Acta Crystallographica Section E
**Structure Reports Online**
ISSN 1600-5368

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

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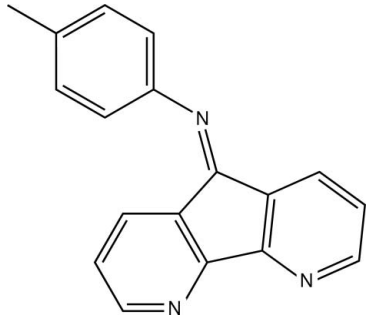
Received 16 November 2008; accepted 25 November 2008

Key indicators: single-crystal X-ray study; $T$ = 293 K; mean $\sigma$(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
$\alpha$ = 77.11 (3)°
$\beta$ = 87.48 (3)°
$\gamma$ = 85.79 (3)°
$V$ = 699.1 (2) Å$^3$
$Z$ = 2
Mo $K\alpha$ radiation
$\mu$ = 0.08 mm$^{-1}$
$T$ = 293 (2) K
0.30 × 0.20 × 0.20 mm

### Data collection

Enraf–Nonius CAD-4 diffractometer
Absorption correction: $\psi$ 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*.

The authors thank the Center of Testing and Analysis, Nanjing University, for support.

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

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# supporting information

*Acta Cryst.* (2009). E**65**, o6 [doi:10.1107/S1600536808039627]

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

**Hui Cang, Dong Jin, Si-Qing Wang, Bin Xu and Jin-Tang Wang**

### S1. 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.

### S2. 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.

### S3. 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.

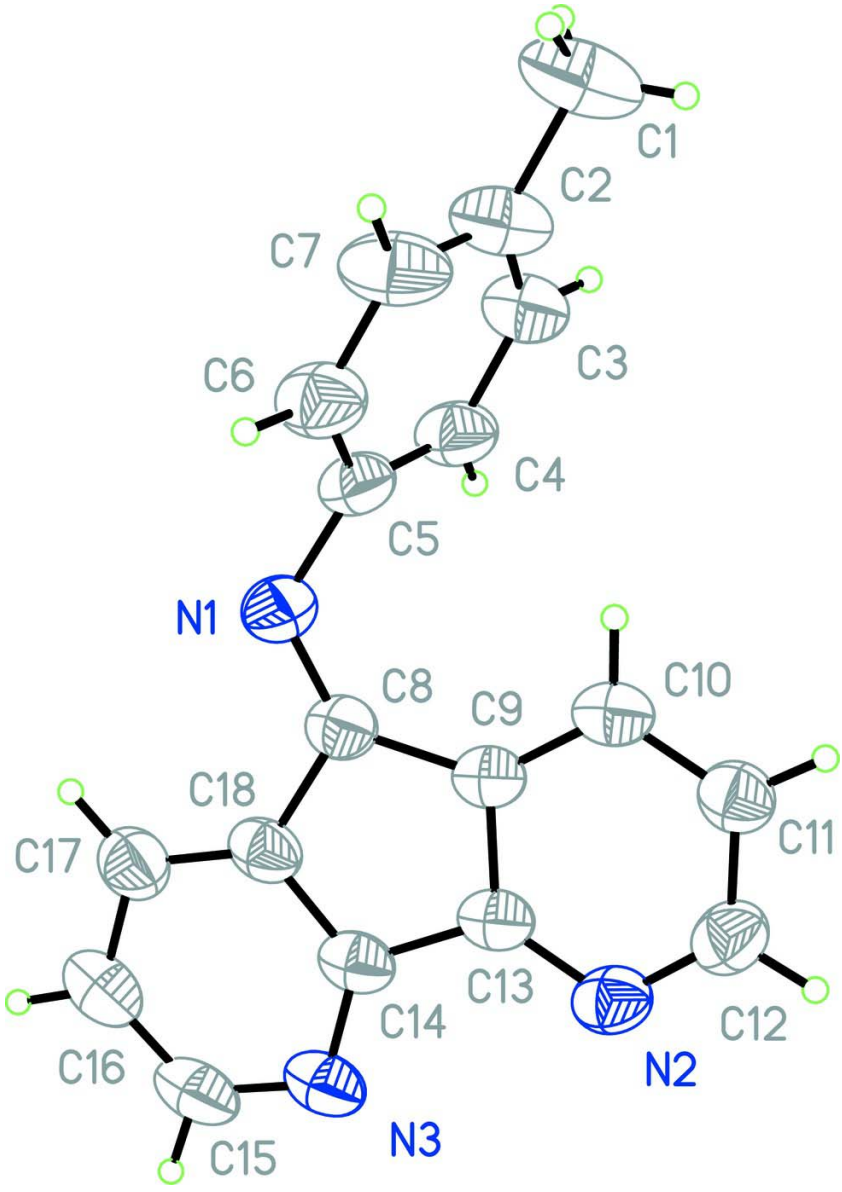


**Figure 1**

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

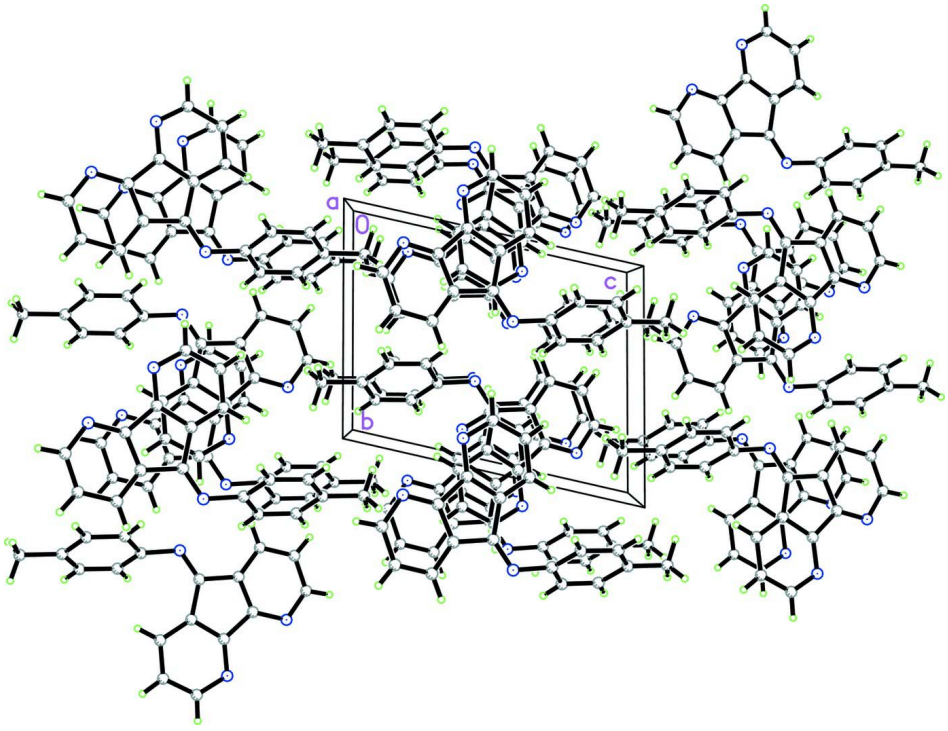


**Figure 2**

A packing diagram for (I).

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

*Crystal data*

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

*Data collection*

Enraf–Nonius CAD-4 diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
$\omega/2\theta$ scans
Absorption correction: $\psi$ scan (North *et al.*, 1968)
$T_{min} = 0.977$, $T_{max} = 0.985$
2742 measured reflections
2534 independent reflections
1829 reflections with $I > 2\sigma(I)$
$R_{int} = 0.026$
$\theta_{max} = 25.3°$, $\theta_{min} = 1.9°$
$h = -9\rightarrow9$
$k = -9\rightarrow10$
$l = 0\rightarrow13$
3 standard reflections every 200 reflections
intensity decay: none

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.058$
$wR(F^2) = 0.167$
$S = 1.01$
2534 reflections
190 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
$w = 1/[\sigma^2(F_o^2) + (0.05P)^2 + 0.85P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.50$ e Å$^{-3}$
$\Delta\rho_{min} = -0.28$ e Å$^{-3}$

*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 (Å²)*

| | *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 (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| 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, º)*

| | | | |
|---|---|---|---|
| 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) |