# 3-Phenyl-1,5-di-2-pyridylpentane-1,5-dione

Leanne James,[a] Glenn E. M. Maguire,[b] Bice S. Martincigh,[b] Vickie McKee[a]* and Nombuso Ndlovu[b]

[a]Chemistry Department, Loughborough University, Loughborough, Leicestershire LE11 3TU, England, and [b]School of Chemistry, University of KwaZulu-Natal, Howard College, Durban 4041, South Africa

Correspondence e-mail: v.mckee@lboro.ac.uk

The title compound, $C_{21}H_{18}N_2O_2$, shows both $\pi$–$\pi$ stacking of the pyridine groups and significant C—H$\cdots$O and C—H$\cdots$N hydrogen bonding.

## Comment

2,2′:6′,2″-Terpyridine and its derivatives have been widely used for complexation of transition metals (Gao *et al.*, 2003; Sharma *et al.*, 2005). In more recent times there has been a lot of interest in these ligands as synthetic building blocks for supramolecular structures (Hofmeier & Schubert, 2004). Multistep syntheses (Kröhnke, 1976; Collin *et al.*, 1996), as well as single-step approaches for these ligands, have been developed (Tu *et al.*, 2005).

The title compound, (I), is a synthetic intermediate for the compound 4′-phenyl-2,2′:6′,2″-terpyridine. The synthesis and characterization of the latter have been described previously along with other derivatives (Constable *et al.*, 1990; Moya *et al.*, 2001). The structure of the fluorinated analogue, 3-(4-fluorophenyl)pentane-1,5-bis(2-pyridyl)-1,5-dione, (II), has been reported previously (Constable *et al.*, 1998). A search of the CSD (Version 5.27) showed that no other members of this family have been structurally characterized (Allen, 2002; Fletcher *et al.*, 1996).



(I)
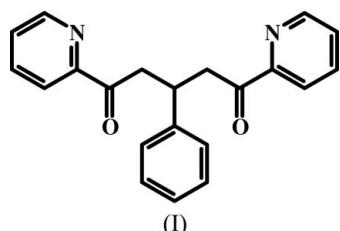
The structure of (I) is shown in Fig. 1. The mean planes of the two pyridine rings are inclined at 11.26 (7)°. The phenyl ring, comprising C16–C21, is inclined by 87.08 (3)° to the mean plane of the rest of the molecule. The fluorinated analogue (II) is essentially isomorphous with the present compound, the fluorinated phenyl group making no significant difference to the molecular packing. Constable *et al.* (1998) stated that there were neither stacking interactions nor significant short intermolecular contacts in the crystal structure of (II). We have perhaps taken a more liberal view, and observed both $\pi$–$\pi$ stacking and C—H$\cdots$O/N hydrogen bonding in (I).

Fig. 2 shows a packing diagram, viewed perpendicular to the *a* axis and illustrates the $\pi$–$\pi$ stacking of the pyridine rings, in which rings comprising N1/C1–C5 alternate with those comprising N2/C11–C15. As mentioned above, the rings are

# organic papers
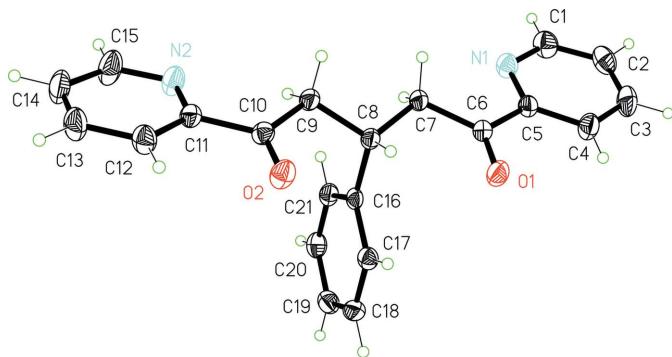


**Figure 1**
The molecular structure of (I); displacement ellipsoids are drawn at the 50% probability level.
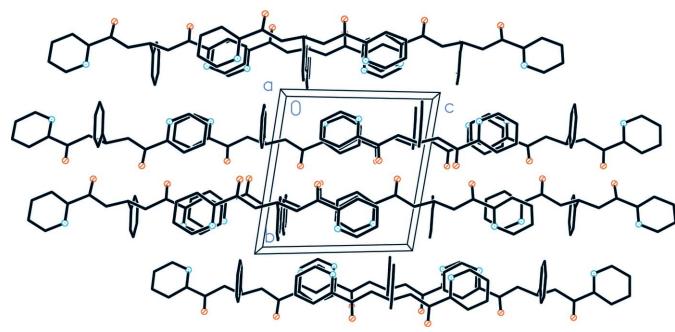


**Figure 2**
Packing diagram viewed down the *a* axis and showing $\pi$–$\pi$ stacking. H atoms have been omitted for clarity.
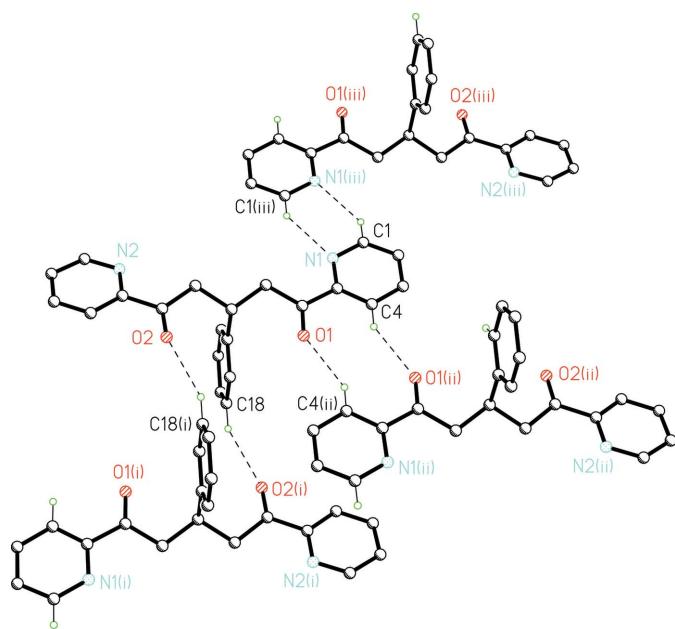


**Figure 3**
Intermolecular C—H$\cdots$O and C—H$\cdots$N hydrogen bonding (dashed lines). H atoms not involved in hydrogen bonding have been omitted for clarity. Symmetry codes as in Table 1.

inclined at 11.26 (7)°; the centroid–centroid distance is 3.907 Å and the centroid of the N1/C1–C5 ring is 3.684 (1) Å

from the mean plane of the N2/C11–C15 ring under symmetry operation $(-1 + x, y, -1 + z)$.

Three sets of intermolecular hydrogen-bonding interactions are shown in Fig. 3 and listed in Table 1. Each of the interactions is paired by symmetry, giving rise to cyclic patterns which can be described as $R_2^2 (16)$, $R_2^2 (10)$ and $R_2^2(6)$ for C18—H18$\cdots$O2$^{i}$, C4—H4$\cdots$O1$^{ii}$ and C1—H1$\cdots$N1$^{iii}$, respectively (Etter *et al.*, 1990). The $D\cdots A$ distances and $D$—H$\cdots A$ angles in these interactions are within the ranges generally observed for such weak hydrogen bonds (Desiraju & Steiner, 1999) and, collectively, they are likely to be responsible for the observed packing.

## Experimental

Compound (I) was synthesized as previously reported (Cave & Raston, 2001). The product was recrystallized at room temperature from a methanol–chloroform (1:1) mixture, yielding colourless crystals (m.p. 422–424 K).

### Crystal data

| | |
|---|---|
| $C_{21}H_{18}N_2O_2$ | $V = 836.74$ (9) Å$^3$ |
| $M_r = 330.37$ | $Z = 2$ |
| Triclinic, $P\bar{1}$ | $D_x = 1.311$ Mg m$^{-3}$ |
| $a = 8.3545$ (5) Å | Mo $K\alpha$ radiation |
| $b = 10.3696$ (7) Å | $\mu = 0.09$ mm$^{-1}$ |
| $c = 10.6370$ (7) Å | $T = 150$ (2) K |
| $\alpha = 95.208$ (1)° | Block, colourless |
| $\beta = 110.924$ (1)° | $0.45 \times 0.42 \times 0.17$ mm |
| $\gamma = 99.762$ (1)° | |

### Data collection

| | |
|---|---|
| Bruker SMART APEX-II CCD diffractometer | 8223 measured reflections |
| $\omega$ scans | 3832 independent reflections |
| Absorption correction: multi-scan (*SADABS*; Sheldrick, 2003) | 3149 reflections with $I > 2\sigma(I)$ |
| $T_{min} = 0.963$, $T_{max} = 0.986$ | $R_{int} = 0.019$ |
| | $\theta_{max} = 27.5°$ |

### Refinement

| | |
|---|---|
| Refinement on $F^2$ | $w = 1/[\sigma^2(F_o^2) + (0.0518P)^2$ |
| $R[F^2 > 2\sigma(F^2)] = 0.042$ | $+ 0.1695P]$ |
| $wR(F^2) = 0.111$ | where $P = (F_o^2 + 2F_c^2)/3$ |
| $S = 1.09$ | $(\Delta/\sigma)_{max} < 0.001$ |
| 3832 reflections | $\Delta\rho_{max} = 0.26$ e Å$^{-3}$ |
| 226 parameters | $\Delta\rho_{min} = -0.24$ e Å$^{-3}$ |
| H-atom parameters constrained | |

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

| $D$—H$\cdots A$ | $D$—H | H$\cdots A$ | $D\cdots A$ | $D$—H$\cdots A$ |
|---|---|---|---|---|
| C18—H18$\cdots$O2$^{i}$ | 0.95 | 2.62 | 3.4913 (15) | 153 |
| C4—H4$\cdots$O1$^{ii}$ | 0.95 | 2.42 | 3.2556 (16) | 146 |
| C1—H1$\cdots$N1$^{iii}$ | 0.95 | 2.64 | 3.4588 (18) | 145 |

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

H atoms bonded to C and N were inserted at calculated positions and refined using a riding model. The constrained distances were 0.95, 0.99 and 1.00 Å for aryl, methylene and tertiary H atoms, respectively. They were assigned $U_{iso}$(H) = 1.2$U_{eq}$(carrier atom).

Data collection: *APEX2* (Bruker, 2005); cell refinement: *SAINT* (Bruker, 2005); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Sheldrick, 2001); software used to prepare material for publication: *SHELXTL*.

## References

Allen, F. H. (2002). *Acta Cryst.* B**58**, 380–388.

Bruker (2005). *APEX2* (Version 2.1) and *SAINT* (Version 7.23A). Bruker AXS Inc., Madison, Wisconsin, USA.

Cave, G. W. V. & Raston, C. L. (2001). *J. Chem. Soc. Perkin Trans. 1*, pp. 3258–3264.

Collin, J.-P., Harriman, A., Heitz, V., Odobel, F. & Sauvage, J.-P. (1996). *Coord. Chem. Rev.* **148**, 63–69.

Constable, E. C., Lewis, J., Liptrot, M. C. & Raithby, P. R. (1990). *Inorg. Chim. Acta*, **178**, 47–54.

Constable, E. C., Neuberger, M., Smith, D. R. & Zehnder, M. (1998). *Inorg. Chim. Acta*, **275**, 359–365.

Desiraju, G. & Steiner, T. (1999). *The Weak Hydrogen Bond in Structural Chemistry and Biology.* Oxford University Press.

Etter, M. C., MacDonald, J. C. & Bernstein, J. (1990). *Acta Cryst.* B**46**, 256–262.

Fletcher, D. A., McMeeking, R. F. & Parkin, D. (1996). *J. Chem. Inf. Comput. Sci.* **36**, 746–749.

Gao, J., Reibenspies, J. H. & Martell, A. E. (2003). *J. Inorg. Biochem.* **94**, 272–278.

Hofmeier, H. & Schubert, U. S. (2004). *Chem. Soc. Rev.* **33**, 273–399.

Kröhnke, K. (1976). *Synthesis*, pp. 1–24.

Moya, S. A., Pastene, R., Le Bozec, H., Baricelli, P. J., Pardey, A. J. & Gimeno, J. (2001). *Inorg. Chim. Acta,* **312**, 7–14.

Sharma, S., Singh, S. K., Chandra, M. & Pandey, D. S. (2005). *J. Inorg. Biochem.* **99**, 458–466.

Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.

Sheldrick, G. M. (2001). *SHELXTL.* Version 6.12. Bruker AXS Inc., Madison, Wisconsin, USA.

Sheldrick, G. M. (2003). *SADABS.* Version 2.10. Bruker AXS Inc., Madison, Wisconsin, USA.

Tu, S., Li, T., Shi, F., Wang, Q., Zhang, J., Xu, J., Zhu, X., Zhang, X., Zhu, S. & Shi, D. (2005). *Synthesis*, **18**, 3045–3050.

# 3-Phenyl-1,5-di-2-pyridylpentane-1,5-dione

## Leanne James, Glenn E. M. Maguire, Bice S. Martincigh, Vickie McKee and Nombuso Ndlovu

**3-Phenyl-1,5-di-2-pyridylpentane-1,5-dione**

*Crystal data*

| | |
|---|---|
| $C_{21}H_{18}N_2O_2$ | $Z = 2$ |
| $M_r = 330.37$ | $F(000) = 348$ |
| Triclinic, $P\bar{1}$ | $D_x = 1.311$ Mg m$^{-3}$ |
| $a = 8.3545$ (5) Å | Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å |
| $b = 10.3696$ (7) Å | Cell parameters from 3056 reflections |
| $c = 10.6370$ (7) Å | $\theta = 2.7–31.5°$ |
| $\alpha = 95.208$ (1)° | $\mu = 0.09$ mm$^{-1}$ |
| $\beta = 110.924$ (1)° | $T = 150$ K |
| $\gamma = 99.762$ (1)° | Block, colourless |
| $V = 836.74$ (9) Å$^3$ | $0.45 \times 0.42 \times 0.17$ mm |

*Data collection*

| | |
|---|---|
| Bruker SMART APEX-II CCD diffractometer | 8223 measured reflections |
| Radiation source: fine-focus sealed tube | 3832 independent reflections |
| Graphite monochromator | 3149 reflections with $I > 2\sigma(I)$ |
| $\omega$ scans | $R_{int} = 0.019$ |
| Absorption correction: multi-scan (SADABS; Sheldrick, 2003) | $\theta_{max} = 27.5°$, $\theta_{min} = 2.0°$ |
| | $h = -10 \rightarrow 10$ |
| | $k = -13 \rightarrow 13$ |
| $T_{min} = 0.963$, $T_{max} = 0.986$ | $l = -13 \rightarrow 13$ |

*Refinement*

| | |
|---|---|
| Refinement on $F^2$ | Secondary atom site location: difference Fourier map |
| Least-squares matrix: full | |
| $R[F^2 > 2\sigma(F^2)] = 0.042$ | Hydrogen site location: inferred from neighbouring sites |
| $wR(F^2) = 0.111$ | |
| $S = 1.09$ | H-atom parameters constrained |
| 3832 reflections | $w = 1/[\sigma^2(F_o^2) + (0.0518P)^2 + 0.1695P]$ where $P = (F_o^2 + 2F_c^2)/3$ |
| 226 parameters | |
| 0 restraints | $(\Delta/\sigma)_{max} < 0.001$ |
| Primary atom site location: structure-invariant direct methods | $\Delta\rho_{max} = 0.26$ e Å$^{-3}$ |
| | $\Delta\rho_{min} = -0.24$ e Å$^{-3}$ |

*Special details*

**Experimental**. $^1$H NMR (300 MHz, CDCl$_3$): $\delta$ 8.60 (*d*, J = 4.9 Hz, 2H), 7.92 (*d*, J = 5.8 Hz, 2H), 7.75 (*t*, J = 7.6 Hz, 2H), 7.42–7.34 (*m*, 4H), 7.24–7.19 (*m*, 2H), 7.13–7.08 (*m*, 1H), 4.14 (*quint*, J = 7.1 Hz, 1H) 3.68 (*qd*, 4H). $^{13}$C NMR (75 MHz, CDCl$_3$): $\delta$ C 199.93, 153.28, 148.74, 144.54, 136.94, 128.37, 127.73, 127.08, 126.33, 121.86, 44.18, 36.11.

**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 | −0.35196 (14) | 0.16135 (11) | −0.49191 (11) | 0.0282 (2) |
| C1 | −0.47378 (18) | 0.11329 (14) | −0.61683 (14) | 0.0342 (3) |
| H1 | −0.5567 | 0.0336 | −0.6289 | 0.041* |
| C2 | −0.48469 (19) | 0.17425 (14) | −0.72960 (13) | 0.0344 (3) |
| H2 | −0.5752 | 0.1380 | −0.8160 | 0.041* |
| C3 | −0.3633 (2) | 0.28745 (15) | −0.71475 (13) | 0.0360 (3) |
| H3 | −0.3678 | 0.3303 | −0.7908 | 0.043* |
| C4 | −0.23359 (18) | 0.33848 (14) | −0.58688 (13) | 0.0309 (3) |
| H4 | −0.1465 | 0.4159 | −0.5735 | 0.037* |
| C5 | −0.23508 (16) | 0.27262 (12) | −0.47864 (12) | 0.0228 (3) |
| C6 | −0.10237 (15) | 0.32889 (12) | −0.33738 (12) | 0.0226 (3) |
| O1 | 0.01339 (12) | 0.42531 (9) | −0.31951 (9) | 0.0324 (2) |
| C7 | −0.12369 (15) | 0.26619 (12) | −0.21971 (11) | 0.0214 (2) |
| H7A | −0.1441 | 0.1686 | −0.2427 | 0.026* |
| H7B | −0.2278 | 0.2874 | −0.2055 | 0.026* |
| C8 | 0.03857 (15) | 0.31553 (11) | −0.08751 (11) | 0.0196 (2) |
| H8 | 0.0724 | 0.4142 | −0.0777 | 0.024* |
| C9 | 0.00051 (15) | 0.28325 (12) | 0.03820 (11) | 0.0223 (3) |
| H9A | −0.1103 | 0.3088 | 0.0332 | 0.027* |
| H9B | −0.0155 | 0.1864 | 0.0380 | 0.027* |
| C10 | 0.14735 (15) | 0.35513 (12) | 0.17000 (12) | 0.0216 (2) |
| O2 | 0.24581 (12) | 0.45740 (9) | 0.17391 (9) | 0.0301 (2) |
| C11 | 0.16752 (16) | 0.29902 (12) | 0.29879 (12) | 0.0226 (3) |
| C12 | 0.28339 (17) | 0.37379 (13) | 0.42286 (13) | 0.0287 (3) |
| H12 | 0.3474 | 0.4600 | 0.4267 | 0.034* |
| C13 | 0.30403 (19) | 0.32054 (15) | 0.54078 (13) | 0.0353 (3) |
| H13 | 0.3821 | 0.3694 | 0.6272 | 0.042* |
| C14 | 0.2090 (2) | 0.19514 (15) | 0.53014 (14) | 0.0393 (4) |
| H14 | 0.2202 | 0.1556 | 0.6091 | 0.047* |
| C15 | 0.0971 (2) | 0.12797 (15) | 0.40237 (15) | 0.0426 (4) |
| H15 | 0.0326 | 0.0413 | 0.3962 | 0.051* |
| N2 | 0.07389 (16) | 0.17725 (11) | 0.28674 (11) | 0.0326 (3) |
| C16 | 0.19229 (15) | 0.25953 (11) | −0.09513 (11) | 0.0193 (2) |
| C17 | 0.34421 (15) | 0.34252 (12) | −0.09007 (11) | 0.0220 (2) |
| H17 | 0.3524 | 0.4356 | −0.0810 | 0.026* |
| C18 | 0.48440 (16) | 0.29177 (13) | −0.09804 (12) | 0.0253 (3) |

| | | | |
|---|---|---|---|
| H18 | 0.5876 | 0.3501 | −0.0938 | 0.030* |
| C19 | 0.47380 (17) | 0.15584 (13) | −0.11217 (12) | 0.0275 (3) |
| H19 | 0.5695 | 0.1208 | −0.1176 | 0.033* |
| C20 | 0.32209 (17) | 0.07150 (12) | −0.11837 (12) | 0.0269 (3) |
| H20 | 0.3137 | −0.0217 | −0.1284 | 0.032* |
| C21 | 0.18303 (16) | 0.12285 (12) | −0.10991 (12) | 0.0233 (3) |
| H21 | 0.0799 | 0.0643 | −0.1142 | 0.028* |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| N1 | 0.0292 (6) | 0.0286 (6) | 0.0224 (5) | 0.0024 (4) | 0.0063 (4) | 0.0035 (4) |
| C1 | 0.0339 (7) | 0.0326 (7) | 0.0274 (7) | 0.0020 (6) | 0.0049 (6) | −0.0002 (5) |
| C2 | 0.0368 (7) | 0.0393 (8) | 0.0202 (6) | 0.0131 (6) | 0.0020 (5) | −0.0015 (5) |
| C3 | 0.0480 (8) | 0.0416 (8) | 0.0197 (6) | 0.0156 (7) | 0.0106 (6) | 0.0096 (6) |
| C4 | 0.0360 (7) | 0.0341 (7) | 0.0230 (6) | 0.0061 (6) | 0.0111 (6) | 0.0090 (5) |
| C5 | 0.0233 (6) | 0.0262 (6) | 0.0197 (6) | 0.0071 (5) | 0.0078 (5) | 0.0058 (5) |
| C6 | 0.0211 (6) | 0.0265 (6) | 0.0207 (6) | 0.0057 (5) | 0.0076 (5) | 0.0076 (5) |
| O1 | 0.0285 (5) | 0.0361 (5) | 0.0265 (5) | −0.0038 (4) | 0.0067 (4) | 0.0115 (4) |
| C7 | 0.0193 (6) | 0.0258 (6) | 0.0172 (5) | 0.0026 (5) | 0.0052 (4) | 0.0059 (4) |
| C8 | 0.0197 (5) | 0.0208 (5) | 0.0168 (5) | 0.0021 (4) | 0.0062 (4) | 0.0039 (4) |
| C9 | 0.0207 (6) | 0.0269 (6) | 0.0176 (5) | 0.0017 (5) | 0.0069 (5) | 0.0041 (4) |
| C10 | 0.0231 (6) | 0.0228 (6) | 0.0196 (6) | 0.0047 (5) | 0.0095 (5) | 0.0016 (4) |
| O2 | 0.0347 (5) | 0.0256 (5) | 0.0250 (5) | −0.0034 (4) | 0.0102 (4) | 0.0029 (4) |
| C11 | 0.0232 (6) | 0.0256 (6) | 0.0187 (6) | 0.0044 (5) | 0.0081 (5) | 0.0029 (5) |
| C12 | 0.0318 (7) | 0.0268 (6) | 0.0218 (6) | 0.0019 (5) | 0.0061 (5) | 0.0010 (5) |
| C13 | 0.0398 (8) | 0.0400 (8) | 0.0187 (6) | 0.0045 (6) | 0.0049 (6) | 0.0021 (5) |
| C14 | 0.0467 (9) | 0.0461 (9) | 0.0225 (7) | 0.0042 (7) | 0.0111 (6) | 0.0133 (6) |
| C15 | 0.0522 (9) | 0.0380 (8) | 0.0290 (7) | −0.0087 (7) | 0.0121 (7) | 0.0111 (6) |
| N2 | 0.0369 (6) | 0.0310 (6) | 0.0230 (5) | −0.0049 (5) | 0.0084 (5) | 0.0057 (4) |
| C16 | 0.0205 (5) | 0.0227 (6) | 0.0119 (5) | 0.0030 (4) | 0.0037 (4) | 0.0032 (4) |
| C17 | 0.0223 (6) | 0.0214 (6) | 0.0185 (5) | 0.0020 (5) | 0.0050 (5) | 0.0021 (4) |
| C18 | 0.0202 (6) | 0.0314 (7) | 0.0219 (6) | 0.0018 (5) | 0.0068 (5) | 0.0036 (5) |
| C19 | 0.0258 (6) | 0.0346 (7) | 0.0236 (6) | 0.0121 (5) | 0.0084 (5) | 0.0054 (5) |
| C20 | 0.0344 (7) | 0.0233 (6) | 0.0236 (6) | 0.0090 (5) | 0.0099 (5) | 0.0054 (5) |
| C21 | 0.0252 (6) | 0.0233 (6) | 0.0190 (6) | 0.0020 (5) | 0.0066 (5) | 0.0049 (4) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| N1—C1 | 1.3385 (17) | C10—C11 | 1.5023 (16) |
| N1—C5 | 1.3396 (16) | C11—N2 | 1.3367 (16) |
| C1—C2 | 1.3878 (19) | C11—C12 | 1.3882 (17) |
| C1—H1 | 0.9500 | C12—C13 | 1.3826 (18) |
| C2—C3 | 1.371 (2) | C12—H12 | 0.9500 |
| C2—H2 | 0.9500 | C13—C14 | 1.376 (2) |
| C3—C4 | 1.3879 (19) | C13—H13 | 0.9500 |
| C3—H3 | 0.9500 | C14—C15 | 1.381 (2) |
| C4—C5 | 1.3936 (17) | C14—H14 | 0.9500 |

| | | | |
|---|---|---|---|
| C4—H4 | 0.9500 | C15—N2 | 1.3375 (17) |
| C5—C6 | 1.5030 (16) | C15—H15 | 0.9500 |
| C6—O1 | 1.2154 (14) | C16—C17 | 1.3881 (16) |
| C6—C7 | 1.5129 (16) | C16—C21 | 1.3975 (16) |
| C7—C8 | 1.5327 (15) | C17—C18 | 1.3884 (17) |
| C7—H7A | 0.9900 | C17—H17 | 0.9500 |
| C7—H7B | 0.9900 | C18—C19 | 1.3881 (18) |
| C8—C16 | 1.5204 (16) | C18—H18 | 0.9500 |
| C8—C9 | 1.5330 (15) | C19—C20 | 1.3891 (18) |
| C8—H8 | 1.0000 | C19—H19 | 0.9500 |
| C9—C10 | 1.5158 (16) | C20—C21 | 1.3846 (17) |
| C9—H9A | 0.9900 | C20—H20 | 0.9500 |
| C9—H9B | 0.9900 | C21—H21 | 0.9500 |
| C10—O2 | 1.2144 (14) | | |
| | | | |
| C1—N1—C5 | 117.02 (11) | O2—C10—C11 | 119.74 (10) |
| N1—C1—C2 | 123.15 (13) | O2—C10—C9 | 121.64 (10) |
| N1—C1—H1 | 118.4 | C11—C10—C9 | 118.61 (10) |
| C2—C1—H1 | 118.4 | N2—C11—C12 | 123.42 (11) |
| C3—C2—C1 | 119.19 (12) | N2—C11—C10 | 117.49 (10) |
| C3—C2—H2 | 120.4 | C12—C11—C10 | 119.08 (11) |
| C1—C2—H2 | 120.4 | C13—C12—C11 | 118.81 (12) |
| C2—C3—C4 | 118.98 (12) | C13—C12—H12 | 120.6 |
| C2—C3—H3 | 120.5 | C11—C12—H12 | 120.6 |
| C4—C3—H3 | 120.5 | C14—C13—C12 | 118.51 (12) |
| C3—C4—C5 | 117.98 (13) | C14—C13—H13 | 120.7 |
| C3—C4—H4 | 121.0 | C12—C13—H13 | 120.7 |
| C5—C4—H4 | 121.0 | C13—C14—C15 | 118.67 (13) |
| N1—C5—C4 | 123.66 (11) | C13—C14—H14 | 120.7 |
| N1—C5—C6 | 116.86 (10) | C15—C14—H14 | 120.7 |
| C4—C5—C6 | 119.47 (11) | N2—C15—C14 | 124.11 (13) |
| O1—C6—C5 | 120.13 (10) | N2—C15—H15 | 117.9 |
| O1—C6—C7 | 121.84 (11) | C14—C15—H15 | 117.9 |
| C5—C6—C7 | 117.96 (10) | C11—N2—C15 | 116.48 (11) |
| C6—C7—C8 | 111.75 (9) | C17—C16—C21 | 118.25 (11) |
| C6—C7—H7A | 109.3 | C17—C16—C8 | 120.93 (10) |
| C8—C7—H7A | 109.3 | C21—C16—C8 | 120.82 (10) |
| C6—C7—H7B | 109.3 | C16—C17—C18 | 121.14 (11) |
| C8—C7—H7B | 109.3 | C16—C17—H17 | 119.4 |
| H7A—C7—H7B | 107.9 | C18—C17—H17 | 119.4 |
| C16—C8—C7 | 110.71 (9) | C19—C18—C17 | 120.04 (11) |
| C16—C8—C9 | 111.00 (9) | C19—C18—H18 | 120.0 |
| C7—C8—C9 | 111.88 (9) | C17—C18—H18 | 120.0 |
| C16—C8—H8 | 107.7 | C18—C19—C20 | 119.46 (12) |
| C7—C8—H8 | 107.7 | C18—C19—H19 | 120.3 |
| C9—C8—H8 | 107.7 | C20—C19—H19 | 120.3 |
| C10—C9—C8 | 112.00 (9) | C21—C20—C19 | 120.18 (11) |
| C10—C9—H9A | 109.2 | C21—C20—H20 | 119.9 |

| | | | |
|---|---|---|---|
| C8—C9—H9A | 109.2 | C19—C20—H20 | 119.9 |
| C10—C9—H9B | 109.2 | C20—C21—C16 | 120.93 (11) |
| C8—C9—H9B | 109.2 | C20—C21—H21 | 119.5 |
| H9A—C9—H9B | 107.9 | C16—C21—H21 | 119.5 |

*Hydrogen-bond geometry (Å, º)*

| D—H···A | D—H | H···A | D···A | D—H···A |
|---|---|---|---|---|
| C18—H18···O2[i] | 0.95 | 2.62 | 3.4913 (15) | 153 |
| C4—H4···O1[ii] | 0.95 | 2.42 | 3.2556 (16) | 146 |
| C1—H1···N1[iii] | 0.95 | 2.64 | 3.4588 (18) | 145 |

Symmetry codes: (i) −$x$+1, −$y$+1, −$z$; (ii) −$x$, −$y$+1, −$z$−1; (iii) −$x$−1, −$y$, −$z$−1.