

Acta Crystallographica Section E

## Structure Reports

Online

ISSN 1600-5368

**(Z)-N-[(Z)-3-(2,5-Dimethylphenylimino)-butan-2-ylidene]-2,5-dimethylaniline**

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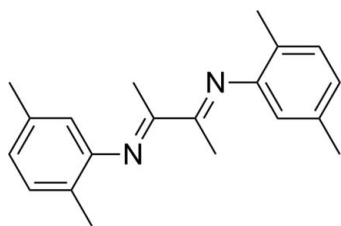
Received 23 December 2013; accepted 15 January 2014

Key indicators: single-crystal X-ray study;  $T = 296$  K; mean  $\sigma(\text{C}-\text{C}) = 0.003$  Å; R factor = 0.051;  $wR$  factor = 0.175; data-to-parameter ratio = 16.0.

The asymmetric unit of the title compound,  $\text{C}_{20}\text{H}_{24}\text{N}_2$ , contains one half-molecule, with the single C—C bond of the 1,4-diazabutadiene fragment situated on a centre of symmetry. The benzene rings are inclined to the 1,4-diazabutadiene mean plane by  $59.5$  ( $1$ )°.

## Related literature

For the crystal structures of related compounds, see: Kuhn *et al.* (2001); Schaub & Radius (2006); Yuan *et al.* (2012).



## Experimental

## Crystal data

 $\text{C}_{20}\text{H}_{24}\text{N}_2$  $M_r = 292.42$ 

Monoclinic,  $P2_1/n$   
 $a = 7.128$  (3) Å  
 $b = 8.304$  (4) Å  
 $c = 15.162$  (7) Å  
 $\beta = 96.528$  (5)°  
 $V = 891.7$  (7) Å<sup>3</sup>

$Z = 2$   
 Mo  $K\alpha$  radiation  
 $\mu = 0.06$  mm<sup>-1</sup>  
 $T = 296$  K  
 $0.23 \times 0.21 \times 0.19$  mm

## Data collection

Bruker APEXII CCD area-detector diffractometer  
 Absorption correction: multi-scan (SADABS; Bruker, 2008)  
 $T_{\min} = 0.986$ ,  $T_{\max} = 0.988$

4080 measured reflections  
 1643 independent reflections  
 1114 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.040$

## Refinement

$R[F^2 > 2\sigma(F^2)] = 0.051$   
 $wR(F^2) = 0.175$   
 $S = 1.07$   
 1643 reflections

103 parameters  
 H-atom parameters constrained  
 $\Delta\rho_{\text{max}} = 0.17$  e Å<sup>-3</sup>  
 $\Delta\rho_{\text{min}} = -0.17$  e Å<sup>-3</sup>

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.

The authors are grateful for financial support from the National Natural Science Foundation of China (grant No. 21062016), the Key Laboratory of Polymer Materials of Gansu Province (Northwest Normal University), the Bioactive Product Engineering Research Centre for Gansu Distinctive Plants and the State Key Laboratory of Applied Organic Chemistry, Lanzhou University.

Supporting information for this paper is available from the IUCr electronic archives (Reference: CV5439).

## References

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 Kuhn, N., Steimann, M. & Walker, I. (2001). *Z. Kristallogr. New Cryst. Struct.* **216**, 318–319.  
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 Yuan, J., Miao, C., Xu, W. & Yuan, B. (2012). *Acta Cryst.* **E68**, o164.

## supporting information

*Acta Cryst.* (2014). E70, o175 [doi:10.1107/S1600536814001020]

**(Z)-N-[(Z)-3-(2,5-Dimethylphenylimino)butan-2-ylidene]-2,5-dimethylaniline****Wen-Xian Lv, Jian Li, Yu-Lai Hu, Ying-Peng Su and Dan-Feng Huang****S1. Comment**

In a continuation of our crystallographic study of the compounds used as  $\alpha$ -diimine ligands for Ni<sup>II</sup>- $\alpha$ -diimine olefin polymerization catalysts (Yuan *et al.*, 2012), we present here the crystal structure of the title compound (I).

The asymmetric unit of (I) contains one half-molecule with the single C—C bond in 1,4-diazabutadiene fragment situated on a centre of symmetry (Fig. 1). The single bond of 1,4-diazabutadiene fragment is (E)-configured. The dihedral angle between the benzene ring and the 1,4-diazabutadiene plane is 59.5 (1). All bond lengths and angles in (I) are normal and correspond well to those observed in the related compounds 2,3-bis(2,6-dimethylphenyl)iminobutane (Kuhn *et al.*, 2001), *N,N'*-dimesitylbutane-2,3-diimine (Schaub & Radius, 2006) and (Z)-N-[(Z)-3-(2,4-dimethylphenylimino)-butan-2-ylidene]-2,4-dimethylaniline (Yuan *et al.*, 2012).

**S2. Experimental**

Formic acid (1 ml) was added to a stirred solution of 2,3-butanedione (0.052 g, 0.6 mmol) and 2,5-dimethylaniline (0.145 g, 1.2 mmol) in methanol (30 ml). The mixture was refluxed for 24 h, and then the solvent was removed. The residue was purified by chromatography on silica gel with petroleum ether/ethyl ester ( $v/v = 20:1$ ). The crude product was recrystallized from ethanol/dichloromethane ( $v/v = 8:1$ ). The pure product was washed and dried in vacuo to give a fine yellow powder. Yield: 0.143 g (82%). Crystals suitable for X-ray structure determination were grown from a solution of the title compound in a mixture of cyclohexane/dichloromethane ( $v/v = 1:2$ ).

**S3. Refinement**

All H atoms were placed in calculated positions (C—H = 0.93–0.96 Å) and included in the refinement in a riding-model approximation, with  $U_{\text{iso}}(\text{H}) = 1.2\text{--}1.5U_{\text{eq}}(\text{C})$ .

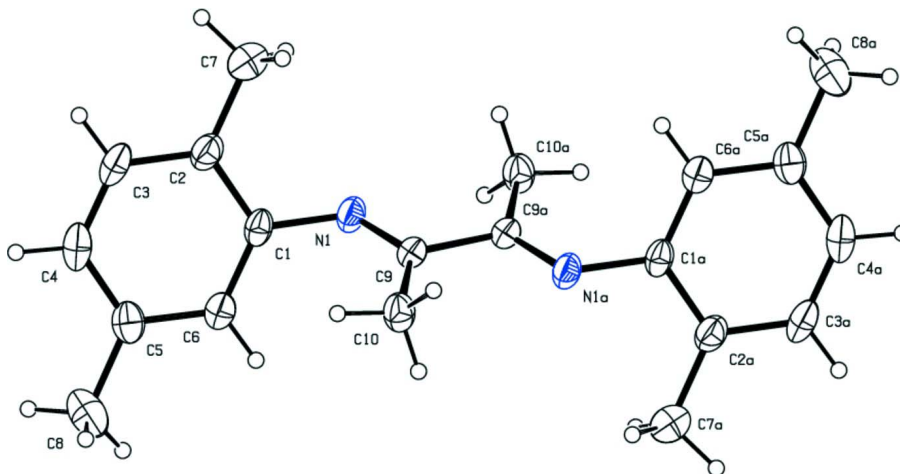


Figure 1

The molecular structure of (I), showing the atomic numbering and 30% probability displacement ellipsoids [symmetry code: (a)  $2 - x, 1 - y, 1 - z$ ].

**(Z)-N-[(Z)-3-(2,5-Dimethylphenylimino)butan-2-ylidene]-2,5-dimethylaniline**

*Crystal data*

$C_{20}H_{24}N_2$

$M_r = 292.42$

Monoclinic,  $P2_1/n$

$a = 7.128$  (3) Å

$b = 8.304$  (4) Å

$c = 15.162$  (7) Å

$\beta = 96.528$  (5)°

$V = 891.7$  (7) Å<sup>3</sup>

$Z = 2$

$F(000) = 316$

$D_x = 1.089$  Mg m<sup>-3</sup>

Mo  $K\alpha$  radiation,  $\lambda = 0.71073$  Å

Cell parameters from 1181 reflections

$\theta = 2.7$ – $25.0$ °

$\mu = 0.06$  mm<sup>-1</sup>

$T = 296$  K

Block, yellow

$0.23 \times 0.21 \times 0.19$  mm

*Data collection*

Bruker APEXII CCD area-detector  
diffractometer

Radiation source: fine-focus sealed tube

Graphite monochromator

$\varphi$  and  $\omega$  scans

Absorption correction: multi-scan

(*SADABS*; Bruker, 2008)

$T_{\min} = 0.986$ ,  $T_{\max} = 0.988$

4080 measured reflections

1643 independent reflections

1114 reflections with  $I > 2\sigma(I)$

$R_{\text{int}} = 0.040$

$\theta_{\max} = 25.5$ °,  $\theta_{\min} = 2.7$ °

$h = -8 \rightarrow 8$

$k = -9 \rightarrow 10$

$l = -12 \rightarrow 18$

*Refinement*

Refinement on  $F^2$

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.051$

$wR(F^2) = 0.175$

$S = 1.07$

1643 reflections

103 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

$w = 1/[\sigma^2(F_o^2) + (0.1P)^2]$

where  $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} < 0.001$

$\Delta\rho_{\max} = 0.17$  e Å<sup>-3</sup>

$\Delta\rho_{\min} = -0.17$  e Å<sup>-3</sup>

*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 ( $\text{\AA}^2$ )*

	$x$	$y$	$z$	$U_{\text{iso}}^*/U_{\text{eq}}$
C1	1.0081 (3)	0.6052 (2)	0.30932 (11)	0.0418 (5)
C2	1.1376 (3)	0.7091 (3)	0.27639 (12)	0.0507 (6)
C3	1.0926 (3)	0.7661 (3)	0.19027 (13)	0.0604 (6)
H3	1.1760	0.8356	0.1665	0.072*
C4	0.9291 (3)	0.7230 (3)	0.13947 (13)	0.0598 (6)
H4	0.9043	0.7635	0.0821	0.072*
C5	0.7995 (3)	0.6200 (2)	0.17187 (13)	0.0513 (6)
C6	0.8429 (3)	0.5612 (2)	0.25790 (12)	0.0470 (5)
H6	0.7594	0.4912	0.2812	0.056*
C7	1.3169 (3)	0.7566 (4)	0.33116 (16)	0.0798 (8)
H7A	1.2882	0.8234	0.3795	0.120*
H7B	1.3955	0.8152	0.2949	0.120*
H7C	1.3822	0.6618	0.3542	0.120*
C8	0.6167 (4)	0.5763 (3)	0.11792 (16)	0.0819 (8)
H8A	0.5215	0.6532	0.1287	0.123*
H8B	0.5782	0.4708	0.1346	0.123*
H8C	0.6337	0.5768	0.0560	0.123*
C9	0.9622 (2)	0.5443 (2)	0.45830 (11)	0.0393 (5)
C10	0.7828 (3)	0.6367 (2)	0.45927 (12)	0.0508 (6)
H10A	0.7584	0.6973	0.4053	0.076*
H10B	0.7942	0.7090	0.5090	0.076*
H10C	0.6805	0.5633	0.4642	0.076*
N1	1.0615 (2)	0.53296 (18)	0.39391 (9)	0.0443 (5)

*Atomic displacement parameters ( $\text{\AA}^2$ )*

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
C1	0.0482 (11)	0.0458 (11)	0.0342 (10)	0.0095 (8)	0.0166 (8)	0.0053 (8)
C2	0.0488 (12)	0.0590 (13)	0.0469 (12)	0.0012 (9)	0.0173 (9)	0.0091 (9)
C3	0.0707 (15)	0.0649 (14)	0.0501 (12)	-0.0011 (11)	0.0271 (11)	0.0180 (10)
C4	0.0818 (16)	0.0625 (14)	0.0369 (11)	0.0159 (12)	0.0150 (11)	0.0128 (10)
C5	0.0641 (13)	0.0483 (12)	0.0415 (11)	0.0107 (9)	0.0065 (10)	0.0004 (9)
C6	0.0523 (11)	0.0492 (11)	0.0411 (11)	0.0016 (9)	0.0117 (9)	0.0056 (9)
C7	0.0618 (15)	0.102 (2)	0.0764 (16)	-0.0188 (13)	0.0104 (13)	0.0217 (14)
C8	0.0931 (19)	0.0822 (17)	0.0637 (15)	0.0037 (14)	-0.0204 (14)	0.0008 (13)
C9	0.0386 (10)	0.0450 (11)	0.0355 (9)	-0.0032 (8)	0.0088 (8)	0.0028 (8)

C10	0.0510 (12)	0.0630 (13)	0.0396 (11)	0.0125 (9)	0.0101 (9)	0.0047 (9)
N1	0.0463 (10)	0.0532 (10)	0.0353 (9)	0.0037 (7)	0.0137 (7)	0.0083 (7)

*Geometric parameters (Å, °)*

C1—C6	1.386 (3)	C7—H7A	0.9600
C1—C2	1.396 (3)	C7—H7B	0.9600
C1—N1	1.428 (2)	C7—H7C	0.9600
C2—C3	1.392 (3)	C8—H8A	0.9600
C2—C7	1.496 (3)	C8—H8B	0.9600
C3—C4	1.369 (3)	C8—H8C	0.9600
C3—H3	0.9300	C9—N1	1.273 (2)
C4—C5	1.389 (3)	C9—C10	1.493 (3)
C4—H4	0.9300	C9—C9 <sup>i</sup>	1.508 (3)
C5—C6	1.394 (3)	C10—H10A	0.9600
C5—C8	1.502 (3)	C10—H10B	0.9600
C6—H6	0.9300	C10—H10C	0.9600
C6—C1—C2	121.04 (16)	H7A—C7—H7B	109.5
C6—C1—N1	121.17 (17)	C2—C7—H7C	109.5
C2—C1—N1	117.46 (16)	H7A—C7—H7C	109.5
C3—C2—C1	117.04 (18)	H7B—C7—H7C	109.5
C3—C2—C7	121.47 (19)	C5—C8—H8A	109.5
C1—C2—C7	121.49 (18)	C5—C8—H8B	109.5
C4—C3—C2	121.9 (2)	H8A—C8—H8B	109.5
C4—C3—H3	119.0	C5—C8—H8C	109.5
C2—C3—H3	119.0	H8A—C8—H8C	109.5
C3—C4—C5	121.32 (18)	H8B—C8—H8C	109.5
C3—C4—H4	119.3	N1—C9—C10	126.66 (16)
C5—C4—H4	119.3	N1—C9—C9 <sup>i</sup>	115.49 (19)
C4—C5—C6	117.43 (19)	C10—C9—C9 <sup>i</sup>	117.84 (19)
C4—C5—C8	121.79 (19)	C9—C10—H10A	109.5
C6—C5—C8	120.8 (2)	C9—C10—H10B	109.5
C1—C6—C5	121.21 (19)	H10A—C10—H10B	109.5
C1—C6—H6	119.4	C9—C10—H10C	109.5
C5—C6—H6	119.4	H10A—C10—H10C	109.5
C2—C7—H7A	109.5	H10B—C10—H10C	109.5
C2—C7—H7B	109.5	C9—N1—C1	122.91 (15)
C6—C1—C2—C3	0.2 (3)	C2—C1—C6—C5	-0.5 (3)
N1—C1—C2—C3	173.73 (16)	N1—C1—C6—C5	-173.76 (16)
C6—C1—C2—C7	-179.7 (2)	C4—C5—C6—C1	0.6 (3)
N1—C1—C2—C7	-6.1 (3)	C8—C5—C6—C1	-177.77 (18)
C1—C2—C3—C4	-0.1 (3)	C10—C9—N1—C1	-2.7 (3)
C7—C2—C3—C4	179.8 (2)	C9 <sup>i</sup> —C9—N1—C1	178.12 (18)
C2—C3—C4—C5	0.2 (3)	C6—C1—N1—C9	-61.6 (2)

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C3—C4—C5—C6	-0.4 (3)	C2—C1—N1—C9	124.9 (2)
C3—C4—C5—C8	177.9 (2)		

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Symmetry code: (i)  $-x+2, -y+1, -z+1$ .