—C2	107.2 (9)
C1 ⁱⁱⁱ —Yb1—C1	30.4 (3)	C2 ⁱⁱⁱ —C3—Yb1	74.9 (3)
N1 ⁱ —Yb1—C1 ⁱⁱ	102.21 (14)	C2—C3—Yb1	74.9 (3)
N1—Yb1—C1 ⁱⁱ	82.80 (12)	C2 ⁱⁱⁱ —C3—H3	126.4
C3 ⁱⁱ —Yb1—C1 ⁱⁱ	50.0 (2)	C2—C3—H3	126.4
C3—Yb1—C1 ⁱⁱ	123.7 (3)	Yb1—C3—H3	116.0
C2 ⁱⁱⁱ —Yb1—C1 ⁱⁱ	126.0 (2)		

Symmetry codes: (i) $-x+1, -y, -z+1$; (ii) $-x+1, y, -z+1$; (iii) $x, y, -z+1$; (iv) $-x+1, y, z$.

Fig. 1

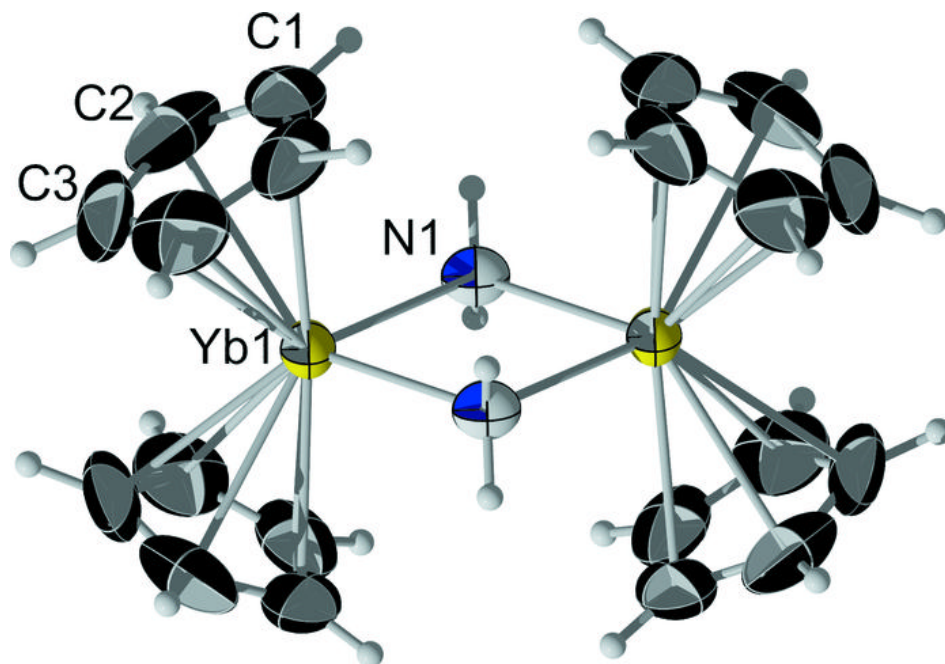


Fig. 2

