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2,3-Bis(2-ethylphenylimino)butane

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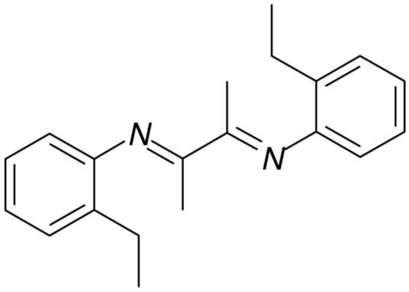
Key indicators: single-crystal X-ray study; T = 295 K; mean σ(C–C) = 0.002 Å; R factor = 0.042; wR factor = 0.131; data-to-parameter ratio = 15.1.

The title molecule, $C_{20}H_{24}N_2$, is centrosymmetric, crystallizing with the $-N{=}C(CH_3)-C(CH_3){=}N-$ diimino group in a *trans* configuration. The crystal structure is stabilized by an intramolecular C—H···N hydrogen-bonding interaction.

Related literature

For related literature, see: Brookhart *et al.* (2000); Bomfim *et al.*. (2007); Ferreira *et al.* (2006); Van Koten & Vrieze (1982); Vrieze *et al.* (1987). Hydrogen-bond calculations were carried out using *PLATON* (Spek, 2003).



Experimental

Crystal data

$C_{20}H_{24}N_2$
M_r = 292.41
Monoclinic, $P2_1/n$
a = 7.1898 (14) Å
b = 8.3807 (17) Å
c = 14.784 (3) Å
β = 99.76 (3)°
V = 877.9 (3) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.07 mm^{-1}
T = 295 (2) K
0.59 × 0.42 × 0.20 mm

Data collection

Nonius KappaCCD diffractometer
Absorption correction: none
14591 measured reflections
1541 independent reflections
1295 reflections with $I > 2\sigma(I)$
$R_{\rm int}$ = 0.032

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.042
$wR(F^2)$ = 0.131
S = 1.11
1541 reflections
102 parameters
H-atom parameters constrained
$\Delta\rho_{\rm max}$ = 0.17 e Å^{-3}
$\Delta\rho_{\rm min}$ = −0.15 e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
C9—H9B···N1	0.97	2.52	2.885 (2)	102

Data collection: *COLLECT* (Nonius, 1998); cell refinement: *PHICHI* (Duisenberg *et al.*, 2000); data reduction: *EvalCCD* (Duisenberg *et al.*, 2003); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: OM2165).

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supplementary materials

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2,3-Bis(2-ethylphenylimino)butane

R. S. Martins, C. A. L. Filgueiras, L. do C. Visentin, J. Bordinhão and **L. C. Ferreira**

Comment

Coordinated 1,4-diaza-1,3-butadiene groups have attracted much interest in relation to their electron donor and acceptor properties (Van Koten & Vrieze, 1982; Vrieze *et al.*, 1987) and, more recently, due to their application to olefin homo- and co- polymerizations [Brookhart *et al.*, 2000].

The present communication describes the chief characteristics of 2,3-bis(2-ethylphenylimino)butane, Scheme 1. The molecule is centrosymmetric, crystallizing with the diimino group –N=C(CH_3)-C(CH_3)=N– in a *trans* configuration, Fig. 1.

The carbon and nitrogen atoms in the –N=C(CH_3)-C(CH_3)=N– moiety present bond lengths of 1.2780 (16) Å for C9=N1, 1.4279 (17) Å for C8—N1, and 1.514 (2) Å for C9—C9^i [Symmetry code: (i)= $-x,-y+1,-z$]. These values are in good agreement with the distances found in the recently reported crystalline structure of the compound 2,3-bis(2-trifluoromethylphenylimino)butane in which the following bond lengths were observed: 1.423 (3) Å for C—N, 1.280 (3) Å for C=N, and 1.513 (4) Å for C—C (Bomfim *et al.*, 2007). The isolated molecules show intramolecular non-classical hydrogen bonds including the nitrogen atom (N1) of the –N=C(CH_3)-C(CH_3)=N– moiety as acceptor, (Fig. 1). The C9—H9B···N1 distance is 2.885 (2) Å and the angle is 102° (Spek, 2003).

The observed torsion angles are −4.4 (2)° for C10—C9—N1—C8, −9.0 (2)° for N1—C8—C3—C2, 115.32 (15)° for C9—N1—C8—C3, and −86.88 (19)° for C8—C3—C2—C1. These values can be considered as consequence of the intramolecular interaction described above. The observed deviations between the C8/N1/C9/C10 and C3/C4/C5/C6/C7/C8 planes [70,8(1)°] can be related to steric factors because the presence of the ethyl groups in the phenyl rings. This kind of deviation was also observed in the crystalline structure of the similar compound 2,3-bis(2-*tert*-butylphenylimino)butane, 69.6 (1)° (Ferreira *et al.*, 2006).

Experimental

To a solution of 3.72 g (40 mmol) of aniline in 40 ml of dichloromethane, were added 1.29 g (15 mmol) of 2,3-butanedione and a catalytic amount of *p*-toluene sulfonic acid. The mixture was heated under reflux for 24 h, after which the reaction mixture was cooled to room temperature and filtered. After removing the solvent under reduced pressure, the product was precipitated with cold methanol. The product was filtered and washed with cold ethanol (5 *x* 10 ml). X-ray diffraction quality crystals were obtained by recrystallization from a methanol/ethanol (1:1) mixture. The diimine was obtained as yellow crystals. Yield: 70%; Mp: 367 K; The compound was characterized by ^1H, ^{13}C NMR spectroscopy and infrared spectroscopy.

Refinement

The hydrogen atoms were fixed geometrically and allowed to ride on their parent atoms; the carbon rings and their respective atoms have been refined with C—H of 0.93 Å and U_{iso}(H) = 1.2 U_{eq}(Csp^2)·The hydrogen atoms of the methyl group hydrogen atoms were fixed geometrically at a distance of 0.96 Å and refined with U_{iso}(H) = 1.5U_{eq}(Csp^3).

Figures

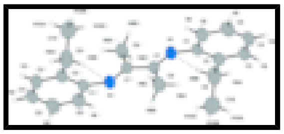


Fig. 1. *ORTEP* of the title compound with thermal parameters at a level of 50% probability. [Symmetry code: (i) = $-x$, $-y + 1$, $-z$]. In addition, intramolecular interactions are shown.

2,3-Bis(2-ethylphenylimino)butane

Crystal data

$C_{20}H_{24}N_2$
M_r = 292.41
Monoclinic, $P2_1/n$
Hall symbol: -P 2yn
a = 7.1898 (14) Å
b = 8.3807 (17) Å
c = 14.784 (3) Å
β = 99.76 (3)°
V = 877.9 (3) Å^3

Z = 2
F_{000} = 316
D_x = 1.106 Mg m^{-3}
Mo $K\alpha$ radiation
λ = 0.71073 Å
θ = 1–27.5°
μ = 0.07 mm^{-1}
T = 295 (2) K
Block, yellow
0.59 × 0.42 × 0.20 mm

Data collection

Nonius KappaCCD diffractometer
Radiation source: fine-focus sealed tube
Monochromator: graphite
T = 295(2) K
φ scans, and ω scans with κ
Absorption correction: none
14591 measured reflections
1541 independent reflections

1295 reflections with $I > 2\sigma(I)$
R_{int} = 0.032
θ_{max} = 25.0°
θ_{min} = 2.8°
$h = -8 \rightarrow 8$
$k = -9 \rightarrow 9$
$l = -17 \rightarrow 17$

Refinement

Refinement on F^2
Least-squares matrix: full

Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites

$R[F^2 > 2\sigma(F^2)] = 0.042$	H-atom parameters constrained
$wR(F^2) = 0.131$	$w = 1/[\sigma^2(F_o^2) + (0.0709P)^2 + 0.1453P]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.11$	$(\Delta/\sigma)_{max} < 0.001$
1541 reflections	$\Delta\rho_{max} = 0.17$ e Å^{-3}
102 parameters	$\Delta\rho_{min} = -0.15$ e Å^{-3}
Primary atom site location: structure-invariant direct methods	Extinction correction: none

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. Least-squares planes (*x*,*y*,*z* in crystal coordinates) and deviations from them (* indicates atom used to define plane)

− 4.2173 (0.0061) *x* + 6.2054 (0.0072) *y* − 3.3116 (0.0107) *z* = 3.1390 (0.0047)

* −0.0075 (0.0003) C1 * 0.0171 (0.0008) N1 * −0.0170 (0.0008) C7 * 0.0074 (0.0003) C8

Rms deviation of fitted atoms = 0.0132

4.1020 (0.0043) *x* + 6.1247 (0.0048) *y* − 6.8890 (0.0086) *z* = 2.4256 (0.0042)

Angle to previous plane (with approximate e.s.d.) = 70.77 (0.06)

* 0.0027 (0.0010) C1 * −0.0047 (0.0010) C2 * 0.0029 (0.0010) C3 * 0.0010 (0.0011) C4 * −0.0031 (0.0011) C5 * 0.0012 (0.0011) C6

Rms deviation of fitted atoms = 0.0029

− 2.2590 (0.0157) *x* + 5.3706 (0.0113) *y* + 10.9924 (0.0126) *z* = 6.6238 (0.0055)

Angle to previous plane (with approximate e.s.d.) = 88.48 (0.10)

* 0.0000 (0.0000) C2 * 0.0000 (0.0000) C9 * 0.0000 (0.0000) C10

Rms deviation of fitted atoms = 0.0000

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 $F^2 > 2\text{sigma}(F^2)$ 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	0.00541 (18)	0.61257 (16)	0.19534 (8)	0.0418 (3)
C2	−0.11601 (18)	0.72533 (17)	0.22436 (9)	0.0453 (4)
C3	−0.0656 (2)	0.79142 (18)	0.31205 (9)	0.0525 (4)

H3	−0.1439	0.8673	0.3320	0.063*
C4	0.0974 (2)	0.74664 (19)	0.36952 (9)	0.0574 (4)
H4	0.1280	0.7917	0.4276	0.069*
C5	0.2150 (2)	0.6345 (2)	0.34047 (10)	0.0606 (4)
H5	0.3248	0.6036	0.3792	0.073*
C6	0.1701 (2)	0.56783 (19)	0.25387 (10)	0.0542 (4)
H6	0.2503	0.4928	0.2346	0.065*
C7	0.03553 (16)	0.54972 (15)	0.04211 (8)	0.0388 (3)
C8	0.1974 (2)	0.6616 (2)	0.03814 (10)	0.0566 (4)
H8A	0.2124	0.7317	0.0902	0.085*
H8B	0.1721	0.7233	−0.0173	0.085*
H8C	0.3111	0.6011	0.0390	0.085*
C9	−0.2913 (2)	0.7804 (2)	0.16144 (12)	0.0681 (5)
H9A	−0.3851	0.8122	0.1979	0.082*
H9B	−0.3429	0.6923	0.1225	0.082*
C10	−0.2522 (3)	0.9194 (3)	0.10155 (13)	0.0905 (7)
H10A	−0.2031	1.0075	0.1398	0.136*
H10B	−0.3672	0.9509	0.0628	0.136*
H10C	−0.1616	0.8877	0.0642	0.136*
N1	−0.05162 (15)	0.53221 (14)	0.10996 (7)	0.0434 (3)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0463 (7)	0.0458 (7)	0.0351 (7)	−0.0058 (5)	0.0123 (5)	−0.0023 (5)
C2	0.0473 (7)	0.0491 (8)	0.0422 (7)	−0.0015 (6)	0.0155 (6)	−0.0036 (6)
C3	0.0656 (9)	0.0501 (8)	0.0455 (8)	−0.0015 (7)	0.0205 (7)	−0.0081 (6)
C4	0.0792 (10)	0.0572 (9)	0.0364 (7)	−0.0109 (8)	0.0112 (7)	−0.0061 (6)
C5	0.0645 (9)	0.0701 (10)	0.0435 (8)	0.0023 (8)	−0.0013 (7)	0.0013 (7)
C6	0.0555 (8)	0.0607 (9)	0.0465 (8)	0.0102 (7)	0.0087 (6)	−0.0034 (7)
C7	0.0390 (6)	0.0410 (7)	0.0370 (6)	0.0026 (5)	0.0076 (5)	−0.0020 (5)
C8	0.0607 (9)	0.0634 (9)	0.0480 (8)	−0.0185 (7)	0.0155 (6)	−0.0104 (7)
C9	0.0516 (8)	0.0871 (12)	0.0645 (10)	0.0169 (8)	0.0069 (7)	−0.0202 (9)
C10	0.1110 (16)	0.0836 (14)	0.0669 (11)	0.0429 (12)	−0.0134 (11)	−0.0018 (10)
N1	0.0452 (6)	0.0483 (7)	0.0382 (6)	−0.0015 (5)	0.0112 (5)	−0.0064 (5)

Geometric parameters (Å, °)

C1—C6	1.394 (2)	C7—N1	1.2780 (16)
C1—C2	1.4023 (19)	C7—C8	1.5032 (19)
C1—N1	1.4280 (16)	C7—C7^{i}	1.513 (2)
C2—C3	1.3998 (19)	C8—H8A	0.9600
C2—C9	1.506 (2)	C8—H8B	0.9600
C3—C4	1.378 (2)	C8—H8C	0.9600
C3—H3	0.9300	C9—C10	1.518 (3)
C4—C5	1.381 (2)	C9—H9A	0.9700
C4—H4	0.9300	C9—H9B	0.9700
C5—C6	1.384 (2)	C10—H10A	0.9600

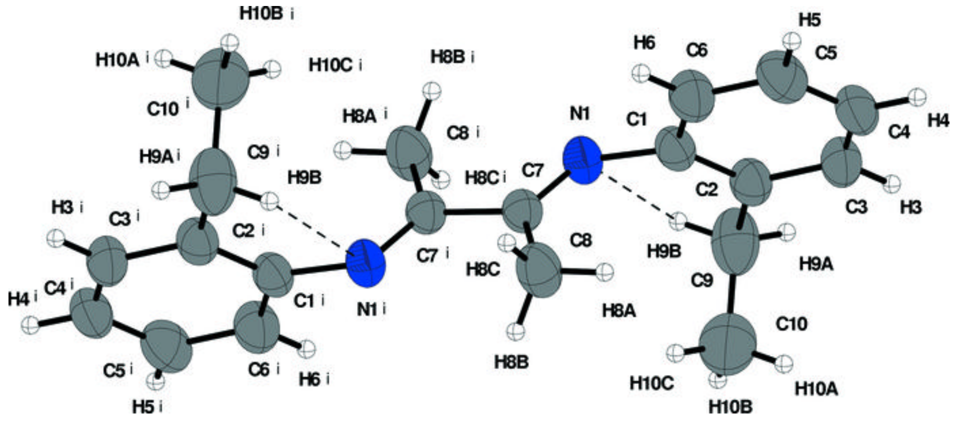
C5—H5	0.9300	C10—H10B	0.9600
C6—H6	0.9300	C10—H10C	0.9600
C6—C1—C2	119.91 (12)	C8—C7—C7^{i}	117.84 (13)
C6—C1—N1	120.54 (12)	C7—C8—H8A	109.5
C2—C1—N1	119.20 (12)	C7—C8—H8B	109.5
C3—C2—C1	118.23 (13)	H8A—C8—H8B	109.5
C3—C2—C9	120.60 (13)	C7—C8—H8C	109.5
C1—C2—C9	121.10 (12)	H8A—C8—H8C	109.5
C4—C3—C2	121.54 (14)	H8B—C8—H8C	109.5
C4—C3—H3	119.2	C2—C9—C10	112.15 (15)
C2—C3—H3	119.2	C2—C9—H9A	109.2
C3—C4—C5	119.69 (13)	C10—C9—H9A	109.2
C3—C4—H4	120.2	C2—C9—H9B	109.2
C5—C4—H4	120.2	C10—C9—H9B	109.2
C4—C5—C6	120.22 (14)	H9A—C9—H9B	107.9
C4—C5—H5	119.9	C9—C10—H10A	109.5
C6—C5—H5	119.9	C9—C10—H10B	109.5
C5—C6—C1	120.39 (14)	H10A—C10—H10B	109.5
C5—C6—H6	119.8	C9—C10—H10C	109.5
C1—C6—H6	119.8	H10A—C10—H10C	109.5
N1—C7—C8	126.02 (12)	H10B—C10—H10C	109.5
N1—C7—C7^{i}	116.13 (13)	C7—N1—C1	122.28 (11)
C6—C1—C2—C3	0.8 (2)	C2—C1—C6—C5	−0.2 (2)
N1—C1—C2—C3	173.99 (12)	N1—C1—C6—C5	−173.36 (13)
C6—C1—C2—C9	177.77 (14)	C3—C2—C9—C10	90.00 (18)
N1—C1—C2—C9	−9.0 (2)	C1—C2—C9—C10	−86.93 (19)
C1—C2—C3—C4	−0.8 (2)	C8—C7—N1—C1	−4.4 (2)
C9—C2—C3—C4	−177.81 (14)	C7^{i}—C7—N1—C1	176.99 (13)
C2—C3—C4—C5	0.3 (2)	C6—C1—N1—C7	−71.49 (18)
C3—C4—C5—C6	0.3 (2)	C2—C1—N1—C7	115.32 (14)
C4—C5—C6—C1	−0.3 (2)		

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

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C9—H9B···N1	0.97	2.52	2.885 (2)	102

Fig. 1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H10A i
H10C i
H8B i
H6
C6
H5
C5
C10 i
H8A i
C8 i
N1
C1
C4
H4
H9A i
C9 i
H9B
H8C i
C7
C2
H3 i
C3 i
C2 i
C3
H3
C7 i
H8C
C8
H9B
H9A
C9
H4 i
C4 i
C1 i
N1 i
H8A
C10
H8B
C5 i
C6 i
H6 i
H10C
H10A
H10B
H5 i