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# Diethyl 4-[4-(dimethylamino)phenyl]-2,6-dimethyl-1,4-dihydropyridine-3,5-dicarboxylate

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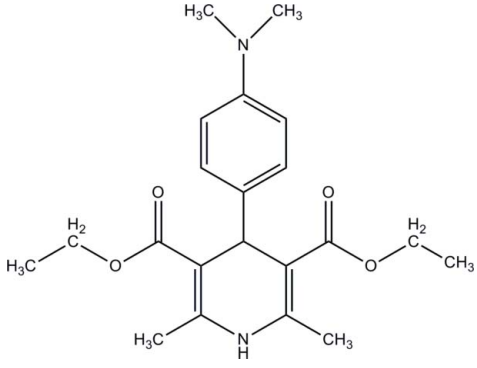
Key indicators: single-crystal X-ray study; $T$ = 298 K; mean $\sigma$(C–C) = 0.005 Å; disorder in main residue; $R$ factor = 0.061; $wR$ factor = 0.202; data-to-parameter ratio = 13.2.

In the title compound, $C_{21}H_{28}N_2O_4$, the dihydropyridine ring adopts a flattened boat conformation. The mean plane of the dihydropyridine ring and the attached benzene ring form a dihedral angle of 85.1 (1) Å. One of two ethyl fragments is disordered between two conformations in a 0.67 (4):0.33 (4) ratio. In the crystal structure, molecules related by translation along the *a* axis are linked into chains *via* intermolecular N—H···O hydrogen bonds.

## Related literature

For the pharmacological activity of compounds containing substituted 1,4-dihydropyridine ring systems, see: Triggle *et al.*(1980); Henry (2004). For a related structure, see Sun *et al.* (2006).



## Experimental

### Crystal data

$C_{21}H_{28}N_2O_4$
$M_r$ = 372.45
Monoclinic, $P2_1/n$
$a$ = 7.5023 (7) Å
$b$ = 14.9797 (14) Å
$c$ = 18.0691 (19) Å
$\beta$ = 91.021 (1)°
$V$ = 2030.3 (3) Å$^3$
$Z$ = 4
Mo $K\alpha$ radiation
$\mu$ = 0.08 mm$^{-1}$
$T$ = 298 K
0.49 × 0.48 × 0.47 mm

### Data collection

Bruker SMART APEX CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{min}$ = 0.960, $T_{max}$ = 0.961
10007 measured reflections
3572 independent reflections
1680 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.067

### Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.061
$wR(F^2)$ = 0.202
$S$ = 1.02
3572 reflections
270 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.19 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.18 e Å$^{-3}$

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

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| N1—H1···O4$^{i}$ | 0.86 | 2.18 | 3.036 (4) | 173 |

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

Data collection: *SMART* (Siemens, 1996); cell refinement: *SAINT* (Siemens, 1996); data reduction: *SAINT*; 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: CV2687).

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# supporting information

*Acta Cryst.* (2010). E**66**, o519 [doi:10.1107/S1600536810003508]

# Diethyl 4-[4-(dimethylamino)phenyl]-2,6-dimethyl-1,4-dihydropyridine-3,5-dicarboxylate

**Xiao-Ling Ji, Wang-Bin Sun, Qian Zhang, Yan-Ping Cao and Ming-Sheng Bai**

## S1. Comment

Substituted 1,4-dihydropyridine ring system exhibit diverse pharmacological activities (Triggle *et al.*, 1980). In addition, several of these compounds were discovered to be highly selective ligands for adenosine receptors, which were recently recognized as potential targets for the development of new drugs for the treatment of Parkinson's disease, hypoxia/ischemia, asthma, kidney disease, epilepsy, and cancer (Henry *et al.*, 2004). In continuation of our ongoing program directed to the development of pyridine chemistry, we present here the crystal structure of the title compound, (I).

In (I) (Fig. 1), the bond lengths an angles are normal and are comparable to the values observed in similar compound ethyl 5-carboxy-2,6-dimethyl-4-(4-nitrophenyl) -1,4-dihydropyridine-3-carboxylate (Sun *et al.*, 2006). The carboxylate and methyl groups lie to the each side of the dihydropyridine ring. The dihedral angle between the planes C2/C3/C4 and C1/N1/C5 is 36.9 (3) Å. The dihydropyridine ring adopts a flattened boat conformation and the plane of the base of the boat (C1/C2/C4/C5) forms a dihedral angle of 83.73 (13) Å with the benzene ring.

In the crystal structure, intermolecular N—H···O hydrogen bonds (Table 1) link the molecules into chains propagated in direction [100].

## S2. Experimental

4-(N, N-dimethyl)benzaldehyde (10.0 mmol), 20 ml methanol, ethyl acetoacetate (20 mmol) and $NH_4HCO_3$ (6 mmol) were mixed in 50 ml flash. After refluxing 3 h, the resulting mixture was cooled to room temperature, and recrystalized from methanol, and afforded the title compound as a crystalline solid. Elemental analysis: calculated for $C_{21}H_{28}N_2O_4$: C 67.72, H 7.58, N 7.52%; found: C 67.82, H 7.73, N 7.43%.

## S3. Refinement

All H atoms were placed in geometrically idealized positions ( C-H of methyl distances is 0.96 Å , C-H of methylene distances is 0.97 Å , C-H of methine distances is 0.98 Å and aromatic C-H distances is 0.93 Å, N-H distances is 0.86 Å) and treated as riding on their parent atoms, with $U_{iso}$(H) = 1.2 U-1.5$_{eq}$(C, N) . Atoms $C_8$, $C_9$ were treated as disordered between two positions, with refined occupancies of 0.33 (4) and 0.67 (4).

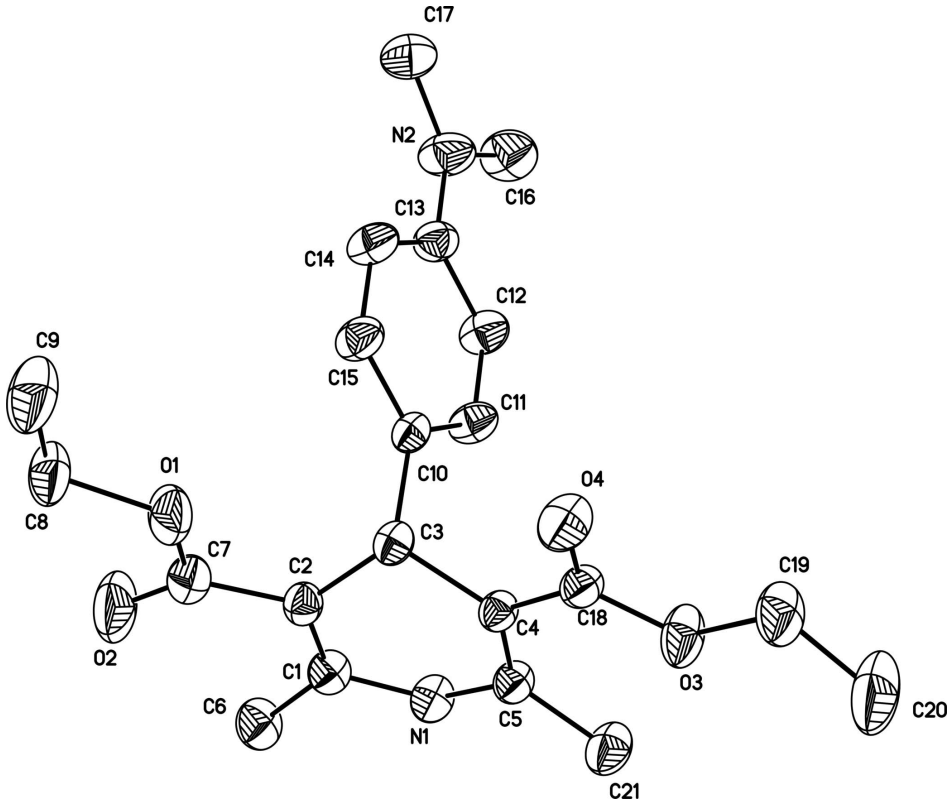


**Figure 1**

A view of (I) showing the atomic numbering scheme and 30% probability displacement ellipsoids. Only major parts of disordered atoms are shown. H atoms omitted for clarity.

**Diethyl 4-[4-(dimethylamino)phenyl]-2,6-dimethyl-1,4-dihydropyridine-3,5-dicarboxylate**

*Crystal data*

$C_{21}H_{28}N_2O_4$
$M_r = 372.45$
Monoclinic, $P2_1/n$
$a = 7.5023\ (7)$ Å
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$c = 18.0691\ (19)$ Å
$\beta = 91.021\ (1)°$
$V = 2030.3\ (3)$ Å$^3$
$Z = 4$

$F(000) = 800$
$D_x = 1.218$ Mg m$^{-3}$
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 1665 reflections
$\theta = 2.6–20.9°$
$\mu = 0.08$ mm$^{-1}$
$T = 298$ K
Needle, red
0.49 × 0.48 × 0.47 mm

*Data collection*

Bruker SMART APEX CCD area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
phi and $\omega$ scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{min} = 0.960$, $T_{max} = 0.961$

10007 measured reflections
3572 independent reflections
1680 reflections with $I > 2\sigma(I)$
$R_{int} = 0.067$
$\theta_{max} = 25.0°$, $\theta_{min} = 1.8°$
$h = -8\rightarrow8$
$k = -11\rightarrow17$
$l = -21\rightarrow21$

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.061$
$wR(F^2) = 0.202$
$S = 1.02$
3572 reflections
270 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
$w = 1/[\sigma^2(F_o^2) + (0.0653P)^2 + 1.4769P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.19$ e Å$^{-3}$
$\Delta\rho_{min} = -0.18$ e Å$^{-3}$

*Special details*

**Geometry**. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds 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 (Å²)*

| | *x* | *y* | *z* | $U_{iso}$*/$U_{eq}$ | Occ. (<1) |
|---|---|---|---|---|---|
| N1 | 1.0562 (4) | 0.1513 (2) | 1.06111 (16) | 0.0535 (9) | |
| H1 | 1.1638 | 0.1618 | 1.0758 | 0.064* | |
| N2 | 0.6296 (5) | 0.5330 (2) | 0.8972 (2) | 0.0785 (11) | |
| O1 | 0.6518 (4) | 0.0907 (2) | 0.86907 (16) | 0.0810 (10) | |
| O2 | 0.9341 (5) | 0.0681 (3) | 0.84207 (18) | 0.1139 (14) | |
| O3 | 0.6311 (3) | 0.1834 (2) | 1.20424 (14) | 0.0693 (9) | |
| O4 | 0.4439 (3) | 0.1727 (2) | 1.10883 (14) | 0.0679 (9) | |
| C1 | 1.0290 (5) | 0.1244 (2) | 0.9891 (2) | 0.0495 (10) | |
| C2 | 0.8620 (5) | 0.1274 (2) | 0.96009 (18) | 0.0464 (9) | |
| C3 | 0.7131 (5) | 0.1728 (2) | 1.00311 (17) | 0.0436 (9) | |
| H3 | 0.6020 | 0.1402 | 0.9931 | 0.052* | |
| C4 | 0.7524 (4) | 0.1684 (2) | 1.08551 (17) | 0.0414 (9) | |
| C5 | 0.9222 (5) | 0.1625 (3) | 1.11124 (18) | 0.0474 (9) | |
| C6 | 1.1942 (5) | 0.0920 (3) | 0.9527 (2) | 0.0677 (12) | |
| H6A | 1.1736 | 0.0334 | 0.9327 | 0.102* | |
| H6B | 1.2902 | 0.0896 | 0.9885 | 0.102* | |
| H6C | 1.2250 | 0.1321 | 0.9135 | 0.102* | |
| C7 | 0.8257 (7) | 0.0925 (3) | 0.8863 (2) | 0.0648 (12) | |
| C8 | 0.624 (3) | 0.0697 (13) | 0.7887 (8) | 0.077 (4) | 0.67 (4) |
| H8A | 0.6810 | 0.1141 | 0.7582 | 0.092* | 0.67 (4) |
| H8B | 0.6723 | 0.0115 | 0.7771 | 0.092* | 0.67 (4) |
| C9 | 0.428 (2) | 0.0708 (15) | 0.7756 (8) | 0.095 (5) | 0.67 (4) |
| H9A | 0.3732 | 0.0268 | 0.8065 | 0.142* | 0.67 (4) |
| H9B | 0.4022 | 0.0574 | 0.7246 | 0.142* | 0.67 (4) |
| H9C | 0.3820 | 0.1288 | 0.7873 | 0.142* | 0.67 (4) |

| | | | | | |
|---|---|---|---|---|---|
| C8′ | 0.557 (5) | 0.036 (2) | 0.814 (2) | 0.079 (9) | 0.33 (4) |
| H8′1 | 0.4525 | 0.0074 | 0.8345 | 0.094* | 0.33 (4) |
| H8′2 | 0.6338 | −0.0093 | 0.7929 | 0.094* | 0.33 (4) |
| C9′ | 0.508 (6) | 0.107 (3) | 0.7585 (16) | 0.098 (10) | 0.33 (4) |
| H9′1 | 0.4027 | 0.1381 | 0.7745 | 0.146* | 0.33 (4) |
| H9′2 | 0.4844 | 0.0805 | 0.7111 | 0.146* | 0.33 (4) |
| H9′3 | 0.6041 | 0.1491 | 0.7547 | 0.146* | 0.33 (4) |
| C10 | 0.6878 (4) | 0.2688 (2) | 0.97650 (17) | 0.0420 (9) | |
| C11 | 0.7898 (5) | 0.3382 (3) | 1.0039 (2) | 0.0577 (11) | |
| H11 | 0.8738 | 0.3263 | 1.0411 | 0.069* | |
| C12 | 0.7721 (5) | 0.4242 (3) | 0.9787 (2) | 0.0619 (11) | |
| H12 | 0.8445 | 0.4686 | 0.9990 | 0.074* | |
| C13 | 0.6486 (5) | 0.4466 (3) | 0.9232 (2) | 0.0553 (10) | |
| C14 | 0.5446 (5) | 0.3772 (3) | 0.8956 (2) | 0.0629 (11) | |
| H14 | 0.4592 | 0.3888 | 0.8589 | 0.076* | |
| C15 | 0.5659 (5) | 0.2911 (3) | 0.9218 (2) | 0.0584 (11) | |
| H15 | 0.4947 | 0.2462 | 0.9015 | 0.070* | |
| C16 | 0.7323 (7) | 0.6039 (3) | 0.9282 (3) | 0.0911 (16) | |
| H16A | 0.7142 | 0.6064 | 0.9806 | 0.137* | |
| H16B | 0.6959 | 0.6593 | 0.9060 | 0.137* | |
| H16C | 0.8563 | 0.5937 | 0.9189 | 0.137* | |
| C17 | 0.4833 (7) | 0.5557 (3) | 0.8486 (3) | 0.0834 (15) | |
| H17A | 0.4969 | 0.5257 | 0.8021 | 0.125* | |
| H17B | 0.4815 | 0.6190 | 0.8407 | 0.125* | |
| H17C | 0.3735 | 0.5374 | 0.8705 | 0.125* | |
| C18 | 0.5961 (5) | 0.1746 (2) | 1.1321 (2) | 0.0467 (9) | |
| C19 | 0.4839 (5) | 0.1922 (3) | 1.2541 (2) | 0.0735 (14) | |
| H19A | 0.3974 | 0.1452 | 1.2448 | 0.088* | |
| H19B | 0.4253 | 0.2493 | 1.2466 | 0.088* | |
| C20 | 0.5528 (7) | 0.1859 (4) | 1.3293 (3) | 0.113 (2) | |
| H20A | 0.6072 | 0.1285 | 1.3367 | 0.170* | |
| H20B | 0.4570 | 0.1932 | 1.3632 | 0.170* | |
| H20C | 0.6400 | 0.2319 | 1.3377 | 0.170* | |
| C21 | 0.9918 (5) | 0.1636 (3) | 1.1893 (2) | 0.0708 (13) | |
| H21A | 0.9342 | 0.2103 | 1.2162 | 0.106* | |
| H21B | 1.1181 | 0.1739 | 1.1895 | 0.106* | |
| H21C | 0.9677 | 0.1071 | 1.2123 | 0.106* | |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| N1 | 0.0339 (17) | 0.075 (2) | 0.0513 (19) | −0.0034 (15) | 0.0033 (15) | −0.0005 (16) |
| N2 | 0.089 (3) | 0.055 (2) | 0.091 (3) | −0.002 (2) | −0.019 (2) | 0.017 (2) |
| O1 | 0.085 (2) | 0.086 (2) | 0.071 (2) | 0.0062 (18) | −0.0216 (17) | −0.0345 (17) |
| O2 | 0.108 (3) | 0.169 (4) | 0.065 (2) | 0.033 (3) | 0.006 (2) | −0.044 (2) |
| O3 | 0.0432 (16) | 0.118 (3) | 0.0468 (17) | 0.0008 (15) | 0.0103 (13) | −0.0104 (15) |
| O4 | 0.0372 (16) | 0.109 (2) | 0.0575 (17) | 0.0035 (15) | 0.0019 (13) | 0.0050 (15) |
| C1 | 0.043 (2) | 0.056 (2) | 0.050 (2) | −0.0050 (19) | 0.0102 (18) | 0.0038 (19) |

| | | | | | | |
|---|---|---|---|---|---|---|
| C2 | 0.051 (2) | 0.048 (2) | 0.041 (2) | −0.0027 (18) | 0.0047 (18) | −0.0003 (17) |
| C3 | 0.039 (2) | 0.053 (2) | 0.0386 (19) | −0.0056 (17) | 0.0000 (16) | 0.0004 (17) |
| C4 | 0.037 (2) | 0.050 (2) | 0.0371 (19) | 0.0003 (17) | 0.0017 (16) | 0.0013 (16) |
| C5 | 0.040 (2) | 0.060 (3) | 0.043 (2) | 0.0017 (18) | 0.0032 (18) | 0.0021 (18) |
| C6 | 0.056 (3) | 0.084 (3) | 0.064 (3) | 0.004 (2) | 0.020 (2) | −0.003 (2) |
| C7 | 0.070 (3) | 0.070 (3) | 0.054 (3) | 0.007 (2) | −0.007 (2) | −0.009 (2) |
| C8 | 0.081 (10) | 0.092 (9) | 0.058 (7) | −0.008 (6) | −0.009 (6) | −0.029 (6) |
| C9 | 0.079 (10) | 0.134 (13) | 0.071 (7) | −0.020 (7) | −0.011 (6) | −0.032 (7) |
| C8′ | 0.085 (18) | 0.079 (17) | 0.071 (16) | −0.012 (11) | −0.005 (13) | −0.023 (13) |
| C9′ | 0.10 (3) | 0.12 (2) | 0.075 (14) | −0.004 (16) | −0.022 (15) | 0.003 (14) |
| C10 | 0.041 (2) | 0.051 (2) | 0.0346 (19) | −0.0020 (18) | 0.0004 (16) | 0.0033 (16) |
| C11 | 0.054 (2) | 0.059 (3) | 0.059 (2) | −0.006 (2) | −0.0139 (19) | 0.009 (2) |
| C12 | 0.063 (3) | 0.055 (3) | 0.066 (3) | −0.012 (2) | −0.016 (2) | 0.006 (2) |
| C13 | 0.058 (3) | 0.054 (3) | 0.053 (2) | 0.003 (2) | 0.002 (2) | 0.010 (2) |
| C14 | 0.061 (3) | 0.066 (3) | 0.061 (3) | 0.000 (2) | −0.018 (2) | 0.010 (2) |
| C15 | 0.062 (3) | 0.056 (3) | 0.056 (2) | −0.005 (2) | −0.018 (2) | 0.002 (2) |
| C16 | 0.106 (4) | 0.065 (3) | 0.102 (4) | −0.010 (3) | −0.005 (3) | 0.013 (3) |
| C17 | 0.100 (4) | 0.069 (3) | 0.081 (3) | 0.014 (3) | −0.007 (3) | 0.019 (3) |
| C18 | 0.041 (2) | 0.057 (3) | 0.041 (2) | 0.0039 (18) | 0.0010 (18) | 0.0046 (18) |
| C19 | 0.048 (3) | 0.113 (4) | 0.059 (3) | 0.006 (2) | 0.018 (2) | −0.006 (2) |
| C20 | 0.095 (4) | 0.186 (7) | 0.059 (3) | 0.004 (4) | 0.022 (3) | −0.018 (3) |
| C21 | 0.048 (2) | 0.112 (4) | 0.052 (2) | 0.013 (2) | −0.005 (2) | −0.001 (2) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| N1—C1 | 1.374 (4) | C8′—C9′ | 1.51 (7) |
| N1—C5 | 1.375 (4) | C8′—H8′1 | 0.9700 |
| N1—H1 | 0.8600 | C8′—H8′2 | 0.9700 |
| N2—C13 | 1.384 (5) | C9′—H9′1 | 0.9600 |
| N2—C16 | 1.421 (5) | C9′—H9′2 | 0.9600 |
| N2—C17 | 1.434 (5) | C9′—H9′3 | 0.9600 |
| O1—C7 | 1.336 (5) | C10—C15 | 1.376 (5) |
| O1—C8′ | 1.47 (2) | C10—C11 | 1.377 (5) |
| O1—C8 | 1.497 (11) | C11—C12 | 1.372 (5) |
| O2—C7 | 1.208 (5) | C11—H11 | 0.9300 |
| O3—C18 | 1.332 (4) | C12—C13 | 1.393 (5) |
| O3—C19 | 1.444 (4) | C12—H12 | 0.9300 |
| O4—C18 | 1.210 (4) | C13—C14 | 1.388 (5) |
| C1—C2 | 1.350 (5) | C14—C15 | 1.381 (5) |
| C1—C6 | 1.495 (5) | C14—H14 | 0.9300 |
| C2—C7 | 1.453 (5) | C15—H15 | 0.9300 |
| C2—C3 | 1.531 (5) | C16—H16A | 0.9600 |
| C3—C4 | 1.514 (4) | C16—H16B | 0.9600 |
| C3—C10 | 1.528 (5) | C16—H16C | 0.9600 |
| C3—H3 | 0.9800 | C17—H17A | 0.9600 |
| C4—C5 | 1.352 (5) | C17—H17B | 0.9600 |
| C4—C18 | 1.459 (5) | C17—H17C | 0.9600 |
| C5—C21 | 1.495 (5) | C19—C20 | 1.447 (6) |

| | | | |
|---|---|---|---|
| C6—H6A | 0.9600 | C19—H19A | 0.9700 |
| C6—H6B | 0.9600 | C19—H19B | 0.9700 |
| C6—H6C | 0.9600 | C20—H20A | 0.9600 |
| C8—C9 | 1.48 (3) | C20—H20B | 0.9600 |
| C8—H8A | 0.9700 | C20—H20C | 0.9600 |
| C8—H8B | 0.9700 | C21—H21A | 0.9600 |
| C9—H9A | 0.9600 | C21—H21B | 0.9600 |
| C9—H9B | 0.9600 | C21—H21C | 0.9600 |
| C9—H9C | 0.9600 | | |

| | | | |
|---|---|---|---|
| C1—N1—C5 | 124.2 (3) | H9′1—C9′—H9′3 | 109.5 |
| C1—N1—H1 | 117.9 | H9′2—C9′—H9′3 | 109.5 |
| C5—N1—H1 | 117.9 | C15—C10—C11 | 115.7 (3) |
| C13—N2—C16 | 120.9 (4) | C15—C10—C3 | 122.1 (3) |
| C13—N2—C17 | 120.2 (4) | C11—C10—C3 | 122.2 (3) |
| C16—N2—C17 | 117.9 (4) | C12—C11—C10 | 122.6 (4) |
| C7—O1—C8′ | 129.0 (13) | C12—C11—H11 | 118.7 |
| C7—O1—C8 | 110.6 (8) | C10—C11—H11 | 118.7 |
| C8′—O1—C8 | 33.4 (12) | C11—C12—C13 | 121.7 (4) |
| C18—O3—C19 | 118.7 (3) | C11—C12—H12 | 119.2 |
| C2—C1—N1 | 118.6 (3) | C13—C12—H12 | 119.2 |
| C2—C1—C6 | 127.7 (4) | N2—C13—C14 | 121.7 (4) |
| N1—C1—C6 | 113.7 (3) | N2—C13—C12 | 122.2 (4) |
| C1—C2—C7 | 120.1 (4) | C14—C13—C12 | 116.1 (4) |
| C1—C2—C3 | 119.9 (3) | C15—C14—C13 | 121.0 (4) |
| C7—C2—C3 | 120.0 (3) | C15—C14—H14 | 119.5 |
| C4—C3—C10 | 111.8 (3) | C13—C14—H14 | 119.5 |
| C4—C3—C2 | 110.4 (3) | C10—C15—C14 | 122.9 (4) |
| C10—C3—C2 | 110.2 (3) | C10—C15—H15 | 118.5 |
| C4—C3—H3 | 108.1 | C14—C15—H15 | 118.5 |
| C10—C3—H3 | 108.1 | N2—C16—H16A | 109.5 |
| C2—C3—H3 | 108.1 | N2—C16—H16B | 109.5 |
| C5—C4—C18 | 124.6 (3) | H16A—C16—H16B | 109.5 |
| C5—C4—C3 | 120.5 (3) | N2—C16—H16C | 109.5 |
| C18—C4—C3 | 114.9 (3) | H16A—C16—H16C | 109.5 |
| C4—C5—N1 | 118.5 (3) | H16B—C16—H16C | 109.5 |
| C4—C5—C21 | 129.4 (3) | N2—C17—H17A | 109.5 |
| N1—C5—C21 | 112.1 (3) | N2—C17—H17B | 109.5 |
| C1—C6—H6A | 109.5 | H17A—C17—H17B | 109.5 |
| C1—C6—H6B | 109.5 | N2—C17—H17C | 109.5 |
| H6A—C6—H6B | 109.5 | H17A—C17—H17C | 109.5 |
| C1—C6—H6C | 109.5 | H17B—C17—H17C | 109.5 |
| H6A—C6—H6C | 109.5 | O4—C18—O3 | 120.7 (3) |
| H6B—C6—H6C | 109.5 | O4—C18—C4 | 124.1 (3) |
| O2—C7—O1 | 120.4 (4) | O3—C18—C4 | 115.1 (3) |
| O2—C7—C2 | 126.8 (4) | O3—C19—C20 | 108.4 (4) |
| O1—C7—C2 | 112.8 (4) | O3—C19—H19A | 110.0 |
| C9—C8—O1 | 106.0 (14) | C20—C19—H19A | 110.0 |

| | | | |
|---|---|---|---|
| C9—C8—H8A | 110.5 | O3—C19—H19B | 110.0 |
| O1—C8—H8A | 110.5 | C20—C19—H19B | 110.0 |
| C9—C8—H8B | 110.5 | H19A—C19—H19B | 108.4 |
| O1—C8—H8B | 110.5 | C19—C20—H20A | 109.5 |
| H8A—C8—H8B | 108.7 | C19—C20—H20B | 109.5 |
| O1—C8′—C9′ | 99 (3) | H20A—C20—H20B | 109.5 |
| O1—C8′—H8′1 | 111.9 | C19—C20—H20C | 109.5 |
| C9′—C8′—H8′1 | 111.9 | H20A—C20—H20C | 109.5 |
| O1—C8′—H8′2 | 111.9 | H20B—C20—H20C | 109.5 |
| C9′—C8′—H8′2 | 111.9 | C5—C21—H21A | 109.5 |
| H8′1—C8′—H8′2 | 109.6 | C5—C21—H21B | 109.5 |
| C8′—C9′—H9′1 | 109.5 | H21A—C21—H21B | 109.5 |
| C8′—C9′—H9′2 | 109.5 | C5—C21—H21C | 109.5 |
| H9′1—C9′—H9′2 | 109.5 | H21A—C21—H21C | 109.5 |
| C8′—C9′—H9′3 | 109.5 | H21B—C21—H21C | 109.5 |

*Hydrogen-bond geometry (Å, º)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| N1—H1···O4$^{i}$ | 0.86 | 2.18 | 3.036 (4) | 173 |

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