Acta Crystallographica Section E
Structure Reports
Online

ISSN 1600-5368

(Z)-N-[(Z)-3-(2,4-Dimethylphenylimino)-butan-2-ylidene]-2,4-dimethylaniline

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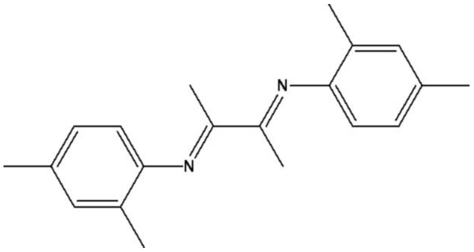
Received 15 November 2011; accepted 10 December 2011

Key indicators: single-crystal X-ray study; T = 296 K; mean σ(C–C) = 0.003 Å; R factor = 0.052; wR factor = 0.175; data-to-parameter ratio = 15.3.

The asymmetric unit of the title compound, $C_{20}H_{24}N_2$, contains one half -molecule which exhibits a crystallographically imposed center of symmetry. The benzene rings are inclined to the 1,4-diazabutadiene mean plane by 78.3 (2)°.

Related literature

The title compound was synthesized as a α-diimine ligand for Ni^{II}-α-diimine olefin polymerization catalysts. For applications of α-diimine ligands, see: Johnson *et al.* (1995); Killian *et al.* (1996). For the design and synthesis of new α-diimine derivatives, see: Yuan *et al.* (2005); Popeney & Guan (2005, 2010); Popeney *et al.* (2011). The crystal structures of Re and Ni complexes with the title ligand were reported by Kia *et al.* (2005) and Yuan *et al.* (2011), respectively.



Experimental

Crystal data

$C_{20}H_{24}N_2$
M_r = 292.41
Orthorhombic, $Pbca$
a = 13.50 (1) Å
b = 7.571 (6) Å
c = 16.738 (12) Å
V = 1711 (2) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.07 mm^{-1}
T = 296 K
0.23 × 0.20 × 0.14 mm

Data collection

Bruker APEXII CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2008)
T_{min} = 0.985, T_{max} = 0.991
5143 measured reflections
1592 independent reflections
1043 reflections with $I > 2\sigma(I)$
R_{int} = 0.031

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.052
$wR(F^2)$ = 0.175
S = 1.05
1592 reflections
104 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.21 e Å^{-3}
$\Delta\rho_{min}$ = −0.15 e Å^{-3}

Data collection: *APEX2* (Bruker, 2008); cell refinement: *SAINT* (Bruker, 2008); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXTL*.

We thank the National Natural Science Foundation of China (grant No. 20964003) for funding. We also thank the Key Laboratory of Eco Environment-Related Polymer Materials of the Ministry of Education and Key Laboratory of Polymer Materials of Gansu Province (Northwest Normal University) for financial support.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: CV5204).

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supporting information

Acta Cryst. (2012). E**68**, o164 [doi:10.1107/S1600536811053244]

(*Z*)-*N*-[(*Z*)-3-(2,4-Dimethylphenylimino)butan-2-ylidene]-2,4-dimethylaniline

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S1. Comment

α-Diimine ligand nickel catalysts greatly attracted attention due to their high catalytic activity in ethylene polymerization (Johnson *et al.*, 1995; Killian *et al.*, 1996). Design and synthesis of the ligands is crucial (Popeney *et al.*, 2005, 2010, 2011; Yuan *et al.*, 2005). Herewith we present the title compound (I).

In (I) (Fig. 1), the single C—C bond in 1,4-diazabutadiene fragment is *trans*-configured and situated on inversion center. The dihedral angle between the benzene ring and 1,4-diazabutadiene plane is 78.3 (2)°. However, the *trans*-configured ligand can be transformed into *cis*-configured ligand in order to facilitate the formation of *α*-diimine-metal complexes, for examples, see Yuan *et al.* (2011) for Ni complex, and Kia *et al.* (2005) for Re complex.

S2. Experimental

Formic acid (1 ml) was added to a stirred solution of 2,3-butanedione (0.052 g, 0.6 mmol) and 2,4-dimethylaniline (0.144 g, 1.2 mmol) in methanol (30 ml). The mixture was refluxed for 24 h, then cooled and the precipitate was separated by filtration. The solid was recrystallized from ethanol/dichloromethane (*v*/*v* = 8:1), washed and dried under vacuum. Yield: 0.160 g (82%). Crystals suitable for X-ray structure determination were grown from a solution of the title compound in a mixture of cyclohexane/dichloromethane (1:2, *v*/*v*).

S3. Refinement

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

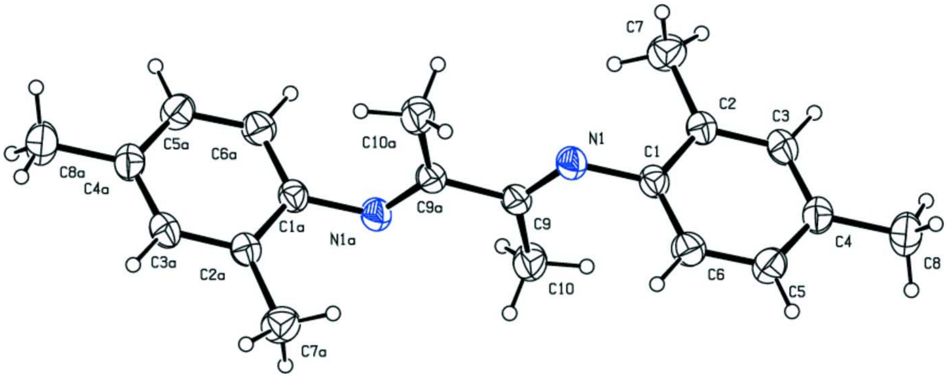

Figure 1

The molecular structure of the title compound, with the atom-labelling scheme [symmetry code: (*a*) 1 - *x*, 2 - *y*, 1 - *z*]. Displacement ellipsoids are shown at the 30% probability level.

(*Z*)-*N*-[(*Z*)-3-(2,4-Dimethylphenylimino)butan-2-ylidene]- 2,4-dimethylaniline

Crystal data

$C_{20}H_{24}N_2$
$M_r = 292.41$
Orthorhombic, *Pbca*
$a = 13.50$ (1) Å
$b = 7.571$ (6) Å
$c = 16.738$ (12) Å
$V = 1711$ (2) Å^3
$Z = 4$
$F(000) = 632$
$D_x = 1.135$ Mg m^{-3}
Mo *Kα* radiation, $\lambda = 0.71073$ Å
Cell parameters from 1144 reflections
$\theta = 2.9$–23.2°
$\mu = 0.07$ mm^{-1}
$T = 296$ K
Block, yellow
0.23 × 0.20 × 0.14 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)
$T_{min} = 0.985$, $T_{max} = 0.991$
5143 measured reflections
1592 independent reflections
1043 reflections with $I > 2\sigma(I)$
$R_{int} = 0.031$
$\theta_{max} = 25.5°$, $\theta_{min} = 2.4°$
$h = -8 \rightarrow 16$
$k = -6 \rightarrow 9$
$l = -16 \rightarrow 20$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.052$
$wR(F^2) = 0.175$
$S = 1.05$
1592 reflections
104 parameters
0 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
$w = 1/[\sigma^2(F_o^2) + (0.0962P)^2 + 0.2091P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.21$ e Å^{-3}
$\Delta\rho_{min} = -0.15$ e Å^{-3}
Extinction correction: *SHELXL97* (Sheldrick, 2008), $Fc^* = kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.009 (4)

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 (Å^2)

	x	*y*	*z*	$U_{iso}*/U_{eq}$
C1	0.60082 (15)	0.9674 (3)	0.65918 (13)	0.0471 (6)
C2	0.69975 (15)	0.9165 (3)	0.66744 (13)	0.0464 (6)
C3	0.72926 (16)	0.8473 (3)	0.74028 (13)	0.0528 (6)

H3	0.7946	0.8104	0.7460	0.063*
C4	0.66613 (18)	0.8305 (3)	0.80502 (13)	0.0548 (6)
C5	0.56961 (18)	0.8859 (3)	0.79551 (14)	0.0596 (7)
H5	0.5257	0.8782	0.8382	0.072*
C6	0.53728 (17)	0.9527 (3)	0.72328 (15)	0.0579 (7)
H6	0.4717	0.9882	0.7178	0.070*
C7	0.77174 (18)	0.9360 (3)	0.59996 (15)	0.0669 (8)
H7A	0.7403	0.9026	0.5508	0.100*
H7B	0.8280	0.8612	0.6092	0.100*
H7C	0.7931	1.0567	0.5966	0.100*
C8	0.7013 (2)	0.7515 (4)	0.88279 (14)	0.0795 (9)
H8A	0.7642	0.6951	0.8747	0.119*
H8B	0.6540	0.6659	0.9011	0.119*
H8C	0.7081	0.8432	0.9220	0.119*
C9	0.51658 (15)	0.9537 (3)	0.53705 (12)	0.0467 (6)
C10	0.48786 (19)	0.7641 (3)	0.54753 (15)	0.0662 (7)
H10A	0.5191	0.7177	0.5946	0.099*
H10B	0.5088	0.6976	0.5017	0.099*
H10C	0.4172	0.7555	0.5530	0.099*
N1	0.56812 (12)	1.0428 (2)	0.58600 (11)	0.0516 (6)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0501 (13)	0.0426 (12)	0.0487 (13)	−0.0029 (9)	−0.0084 (10)	0.0010 (10)
C2	0.0488 (13)	0.0429 (12)	0.0476 (13)	0.0017 (9)	−0.0048 (9)	−0.0027 (10)
C3	0.0475 (12)	0.0526 (13)	0.0583 (14)	0.0050 (10)	−0.0116 (10)	0.0000 (11)
C4	0.0658 (15)	0.0504 (14)	0.0483 (14)	−0.0026 (12)	−0.0108 (11)	0.0014 (11)
C5	0.0630 (15)	0.0636 (16)	0.0523 (14)	0.0035 (12)	0.0046 (11)	0.0049 (12)
C6	0.0482 (12)	0.0610 (16)	0.0646 (15)	0.0054 (11)	−0.0008 (11)	0.0083 (12)
C7	0.0624 (15)	0.0685 (17)	0.0697 (16)	0.0067 (12)	0.0087 (12)	0.0054 (13)
C8	0.0911 (19)	0.089 (2)	0.0584 (16)	−0.0039 (15)	−0.0191 (14)	0.0118 (15)
C9	0.0401 (11)	0.0499 (14)	0.0501 (13)	0.0004 (9)	−0.0031 (9)	0.0045 (10)
C10	0.0783 (17)	0.0551 (15)	0.0652 (16)	−0.0118 (12)	−0.0161 (12)	0.0118 (12)
N1	0.0503 (11)	0.0505 (11)	0.0540 (12)	−0.0017 (8)	−0.0086 (9)	0.0083 (9)

Geometric parameters (Å, º)

C1—C6	1.378 (3)	C7—H7A	0.9600
C1—C2	1.397 (3)	C7—H7B	0.9600
C1—N1	1.421 (3)	C7—H7C	0.9600
C2—C3	1.385 (3)	C8—H8A	0.9600
C2—C7	1.497 (3)	C8—H8B	0.9600
C3—C4	1.384 (3)	C8—H8C	0.9600
C3—H3	0.9300	C9—N1	1.269 (3)
C4—C5	1.378 (3)	C9—C9$^{\rm i}$	1.494 (4)
C4—C8	1.509 (3)	C9—C10	1.497 (3)
C5—C6	1.381 (3)	C10—H10A	0.9600

C5—H5	0.9300	C10—H10B	0.9600
C6—H6	0.9300	C10—H10C	0.9600

C6—C1—C2	119.7 (2)	H7A—C7—H7B	109.5
C6—C1—N1	120.67 (19)	C2—C7—H7C	109.5
C2—C1—N1	119.5 (2)	H7A—C7—H7C	109.5
C3—C2—C1	117.8 (2)	H7B—C7—H7C	109.5
C3—C2—C7	121.0 (2)	C4—C8—H8A	109.5
C1—C2—C7	121.2 (2)	C4—C8—H8B	109.5
C4—C3—C2	123.1 (2)	H8A—C8—H8B	109.5
C4—C3—H3	118.4	C4—C8—H8C	109.5
C2—C3—H3	118.4	H8A—C8—H8C	109.5
C5—C4—C3	117.6 (2)	H8B—C8—H8C	109.5
C5—C4—C8	121.2 (2)	N1—C9—C9^{i}	116.8 (2)
C3—C4—C8	121.2 (2)	N1—C9—C10	125.18 (19)
C4—C5—C6	120.7 (2)	C9^{i}—C9—C10	118.0 (2)
C4—C5—H5	119.6	C9—C10—H10A	109.5
C6—C5—H5	119.6	C9—C10—H10B	109.5
C1—C6—C5	121.0 (2)	H10A—C10—H10B	109.5
C1—C6—H6	119.5	C9—C10—H10C	109.5
C5—C6—H6	119.5	H10A—C10—H10C	109.5
C2—C7—H7A	109.5	H10B—C10—H10C	109.5
C2—C7—H7B	109.5	C9—N1—C1	120.87 (19)

C6—C1—C2—C3	1.9 (3)	C8—C4—C5—C6	−177.9 (2)
N1—C1—C2—C3	178.55 (19)	C2—C1—C6—C5	−0.9 (4)
C6—C1—C2—C7	−178.0 (2)	N1—C1—C6—C5	−177.5 (2)
N1—C1—C2—C7	−1.4 (3)	C4—C5—C6—C1	−0.7 (4)
C1—C2—C3—C4	−1.5 (3)	C9^{i}—C9—N1—C1	178.3 (2)
C7—C2—C3—C4	178.4 (2)	C10—C9—N1—C1	−2.4 (3)
C2—C3—C4—C5	0.0 (3)	C6—C1—N1—C9	−78.8 (3)
C2—C3—C4—C8	179.0 (2)	C2—C1—N1—C9	104.6 (2)
C3—C4—C5—C6	1.1 (4)		

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