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# 1,2-Bis(2-*tert*-butylphenylimino)-1,2-dihydroacenaphthylene

**L. C. Ferreira,[a]* Carlos A. L. Filgueiras,[a] M. Horner,[b] Lorenzo do C. Visentin[a] and Jairo Bordinhao[a]**

[a]Instituto de Química, Universidade Federal do Rio de Janeiro, Caixa Postal 68563, 21949-900 Rio de Janeiro, RJ, Brazil, and [b]Departamento de Química, Universidade Federal de Santa Maria, Caixa Postal 5031, 97110-900 Santa Maria, RS, Brazil
Correspondence e-mail: leonardocf@iq.ufrj.br

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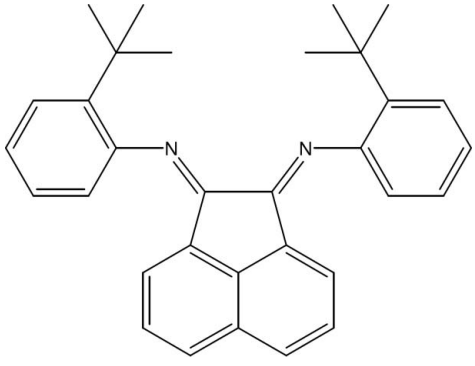
Key indicators: single-crystal X-ray study; $T$ = 291 K; mean $\sigma$(C–C) = 0.003 Å; $R$ factor = 0.044; $wR$ factor = 0.079; data-to-parameter ratio = 15.1.

The crystal structure of the title compound, $C_{32}H_{32}N_2$, reveals a dimeric association of $\alpha$-diimine units into centrosymmetric dimers *via* intermolecular non-classical C—H···N hydrogen bonds. There is also intramolecular non-classical C—H···N bonding between the methyl groups and the N atoms of the $\alpha$-diimine unit. In addition, the crystal packing indicates $\pi$–$\pi$ interactions between acenaphthylene rings [3.888 (7) Å]. The deviation from a planar arrangement of the molecule can be attributed to both inter- and intramolecular non-classical hydrogen bonding.

## Related literature

For related literature, see: Aoki & Salam (2001); Jeffrey *et al.* (1985).




## Experimental

### Crystal data

$C_{32}H_{32}N_2$
$M_r$ = 444.60
Triclinic, $P\overline{1}$
$a$ = 10.0996 (6) Å
$b$ = 12.3843 (7) Å
$c$ = 12.4174 (7) Å
$\alpha$ = 60.771 (3)°
$\beta$ = 73.256 (4)°
$\gamma$ = 71.768 (3)°
$V$ = 1270.06 (13) Å$^3$
$Z$ = 2
Mo $K\alpha$ radiation
$\mu$ = 0.07 mm$^{-1}$
$T$ = 291 (2) K
0.54 × 0.10 × 0.08 mm

### Data collection

Bruker APEXII CCD area-detector diffractometer
Absorption correction: none
21526 measured reflections
4709 independent reflections
2066 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.068

### Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.044
$wR(F^2)$ = 0.079
$S$ = 1.03
4709 reflections
312 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.15 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.15 e Å$^{-3}$

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

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| C23—H23···N1$^{i}$ | 0.97 (2) | 2.62 (2) | 3.331 (3) | 130.6 (17) |
| C28—H28*C*···N1 | 0.96 | 2.40 | 3.048 (3) | 125 |
| C29—H29*B*···N1 | 0.96 | 2.35 | 3.010 (3) | 125 |
| C31—H31*B*···N2 | 0.96 | 2.30 | 2.975 (3) | 127 |

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

Data collection: *COSMO* (Bruker, 2004) and *APEX2* (Bruker, 2004); cell refinement: *SAINT* (Bruker, 2004); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3* (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: OM2141).

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# supplementary materials

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# 1,2-Bis(2-*tert*-butylphenylimino)-1,2-dihydroacenaphthylene

**L. C. Ferreira, C. A. L. Filgueiras, M. Horner, L. do C. Visentin and J. Bordinhao**

## Comment

The present report describes the main structural features of a novel α-diimine, as given in Scheme 1 and Figure 1. This molecule is bis(*N*-2-*tert*-butylphenyl)imineacenaphthene, in which the two *tert*-butyl substituents on the aromatic rings are *cis* to each other.

In addition, another strong feature of this molecular structure is the presence of molecular interactions *via* non-classical hydrogen bonds (Jeffrey *et al.*, 1985), represented as C—H···N. Each α-diimine unit presents two intermolecular interactions, generating a dimeric structure with an inversion center, as shown in Figure 2. The geometric parameters of all non-classical interactions are listed in Table 1.

The dimers are related by translation along the crystallographic axis a. The cell packing shows that the dimers present π-π interactions between the acenaphthenequine rings [C4=C9···C9$^{ii}$=C4$^{ii}$=3.880 (7) Å; symmetry code: (ii) 1 − *x*, 1 − *y*, 1 − *z*] (Fig. 3), in agreement with results found in the literature (Aoki & Salam, 2001).

Lastly, it is believed that both the inter- as well as the intramolecular interactions are partly responsible for the large deviation of planarity shown between the aromatic rings and the *tert*-butyl substituents, C13—C14—C15—C16—C17—C18 (r.m.s. = 0.0118) and C19—C20—C21—C22—C23—C24 (r.m.s. = 0.0030), of 28.84 (7)°. In addition, the torsion angles formed by the atoms C5—C1—N1—C13, of −7.7 (4)°, and C3—C2—N2—C19, of 10.8 (4)°, are also ascribed to the aforementioned deviation.

## Experimental

0.5465 g (3 mmole) of acenaphthenequinone was placed in a 100 ml round bottom flask, as well as 0.935 ml (6 mmole) of 2-*tert*-butylaniline, with MeOH (30 ml) as the solvent and glacial acetic acid as catalyst (5 drops). The flask was connected to a Liebig condenser and the reaction mixture was kept under reflux for 3 h, after which heating was stopped. After cooling to room temperature, a red solid precipitated. The mixture was filtered and the solid washed with MeOH (20 ml). The red precipitate was recrystallized from MeOH, giving small red crystals suitable for *X* ray crystallography. (Yield: 85%, Melting point: 485 K)

## Refinement

The hydrogen atoms of phenyl rings and acenaphthene ring were fixed geometrically to a distance 0.93 Å; the carbon rings and its respective atoms have been dealt with $U_{iso}$(H) 1.2 $U_{eq}$ (C$sp^2$). The hydrogen atom H23 was located in the Fourier map and refined. The hydrogen atoms of methyl group were fixed geometrically to a distance 0.96 Å; the carbon methyl and its respective atoms have been dealt with $U_{iso}$(H) 1.5 $U_{eq}$ (C$sp^3$).

## Figures

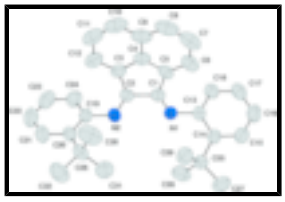


Fig. 1. *ORTEP* drawing of bis(*N*-2-*tert*-butylphenyl)imineacenaphthene. Thermal ellipsoids with 50% probability. Hydrogen atoms were omitted so as not to encumber the drawing.

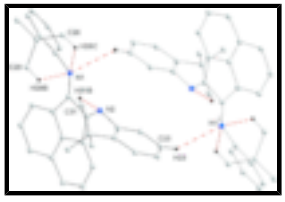


Fig. 2. Representation of all non-classical inter- and intramolecular interactions in the dimer.[symmetry code: (i) −*x*, 1 − *y*, 1 − *z*].

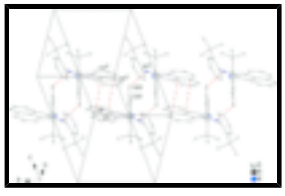


Fig. 3. Representation of the packing along of crystallographic axis a. [symmetry code: (ii) 1 − *x*, 1 − *y*, 1 − *z*].

### 1,2-Bis(2-*tert*-butylphenylimino)-1,2-dihydroacenaphthylene

*Crystal data*

| | |
|---|---|
| $C_{32}H_{32}N_2$ | $Z = 2$ |
| $M_r = 444.60$ | $F_{000} = 476$ |
| Triclinic, $P\overline{1}$ | $D_x = 1.163$ Mg m$^{-3}$ |
| Hall symbol: -P 1 | Melting point: 485 K |
| $a = 10.0996$ (6) Å | Mo $K\alpha$ radiation<br>$\lambda = 0.71073$ Å |
| $b = 12.3843$ (7) Å | $\theta = 1$–30º |
| $c = 12.4174$ (7) Å | $\mu = 0.07$ mm$^{-1}$ |
| $\alpha = 60.771$ (3)º | $T = 291$ (2) K |
| $\beta = 73.256$ (4)º | Block, red |
| $\gamma = 71.768$ (3)º | 0.54 × 0.10 × 0.08 mm |
| $V = 1270.06$ (13) Å$^3$ | |

*Data collection*

| | |
|---|---|
| Bruker APEX II CCD area-detector diffractometer | 2066 reflections with $I > 2\sigma(I)$ |
| Radiation source: fine-focus sealed tube | $R_{int} = 0.068$ |
| Monochromator: graphite | $\theta_{max} = 25.5$º |
| $T = 291(2)$ K | $\theta_{min} = 3.1$º |
| φ and ω scans | $h = -12 \rightarrow 12$ |
| Absorption correction: none | $k = -14 \rightarrow 14$ |
| 21526 measured reflections | $l = -15 \rightarrow 15$ |

4709 independent reflections

*Refinement*

| | |
|---|---|
| Refinement on $F^2$ | Hydrogen site location: inferred from neighbouring sites |
| Least-squares matrix: full | H atoms treated by a mixture of independent and constrained refinement |
| $R[F^2 > 2\sigma(F^2)] = 0.044$ | $w = 1/[\sigma^2(F_o^2) + (0.02P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$ |
| $wR(F^2) = 0.079$ | $(\Delta/\sigma)_{max} < 0.001$ |
| $S = 1.03$ | $\Delta\rho_{max} = 0.15$ e Å$^{-3}$ |
| 4709 reflections | $\Delta\rho_{min} = -0.15$ e Å$^{-3}$ |
| 312 parameters | Extinction correction: SHELXL97, $Fc^* = kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$ |
| Primary atom site location: structure-invariant direct methods | Extinction coefficient: 0.0177 (11) |
| Secondary atom site location: difference Fourier map | |

*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 $F^2 > 2$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 | 0.3511 (2) | 0.59137 (18) | 0.23428 (16) | 0.0499 (5) |
| C2 | 0.3089 (2) | 0.47043 (17) | 0.34154 (16) | 0.0499 (5) |
| C3 | 0.4264 (2) | 0.40396 (19) | 0.41384 (18) | 0.0539 (5) |
| C4 | 0.5341 (2) | 0.47330 (19) | 0.34771 (19) | 0.0537 (5) |
| C5 | 0.4974 (2) | 0.58409 (18) | 0.24060 (18) | 0.0518 (5) |
| C6 | 0.5966 (2) | 0.65484 (19) | 0.16418 (19) | 0.0682 (6) |
| H6 | 0.5753 | 0.7279 | 0.0920 | 0.082* |
| C7 | 0.7324 (3) | 0.6146 (3) | 0.1972 (3) | 0.0865 (8) |
| H7 | 0.8006 | 0.6623 | 0.1454 | 0.104* |
| C8 | 0.7667 (3) | 0.5086 (3) | 0.3021 (3) | 0.0865 (8) |
| H8 | 0.8572 | 0.4856 | 0.3207 | 0.104* |
| C9 | 0.6671 (3) | 0.4327 (2) | 0.3833 (2) | 0.0707 (6) |
| C10 | 0.6881 (3) | 0.3190 (3) | 0.4922 (3) | 0.0854 (8) |
| H10 | 0.7746 | 0.2886 | 0.5200 | 0.102* |

| | | | | |
|---|---|---|---|---|
| C11 | 0.5831 (3) | 0.2532 (2) | 0.5570 (2) | 0.0856 (7) |
| H11 | 0.5992 | 0.1791 | 0.6296 | 0.103* |
| C12 | 0.4509 (2) | 0.29298 (19) | 0.51840 (18) | 0.0705 (6) |
| H12 | 0.3819 | 0.2448 | 0.5631 | 0.085* |
| C13 | 0.28881 (17) | 0.78949 (17) | 0.06525 (17) | 0.0485 (5) |
| C14 | 0.27507 (17) | 0.82530 (17) | −0.05757 (16) | 0.0475 (5) |
| C15 | 0.29109 (19) | 0.94722 (18) | −0.14324 (17) | 0.0630 (6) |
| H15 | 0.2858 | 0.9738 | −0.2263 | 0.076* |
| C16 | 0.3145 (2) | 1.03100 (18) | −0.11115 (19) | 0.0749 (6) |
| H16 | 0.3251 | 1.1118 | −0.1720 | 0.090* |
| C17 | 0.3221 (2) | 0.99505 (19) | 0.0098 (2) | 0.0726 (6) |
| H17 | 0.3353 | 1.0518 | 0.0325 | 0.087* |
| C18 | 0.31013 (19) | 0.87446 (19) | 0.09798 (17) | 0.0644 (6) |
| H18 | 0.3163 | 0.8494 | 0.1805 | 0.077* |
| C19 | 0.14738 (18) | 0.33474 (17) | 0.44951 (17) | 0.0498 (5) |
| C20 | 0.13939 (17) | 0.23113 (17) | 0.43590 (17) | 0.0499 (5) |
| C21 | 0.07626 (19) | 0.13984 (17) | 0.5408 (2) | 0.0659 (6) |
| H21 | 0.0698 | 0.0693 | 0.5355 | 0.079* |
| C22 | 0.0227 (2) | 0.1477 (2) | 0.6522 (2) | 0.0756 (6) |
| H22 | −0.0191 | 0.0838 | 0.7198 | 0.091* |
| C23 | 0.0310 (2) | 0.2494 (2) | 0.6637 (2) | 0.0717 (7) |
| C24 | 0.0927 (2) | 0.34239 (18) | 0.56205 (19) | 0.0665 (6) |
| H24 | 0.0978 | 0.4124 | 0.5689 | 0.080* |
| C25 | 0.23720 (19) | 0.73914 (17) | −0.09427 (15) | 0.0519 (5) |
| C26 | 0.2008 (2) | 0.21503 (18) | 0.31614 (18) | 0.0629 (5) |
| C27 | 0.2373 (2) | 0.79766 (17) | −0.23541 (15) | 0.0765 (6) |
| H27A | 0.2133 | 0.7405 | −0.2545 | 0.115* |
| H27B | 0.3295 | 0.8131 | −0.2800 | 0.115* |
| H27C | 0.1692 | 0.8760 | −0.2600 | 0.115* |
| C28 | 0.08905 (19) | 0.71504 (18) | −0.02588 (17) | 0.0797 (7) |
| H28A | 0.0647 | 0.6608 | −0.0487 | 0.120* |
| H28B | 0.0219 | 0.7940 | −0.0489 | 0.120* |
| H28C | 0.0878 | 0.6752 | 0.0628 | 0.120* |
| C29 | 0.3438 (2) | 0.61238 (15) | −0.06099 (16) | 0.0699 (6) |
| H29A | 0.3175 | 0.5603 | −0.0853 | 0.105* |
| H29B | 0.3440 | 0.5703 | 0.0276 | 0.105* |
| H29C | 0.4365 | 0.6273 | −0.1044 | 0.105* |
| C30 | 0.3615 (2) | 0.1955 (2) | 0.2940 (2) | 0.1053 (8) |
| H30A | 0.4003 | 0.1855 | 0.2184 | 0.158* |
| H30B | 0.3983 | 0.1211 | 0.3632 | 0.158* |
| H30C | 0.3869 | 0.2677 | 0.2865 | 0.158* |
| C31 | 0.1421 (2) | 0.33014 (19) | 0.20334 (17) | 0.0930 (7) |
| H31A | 0.1826 | 0.3173 | 0.1294 | 0.139* |
| H31B | 0.1656 | 0.4043 | 0.1932 | 0.139* |
| H31C | 0.0413 | 0.3410 | 0.2167 | 0.139* |
| C32 | 0.1652 (2) | 0.09977 (19) | 0.3219 (2) | 0.0984 (8) |
| H32A | 0.2059 | 0.0936 | 0.2445 | 0.148* |
| H32B | 0.0645 | 0.1098 | 0.3350 | 0.148* |
| H32C | 0.2031 | 0.0243 | 0.3896 | 0.148* |

| | | | | |
|---|---|---|---|---|
| N1 | 0.26365 (15) | 0.67005 (15) | 0.16129 (13) | 0.0526 (4) |
| N2 | 0.19586 (17) | 0.44176 (13) | 0.34750 (13) | 0.0545 (4) |
| H23 | −0.0036 (17) | 0.2586 (14) | 0.7406 (15) | 0.077 (6)* |

*Atomic displacement parameters (Å$^2$)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| C1 | 0.0590 (15) | 0.0515 (14) | 0.0470 (12) | −0.0150 (11) | −0.0098 (11) | −0.0246 (11) |
| C2 | 0.0603 (14) | 0.0464 (13) | 0.0476 (12) | −0.0128 (12) | −0.0106 (11) | −0.0220 (11) |
| C3 | 0.0620 (15) | 0.0531 (14) | 0.0510 (13) | −0.0051 (13) | −0.0178 (12) | −0.0255 (12) |
| C4 | 0.0534 (15) | 0.0582 (15) | 0.0639 (14) | −0.0006 (13) | −0.0151 (13) | −0.0410 (13) |
| C5 | 0.0519 (15) | 0.0527 (14) | 0.0637 (14) | −0.0147 (12) | −0.0042 (12) | −0.0355 (12) |
| C6 | 0.0616 (16) | 0.0692 (16) | 0.0895 (16) | −0.0187 (14) | −0.0046 (14) | −0.0475 (13) |
| C7 | 0.0604 (19) | 0.102 (2) | 0.131 (2) | −0.0234 (16) | 0.0007 (16) | −0.081 (2) |
| C8 | 0.0552 (17) | 0.114 (2) | 0.135 (2) | 0.0018 (18) | −0.0235 (18) | −0.095 (2) |
| C9 | 0.0625 (19) | 0.085 (2) | 0.0918 (19) | 0.0030 (17) | −0.0241 (16) | −0.0637 (17) |
| C10 | 0.074 (2) | 0.106 (2) | 0.099 (2) | 0.0239 (17) | −0.0439 (17) | −0.070 (2) |
| C11 | 0.094 (2) | 0.087 (2) | 0.0741 (17) | 0.0171 (18) | −0.0405 (17) | −0.0409 (15) |
| C12 | 0.0810 (18) | 0.0700 (17) | 0.0610 (14) | −0.0009 (13) | −0.0252 (13) | −0.0306 (13) |
| C13 | 0.0505 (12) | 0.0440 (13) | 0.0474 (12) | −0.0133 (10) | −0.0054 (9) | −0.0166 (11) |
| C14 | 0.0463 (12) | 0.0420 (12) | 0.0493 (12) | −0.0056 (10) | −0.0086 (10) | −0.0179 (11) |
| C15 | 0.0772 (15) | 0.0567 (14) | 0.0542 (13) | −0.0144 (12) | −0.0136 (11) | −0.0216 (12) |
| C16 | 0.0986 (18) | 0.0529 (14) | 0.0649 (15) | −0.0220 (13) | −0.0146 (13) | −0.0152 (13) |
| C17 | 0.0955 (17) | 0.0526 (15) | 0.0803 (15) | −0.0278 (12) | −0.0167 (13) | −0.0285 (13) |
| C18 | 0.0853 (15) | 0.0587 (14) | 0.0577 (13) | −0.0263 (12) | −0.0113 (11) | −0.0253 (12) |
| C19 | 0.0511 (13) | 0.0417 (13) | 0.0487 (12) | −0.0107 (10) | −0.0096 (10) | −0.0124 (11) |
| C20 | 0.0425 (12) | 0.0440 (12) | 0.0571 (13) | −0.0100 (10) | −0.0073 (10) | −0.0174 (11) |
| C21 | 0.0640 (15) | 0.0515 (14) | 0.0719 (15) | −0.0175 (11) | −0.0094 (12) | −0.0169 (13) |
| C22 | 0.0644 (15) | 0.0648 (17) | 0.0693 (16) | −0.0176 (12) | −0.0058 (12) | −0.0082 (13) |
| C23 | 0.0690 (16) | 0.0757 (18) | 0.0520 (15) | −0.0042 (14) | −0.0064 (12) | −0.0224 (15) |
| C24 | 0.0809 (16) | 0.0569 (14) | 0.0582 (13) | −0.0100 (12) | −0.0130 (12) | −0.0241 (12) |
| C25 | 0.0524 (13) | 0.0535 (13) | 0.0515 (12) | −0.0087 (11) | −0.0116 (10) | −0.0240 (11) |
| C26 | 0.0602 (15) | 0.0613 (14) | 0.0761 (15) | −0.0225 (11) | −0.0038 (12) | −0.0349 (12) |
| C27 | 0.0948 (16) | 0.0786 (15) | 0.0643 (14) | −0.0084 (12) | −0.0275 (12) | −0.0353 (12) |
| C28 | 0.0692 (16) | 0.1032 (18) | 0.0887 (15) | −0.0307 (13) | −0.0112 (12) | −0.0518 (14) |
| C29 | 0.0849 (16) | 0.0529 (13) | 0.0719 (13) | −0.0028 (12) | −0.0164 (11) | −0.0321 (11) |
| C30 | 0.0646 (17) | 0.149 (2) | 0.139 (2) | −0.0252 (15) | 0.0108 (15) | −0.1028 (19) |
| C31 | 0.130 (2) | 0.0931 (18) | 0.0710 (15) | −0.0391 (16) | −0.0202 (14) | −0.0355 (14) |
| C32 | 0.117 (2) | 0.0886 (17) | 0.1186 (19) | −0.0389 (15) | −0.0052 (15) | −0.0640 (15) |
| N1 | 0.0646 (12) | 0.0513 (11) | 0.0452 (9) | −0.0199 (10) | −0.0116 (9) | −0.0176 (9) |
| N2 | 0.0655 (12) | 0.0464 (11) | 0.0532 (10) | −0.0191 (9) | −0.0117 (9) | −0.0174 (9) |

*Geometric parameters (Å, °)*

| | | | |
|---|---|---|---|
| C1—N1 | 1.2692 (19) | C20—C21 | 1.384 (2) |
| C1—C5 | 1.474 (2) | C20—C26 | 1.522 (2) |
| C1—C2 | 1.524 (2) | C21—C22 | 1.372 (2) |
| C2—N2 | 1.271 (2) | C21—H21 | 0.9300 |
| C2—C3 | 1.474 (2) | C22—C23 | 1.364 (3) |

| | | | |
|---|---|---|---|
| C3—C12 | 1.366 (2) | C22—H22 | 0.9300 |
| C3—C4 | 1.406 (2) | C23—C24 | 1.373 (2) |
| C4—C9 | 1.398 (2) | C23—N1$^{i}$ | 3.333 (3) |
| C4—C5 | 1.405 (2) | C23—H23 | 0.968 (15) |
| C5—C6 | 1.365 (2) | C24—H24 | 0.9300 |
| C6—C7 | 1.412 (3) | C25—C28 | 1.533 (2) |
| C6—H6 | 0.9300 | C25—C27 | 1.535 (2) |
| C7—C8 | 1.358 (3) | C25—C29 | 1.536 (2) |
| C7—H7 | 0.9300 | C26—C30 | 1.526 (2) |
| C8—C9 | 1.412 (3) | C26—C31 | 1.531 (2) |
| C8—H8 | 0.9300 | C26—C32 | 1.542 (2) |
| C9—C10 | 1.405 (3) | C27—H27A | 0.9600 |
| C10—C11 | 1.358 (3) | C27—H27B | 0.9600 |
| C10—H10 | 0.9300 | C27—H27C | 0.9600 |
| C11—C12 | 1.408 (3) | C28—N1 | 3.050 (2) |
| C11—H11 | 0.9300 | C28—H28A | 0.9600 |
| C12—H12 | 0.9300 | C28—H28B | 0.9600 |
| C13—C18 | 1.389 (2) | C28—H28C | 0.9600 |
| C13—C14 | 1.402 (2) | C29—N1 | 3.010 (2) |
| C13—N1 | 1.4187 (19) | C29—H29A | 0.9600 |
| C14—C15 | 1.387 (2) | C29—H29B | 0.9600 |
| C14—C25 | 1.524 (2) | C29—H29C | 0.9600 |
| C15—C16 | 1.380 (2) | C30—H30A | 0.9600 |
| C15—H15 | 0.9300 | C30—H30B | 0.9600 |
| C16—C17 | 1.360 (2) | C30—H30C | 0.9600 |
| C16—H16 | 0.9300 | C31—N2 | 2.976 (2) |
| C17—C18 | 1.373 (2) | C31—H31A | 0.9600 |
| C17—H17 | 0.9300 | C31—H31B | 0.9600 |
| C18—H18 | 0.9300 | C31—H31C | 0.9600 |
| C19—C24 | 1.385 (2) | C32—H32A | 0.9600 |
| C19—C20 | 1.402 (2) | C32—H32B | 0.9600 |
| C19—N2 | 1.4178 (19) | C32—H32C | 0.9600 |
| N1—C1—C5 | 134.72 (18) | C14—C25—C27 | 112.17 (15) |
| N1—C1—C2 | 119.00 (18) | C28—C25—C27 | 108.18 (15) |
| C5—C1—C2 | 106.24 (17) | C14—C25—C29 | 111.31 (15) |
| N2—C2—C3 | 133.50 (18) | C28—C25—C29 | 109.42 (15) |
| N2—C2—C1 | 119.94 (18) | C27—C25—C29 | 106.54 (14) |
| C3—C2—C1 | 106.31 (17) | C20—C26—C30 | 109.44 (16) |
| C12—C3—C4 | 119.1 (2) | C20—C26—C31 | 111.36 (15) |
| C12—C3—C2 | 134.1 (2) | C30—C26—C31 | 109.69 (18) |
| C4—C3—C2 | 106.60 (18) | C20—C26—C32 | 112.48 (16) |
| C9—C4—C3 | 122.8 (2) | C30—C26—C32 | 107.58 (16) |
| C9—C4—C5 | 123.4 (2) | C31—C26—C32 | 106.17 (16) |
| C3—C4—C5 | 113.8 (2) | C25—C27—H27A | 109.5 |
| C6—C5—C4 | 119.2 (2) | C25—C27—H27B | 109.5 |
| C6—C5—C1 | 134.0 (2) | H27A—C27—H27B | 109.5 |
| C4—C5—C1 | 106.69 (18) | C25—C27—H27C | 109.5 |
| C5—C6—C7 | 118.5 (2) | H27A—C27—H27C | 109.5 |

| | | | |
|---|---|---|---|
| C5—C6—H6 | 120.8 | H27B—C27—H27C | 109.5 |
| C7—C6—H6 | 120.8 | C25—C28—N1 | 70.73 (10) |
| C8—C7—C6 | 122.2 (2) | C25—C28—H28A | 109.5 |
| C8—C7—H7 | 118.9 | N1—C28—H28A | 134.2 |
| C6—C7—H7 | 118.9 | C25—C28—H28B | 109.5 |
| C7—C8—C9 | 121.0 (2) | N1—C28—H28B | 113.3 |
| C7—C8—H8 | 119.5 | H28A—C28—H28B | 109.5 |
| C9—C8—H8 | 119.5 | C25—C28—H28C | 109.5 |
| C4—C9—C10 | 116.6 (2) | H28A—C28—H28C | 109.5 |
| C4—C9—C8 | 115.8 (2) | H28B—C28—H28C | 109.5 |
| C10—C9—C8 | 127.5 (3) | C25—C29—N1 | 72.04 (9) |
| C11—C10—C9 | 120.4 (2) | C25—C29—H29A | 109.5 |
| C11—C10—H10 | 119.8 | N1—C29—H29A | 136.4 |
| C9—C10—H10 | 119.8 | C25—C29—H29B | 109.5 |
| C10—C11—C12 | 122.5 (2) | H29A—C29—H29B | 109.5 |
| C10—C11—H11 | 118.7 | C25—C29—H29C | 109.5 |
| C12—C11—H11 | 118.7 | N1—C29—H29C | 110.7 |
| C3—C12—C11 | 118.5 (2) | H29A—C29—H29C | 109.5 |
| C3—C12—H12 | 120.8 | H29B—C29—H29C | 109.5 |
| C11—C12—H12 | 120.8 | C26—C30—H30A | 109.5 |
| C18—C13—C14 | 121.09 (17) | C26—C30—H30B | 109.5 |
| C18—C13—N1 | 118.71 (16) | H30A—C30—H30B | 109.5 |
| C14—C13—N1 | 119.75 (16) | C26—C30—H30C | 109.5 |
| C15—C14—C13 | 115.71 (16) | H30A—C30—H30C | 109.5 |
| C15—C14—C25 | 121.64 (16) | H30B—C30—H30C | 109.5 |
| C13—C14—C25 | 122.58 (16) | C26—C31—N2 | 76.07 (10) |
| C16—C15—C14 | 123.15 (18) | C26—C31—H31A | 109.5 |
| C16—C15—H15 | 118.4 | N2—C31—H31A | 139.8 |
| C14—C15—H15 | 118.4 | C26—C31—H31B | 109.5 |
| C17—C16—C15 | 119.76 (19) | H31A—C31—H31B | 109.5 |
| C17—C16—H16 | 120.1 | C26—C31—H31C | 109.5 |
| C15—C16—H16 | 120.1 | N2—C31—H31C | 105.3 |
| C16—C17—C18 | 119.52 (18) | H31A—C31—H31C | 109.5 |
| C16—C17—H17 | 120.2 | H31B—C31—H31C | 109.5 |
| C18—C17—H17 | 120.2 | C26—C32—H32A | 109.5 |
| C17—C18—C13 | 120.68 (17) | C26—C32—H32B | 109.5 |
| C17—C18—H18 | 119.7 | H32A—C32—H32B | 109.5 |
| C13—C18—H18 | 119.7 | C26—C32—H32C | 109.5 |
| C24—C19—C20 | 120.40 (17) | H32A—C32—H32C | 109.5 |
| C24—C19—N2 | 116.80 (17) | H32B—C32—H32C | 109.5 |
| C20—C19—N2 | 122.43 (16) | C1—N1—C13 | 123.03 (16) |
| C21—C20—C19 | 115.78 (17) | C1—N1—H29B | 92.7 |
| C21—C20—C26 | 121.07 (18) | C13—N1—H29B | 94.2 |
| C19—C20—C26 | 123.11 (17) | C1—N1—H28C | 140.3 |
| C22—C21—C20 | 123.55 (19) | C13—N1—H28C | 91.9 |
| C22—C21—H21 | 118.2 | H29B—N1—H28C | 64.2 |
| C20—C21—H21 | 118.2 | C1—N1—C29 | 105.62 (11) |
| C23—C22—C21 | 119.9 (2) | C13—N1—C29 | 80.55 (9) |
| C23—C22—H22 | 120.1 | H28C—N1—C29 | 58.5 |

| | | | |
|---|---|---|---|
| C21—C22—H22 | 120.1 | C1—N1—C28 | 146.13 (12) |
| C22—C23—C24 | 118.6 (2) | C13—N1—C28 | 79.23 (10) |
| C22—C23—N1$^{i}$ | 109.37 (16) | H29B—N1—C28 | 57.9 |
| C24—C23—N1$^{i}$ | 119.39 (15) | C29—N1—C28 | 48.82 (5) |
| C22—C23—H23 | 123.1 (10) | C2—N2—C19 | 122.07 (16) |
| C24—C23—H23 | 118.3 (10) | C2—N2—H31B | 121.2 |
| C23—C24—C19 | 121.7 (2) | C19—N2—H31B | 96.2 |
| C23—C24—H24 | 119.1 | C2—N2—C31 | 128.81 (12) |
| C19—C24—H24 | 119.1 | C19—N2—C31 | 81.48 (11) |
| C14—C25—C28 | 109.13 (14) | | |

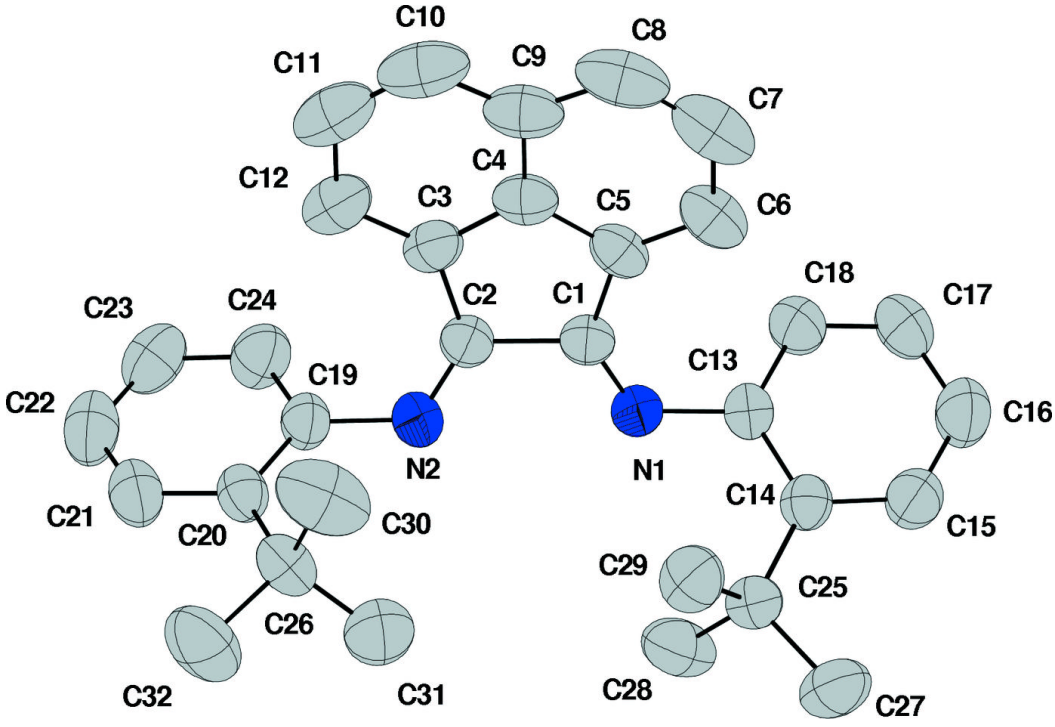
Symmetry codes: (i) −*x*, −*y*+1, −*z*+1.

*Hydrogen-bond geometry (Å, °)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| C23—H23···N1$^{i}$ | 0.97 (2) | 2.62 (2) | 3.331 (3) | 130.6 (17) |
| C28—H28C···N1 | 0.96 | 2.40 | 3.048 (3) | 125 |
| C29—H29B···N1 | 0.96 | 2.35 | 3.010 (3) | 125 |
| C31—H31B···N2 | 0.96 | 2.30 | 2.975 (3) | 127 |

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

Fig. 1


C10
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C19
C13
C22
C16
N2
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C32
C31

Fig. 2

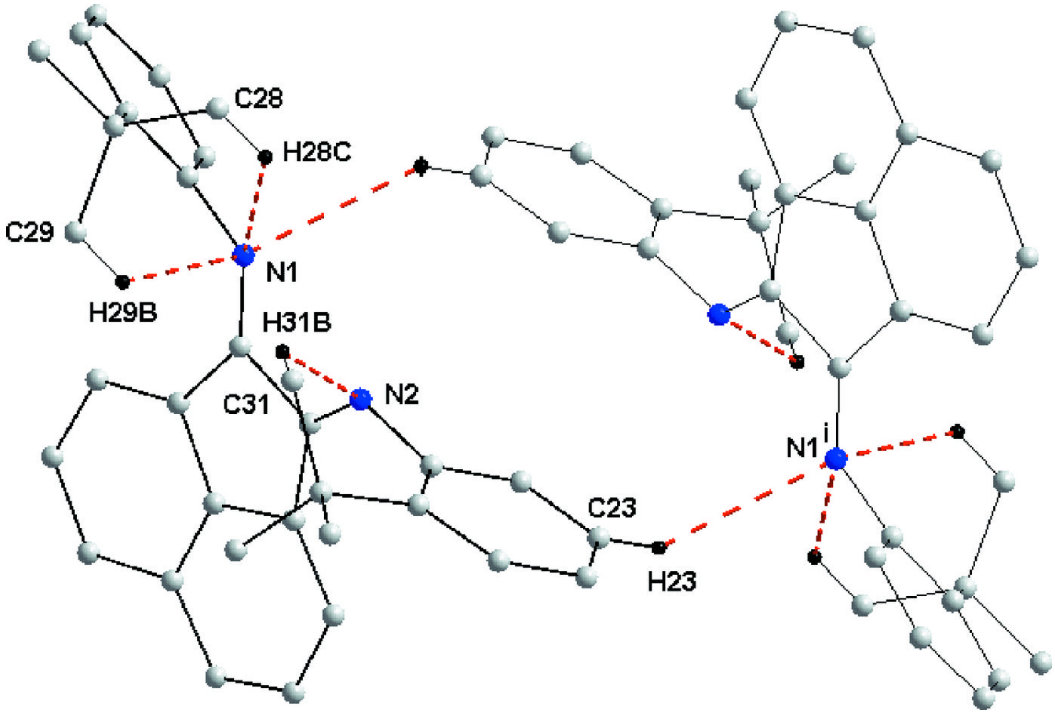

**Fig. 3**