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Crystal structure of 2,3-bis[(4-*tert*-butyl-2,6-dimethylphenyl)imino]butane

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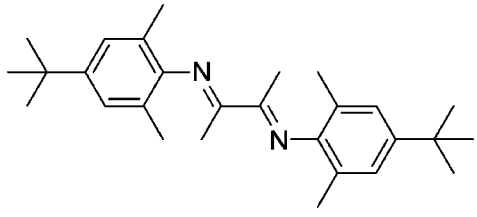
The title compound, $C_{28}H_{40}N_2$, was obtained from the condensation reaction of 4-*tert*-butyl-2,6-dimethylaniline and butane-2,3-dione. The molecule lies on an inversion centre. The C=N bond has an *E* conformation. The plane of the benzene ring is almost perpendicular to the 1,4-diaza-butadiene mean plane [dihedral angle = 89.8 (9)°].

Keywords: crystal structure; α-diimine ligand; catalyst; aniline; diimino-butane.

CCDC reference: 1054707

1. Related literature

The title compound was synthesized as an α-diimine ligand for applications in olefin polymerization Ni^{II}–α-diimine catalysts, see: Cotts *et al.* (2000); Johnson *et al.*(1995); Ittel *et al.* (2000); Mecking *et al.* (1998) . For the effect of the ligand structure on the activity of the catalyst and the properties of the products, see: Gates *et al.* (2000); Meinhard *et al.* (2007); For related structures, see: Yuan *et al.* (2005).



2. Experimental

2.1. Crystal data

$C_{28}H_{40}N_2$ M_r = 404.62

Triclinic, $P\overline{1}$
a = 5.993 (6) Å
b = 10.064 (9) Å
c = 11.614 (11) Å
α = 107.913 (9)°
β = 100.484 (10)°
γ = 99.260 (9)°
V = 637.5 (10) Å^3
Z = 1
Mo $K\alpha$ radiation
μ = 0.06 mm^{-1}
T = 296 K
0.23 × 0.21 × 0.18 mm

2.2. Data collection

Bruker APEXII CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2008)
T_{min} = 0.986, T_{max} = 0.989
4557 measured reflections
2310 independent reflections
1348 reflections with $I > 2\sigma(I)$
R_{int} = 0.030

2.3. Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.102
$wR(F^2)$ = 0.220
S = 1.05
2310 reflections
142 parameters
42 restraints
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.52 e Å^{-3}
$\Delta\rho_{min}$ = −0.21 e Å^{-3}

Data collection: *APEX2* (Bruker, 2008); cell refinement: *SAINT* (Bruker, 2008); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 2008); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

Acknowledgements

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Supporting information for this paper is available from the IUCr electronic archives (Reference: XU5841).

References

Bruker (2008). *APEX2*, *SAINT* and *SADABS*. Bruker AXS Inc., Madison, Wisconsin, USA.
Cotts, P. M., Guan, Z., McCord, E. & McLain, S. (2000). *Macromolecules*, **33**, 6945–6952.
Gates, D. P., Svejda, S. A., Oñate, E., Killian, C. M., Johnson, L. K., White, P. S. & Brookhart, M. (2000). *Macromolecules*, **33**, 2320–2334.
Ittel, S. D., Johnson, L. K. & Brookhart, M. (2000). *Chem. Rev.* **100**, 1169–1204.
Johnson, L. K., Killian, C. M. & Brookhart, M. (1995). *J. Am. Chem. Soc.* **117**, 6414–6415.
Mecking, S., Johnson, L. K., Wang, L. & Brookhart, M. (1998). *J. Am. Chem. Soc.* **120**, 888–899.
Meinhard, D., Wegner, M., Kipiani, G., Hearley, A., Reuter, P., Fischer, S., Marti, O. & Rieger, B. (2007). *J. Am. Chem. Soc.* **129**, 9182–9191.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Yuan, J. C., Silva, L. C., Gomes, P. T., Valerga, P., Campos, J. M., Ribeiro, M. R., Chien, J. C. W. & Marques, M. M. (2005). *Polymer*, **46**, 2122–2132.

supporting information

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Crystal structure of 2,3-bis[(4-*tert*-butyl-2,6-dimethylphenyl)imino]butane

Sheng-Lan Zhao, Jian-Chao Yuan and Yan Zhao

S1. Introduction

Since Brookhart and co-workers discovered Ni^{II} and Pd^{II} aryl-substituted α-diimine complexes for olefin polymerization (Cotts *et al.*, 2000; Gates *et al.*, 2000; Johnson *et al.*, 1995; Meinhard *et al.*, 2007; Mecking *et al.*, 1998), late transition metal catalysts have attracted increseaing attention from their high functionality. It is well known that the ligand structure had significant influence on the product properties and polymerization activities (Ittel *et al.*, 2000; Yuan *et al.*, 2005). In this study, we designed and synthesized the title compound as a bidentate ligand, and its molecular structure was characterized by X-ray diffraction. In the crystal structure of the title compound, $C_{28}H_{40}N_2$, the complete molecule is generated by the application of C_2 symmetry. The single bond of 1,4-diazabutadiene fragment is (E)-configured. The dihedral angle between the benzene ring and 1,4-diazabutadiene plane is 89.8 (9)°.

S2. Synthesis and crystallization

Formic acid (0.2 ml) was added to a stirred solution of butane-2,3-dione (0.09 g, 1.00 mmol) and 4-(*tert*-butyl)-2,6-dimethylaniline (0.39 g, 2.2 mmol) in ethanol (10 ml). The mixture was refluxed for 24 h, then cooled and the precipitate was separated by filtration. The solid was recrystallized from MeOH/CH_2Cl_2 (v/v = 10:1), than washed with cold ethanol and dried under vacuum (0.35 g, 87% yield). Anal. Calc. for $C_{28}H_{40}N_2$: C, 83.11; H, 9.96; N, 6.92. Found: C, 83.23; H, 10.03; N, 6.89.

S3. Refinement

All hydrogen atoms were placed in calculated positions with C—H distances of 0.93Å and 0.96Å for aryl and methyl H atoms. They were included in the refinement in a riding model approximation, respectively. The H atoms were assigned U_{iso} = 1.2U_{eq}(C) for aryl H and U_{iso} = 1.5U_{eq}(C) for methyl H.

S4. Results and discussion

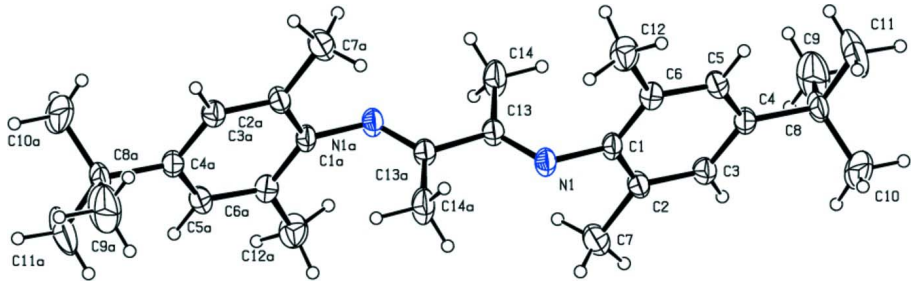

Figure 1

Molecular structure of the title compound, using 30% probability level ellipsoids (the hydrogens have been omitted for clarity). Primed atoms are related by the symmetry code (-x+1, -y+2, -z+1).

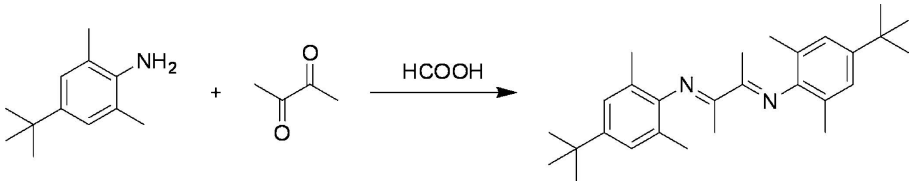

Figure 2

Synthesis of 2,3-bis{[4-(*tert*-butyl)-2,6-dimethyl]imino}butane.

4-*tert*-Butyl-*N*-{3-[(4-*tert*-butyl-2,6-dimethylphenyl)imino]butan-2-ylidene}-2,6-dimethylaniline

Crystal data

$C_{28}H_{40}N_2$
$M_r = 404.62$
Triclinic, $P\overline{1}$
Hall symbol: -P 1
$a = 5.993$ (6) Å
$b = 10.064$ (9) Å
$c = 11.614$ (11) Å
$\alpha = 107.913$ (9)°
$\beta = 100.484$ (10)°
$\gamma = 99.260$ (9)°
$V = 637.5$ (10) Å^3
$Z = 1$
$F(000) = 222$
$D_x = 1.054$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 1229 reflections
$\theta = 2.4$–$28.3°$
$\mu = 0.06$ mm^{-1}
$T = 296$ K
Block, yellow
0.23 × 0.21 × 0.18 mm

Data collection

Bruker APEXII CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2008)
$T_{min} = 0.986$, $T_{max} = 0.989$
4557 measured reflections
2310 independent reflections
1348 reflections with $I > 2\sigma(I)$
$R_{int} = 0.030$
$\theta_{max} = 25.5°$, $\theta_{min} = 2.4°$
$h = -7 \rightarrow 7$
$k = -12 \rightarrow 11$
$l = -14 \rightarrow 13$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.102$
$wR(F^2) = 0.220$
$S = 1.05$
2310 reflections
142 parameters
42 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.040P)^2 + 1.2419P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.52$ e Å^{-3}
$\Delta\rho_{min} = -0.21$ e Å^{-3}

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 (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
C1	0.4381 (7)	0.6834 (4)	0.3770 (4)	0.0394 (10)
C2	0.2730 (7)	0.6166 (4)	0.2638 (4)	0.0434 (11)
C3	0.2889 (7)	0.4854 (4)	0.1863 (4)	0.0439 (11)
H3	0.1786	0.4407	0.1104	0.053*
C4	0.4620 (7)	0.4180 (4)	0.2170 (4)	0.0413 (11)
C5	0.6199 (8)	0.4869 (4)	0.3302 (4)	0.0466 (11)
H5	0.7373	0.4430	0.3531	0.056*
C6	0.6132 (8)	0.6185 (4)	0.4121 (4)	0.0431 (11)
C7	0.0812 (9)	0.6857 (5)	0.2255 (5)	0.0642 (15)
H7A	−0.0024	0.7076	0.2894	0.096*
H7B	−0.0239	0.6209	0.1487	0.096*
H7C	0.1475	0.7725	0.2138	0.096*
C8	0.4746 (9)	0.2729 (4)	0.1263 (4)	0.0540 (13)
C9	0.5397 (13)	0.2937 (6)	0.0139 (6)	0.107 (2)
H9A	0.5464	0.2030	−0.0431	0.160*
H9B	0.6895	0.3591	0.0387	0.160*
H9C	0.4250	0.3322	−0.0264	0.160*
C10	0.2528 (12)	0.1679 (6)	0.0939 (8)	0.138 (3)
H10A	0.1292	0.2030	0.0567	0.208*
H10B	0.2225	0.1533	0.1680	0.208*
H10C	0.2614	0.0785	0.0357	0.208*
C11	0.6673 (13)	0.2145 (6)	0.1849 (6)	0.112 (2)
H11A	0.6383	0.2030	0.2603	0.167*
H11B	0.8149	0.2805	0.2039	0.167*
H11C	0.6703	0.1233	0.1274	0.167*
C12	0.7936 (9)	0.6874 (5)	0.5336 (4)	0.0648 (15)
H12A	0.9289	0.7412	0.5211	0.097*
H12B	0.8363	0.6146	0.5645	0.097*
H12C	0.7313	0.7507	0.5930	0.097*
C13	0.5168 (8)	0.9330 (4)	0.4552 (4)	0.0404 (10)
C14	0.6647 (10)	0.9478 (5)	0.3680 (5)	0.0671 (16)
H14A	0.6877	0.8553	0.3238	0.101*
H14B	0.8131	1.0115	0.4145	0.101*
H14C	0.5890	0.9860	0.3095	0.101*
N1	0.4167 (6)	0.8151 (3)	0.4603 (3)	0.0440 (10)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.052 (3)	0.026 (2)	0.042 (2)	0.0086 (19)	0.023 (2)	0.0084 (18)

C2	0.050 (3)	0.034 (2)	0.051 (3)	0.016 (2)	0.020 (2)	0.013 (2)
C3	0.047 (3)	0.037 (2)	0.044 (3)	0.010 (2)	0.010 (2)	0.009 (2)
C4	0.049 (3)	0.031 (2)	0.047 (3)	0.010 (2)	0.019 (2)	0.0123 (19)
C5	0.050 (3)	0.037 (2)	0.054 (3)	0.018 (2)	0.013 (2)	0.013 (2)
C6	0.051 (3)	0.036 (2)	0.043 (3)	0.009 (2)	0.013 (2)	0.013 (2)
C7	0.063 (3)	0.053 (3)	0.072 (4)	0.026 (3)	0.012 (3)	0.013 (3)
C8	0.068 (3)	0.033 (2)	0.061 (3)	0.021 (2)	0.029 (2)	0.005 (2)
C9	0.164 (6)	0.077 (4)	0.080 (4)	0.040 (4)	0.058 (4)	0.008 (3)
C10	0.107 (5)	0.049 (3)	0.198 (7)	−0.008 (3)	0.068 (5)	−0.046 (4)
C11	0.148 (6)	0.067 (4)	0.108 (5)	0.068 (4)	0.018 (4)	0.000 (3)
C12	0.072 (4)	0.054 (3)	0.052 (3)	0.012 (3)	−0.002 (3)	0.007 (2)
C13	0.050 (3)	0.032 (2)	0.042 (2)	0.0111 (19)	0.018 (2)	0.0119 (18)
C14	0.102 (4)	0.035 (2)	0.074 (3)	0.017 (3)	0.053 (3)	0.013 (2)
N1	0.057 (2)	0.0297 (19)	0.047 (2)	0.0110 (17)	0.0237 (18)	0.0097 (16)

Geometric parameters (Å, º)

C1—C2	1.388 (6)	C9—H9A	0.9600
C1—C6	1.386 (6)	C9—H9B	0.9600
C1—N1	1.421 (5)	C9—H9C	0.9600
C2—C3	1.379 (5)	C10—H10A	0.9600
C2—C7	1.505 (6)	C10—H10B	0.9600
C3—C4	1.380 (6)	C10—H10C	0.9600
C3—H3	0.9300	C11—H11A	0.9600
C4—C5	1.372 (6)	C11—H11B	0.9600
C4—C8	1.538 (5)	C11—H11C	0.9600
C5—C6	1.384 (6)	C12—H12A	0.9600
C5—H5	0.9300	C12—H12B	0.9600
C6—C12	1.497 (6)	C12—H12C	0.9600
C7—H7A	0.9600	C13—N1	1.264 (5)
C7—H7B	0.9600	C13—C14	1.486 (6)
C7—H7C	0.9600	C13—C13^{i}	1.497 (7)
C8—C10	1.465 (7)	C14—H14A	0.9600
C8—C9	1.493 (7)	C14—H14B	0.9600
C8—C11	1.523 (7)	C14—H14C	0.9600
C2—C1—C6	120.6 (3)	H9A—C9—H9B	109.5
C2—C1—N1	119.1 (4)	C8—C9—H9C	109.5
C6—C1—N1	120.2 (4)	H9A—C9—H9C	109.5
C1—C2—C3	118.6 (4)	H9B—C9—H9C	109.5
C1—C2—C7	121.0 (4)	C8—C10—H10A	109.5
C3—C2—C7	120.4 (4)	C8—C10—H10B	109.5
C4—C3—C2	122.6 (4)	H10A—C10—H10B	109.5
C4—C3—H3	118.7	C8—C10—H10C	109.5
C2—C3—H3	118.7	H10A—C10—H10C	109.5
C5—C4—C3	116.9 (4)	H10B—C10—H10C	109.5
C5—C4—C8	122.5 (4)	C8—C11—H11A	109.5
C3—C4—C8	120.6 (4)	C8—C11—H11B	109.5

C4—C5—C6	123.2 (4)	H11A—C11—H11B	109.5
C4—C5—H5	118.4	C8—C11—H11C	109.5
C6—C5—H5	118.4	H11A—C11—H11C	109.5
C1—C6—C5	118.0 (4)	H11B—C11—H11C	109.5
C1—C6—C12	122.0 (4)	C6—C12—H12A	109.5
C5—C6—C12	120.0 (4)	C6—C12—H12B	109.5
C2—C7—H7A	109.5	H12A—C12—H12B	109.5
C2—C7—H7B	109.5	C6—C12—H12C	109.5
H7A—C7—H7B	109.5	H12A—C12—H12C	109.5
C2—C7—H7C	109.5	H12B—C12—H12C	109.5
H7A—C7—H7C	109.5	N1—C13—C14	124.9 (4)
H7B—C7—H7C	109.5	N1—C13—C13^{i}	116.8 (5)
C10—C8—C9	112.2 (6)	C14—C13—C13^{i}	118.2 (5)
C10—C8—C11	108.4 (5)	C13—C14—H14A	109.5
C9—C8—C11	105.7 (5)	C13—C14—H14B	109.5
C10—C8—C4	110.2 (4)	H14A—C14—H14B	109.5
C9—C8—C4	109.4 (4)	C13—C14—H14C	109.5
C11—C8—C4	110.9 (4)	H14A—C14—H14C	109.5
C8—C9—H9A	109.5	H14B—C14—H14C	109.5
C8—C9—H9B	109.5	C13—N1—C1	120.1 (3)

C6—C1—C2—C3	−0.9 (6)	N1—C1—C6—C12	−3.9 (6)
N1—C1—C2—C3	−177.0 (4)	C4—C5—C6—C1	−0.2 (6)
C6—C1—C2—C7	179.5 (4)	C4—C5—C6—C12	−179.4 (4)
N1—C1—C2—C7	3.5 (6)	C5—C4—C8—C10	125.0 (6)
C1—C2—C3—C4	0.1 (6)	C3—C4—C8—C10	−55.4 (7)
C7—C2—C3—C4	179.7 (4)	C5—C4—C8—C9	−111.2 (5)
C2—C3—C4—C5	0.6 (6)	C3—C4—C8—C9	68.3 (6)
C2—C3—C4—C8	−179.0 (4)	C5—C4—C8—C11	5.0 (6)
C3—C4—C5—C6	−0.5 (6)	C3—C4—C8—C11	−175.5 (5)
C8—C4—C5—C6	179.0 (4)	C14—C13—N1—C1	−1.2 (7)
C2—C1—C6—C5	0.9 (6)	C13^{i}—C13—N1—C1	179.2 (5)
N1—C1—C6—C5	176.9 (4)	C2—C1—N1—C13	−91.4 (5)
C2—C1—C6—C12	−179.9 (4)	C6—C1—N1—C13	92.6 (5)

Symmetry code: (i) −*x*+1, −*y*+2, −*z*+1.