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N-(4,5-Diazafluoren-9-ylidene)-4-methylaniline

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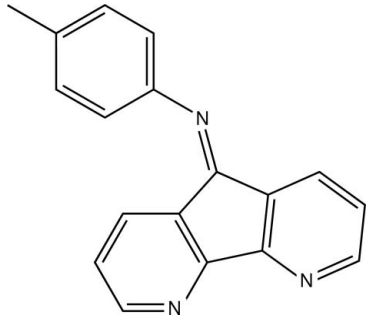
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.004 Å; R factor = 0.058; wR factor = 0.167; data-to-parameter ratio = 13.3.

In the molecule of the title compound, $C_{18}H_{13}N_3$, the 4,5-diazafluorenylidene unit is nearly planar and is oriented at a dihedral angle of 66.31 (1)° with respect to the benzene ring. In the crystal structure, molecules are stacked regularly along the c axis.

Related literature

For the photochemical properties of 4-methyl-*N*-(4,5-diazafluorenylidene)benzenamine, see: Wang & Rillema (1997). For related structures, see: Glagovich *et al.* (2004*a*,*b*); Peters *et al.* (1998); Wang *et al.* (2006).



Experimental

Crystal data

$C_{18}H_{13}N_3$
M_r = 271.31
Triclinic, $P\overline{1}$
a = 7.5970 (15) Å
b = 8.6100 (17) Å
c = 10.998 (2) Å
α = 77.11 (3)°
β = 87.48 (3)°
γ = 85.79 (3)°
V = 699.1 (2) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.08 mm^{-1}
T = 293 (2) K
0.30 × 0.20 × 0.20 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
Absorption correction: ψ scan (North *et al.*, 1968)
T_{min} = 0.977, T_{max} = 0.985
2742 measured reflections
2534 independent reflections
1829 reflections with $I > 2\sigma(I)$
R_{int} = 0.025
3 standard reflections
every 200 reflections
intensity decay: none

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.058
$wR(F^2)$ = 0.167
S = 1.00
2534 reflections
190 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.50 e Å^{-3}
$\Delta\rho_{min}$ = −0.28 e Å^{-3}

Data collection: *CAD-4 Software* (Enraf–Nonius, 1985); cell refinement: *CAD-4 Software*; data reduction: *XCAD4* (Harms & Wocadlo, 1995); 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*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BX2187).

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supplementary materials

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N-(4,5-Diazafluoren-9-ylidene)-4-methylaniline

H. Cang, D. Jin, S.-Q. Wang, B. Xu and J.-T. Wang

Comment

4-Methyl-*N*-(4,5-diazafluorenylidene)benzenamine, is one of the important ligands, being utilized to synthesize complexes with interesting photochemical properties (Wang & Rillema, 1997). The crystal structure of 4-methyl-*N*-(4,5-diazafluorenylidene)benzenamine monohydrate, (II) (Wang *et al.*, 2006) was reported, previously. We report herein the crystal structure of the title compound, (I), Fig. 1.The bond lengths and angles are comparable with the solvated form (II), and with other fluorenylidene compounds : *N*-fluorenylidene-aniline-benzene (4/1) (III) (Peters *et al.*, 1998), *N*-(9*H*-fluoren-9-ylidene)-*N*-(4-methoxyphenyl)amine, (IV) (Glagovich *et al.*, 2004*a*) and *N*-9*H*-fluoren-9-ylidene-3,4-dimethyl- aniline, (V) (Glagovich *et al.*, 2004*b*). The coplanar ring system is oriented with respect to benzene ring at a dihedral angle of 66.31 (1)°.In the crystal of the title compound, no obvious hydrogen bond is observed, and molecules are stacked regularly along *c* axis, Fig. 2.

Experimental

The title compound was synthesized by a method reported in literature (Wang & Rillema, 1997). The crystals were obtained by dissolving compound (I) (2.0 g, 6.3 mmol) into solution of acetic ether (50 ml, 1.0 mol/*L*), and evaporating the solvent slowly at room temperature for about 5 d.

Refinement

H atoms were positioned geometrically, with O—H = 0.82 and C—H = 0.93Å for aromatic H, and constrained to ride on their parent atoms, with U_{iso}(H) = xU_{eq}(C/O), where x = 1.2 for aromatic H and x = 1.5 for other H.

Figures

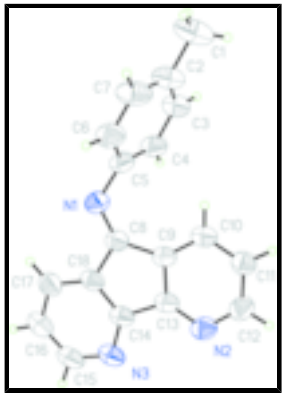

Fig. 1. A drawing of the title molecular structure, with the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level.

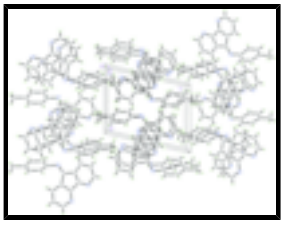

Fig. 2. A packing diagram for (I).

N-(4,5-Diazafluoren-9-ylidene)-4-methylaniline

Crystal data

$C_{18}H_{13}N_3$	$Z = 2$
$M_r = 271.31$	$F_{000} = 284$
Triclinic, $P\overline{1}$	$D_x = 1.289$ Mg m^{-3}
Hall symbol: -P 1	Mo $K\alpha$ radiation $\lambda = 0.71073$ Å
$a = 7.5970$ (15) Å	Cell parameters from 25 reflections
$b = 8.6100$ (17) Å	$\theta = 10$–13^o
$c = 10.998$ (2) Å	$\mu = 0.08$ mm^{-1}
$\alpha = 77.11$ (3)º	$T = 293$ (2) K
$\beta = 87.48$ (3)º	Plate, yellow
$\gamma = 85.79$ (3)º	0.30 × 0.20 × 0.20 mm
$V = 699.1$ (2) Å^3	

Data collection

Enraf–Nonius CAD-4 diffractometer	$R_{int} = 0.026$
Radiation source: fine-focus sealed tube	$\theta_{max} = 25.3^o$
Monochromator: graphite	$\theta_{min} = 1.9^o$
$T = 293$(2) K	$h = -9 \rightarrow 9$
$\omega/2\theta$ scans	$k = -9 \rightarrow 10$
Absorption correction: ψ scan (North *et al.*, 1968)	$l = 0 \rightarrow 13$
$T_{min} = 0.977$, $T_{max} = 0.985$	3 standard reflections
2742 measured reflections	every 200 reflections
2534 independent reflections	intensity decay: none
1829 reflections with $I > 2\sigma(I)$	

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.058$	H-atom parameters constrained
$wR(F^2) = 0.167$	$w = 1/[\sigma^2(F_o^2) + (0.05P)^2 + 0.85P]$ where $P = (F_o^2 + 2F_c^2)/3$

S = 1.01	$(\Delta/\sigma)_{max} < 0.001$
2534 reflections	$\Delta\rho_{max}$ = 0.50 e Å^{-3}
190 parameters	$\Delta\rho_{min}$ = −0.28 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.

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}
N1	0.2577 (3)	1.3579 (3)	0.5598 (2)	0.0539 (7)
C1	0.0220 (6)	1.2134 (6)	1.0717 (3)	0.0953 (14)
H1B	−0.0898	1.2705	1.0794	0.143*
H1C	0.1063	1.2457	1.1223	0.143*
H1D	0.0091	1.1008	1.0992	0.143*
N2	0.2970 (3)	0.8734 (3)	0.3984 (2)	0.0507 (6)
C2	0.0855 (5)	1.2499 (5)	0.9378 (3)	0.0658 (9)
N3	0.4535 (3)	1.1454 (3)	0.2071 (2)	0.0526 (6)
C3	0.2450 (4)	1.1836 (4)	0.8993 (3)	0.0584 (8)
H3B	0.3152	1.1156	0.9583	0.070*
C4	0.3018 (4)	1.2158 (4)	0.7759 (3)	0.0551 (8)
H4A	0.4094	1.1703	0.7528	0.066*
C5	0.1987 (4)	1.3158 (3)	0.6866 (3)	0.0501 (7)
C6	0.0411 (4)	1.3877 (4)	0.7236 (3)	0.0602 (8)
H6A	−0.0268	1.4590	0.6650	0.072*
C7	−0.0137 (5)	1.3531 (5)	0.8469 (3)	0.0723 (10)
H7A	−0.1203	1.4002	0.8701	0.087*
C8	0.2867 (4)	1.2521 (3)	0.4951 (2)	0.0432 (6)
C9	0.2518 (3)	1.0803 (3)	0.5183 (2)	0.0398 (6)
C10	0.1712 (4)	0.9757 (3)	0.6156 (3)	0.0463 (7)
H10A	0.1279	1.0086	0.6868	0.056*
C11	0.1576 (4)	0.8219 (4)	0.6030 (3)	0.0490 (7)
H11A	0.1044	0.7485	0.6663	0.059*
C12	0.2236 (4)	0.7763 (4)	0.4952 (3)	0.0537 (8)
H12A	0.2157	0.6705	0.4907	0.064*
C13	0.3085 (3)	1.0227 (3)	0.4114 (2)	0.0421 (6)
C14	0.3829 (3)	1.1547 (3)	0.3185 (2)	0.0429 (6)

C15	0.5163 (4)	1.2817 (4)	0.1427 (3)	0.0586 (8)
H15A	0.5670	1.2820	0.0641	0.070*
C16	0.5114 (4)	1.4213 (4)	0.1839 (3)	0.0617 (9)
H16A	0.5590	1.5111	0.1338	0.074*
C17	0.4364 (4)	1.4296 (4)	0.2994 (3)	0.0545 (8)
H17A	0.4317	1.5228	0.3292	0.065*
C18	0.3691 (4)	1.2917 (3)	0.3673 (2)	0.0457 (7)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.0653 (17)	0.0495 (14)	0.0480 (14)	0.0060 (12)	0.0035 (12)	−0.0170 (11)
C1	0.086 (3)	0.149 (4)	0.054 (2)	−0.014 (3)	0.016 (2)	−0.029 (2)
N2	0.0502 (15)	0.0541 (15)	0.0501 (14)	0.0092 (11)	−0.0023 (11)	−0.0200 (12)
C2	0.058 (2)	0.097 (3)	0.0483 (18)	−0.0093 (18)	0.0060 (16)	−0.0284 (18)
N3	0.0500 (15)	0.0690 (17)	0.0361 (13)	0.0112 (12)	−0.0010 (11)	−0.0111 (12)
C3	0.059 (2)	0.072 (2)	0.0459 (17)	0.0002 (16)	−0.0022 (14)	−0.0174 (15)
C4	0.0563 (19)	0.0615 (19)	0.0507 (18)	0.0054 (15)	0.0036 (14)	−0.0233 (15)
C5	0.0569 (18)	0.0489 (16)	0.0487 (17)	0.0000 (13)	0.0049 (14)	−0.0220 (13)
C6	0.0562 (19)	0.068 (2)	0.057 (2)	0.0105 (15)	0.0007 (15)	−0.0220 (16)
C7	0.055 (2)	0.106 (3)	0.060 (2)	0.0086 (19)	0.0085 (17)	−0.036 (2)
C8	0.0416 (15)	0.0473 (15)	0.0401 (15)	0.0086 (12)	−0.0030 (12)	−0.0117 (12)
C9	0.0340 (14)	0.0480 (15)	0.0382 (14)	0.0065 (11)	−0.0050 (11)	−0.0135 (12)
C10	0.0424 (15)	0.0585 (18)	0.0401 (15)	0.0051 (13)	−0.0046 (12)	−0.0171 (13)
C11	0.0469 (17)	0.0540 (18)	0.0452 (16)	0.0011 (13)	−0.0054 (13)	−0.0096 (13)
C12	0.0573 (19)	0.0476 (17)	0.0589 (19)	0.0029 (14)	−0.0046 (15)	−0.0186 (15)
C13	0.0368 (15)	0.0523 (16)	0.0375 (14)	0.0120 (12)	−0.0069 (11)	−0.0142 (12)
C14	0.0348 (14)	0.0567 (17)	0.0363 (14)	0.0108 (12)	−0.0068 (11)	−0.0116 (12)
C15	0.0571 (19)	0.081 (2)	0.0335 (15)	0.0107 (17)	0.0004 (13)	−0.0087 (15)
C16	0.062 (2)	0.074 (2)	0.0424 (17)	0.0018 (16)	−0.0010 (15)	−0.0003 (15)
C17	0.0590 (19)	0.0542 (18)	0.0468 (17)	0.0046 (14)	−0.0015 (14)	−0.0062 (14)
C18	0.0436 (16)	0.0542 (17)	0.0366 (14)	0.0103 (12)	−0.0056 (12)	−0.0078 (12)

Geometric parameters (Å, °)

N1—C8	1.277 (3)	C7—H7A	0.9300
N1—C5	1.422 (4)	C8—C9	1.486 (4)
C1—C2	1.501 (5)	C8—C18	1.491 (4)
C1—H1B	0.9600	C9—C10	1.387 (4)
C1—H1C	0.9600	C9—C13	1.414 (4)
C1—H1D	0.9600	C10—C11	1.373 (4)
N2—C12	1.327 (4)	C10—H10A	0.9300
N2—C13	1.334 (4)	C11—C12	1.392 (4)
C2—C7	1.388 (5)	C11—H11A	0.9300
C2—C3	1.391 (5)	C12—H12A	0.9300
N3—C14	1.332 (3)	C13—C14	1.477 (4)
N3—C15	1.337 (4)	C14—C18	1.397 (4)
C3—C4	1.380 (4)	C15—C16	1.374 (5)
C3—H3B	0.9300	C15—H15A	0.9300

C4—C5	1.382 (4)	C16—C17	1.383 (4)
C4—H4A	0.9300	C16—H16A	0.9300
C5—C6	1.394 (4)	C17—C18	1.375 (4)
C6—C7	1.374 (4)	C17—H17A	0.9300
C6—H6A	0.9300		
C8—N1—C5	121.0 (3)	C10—C9—C13	117.7 (3)
C2—C1—H1B	109.5	C10—C9—C8	133.8 (2)
C2—C1—H1C	109.5	C13—C9—C8	108.3 (2)
H1B—C1—H1C	109.5	C11—C10—C9	117.7 (3)
C2—C1—H1D	109.5	C11—C10—H10A	121.2
H1B—C1—H1D	109.5	C9—C10—H10A	121.2
H1C—C1—H1D	109.5	C10—C11—C12	119.8 (3)
C12—N2—C13	115.1 (2)	C10—C11—H11A	120.1
C7—C2—C3	117.2 (3)	C12—C11—H11A	120.1
C7—C2—C1	120.8 (3)	N2—C12—C11	124.6 (3)
C3—C2—C1	122.0 (4)	N2—C12—H12A	117.7
C14—N3—C15	114.0 (3)	C11—C12—H12A	117.7
C4—C3—C2	121.8 (3)	N2—C13—C9	125.1 (3)
C4—C3—H3B	119.1	N2—C13—C14	126.4 (2)
C2—C3—H3B	119.1	C9—C13—C14	108.5 (2)
C3—C4—C5	119.9 (3)	N3—C14—C18	125.3 (3)
C3—C4—H4A	120.0	N3—C14—C13	126.1 (3)
C5—C4—H4A	120.0	C18—C14—C13	108.6 (2)
C4—C5—C6	119.2 (3)	N3—C15—C16	124.8 (3)
C4—C5—N1	121.3 (3)	N3—C15—H15A	117.6
C6—C5—N1	119.3 (3)	C16—C15—H15A	117.6
C7—C6—C5	119.9 (3)	C15—C16—C17	120.6 (3)
C7—C6—H6A	120.1	C15—C16—H16A	119.7
C5—C6—H6A	120.1	C17—C16—H16A	119.7
C6—C7—C2	121.9 (3)	C18—C17—C16	115.9 (3)
C6—C7—H7A	119.1	C18—C17—H17A	122.0
C2—C7—H7A	119.1	C16—C17—H17A	122.0
N1—C8—C9	133.1 (3)	C17—C18—C14	119.4 (3)
N1—C8—C18	121.3 (3)	C17—C18—C8	131.6 (3)
C9—C8—C18	105.6 (2)	C14—C18—C8	108.9 (2)
C7—C2—C3—C4	−1.2 (5)	C12—N2—C13—C14	−179.6 (3)
C1—C2—C3—C4	179.2 (3)	C10—C9—C13—N2	−2.8 (4)
C2—C3—C4—C5	−0.4 (5)	C8—C9—C13—N2	−179.6 (2)
C3—C4—C5—C6	2.5 (5)	C10—C9—C13—C14	177.7 (2)
C3—C4—C5—N1	176.7 (3)	C8—C9—C13—C14	0.8 (3)
C8—N1—C5—C4	63.1 (4)	C15—N3—C14—C18	1.2 (4)
C8—N1—C5—C6	−122.7 (3)	C15—N3—C14—C13	−178.0 (3)
C4—C5—C6—C7	−2.9 (5)	N2—C13—C14—N3	−1.2 (4)
N1—C5—C6—C7	−177.3 (3)	C9—C13—C14—N3	178.4 (2)
C5—C6—C7—C2	1.3 (6)	N2—C13—C14—C18	179.5 (3)
C3—C2—C7—C6	0.7 (6)	C9—C13—C14—C18	−1.0 (3)
C1—C2—C7—C6	−179.7 (4)	C14—N3—C15—C16	0.0 (4)
C5—N1—C8—C9	8.7 (5)	N3—C15—C16—C17	−0.6 (5)

C5—N1—C8—C18	−172.5 (3)	C15—C16—C17—C18	−0.1 (4)
N1—C8—C9—C10	2.4 (5)	C16—C17—C18—C14	1.2 (4)
C18—C8—C9—C10	−176.5 (3)	C16—C17—C18—C8	177.0 (3)
N1—C8—C9—C13	178.6 (3)	N3—C14—C18—C17	−1.9 (4)
C18—C8—C9—C13	−0.3 (3)	C13—C14—C18—C17	177.4 (2)
C13—C9—C10—C11	2.2 (4)	N3—C14—C18—C8	−178.6 (2)
C8—C9—C10—C11	178.1 (3)	C13—C14—C18—C8	0.7 (3)
C9—C10—C11—C12	0.0 (4)	N1—C8—C18—C17	4.5 (5)
C13—N2—C12—C11	1.5 (4)	C9—C8—C18—C17	−176.4 (3)
C10—C11—C12—N2	−2.0 (5)	N1—C8—C18—C14	−179.3 (3)
C12—N2—C13—C9	0.9 (4)	C9—C8—C18—C14	−0.3 (3)

Fig. 1

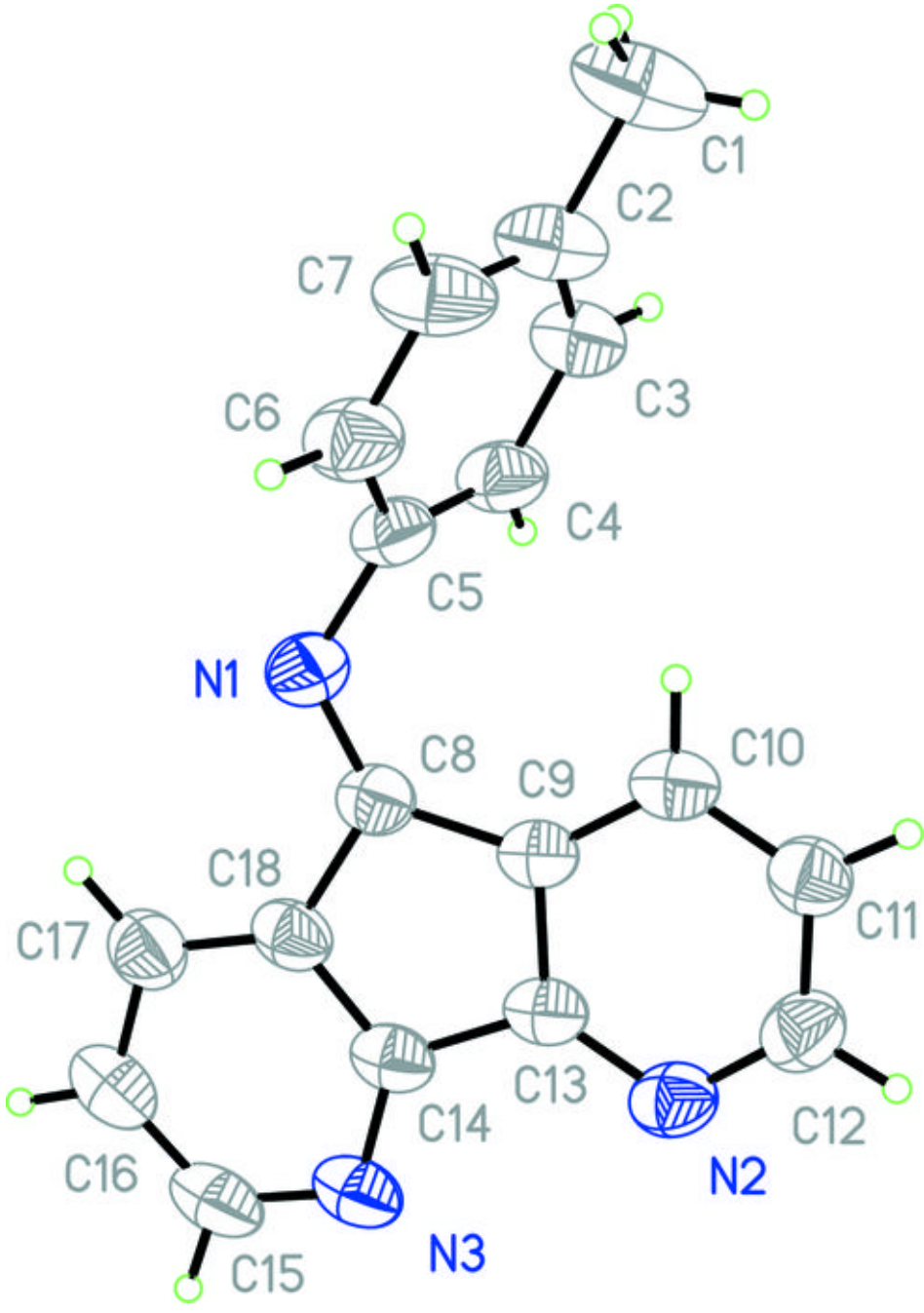
C1
C2
C7
C3
C6
C4
C5
N1
C8
C9
C10
C18
C11
C17
C13
C14
C12
C16
N2
N3
C15

Fig. 2