Crystal structure of bis(cyclopentadienyl)titanium-*N*,*N*-bis(cyclohexyl)-diazabuta-1,3-diene, $C_{24}H_{34}N_2Ti$

A. Spannenberg*, T. Zippel and U. Rosenthal

Institut für Organische Katalyseforschung an der Universität Rostock e.V., Buchbinderstr. 5-6, D-18055 Rostock, Germany

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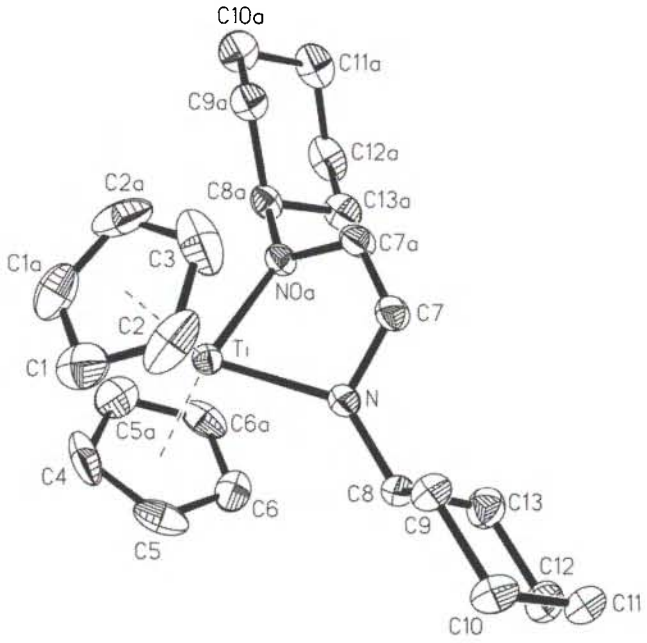

Abstract

$C_{24}H_{34}N_2Ti$, orthorhombic, $Cmc2_1$ (No. 36), a = 19.000(4) Å, b = 14.383(3) Å, c = 7.907(2) Å, V = 2160.8 Å^3, Z = 4, $R_{obs}(F)$ = 0.029, $wR_{all}(F^2)$ = 0.066, T = 293 K.

Source of material

To a solution of 520 mg (1.49 mmol) of $Cp_2Ti(Me_3SiC_2SiMe_3)$ in 20 ml of toluene was added 330 mg (1.50 mmol) of crystalline glyoxal-bis-cyclohexylimine. As the yellowish brown reaction mixture was stirred for 20 hours at 373 K the colour changed to dark green. The solvent was evaporated in vacuo and the residue crystallized at 195 K from n-hexane. After 5 days dark green crystals were obtained, washed with n-hexane and dried in vacuo to yield 520 mg (88%) of the title compound.

Discussion

The chemistry and structural aspects of chelate bonded late-metal diazadiene complexes have been well explored in contrast to the properties of early-metal diazadiene complexes [1]. In the last years diazadiene complexes of the group (IV) metals $Cp_2M(RNC(R')C(R')NR)$ with varying substituents were synthesized[2-4]. Herein we report on the structure of a titanocene diazadiene complex with substituents R = Cy and R'= H). The five membered ring TiNC7C7aN0a forms an envelope conformation. The angle between the planes TiNNOa and NC7C7aN0a are 37.7°.

Table 1. Data collection and handling.

Crystal:	dark green prism, size 0.3 × 0.4 × 0.5 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	4.07 cm^{-1}
Diffractometer, scan mode:	Stoe IPDS, 100 exposures, $\Delta\varphi$ = 2°
$2\theta_{max}$:	48.52°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	3176, 1678
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 1488
$N(param)_{refined}$:	131
Programs:	SHELXS-86 [5], SHELXL-93 [6]

Table 2. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	x	y	z	U_{iso}
H(1)	8*b*	0.0628(2)	0.3748(3)	–0.2409(5)	0.086
H(2)	8*b*	0.1056(2)	0.2182(3)	–0.2199(4)	0.095
H(3)	4*a*	0	0.1116(3)	–0.2137(7)	0.118
H(4)	4*a*	0	0.4701(2)	–0.0171(7)	0.088
H(5)	8*b*	0.1056(2)	0.4096(2)	0.1326(5)	0.080
H(6)	8*b*	0.0644(2)	0.3196(2)	0.3783(5)	0.068
H(7)	8*b*	0.061(1)	0.035(2)	0.117(3)	0.038(6)
H(8)	8*b*	0.1602(1)	0.2379(2)	0.2071(3)	0.048
H(9A)	8*b*	0.1910(1)	0.1577(2)	–0.0341(5)	0.059
H(9B)	8*b*	0.1762(1)	0.0621(2)	0.0545(5)	0.059
H(10A)	8*b*	0.2973(1)	0.0869(2)	0.0562(5)	0.073
H(10B)	8*b*	0.2875(1)	0.1847(2)	0.1411(5)	0.073
H(11A)	8*b*	0.2593(2)	0.0129(2)	0.3016(5)	0.078
H(11B)	8*b*	0.3223(2)	0.0789(2)	0.3474(5)	0.078
H(12A)	8*b*	0.2452(2)	0.1907(2)	0.4469(5)	0.076
H(12B)	8*b*	0.2307(2)	0.0968(2)	0.5418(5)	0.076
H(13A)	8*b*	0.1243(2)	0.1660(2)	0.4543(4)	0.064
H(13B)	8*b*	0.1333(2)	0.0674(2)	0.3723(4)	0.064

* Correspondence author
(e-mail: anke.spannenberg@ifok.uni-rostock.de)

Table 3. Atomic coordinates and displacement parameters (in $Å^2$).

Atom	Site	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
C(1)	8*b*	0.0348(2)	0.3220(3)	–0.2343(5)	0.075(2)	0.102(3)	0.039(2)	–0.022(2)	0.002(2)	0.008(2)
C(2)	8*b*	0.0586(2)	0.2360(3)	–0.2236(4)	0.055(2)	0.146(4)	0.036(2)	0.039(2)	0.007(2)	0.002(2)
C(3)	4*a*	0.0	0.1762(3)	–0.2189(7)	0.229(8)	0.043(3)	0.024(3)	0	0	–0.012(2)
C(4)	4*a*	0.0	0.4325(2)	0.0787(7)	0.147(4)	0.025(2)	0.048(4)	0	0	0.004(2)
C(5)	8*b*	0.0589(2)	0.3994(2)	0.1630(5)	0.070(2)	0.051(2)	0.080(3)	–0.024(2)	0.016(2)	–0.030(2)
C(6)	8*b*	0.0358(2)	0.3490(2)	0.2991(5)	0.084(2)	0.041(2)	0.046(3)	0.017(2)	–0.020(2)	–0.021(2)
C(7)	8*b*	0.0361(1)	0.0933(2)	0.1260(4)	0.040(1)	0.031(1)	0.055(2)	0.003(1)	–0.001(1)	–0.005(1)
C(8)	8*b*	0.1444(1)	0.1732(2)	0.1985(3)	0.039(1)	0.032(1)	0.048(2)	–0.002(1)	–0.010(1)	–0.002(1)
C(9)	8*b*	0.1928(1)	0.1250(2)	0.0732(5)	0.038(1)	0.052(1)	0.057(2)	0.000(1)	–0.010(1)	0.002(1)
C(10)	8*b*	0.2689(1)	0.1219(2)	0.1357(5)	0.039(2)	0.067(2)	0.077(2)	–0.006(1)	–0.012(2)	0.002(2)
C(11)	8*b*	0.2738(2)	0.0774(2)	0.3088(5)	0.047(2)	0.060(2)	0.090(3)	–0.004(2)	–0.028(2)	0.009(2)
C(12)	8*b*	0.2280(2)	0.1277(2)	0.4329(5)	0.078(2)	0.055(2)	0.058(2)	–0.002(1)	–0.033(2)	0.005(2)
C(13)	8*b*	0.1519(2)	0.1302(2)	0.3742(4)	0.060(2)	0.052(2)	0.047(2)	0.005(1)	–0.008(1)	–0.001(1)
N	8*b*	0.07017(9)	0.1755(1)	0.1444(3)	0.038(1)	0.029(1)	0.043(1)	–0.0004(8)	–0.0025(9)	–0.0013(9)
Ti	4*a*	0	0.26731(3)	0.04527(9)	0.0369(3)	0.0283(3)	0.0275(3)	0	0	–0.0019(4)

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