

Acta Crystallographica Section E

Structure Reports

Online

ISSN 1600-5368

2,3-Bis[(3-methylbiphenyl-4-yl)imino]-butane

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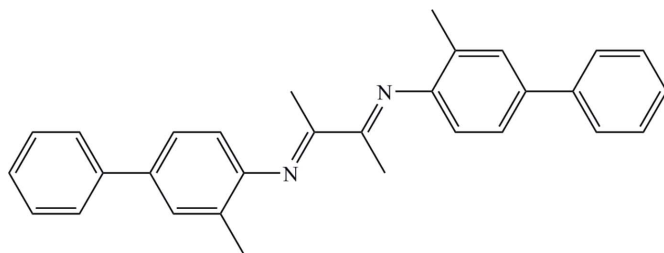
Received 3 March 2014; accepted 13 March 2014

Key indicators: single-crystal X-ray study; $T = 293$ K; mean $\sigma(\text{C}-\text{C}) = 0.004$ Å; R factor = 0.069; wR factor = 0.135; data-to-parameter ratio = 14.9.

The title compound, $\text{C}_{30}\text{H}_{28}\text{N}_2$, is a product of the condensation reaction of 2-methyl-4-phenylaniline and butane-2,3-dione. The molecule lies on a crystallographic inversion centre. The $\text{C}=\text{N}$ bond has an *E* conformation. The dihedral angle between the two benzene rings of the 4-phenyl-2-methylphenyl group is 29.19 (76°). The 1,4-diazabutadiene plane makes an angle of 70.1 (10°) with the N-bonded methylphenyl ring and an angle of 81.08 (97°) with the terminal phenyl group.

Related literature

The title compound was synthesized as an α -diimine ligand for applications in olefin polymerization Ni(II)- α -diimine catalysts, see: Johnson *et al.* (1995); Killian *et al.* (1996); Wang *et al.* (2013); Ionkin & Marshall (2004); Meinhard *et al.* (2007). For the effect of the ligand structure on the activity of the catalyst and the properties of the products, see: Popeney & Guan (2005); Yuan *et al.* (2005); Helldörfer *et al.* (2003). For related structures, see: Yuan *et al.* (2013).



Experimental

Crystal data

$\text{C}_{30}\text{H}_{28}\text{N}_2$
 $M_r = 416.54$
 Orthorhombic, *Pbca*
 $a = 8.347$ (3) Å
 $b = 7.063$ (3) Å
 $c = 39.946$ (16) Å

$V = 2355.0$ (16) Å³
 $Z = 4$
 Mo $K\alpha$ radiation
 $\mu = 0.07$ mm⁻¹
 $T = 293$ K
 $0.19 \times 0.18 \times 0.15$ mm

Data collection

Bruker APEXII CCD diffractometer
 Absorption correction: multi-scan (*SADABS*; Sheldrick, 2004)
 $T_{\min} = 0.987$, $T_{\max} = 0.990$

15668 measured reflections
 2200 independent reflections
 1251 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.088$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.069$
 $wR(F^2) = 0.135$
 $S = 1.01$
 2200 reflections

148 parameters
 H-atom parameters constrained
 $\Delta\rho_{\text{max}} = 0.15$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.16$ 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*.

We thank the National Natural Science Foundation of China (20964003) for funding. We also thank the Key Laboratory of Eco Environment-Related Polymer Materials of the Ministry of Education, Key Laboratory of Polymer Materials of Gansu Province (Northwest Normal University), for financial support.

Supporting information for this paper is available from the IUCr electronic archives (Reference: FK2080).

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supporting information

Acta Cryst. (2014). E70, o455 [doi:10.1107/S1600536814005686]

2,3-Bis[(3-methylbiphenyl-4-yl)imino]butane

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S1. Comment

In recent years, a variety of α -diimine ligands containing various *ortho* and *para* position substituted *N*-aryl rings (Johnson *et al.* 1995; Killian *et al.* 1996; Popeney *et al.* 2005; Yuan *et al.*, 2005; Wang *et al.* 2013) and backbone effects (Helldörfer *et al.* 2003) and teraryl substituted- α -diimine ligands (Ionkin *et al.* 2004; Meinhard *et al.* 2007) were employed to study their influence on the catalytic activity of α -diimine-Ni(II) complexes. In this study, we designed and synthesized the title compound as a bidentate ligand. The molecule lies on a crystallographic inversion centre. The single bond of 1,4-diazabutadiene fragment is (E)-configured. The dihedral angles between the 1,4-diazabutadiene plane and the benzene ring bonded to the N atom are 70.12 (96) $^\circ$ and 81.08 (97) $^\circ$ for the terminal phenyl group, resp. The dihedral angle between both aromatic ring planes is 29.19 (76) $^\circ$ (Figure 1). The crystal packing shows stacking of molecules along *a*-axis (Figure 2), however, no significant intermolecular H-bonding is observed. A very similar molecular structure is known from Yuan *et al.* (2013).

S2. Experimental

Formic acid (0.5 ml) was added to a stirred solution of 2-methyl-4-phenylaniline (0.916 g, 2.2 mmol) and 2,3-Butanedione (0.086 g, 1 mmol) in 20 ml anhydrous ethanol (20 ml). The mixture was stirred at 50 $^\circ\text{C}$ for 24 h, then cooled, and the precipitate was separated by filtration. The solid was recrystallized from ethanol/dichloromethane ($v/v=8:1$), washed with cold ethanol and dried under vacuum to give the title compound. Yield is 86%. Crystals suitable for X-ray structure determination were grown from a cyclohexane/dichloromethane ($v:v=1:2$) solution. Anal. Calc. for $\text{C}_{30}\text{H}_{28}\text{N}_2$: C, 86.50; H, 6.78; N, 6.72. Found: C, 86.62; H, 6.57; N, 6.58.

S3. Refinement

Positions of the methyl H atoms were derived from Fourier maps (HFIX 137), with C–H 0.96 Å and $U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{C})$. All other H atoms were placed in geometrically idealized positions and constrained to ride on their parent atoms with C–H distances distances of 0.93 Å and $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$.

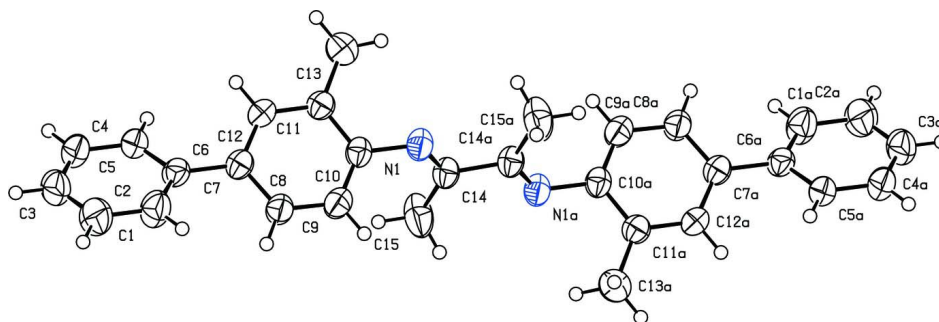


Figure 1

The title molecule with displacement ellipsoids plotted at 50% probability level. Atoms with label "a" are related by the symmetry code $(-x+1, -y+1, -z)$.

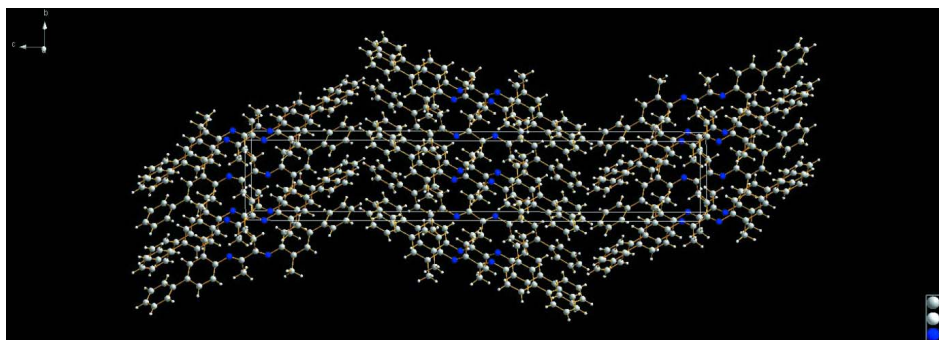


Figure 2

Crystal packing viewed along the a -axis.

2,3-Bis[(3-methylbiphenyl-4-yl)imino]butane

Crystal data

$C_{30}H_{28}N_2$

$M_r = 416.54$

Orthorhombic, $Pbca$

$a = 8.347(3) \text{ \AA}$

$b = 7.063(3) \text{ \AA}$

$c = 39.946(16) \text{ \AA}$

$V = 2355.0(16) \text{ \AA}^3$

$Z = 4$

$F(000) = 888$

$D_x = 1.175 \text{ Mg m}^{-3}$

Mo $K\alpha$ radiation, $\lambda = 0.71073 \text{ \AA}$

Cell parameters from 1674 reflections

$\theta = 2.6\text{--}21.7^\circ$

$\mu = 0.07 \text{ mm}^{-1}$

$T = 293 \text{ K}$

Block, yellow

$0.19 \times 0.18 \times 0.15 \text{ mm}$

Data collection

Bruker APEXII CCD

diffractometer

Radiation source: fine-focus sealed tube

Graphite monochromator

φ and ω scans

Absorption correction: multi-scan

(*SADABS*; Sheldrick, 2004)

$T_{\min} = 0.987$, $T_{\max} = 0.990$

15668 measured reflections

2200 independent reflections

1251 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.088$

$\theta_{\max} = 25.5^\circ$, $\theta_{\min} = 2.6^\circ$

$h = -10 \rightarrow 10$

$k = -8 \rightarrow 8$

$l = -48 \rightarrow 47$

Refinement

Refinement on F^2
 Least-squares matrix: full
 $R[F^2 > 2\sigma(F^2)] = 0.069$
 $wR(F^2) = 0.135$
 $S = 1.01$
 2200 reflections
 148 parameters
 0 restraints
 Primary atom site location: structure-invariant
 direct methods

Secondary atom site location: difference Fourier
 map
 Hydrogen site location: difference Fourier map
 H-atom parameters constrained
 $w = 1/[\sigma^2(F_o^2) + (0.0366P)^2 + 1.4273P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} < 0.001$
 $\Delta\rho_{\max} = 0.15 \text{ e } \text{\AA}^{-3}$
 $\Delta\rho_{\min} = -0.16 \text{ e } \text{\AA}^{-3}$
 Extinction correction: *SHELXTL* (Sheldrick,
 2008), $F_c^* = kF_c[1 + 0.001x F_c^2 \lambda^3 / \sin(2\theta)]^{-1/4}$
 Extinction coefficient: 0.0025 (6)

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)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
C1	0.6218 (4)	-0.0240 (5)	0.17712 (7)	0.0644 (9)
H1	0.7117	-0.0468	0.1640	0.077*
C2	0.6029 (4)	-0.1219 (5)	0.20680 (8)	0.0716 (10)
H2	0.6792	-0.2106	0.2133	0.086*
C3	0.4725 (4)	-0.0893 (5)	0.22677 (7)	0.0663 (9)
H3	0.4597	-0.1548	0.2468	0.080*
C4	0.3612 (4)	0.0413 (4)	0.21680 (7)	0.0594 (9)
H4	0.2726	0.0652	0.2303	0.071*
C5	0.3788 (4)	0.1375 (4)	0.18713 (6)	0.0504 (8)
H5	0.3012	0.2248	0.1807	0.061*
C6	0.5099 (3)	0.1070 (4)	0.16654 (6)	0.0442 (7)
C7	0.5276 (3)	0.2107 (4)	0.13451 (6)	0.0425 (7)
C8	0.6072 (3)	0.1298 (4)	0.10744 (7)	0.0509 (8)
H8	0.6563	0.0124	0.1099	0.061*
C9	0.6142 (3)	0.2216 (4)	0.07704 (7)	0.0532 (8)
H9	0.6678	0.1652	0.0592	0.064*
C10	0.5427 (3)	0.3961 (4)	0.07266 (6)	0.0423 (7)
C11	0.4688 (3)	0.4856 (4)	0.09956 (6)	0.0432 (7)
C12	0.4629 (3)	0.3887 (4)	0.12980 (6)	0.0443 (7)
H12	0.4126	0.4468	0.1479	0.053*
C13	0.3949 (4)	0.6774 (4)	0.09594 (8)	0.0674 (10)
H13A	0.2882	0.6648	0.0873	0.101*
H13B	0.4580	0.7522	0.0808	0.101*

H13C	0.3912	0.7384	0.1174	0.101*
C14	0.4850 (3)	0.4425 (4)	0.01552 (6)	0.0440 (7)
C15	0.3729 (4)	0.2792 (5)	0.01278 (8)	0.0773 (11)
H15A	0.3722	0.2102	0.0335	0.116*
H15B	0.4073	0.1972	-0.0050	0.116*
H15C	0.2670	0.3248	0.0081	0.116*
N1	0.5572 (3)	0.4954 (3)	0.04174 (5)	0.0486 (6)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.068 (2)	0.078 (3)	0.0472 (19)	0.013 (2)	0.0012 (16)	0.0083 (18)
C2	0.086 (3)	0.075 (2)	0.054 (2)	0.016 (2)	-0.0096 (19)	0.012 (2)
C3	0.092 (3)	0.064 (2)	0.0429 (18)	-0.005 (2)	-0.0080 (19)	0.0083 (17)
C4	0.075 (2)	0.063 (2)	0.0400 (17)	-0.0046 (19)	0.0066 (16)	-0.0005 (16)
C5	0.0651 (19)	0.0476 (19)	0.0386 (17)	-0.0013 (16)	0.0034 (14)	0.0006 (15)
C6	0.0502 (17)	0.0475 (18)	0.0349 (15)	0.0018 (15)	-0.0037 (13)	-0.0025 (14)
C7	0.0388 (15)	0.0504 (19)	0.0381 (16)	-0.0011 (15)	-0.0014 (13)	-0.0022 (14)
C8	0.0587 (18)	0.0529 (19)	0.0410 (17)	0.0119 (16)	0.0057 (14)	0.0047 (16)
C9	0.0593 (18)	0.058 (2)	0.0424 (18)	0.0049 (17)	0.0088 (15)	-0.0019 (16)
C10	0.0417 (15)	0.0495 (18)	0.0356 (16)	-0.0052 (15)	-0.0010 (13)	0.0013 (14)
C11	0.0430 (15)	0.0456 (17)	0.0410 (17)	-0.0007 (14)	-0.0023 (13)	-0.0011 (14)
C12	0.0474 (16)	0.0479 (18)	0.0377 (16)	0.0014 (15)	0.0034 (13)	-0.0058 (14)
C13	0.088 (2)	0.055 (2)	0.059 (2)	0.0099 (19)	0.0034 (18)	0.0063 (17)
C14	0.0404 (15)	0.0522 (19)	0.0395 (16)	-0.0047 (14)	0.0022 (13)	0.0024 (13)
C15	0.090 (2)	0.086 (3)	0.055 (2)	-0.041 (2)	-0.0109 (19)	0.0185 (19)
N1	0.0521 (14)	0.0561 (16)	0.0375 (13)	-0.0075 (12)	0.0012 (12)	0.0028 (12)

Geometric parameters (Å, °)

C1—C6	1.381 (4)	C9—C10	1.380 (4)
C1—C2	1.382 (4)	C9—H9	0.9300
C1—H1	0.9300	C10—C11	1.391 (3)
C2—C3	1.369 (4)	C10—N1	1.426 (3)
C2—H2	0.9300	C11—C12	1.389 (3)
C3—C4	1.368 (4)	C11—C13	1.496 (4)
C3—H3	0.9300	C12—H12	0.9300
C4—C5	1.374 (4)	C13—H13A	0.9600
C4—H4	0.9300	C13—H13B	0.9600
C5—C6	1.386 (4)	C13—H13C	0.9600
C5—H5	0.9300	C14—N1	1.265 (3)
C6—C7	1.481 (4)	C14—C15	1.489 (4)
C7—C12	1.381 (4)	C14—C14 ⁱ	1.504 (5)
C7—C8	1.392 (3)	C15—H15A	0.9600
C8—C9	1.378 (4)	C15—H15B	0.9600
C8—H8	0.9300	C15—H15C	0.9600
C6—C1—C2	121.4 (3)	C8—C9—H9	119.6

C6—C1—H1	119.3	C9—C10—C11	120.0 (3)
C2—C1—H1	119.3	C9—C10—N1	120.8 (2)
C3—C2—C1	120.4 (3)	C11—C10—N1	118.9 (3)
C3—C2—H2	119.8	C10—C11—C12	117.6 (3)
C1—C2—H2	119.8	C10—C11—C13	121.3 (3)
C4—C3—C2	118.9 (3)	C12—C11—C13	121.1 (3)
C4—C3—H3	120.5	C7—C12—C11	123.6 (3)
C2—C3—H3	120.5	C7—C12—H12	118.2
C3—C4—C5	120.8 (3)	C11—C12—H12	118.2
C3—C4—H4	119.6	C11—C13—H13A	109.5
C5—C4—H4	119.6	C11—C13—H13B	109.5
C4—C5—C6	121.3 (3)	H13A—C13—H13B	109.5
C4—C5—H5	119.4	C11—C13—H13C	109.5
C6—C5—H5	119.4	H13A—C13—H13C	109.5
C1—C6—C5	117.2 (3)	H13B—C13—H13C	109.5
C1—C6—C7	121.9 (3)	N1—C14—C15	126.1 (3)
C5—C6—C7	120.9 (3)	N1—C14—C14 ⁱ	116.4 (3)
C12—C7—C8	117.0 (3)	C15—C14—C14 ⁱ	117.5 (3)
C12—C7—C6	121.9 (3)	C14—C15—H15A	109.5
C8—C7—C6	121.1 (3)	C14—C15—H15B	109.5
C9—C8—C7	120.8 (3)	H15A—C15—H15B	109.5
C9—C8—H8	119.6	C14—C15—H15C	109.5
C7—C8—H8	119.6	H15A—C15—H15C	109.5
C10—C9—C8	120.9 (3)	H15B—C15—H15C	109.5
C10—C9—H9	119.6	C14—N1—C10	122.1 (2)
C6—C1—C2—C3	-0.8 (5)	C8—C9—C10—C11	3.0 (4)
C1—C2—C3—C4	0.2 (5)	C8—C9—C10—N1	176.7 (3)
C2—C3—C4—C5	0.5 (5)	C9—C10—C11—C12	-3.4 (4)
C3—C4—C5—C6	-0.6 (5)	N1—C10—C11—C12	-177.2 (2)
C2—C1—C6—C5	0.7 (5)	C9—C10—C11—C13	178.0 (3)
C2—C1—C6—C7	-179.0 (3)	N1—C10—C11—C13	4.2 (4)
C4—C5—C6—C1	0.0 (4)	C8—C7—C12—C11	2.3 (4)
C4—C5—C6—C7	179.7 (3)	C6—C7—C12—C11	-176.1 (3)
C1—C6—C7—C12	-152.1 (3)	C10—C11—C12—C7	0.7 (4)
C5—C6—C7—C12	28.3 (4)	C13—C11—C12—C7	179.4 (3)
C1—C6—C7—C8	29.6 (4)	C15—C14—N1—C10	2.3 (5)
C5—C6—C7—C8	-150.0 (3)	C14 ⁱ —C14—N1—C10	-178.4 (3)
C12—C7—C8—C9	-2.7 (4)	C9—C10—N1—C14	71.9 (4)
C6—C7—C8—C9	175.7 (3)	C11—C10—N1—C14	-114.4 (3)
C7—C8—C9—C10	0.1 (4)		

Symmetry code: (i) $-x+1, -y+1, -z$.