



# Crystal structure of (*E*)-*N*-[(2-methoxynaphthalen-1-yl)methylidene]-3-nitroaniline

Devika Bhai R.,<sup>a\*</sup> C. R. Girija,<sup>b</sup> Shalini Suresh<sup>c</sup> and Ramakrishna Reddy<sup>b</sup>

<sup>a</sup>Research & Development Centre, Bharathiar University, Coimbatore 641 046, India,

<sup>b</sup>Govt. Science College, Nrpathunga Road, Bangalore 560 001, India, and <sup>c</sup>SSMRV College, Jayanagar 4th T block, Bangalore 560 041, India. \*Correspondence e-mail: girijashivakumar@rediffmail.com

Received 20 October 2015; accepted 29 October 2015

Edited by W. T. A. Harrison, University of Aberdeen, Scotland

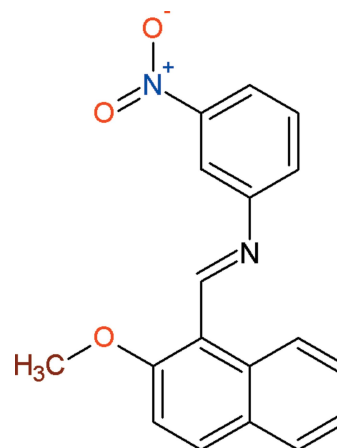
In the title compound, C<sub>18</sub>H<sub>14</sub>N<sub>2</sub>O<sub>3</sub>, the dihedral angle between the naphthalene ring system and the benzene ring is 59.99 (13)°. A short intramolecular C—H...N contact closes an *S*(6) ring. The nitro group is disordered over two orientations in a statistical ratio. In the crystal, weak C—H...O hydrogen bonds and very weak  $\pi$ – $\pi$  stacking interactions [centroid–centroid separation = 3.9168 (17) Å] are observed.

**Keywords:** crystal structure; naphthalimine Schiff base; hydrogen bonding.

**CCDC reference:** 1429914

## 1. Related literature

For background to Schiff bases, see: Tolulope *et al.* (2013).



## 2. Experimental

### 2.1. Crystal data

|   |  |
|---|--|
| C <sub>18</sub> H <sub>14</sub> N <sub>2</sub> O <sub>3</sub> | <i>V</i> = 1494.33 (12) Å <sup>3</sup> |
| <i>M<sub>r</sub></i> = 306.31                                 | <i>Z</i> = 4                           |
| Monoclinic, <i>P</i> 2 <sub>1</sub> / <i>c</i>                | Mo <i>K</i> α radiation                |
| <i>a</i> = 12.8481 (7) Å                                      | $\mu$ = 0.09 mm <sup>-1</sup>          |
| <i>b</i> = 15.4085 (6) Å                                      | <i>T</i> = 293 K                       |
| <i>c</i> = 7.6232 (3) Å                                       | 0.35 × 0.30 × 0.25 mm                  |
| $\beta$ = 98.040 (4)°   |  |

### 2.2. Data collection

|   |   |
|---|---|
| Bruker Kappa APEXII CCD diffractometer                            | 21149 measured reflections                      |
| Absorption correction: multi-scan ( <i>SADABS</i> ; Bruker, 2004) | 2622 independent reflections                    |
| <i>T</i> <sub>min</sub> = 0.957, <i>T</i> <sub>max</sub> = 0.989  | 1646 reflections with <i>I</i> > 2σ( <i>I</i> ) |
|   | <i>R</i> <sub>int</sub> = 0.046                 |

### 2.3. Refinement

|                                 |   |
|---------------------------------|---|
| $R[F^2 > 2\sigma(F^2)] = 0.055$ | 42 restraints                                 |
| $wR(F^2) = 0.143$               | H-atom parameters constrained                 |
| <i>S</i> = 1.14                 | $\Delta\rho_{\max} = 0.16$ e Å <sup>-3</sup>  |
| 2622 reflections                | $\Delta\rho_{\min} = -0.17$ e Å <sup>-3</sup> |
| 227 parameters                  |   |

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

| <i>D</i> —H... <i>A</i>     | <i>D</i> —H | H... <i>A</i> | <i>D</i> ... <i>A</i> | <i>D</i> —H... <i>A</i> |
|-----------------------------|-------------|---------------|-----------------------|-------------------------|
| C16—H16...N2                | 0.93        | 2.31          | 2.961 (3)             | 127                     |
| C13—H13...O1 <sup>ii</sup>  | 0.93        | 2.49          | 3.318 (14)            | 148                     |
| C18—H18A...O2 <sup>ii</sup> | 0.96        | 2.46          | 3.135 (18)            | 127                     |

Symmetry codes: (i)  $-x + 1, y + \frac{1}{2}, -z + \frac{3}{2}$ ; (ii)  $-x, -y, -z + 1$ .

Data collection: *APEX2* (Bruker, 2004); cell refinement: *APEX2* and *SAINT* (Bruker, 2004); data reduction: *SAINT* and *XPREP* (Bruker, 2004); program(s) used to solve structure: *SIR92* (Altomare *et al.*, 1993); program(s) used to refine structure: *SHELXL2014* (Sheldrick, 2015); molecular graphics: *OLEX2* (Dolomanov *et al.*, 2009); software used to prepare material for publication: *SHELXL2014*.

### Acknowledgements

The authors thank the management RSST, R&D Centre Bharathiar University and the Principal of SSMRV Degree College for support. RDB thanks The Oxford College of Science for access to research facilities. The authors also thank the SAIF, IIT, Chennai for providing the XRD facility.

---

Supporting information for this paper is available from the IUCr electronic archives (Reference: HB7529).

---

### References

- Altomare, A., Cascarano, G., Giacovazzo, C., Guagliardi, A., Burla, M. C., Polidori, G. & Camalli, M. (1994). *J. Appl. Cryst.* **27**, 435.
- Bruker (2004). *APEX2*, *SAINT*, *XPREP* and *SADABS*. Bruker AXS Inc., Madison, Wisconsin, USA.
- Dolomanov, O. V., Bourhis, L. J., Gildea, R. J., Howard, J. A. K. & Puschmann, H. (2009). *J. Appl. Cryst.* **42**, 339–341.
- Sheldrick, G. M. (2015). *Acta Cryst.* **C71**, 3–8.
- Tolulope, M., Fasina, R. & Dada, O. (2013). *J. Chem. Pharm. Res.* **5**, 177–181.

## supporting information

*Acta Cryst.* (2015). E71, o941–o942 [https://doi.org/10.1107/S2056989015020502]

### Crystal structure of (*E*)-*N*-[(2-methoxynaphthalen-1-yl)methylidene]-3-nitroaniline

Devika Bhai R., C. R. Girija, Shalini Suresh and Ramakrishna Reddy

#### S1. Chemical context

Schiff bases are considered an important class of organic compounds, which have wide applications. In recent years, they have gained significant interest in the area of drug research and development owing to the broad bioactivities such as insecticidal, antibacterial, antituberculosis and antimicrobial reported for the compounds and their metal complexes. These compounds play an important role in biological systems and are observed in various enzymes such as transaminases, tryptophan synthase etc. The important physical and biological properties of these compounds are related to the presence of the intramolecular hydrogen bond and proton transfer equilibrium. Schiff bases have also been utilized as ligands to synthesize metal complexes with interesting applications. The steric and inductive effects introduced by substituents present on the aromatic portion of the Schiff base can influence the properties of the ligand significantly. In continuation of our efforts in understanding the role of subtle electronic variations such as substituent effects on Chemistry and activity of Schiff bases and their metal complexes, we herein report the crystal structure of Schiff base derived from 3-nitroaniline and 2-methoxy naphthaldehyde (Tolulope *et al.*, 2013).

#### S2. Structural commentary

The molecule of title compound is non-planar, with a dihedral angle between the naphthyl and phenyl aromatic rings of 59.99 (13)°, in which the two rings are twisted from one another. The C9—O3 single bond of 1.358 (3) Å and the C7=N2 double bond of 1.261 (3) Å. The bond angle of C5—N2—C7 of the imine group is 117.5 (2)°, less than 120°. The bond length of the nitro group is N1—O2(1.241 (6)Å) and N1—O1(1.239 (6)Å) and bond angle in O2—N1—O1 is 123.4 (12)° which is more than the planar bond angle of 120°. The torsion angles C8—C7—N2—C5 is 178.1 (2)°. These values support that the configurations about the N2=C7 bond is anti(*E*-form), which is in accordance with the enol-imine tautomeric form.

#### S3. Supramolecular features

The title compound has an intra molecular C16—H16···N2 hydrogen bond forming an S(6) motif (Table 2). Also there is a C—H···O intermolecular interaction, in which a C—H of the naphthyl ring of one molecule and O-atom of the nitro group of another molecule are linked to one another.

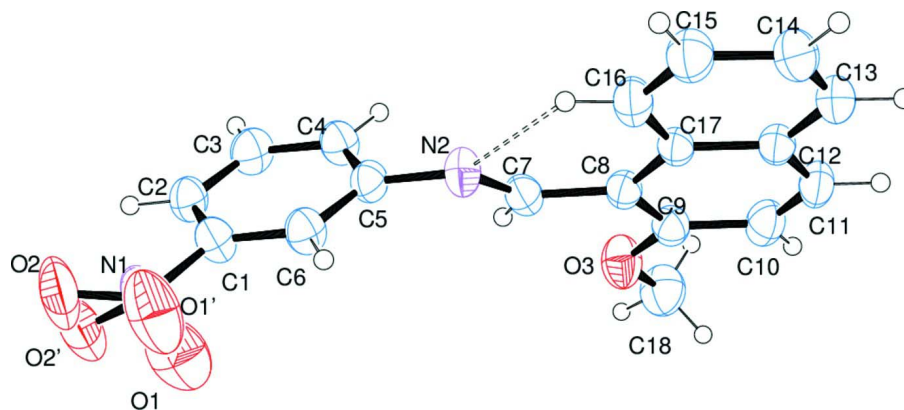
#### S4. Synthesis and crystallization

The block-like, yellow single crystals of the compound C<sub>18</sub> H<sub>14</sub> N<sub>2</sub> O<sub>3</sub>, were grown using 1:1 mixture of CHCl<sub>3</sub> and methanol as solvent by slow evaporation technique.

### S5. Refinement

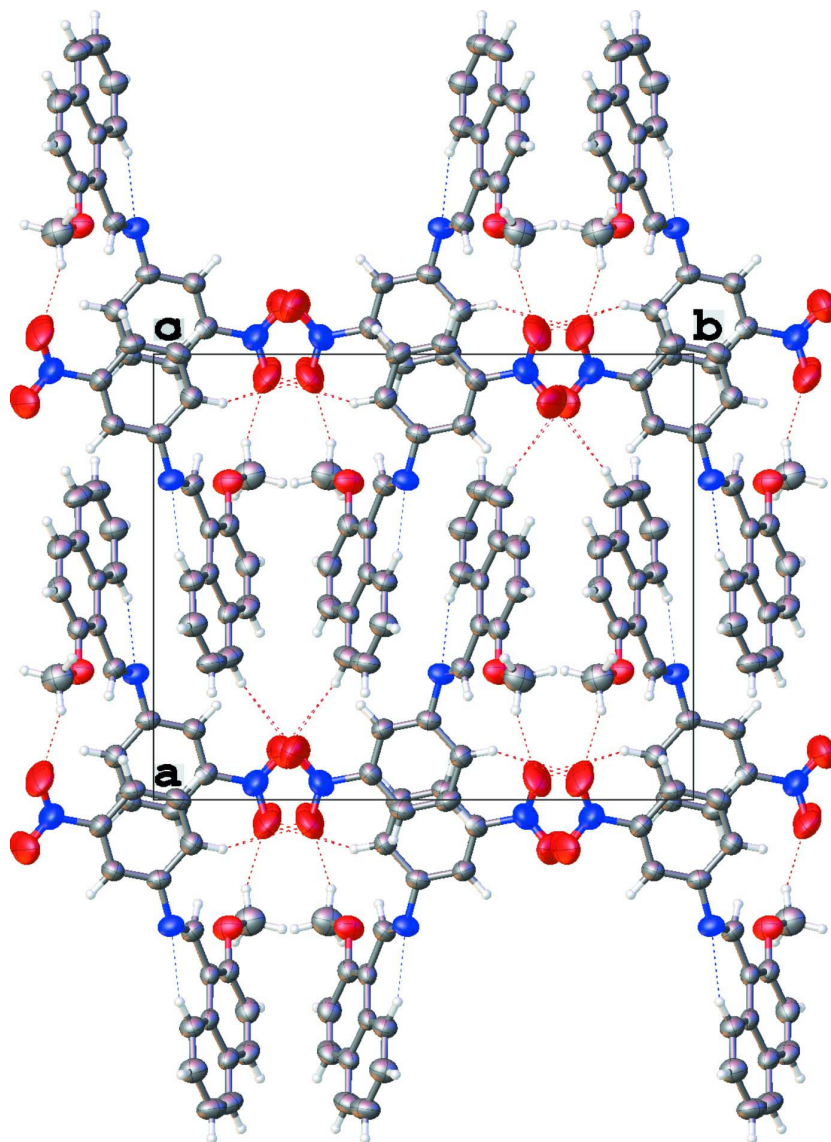
The hydrogen atoms in the structure were positioned geometrically (C—H = 0.93–0.98 Å, N—H = 0.86 Å) and were refined using a riding model with  $U_{iso}(H) = xU_{eq}(C,N)$ , where  $x = 1.5$  for methyl and 1.2 for all other atoms. The two oxygen atoms of the nitro group are disordered over two orientations. The SADI, SIMU, and ISOR commands in SHELXL (Sheldrick, 2015) were used to model the disorder.

Crystal data, data collection and structure refinement details are summarized in Table 1.



**Figure 1**

Plot of the title compound showing the intramolecular C—H...N interaction as a dashed line.



**Figure 2**

Crystal packing diagram showing the C—H...N and C—H...O interactions as dashed lines

*(E)*-*N*-[(2-Methoxynaphthalen-1-yl)methylidene]-3-nitroaniline

*Crystal data*

$C_{18}H_{14}N_2O_3$

$M_r = 306.31$

Monoclinic,  $P2_1/c$

$a = 12.8481 (7) \text{ \AA}$

$b = 15.4085 (6) \text{ \AA}$

$c = 7.6232 (3) \text{ \AA}$

$\beta = 98.040 (4)^\circ$

$V = 1494.33 (12) \text{ \AA}^3$

$Z = 4$

$F(000) = 640$

$D_x = 1.362 \text{ Mg m}^{-3}$

Melting point: 407 K

Mo  $K\alpha$  radiation,  $\lambda = 0.71073 \text{ \AA}$

Cell parameters from 5867 reflections

$\theta = 2.6\text{--}29.9^\circ$

$\mu = 0.09 \text{ mm}^{-1}$

$T = 293 \text{ K}$

Block, yellow

$0.35 \times 0.30 \times 0.25 \text{ mm}$

*Data collection*

|  |  |
|--|--|
| Bruker Kappa APEXII CCD diffractometer                   | 21149 measured reflections   |
| Radiation source: fine-focus sealed tube                 | 2622 independent reflections   |
| Graphite monochromator                                   | 1646 reflections with $I > 2\sigma(I)$                                 |
| $\omega$ and $\varphi$ scan                              | $R_{\text{int}} = 0.046$   |
| Absorption correction: multi-scan (SADABS; Bruker, 2004) | $\theta_{\text{max}} = 25.0^\circ$ , $\theta_{\text{min}} = 2.1^\circ$ |
| $T_{\text{min}} = 0.957$ , $T_{\text{max}} = 0.989$      | $h = -15 \rightarrow 15$   |
|  | $k = -18 \rightarrow 18$   |
|  | $l = -9 \rightarrow 8$   |

*Refinement*

|                                 |  |
|---------------------------------|--|
| Refinement on $F^2$             | Hydrogen site location: inferred from neighbouring sites     |
| Least-squares matrix: full      | H-atom parameters constrained                                |
| $R[F^2 > 2\sigma(F^2)] = 0.055$ | $w = 1/[\sigma^2(F_o^2) + (0.0403P)^2 + 0.9181P]$            |
| $wR(F^2) = 0.143$               | where $P = (F_o^2 + 2F_c^2)/3$                               |
| $S = 1.14$                      | $(\Delta/\sigma)_{\text{max}} < 0.001$                       |
| 2622 reflections                | $\Delta\rho_{\text{max}} = 0.16 \text{ e } \text{\AA}^{-3}$  |
| 227 parameters                  | $\Delta\rho_{\text{min}} = -0.17 \text{ e } \text{\AA}^{-3}$ |
| 42 restraints                   |  |

*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.

*Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters ( $\text{\AA}^2$ )*

|     | <i>x</i>     | <i>y</i>      | <i>z</i>   | $U_{\text{iso}}^*/U_{\text{eq}}$ | Occ. (<1) |
|-----|--------------|---------------|------------|----------------------------------|-----------|
| C1  | 0.0584 (2)   | -0.09762 (18) | 0.7746 (4) | 0.0523 (7)                       |           |
| C2  | -0.0226 (2)  | -0.03905 (19) | 0.7675 (4) | 0.0523 (7)                       |           |
| H2  | -0.0893      | -0.0562       | 0.7877     | 0.063*                           |           |
| C3  | -0.0015 (2)  | 0.0457 (2)    | 0.7296 (4) | 0.0566 (8)                       |           |
| H3  | -0.0545      | 0.0870        | 0.7247     | 0.068*                           |           |
| C4  | 0.0976 (2)   | 0.07014 (18)  | 0.6986 (4) | 0.0517 (7)                       |           |
| H4  | 0.1103       | 0.1276        | 0.6710     | 0.062*                           |           |
| C5  | 0.17862 (19) | 0.01015 (18)  | 0.7081 (3) | 0.0461 (7)                       |           |
| C6  | 0.1585 (2)   | -0.07494 (18) | 0.7487 (4) | 0.0498 (7)                       |           |
| H6  | 0.2118       | -0.1162       | 0.7583     | 0.060*                           |           |
| C7  | 0.2942 (2)   | 0.07229 (17)  | 0.5418 (4) | 0.0475 (7)                       |           |
| H7  | 0.2333       | 0.0823        | 0.4630     | 0.057*                           |           |
| C8  | 0.39198 (19) | 0.10445 (16)  | 0.4875 (3) | 0.0430 (6)                       |           |
| C9  | 0.3839 (2)   | 0.13912 (17)  | 0.3178 (4) | 0.0485 (7)                       |           |
| C10 | 0.4724 (2)   | 0.17407 (18)  | 0.2520 (4) | 0.0571 (8)                       |           |
| H10 | 0.4658       | 0.1972        | 0.1383     | 0.069*                           |           |
| C11 | 0.5665 (2)   | 0.17361 (19)  | 0.3552 (4) | 0.0585 (8)                       |           |
| H11 | 0.6242       | 0.1970        | 0.3107     | 0.070*                           |           |
| C12 | 0.5808 (2)   | 0.13905 (17)  | 0.5278 (4) | 0.0480 (7)                       |           |
| C13 | 0.6798 (2)   | 0.1397 (2)    | 0.6330 (4) | 0.0622 (8)                       |           |
| H13 | 0.7372       | 0.1626        | 0.5870     | 0.075*                           |           |

|      |              |               |            |             |          |
|------|--------------|---------------|------------|-------------|----------|
| C14  | 0.6933 (2)   | 0.1077 (2)    | 0.7988 (5) | 0.0649 (9)  |          |
| H14  | 0.7595       | 0.1082        | 0.8662     | 0.078*      |          |
| C15  | 0.6069 (2)   | 0.0736 (2)    | 0.8696 (4) | 0.0622 (8)  |          |
| H15  | 0.6161       | 0.0521        | 0.9847     | 0.075*      |          |
| C16  | 0.5094 (2)   | 0.07154 (18)  | 0.7716 (4) | 0.0537 (7)  |          |
| H16  | 0.4532       | 0.0484        | 0.8211     | 0.064*      |          |
| C17  | 0.49240 (19) | 0.10383 (16)  | 0.5969 (3) | 0.0424 (6)  |          |
| C18  | 0.2726 (3)   | 0.1775 (2)    | 0.0501 (4) | 0.0753 (10) |          |
| H18A | 0.2006       | 0.1711        | -0.0023    | 0.113*      |          |
| H18B | 0.2897       | 0.2381        | 0.0620     | 0.113*      |          |
| H18C | 0.3176       | 0.1502        | -0.0242    | 0.113*      |          |
| N1   | 0.0376 (2)   | -0.18893 (19) | 0.8121 (5) | 0.0849 (9)  |          |
| N2   | 0.28245 (17) | 0.03270 (16)  | 0.6825 (3) | 0.0544 (6)  |          |
| O3   | 0.28735 (15) | 0.13796 (14)  | 0.2188 (3) | 0.0669 (6)  |          |
| O1   | 0.1053 (10)  | -0.2414 (9)   | 0.778 (4)  | 0.106 (5)   | 0.50 (4) |
| O2   | -0.0433 (13) | -0.2105 (10)  | 0.869 (4)  | 0.108 (5)   | 0.50 (4) |
| O1'  | 0.1105 (8)   | -0.2356 (11)  | 0.876 (4)  | 0.106 (5)   | 0.50 (4) |
| O2'  | -0.0556 (6)  | -0.2107 (9)   | 0.772 (3)  | 0.097 (4)   | 0.50 (4) |

*Atomic displacement parameters (Å<sup>2</sup>)*

|     | $U^{11}$    | $U^{22}$    | $U^{33}$    | $U^{12}$     | $U^{13}$    | $U^{23}$     |
|-----|-------------|-------------|-------------|--------------|-------------|--------------|
| C1  | 0.0460 (16) | 0.0463 (17) | 0.0646 (19) | -0.0062 (13) | 0.0072 (14) | 0.0025 (14)  |
| C2  | 0.0372 (15) | 0.061 (2)   | 0.0598 (18) | -0.0039 (13) | 0.0102 (13) | -0.0005 (15) |
| C3  | 0.0424 (16) | 0.059 (2)   | 0.069 (2)   | 0.0107 (14)  | 0.0099 (14) | 0.0020 (15)  |
| C4  | 0.0476 (16) | 0.0463 (16) | 0.0620 (18) | -0.0013 (13) | 0.0096 (13) | 0.0044 (14)  |
| C5  | 0.0354 (14) | 0.0553 (18) | 0.0477 (16) | -0.0035 (13) | 0.0059 (12) | -0.0008 (13) |
| C6  | 0.0393 (15) | 0.0476 (17) | 0.0625 (18) | 0.0038 (12)  | 0.0077 (13) | 0.0042 (13)  |
| C7  | 0.0418 (15) | 0.0458 (16) | 0.0548 (18) | -0.0055 (12) | 0.0063 (13) | -0.0008 (13) |
| C8  | 0.0444 (16) | 0.0331 (14) | 0.0543 (17) | -0.0011 (11) | 0.0163 (13) | -0.0015 (12) |
| C9  | 0.0487 (17) | 0.0414 (16) | 0.0577 (18) | -0.0013 (12) | 0.0149 (14) | -0.0002 (13) |
| C10 | 0.062 (2)   | 0.0522 (18) | 0.0618 (19) | -0.0004 (14) | 0.0238 (16) | 0.0072 (14)  |
| C11 | 0.0534 (19) | 0.0560 (19) | 0.072 (2)   | -0.0088 (14) | 0.0310 (16) | -0.0021 (15) |
| C12 | 0.0446 (16) | 0.0420 (16) | 0.0609 (18) | -0.0049 (12) | 0.0196 (14) | -0.0074 (13) |
| C13 | 0.0451 (18) | 0.072 (2)   | 0.073 (2)   | -0.0127 (15) | 0.0206 (16) | -0.0146 (17) |
| C14 | 0.0406 (17) | 0.081 (2)   | 0.073 (2)   | -0.0059 (15) | 0.0070 (15) | -0.0106 (18) |
| C15 | 0.0538 (19) | 0.073 (2)   | 0.0598 (19) | -0.0040 (16) | 0.0084 (15) | 0.0028 (16)  |
| C16 | 0.0436 (16) | 0.0553 (18) | 0.0644 (19) | -0.0043 (13) | 0.0146 (14) | 0.0028 (15)  |
| C17 | 0.0420 (15) | 0.0313 (14) | 0.0567 (17) | -0.0024 (11) | 0.0164 (13) | -0.0051 (12) |
| C18 | 0.074 (2)   | 0.091 (3)   | 0.061 (2)   | -0.0056 (19) | 0.0080 (17) | 0.0119 (18)  |
| N1  | 0.062 (2)   | 0.0590 (19) | 0.136 (3)   | -0.0098 (16) | 0.021 (2)   | 0.0112 (19)  |
| N2  | 0.0412 (13) | 0.0627 (16) | 0.0610 (15) | -0.0060 (11) | 0.0132 (11) | 0.0063 (13)  |
| O3  | 0.0566 (13) | 0.0810 (15) | 0.0629 (13) | -0.0057 (11) | 0.0072 (10) | 0.0200 (11)  |
| O1  | 0.086 (5)   | 0.061 (4)   | 0.170 (12)  | 0.014 (3)    | 0.012 (6)   | 0.019 (6)    |
| O2  | 0.094 (6)   | 0.071 (4)   | 0.172 (12)  | -0.023 (4)   | 0.066 (6)   | 0.021 (8)    |
| O1' | 0.083 (5)   | 0.069 (5)   | 0.165 (12)  | 0.003 (4)    | 0.008 (6)   | 0.056 (7)    |
| O2' | 0.074 (4)   | 0.067 (4)   | 0.153 (11)  | -0.025 (3)   | 0.029 (4)   | 0.002 (7)    |

*Geometric parameters (Å, °)*

|           |           |               |           |
|-----------|-----------|---------------|-----------|
| C1—C2     | 1.372 (4) | C11—H11       | 0.9300    |
| C1—C6     | 1.374 (4) | C11—C12       | 1.407 (4) |
| C1—N1     | 1.468 (4) | C12—C13       | 1.406 (4) |
| C2—H2     | 0.9300    | C12—C17       | 1.425 (3) |
| C2—C3     | 1.372 (4) | C13—H13       | 0.9300    |
| C3—H3     | 0.9300    | C13—C14       | 1.345 (4) |
| C3—C4     | 1.380 (4) | C14—H14       | 0.9300    |
| C4—H4     | 0.9300    | C14—C15       | 1.402 (4) |
| C4—C5     | 1.386 (4) | C15—H15       | 0.9300    |
| C5—C6     | 1.380 (4) | C15—C16       | 1.366 (4) |
| C5—N2     | 1.418 (3) | C16—H16       | 0.9300    |
| C6—H6     | 0.9300    | C16—C17       | 1.410 (4) |
| C7—H7     | 0.9300    | C18—H18A      | 0.9600    |
| C7—C8     | 1.463 (3) | C18—H18B      | 0.9600    |
| C7—N2     | 1.261 (3) | C18—H18C      | 0.9600    |
| C8—C9     | 1.390 (4) | C18—O3        | 1.412 (3) |
| C8—C17    | 1.435 (4) | N1—O1         | 1.241 (6) |
| C9—C10    | 1.412 (4) | N1—O2         | 1.227 (6) |
| C9—O3     | 1.358 (3) | N1—O1'        | 1.227 (6) |
| C10—H10   | 0.9300    | N1—O2'        | 1.239 (6) |
| C10—C11   | 1.347 (4) |               |           |
|           |           |               |           |
| C2—C1—C6  | 123.1 (3) | C11—C12—C17   | 118.9 (3) |
| C2—C1—N1  | 118.7 (3) | C13—C12—C11   | 121.4 (3) |
| C6—C1—N1  | 118.2 (3) | C13—C12—C17   | 119.7 (3) |
| C1—C2—H2  | 121.2     | C12—C13—H13   | 119.3     |
| C3—C2—C1  | 117.6 (3) | C14—C13—C12   | 121.4 (3) |
| C3—C2—H2  | 121.2     | C14—C13—H13   | 119.3     |
| C2—C3—H3  | 119.7     | C13—C14—H14   | 120.2     |
| C2—C3—C4  | 120.7 (3) | C13—C14—C15   | 119.6 (3) |
| C4—C3—H3  | 119.7     | C15—C14—H14   | 120.2     |
| C3—C4—H4  | 119.6     | C14—C15—H15   | 119.6     |
| C3—C4—C5  | 120.9 (3) | C16—C15—C14   | 120.9 (3) |
| C5—C4—H4  | 119.6     | C16—C15—H15   | 119.6     |
| C4—C5—N2  | 122.9 (2) | C15—C16—H16   | 119.4     |
| C6—C5—C4  | 118.8 (2) | C15—C16—C17   | 121.1 (3) |
| C6—C5—N2  | 118.2 (2) | C17—C16—H16   | 119.4     |
| C1—C6—C5  | 118.9 (2) | C12—C17—C8    | 118.8 (2) |
| C1—C6—H6  | 120.6     | C16—C17—C8    | 124.0 (2) |
| C5—C6—H6  | 120.6     | C16—C17—C12   | 117.2 (2) |
| C8—C7—H7  | 116.1     | H18A—C18—H18B | 109.5     |
| N2—C7—H7  | 116.1     | H18A—C18—H18C | 109.5     |
| N2—C7—C8  | 127.9 (3) | H18B—C18—H18C | 109.5     |
| C9—C8—C7  | 116.0 (2) | O3—C18—H18A   | 109.5     |
| C9—C8—C17 | 119.2 (2) | O3—C18—H18B   | 109.5     |
| C17—C8—C7 | 124.8 (2) | O3—C18—H18C   | 109.5     |



|               |             |                 |            |
|---------------|-------------|-----------------|------------|
| C8—C9—C10     | 121.2 (3)   | O1—N1—C1        | 115.6 (9)  |
| O3—C9—C8      | 117.1 (2)   | O2—N1—C1        | 121.0 (8)  |
| O3—C9—C10     | 121.7 (3)   | O2—N1—O1        | 123.4 (12) |
| C9—C10—H10    | 120.3       | O1'—N1—C1       | 119.3 (8)  |
| C11—C10—C9    | 119.5 (3)   | O1'—N1—O2'      | 126.6 (12) |
| C11—C10—H10   | 120.3       | O2'—N1—C1       | 114.1 (8)  |
| C10—C11—H11   | 118.7       | C7—N2—C5        | 117.5 (2)  |
| C10—C11—C12   | 122.5 (3)   | C9—O3—C18       | 119.7 (2)  |
| C12—C11—H11   | 118.7       |                 |            |
|               |             |                 |            |
| C1—C2—C3—C4   | 0.5 (4)     | C9—C8—C17—C16   | 179.0 (2)  |
| C2—C1—C6—C5   | -2.1 (4)    | C9—C10—C11—C12  | 0.3 (4)    |
| C2—C1—N1—O1   | 164.6 (15)  | C10—C9—O3—C18   | -4.2 (4)   |
| C2—C1—N1—O2   | -13.9 (19)  | C10—C11—C12—C13 | -179.8 (3) |
| C2—C1—N1—O1'  | -156.0 (17) | C10—C11—C12—C17 | -0.6 (4)   |
| C2—C1—N1—O2'  | 24.5 (14)   | C11—C12—C13—C14 | 179.1 (3)  |
| C2—C3—C4—C5   | -1.1 (4)    | C11—C12—C17—C8  | 0.3 (4)    |
| C3—C4—C5—C6   | 0.2 (4)     | C11—C12—C17—C16 | -178.6 (2) |
| C3—C4—C5—N2   | -178.1 (2)  | C12—C13—C14—C15 | -0.5 (5)   |
| C4—C5—C6—C1   | 1.3 (4)     | C13—C12—C17—C8  | 179.6 (2)  |
| C4—C5—N2—C7   | -53.8 (4)   | C13—C12—C17—C16 | 0.6 (4)    |
| C6—C1—C2—C3   | 1.1 (4)     | C13—C14—C15—C16 | 0.7 (5)    |
| C6—C1—N1—O1   | -15.6 (15)  | C14—C15—C16—C17 | -0.2 (5)   |
| C6—C1—N1—O2   | 165.9 (18)  | C15—C16—C17—C8  | -179.3 (3) |
| C6—C1—N1—O1'  | 23.9 (17)   | C15—C16—C17—C12 | -0.4 (4)   |
| C6—C1—N1—O2'  | -155.6 (13) | C17—C8—C9—C10   | -0.4 (4)   |
| C6—C5—N2—C7   | 127.9 (3)   | C17—C8—C9—O3    | 179.7 (2)  |
| C7—C8—C9—C10  | 178.5 (2)   | C17—C12—C13—C14 | -0.1 (4)   |
| C7—C8—C9—O3   | -1.4 (3)    | N1—C1—C2—C3     | -179.0 (3) |
| C7—C8—C17—C12 | -178.7 (2)  | N1—C1—C6—C5     | 178.1 (3)  |
| C7—C8—C17—C16 | 0.2 (4)     | N2—C5—C6—C1     | 179.7 (2)  |
| C8—C7—N2—C5   | 178.1 (2)   | N2—C7—C8—C9     | 174.4 (3)  |
| C8—C9—C10—C11 | 0.2 (4)     | N2—C7—C8—C17    | -6.8 (4)   |
| C8—C9—O3—C18  | 175.7 (3)   | O3—C9—C10—C11   | -179.9 (3) |
| C9—C8—C17—C12 | 0.1 (4)     |                 |            |

Hydrogen-bond geometry ( $\text{\AA}$ ,  $^\circ$ )

| $D-H\cdots A$                      | $D-H$ | $H\cdots A$ | $D\cdots A$ | $D-H\cdots A$ |
|------------------------------------|-------|-------------|-------------|---------------|
| C16—H16 $\cdots$ N2                | 0.93  | 2.31        | 2.961 (3)   | 127           |
| C13—H13 $\cdots$ O1 <sup>ii</sup>  | 0.93  | 2.49        | 3.318 (14)  | 148           |
| C18—H18A $\cdots$ O2 <sup>ii</sup> | 0.96  | 2.46        | 3.135 (18)  | 127           |

Symmetry codes: (i)  $-x+1, y+1/2, -z+3/2$ ; (ii)  $-x, -y, -z+1$ .