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

# Ethyl 2-methyl-5-oxo-4-(3,4,5-trimethoxyphenyl)-1,4,5,6,7,8-hexahydroquinoline-3-carboxylate

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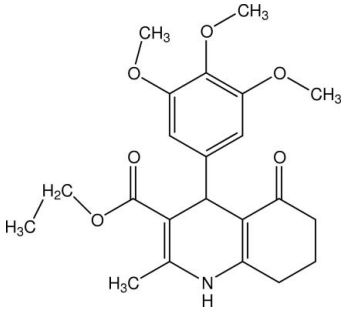
Received 12 July 2010; accepted 29 July 2010

Key indicators: single-crystal X-ray study; $T$ = 294 K; mean $\sigma$(C–C) = 0.003 Å; $R$ factor = 0.043; $wR$ factor = 0.129; data-to-parameter ratio = 13.3.

In the molecular structure of the title compound, $C_{22}H_{27}NO_6$, the dihydropyridine ring adopts a flattened boat conformation while the cyclohexenone ring is in an envelope conformation. In the crystal, molecules stack parallel to the crystallographic $a$ axis linked by intermolecular N—H···O and C—H···O hydrogen bonds.

## Related literature

For general background to the biological activity of quinoline derivatives, see: Baba (1997); Baba *et al.* (1997,1998); Davies *et al.* (2005); Rose & Draeger *et al.* (1992); Warrior *et al.* (2005).



## Experimental

### Crystal data

$C_{22}H_{27}NO_6$
$M_r$ = 401.44
Triclinic, $P\overline{1}$
$a$ = 7.512 (2) Å
$b$ = 10.402 (1) Å
$c$ = 14.568 (3) Å
$\alpha$ = 109.77 (3)°
$\beta$ = 95.42 (1)°
$\gamma$ = 104.41 (2)°
$V$ = 1017.4 (4) Å$^3$
$Z$ = 2
Mo $K\alpha$ radiation
$\mu$ = 0.10 mm$^{-1}$
$T$ = 294 K
0.26 × 0.24 × 0.21 mm

### Data collection

Nonius MACH3 diffractometer
Absorption correction: $\psi$ scan (North *et al.*, 1968)
$T_{min}$ = 0.976, $T_{max}$ = 0.980
4471 measured reflections
3574 independent reflections
2653 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.013
3 standard reflections every 60 min
intensity decay: none

### Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.043
$wR(F^2)$ = 0.129
$S$ = 1.03
3574 reflections
268 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.23 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$N$···O1$^{i}$ | 0.83 (3) | 2.21 (3) | 2.995 (2) | 160 (3) |
| C2—H2$B$···O4$^{ii}$ | 0.97 | 2.55 | 3.340 (3) | 138 |
| C10—H10$B$···O1$^{i}$ | 0.96 | 2.59 | 3.429 (3) | 146 |

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

Data collection: *CAD-4 EXPRESS* (Enraf–Nonius, 1994); cell refinement: *CAD-4 EXPRESS*; data reduction: *XCAD4* (Harms & Wocadlo, 1996); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *PLATON* (Spek, 2009); software used to prepare material for publication: *SHELXL97*.

VV is grateful to the DST-India for funding through the Young Scientist Scheme (Fast Track Proposal).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: JH2183).

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# supplementary materials

*Acta Cryst.* (2010). E**66**, o2240 [ **doi:10.1107/S1600536810030333** ]

# Ethyl 2-methyl-5-oxo-4-(3,4,5-trimethoxyphenyl)-1,4,5,6,7,8-hexahydroquinoline-3-carboxylate

**S. Natarajan, P. Indumathi, B. P. Reddy, V. Vijayakumar and P. L. N. Lakshman**

## Comment

Some derivatives of quinoline are naturally occurring alkaloids and are very attractive for their various bioactivities. For example, they have calcium modulatory properties (Rose &Draeger, 1992), antibacterial activity (Davies *et al.*, 2005),fungicidal activity (Warrior *et al.*, 2005) and selective inhibitor of human immunodeficiency virus type I (HIV-1) transcription (Baba, 1997; Baba *et al.*,1997,1998) *etc*. Due to these significant biological activities, the structure of a quinoline derivative, ethyl 2-methyl-5-oxo -4-(3,4,5-trimethoxyphenyl)-1,4,5,6,7,8-hexahydroquinoline-3-carboxylate is elucidated and reported.

The dihydropyridine ring of the title molecule adopts a flattened boat conformation. The cyclohexenone ring is in an envelope conformation with atom C3 at the flap. The 3,4,5-trimethoxyphenyl ring and the plane of the dihydropyridine ring (N1/C1/C6/C7/C8/C9) are nearly perpendicular to each other, with a dihedral angle of 89.33 (4)°. In the crystal structure, molecules are linked into a sheet (Fig.2) parallel to the *a* axis by N—H···O and C—H···O intra and intermolecular hydrogen bonds (Table 1). Further, it is observed that these sheets are assembled through centrosymmetrically related pairs of molecules by C2—H2B···O4 inter molecular hydrogen bond and weak interactions, which stabilize the structure.

## Experimental

3,4,5-trimethoxy benzaldehyde (10 mmol), 1,3-cyclohexanedione (10 mmol) and ethyl acetoacetate (10 mmol) were mixed along with 20 ml of ethanol. Ammonium acetate (10 mmol) was added to the mixture and refluxed on water bath for about 1 h. The progress of the reaction was monitored by TLC. After confirming that the reaction got completed, the reaction mixture was allowed to cool to room temperature and left aside for a day. Solid crystals started to grow from the mother liquor. It was filtered and washed with diethyl ether to ensure pure crystals [yield: 60%, m.p. 576–578 K].

## Refinement

H atoms were placed at calculated positions and allowed to ride on their carrier atoms with N—H = 0.83 Å, C—H = 0.93–0.97 Å, and $U_{iso}$ = 1.2$U_{eq}$(C) for $CH_2$ and CH groups and $U_{iso}$ = 1.5$U_{eq}$(C) for $CH_3$ group.

## Figures

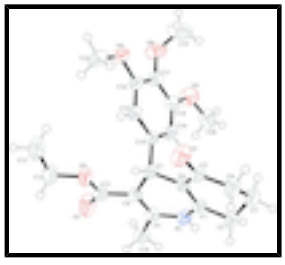

Fig. 1. The molecular structure of (I), showing 50% probability displacement ellipsoids and the atom-numbering scheme.

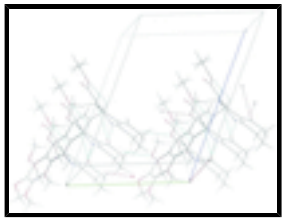

Fig. 2. The packing diagram showing the sheets of hydrogen bonds along the *a* axis

**Ethyl 2-methyl-5-oxo-4-(3,4,5-trimethoxyphenyl)-1,4,5,6,7,8- hexahydroquinoline-3-carboxylate**

*Crystal data*

| | |
|---|---|
| $C_{22}H_{27}NO_6$ | $Z = 2$ |
| $M_r = 401.44$ | $F(000) = 428$ |
| Triclinic, $P\overline{1}$ | $D_x = 1.310$ Mg m$^{-3}$ |
| Hall symbol: -P 1 | Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å |
| $a = 7.512$ (2) Å | Cell parameters from 25 reflections |
| $b = 10.402$ (1) Å | $\theta = 2–25°$ |
| $c = 14.568$ (3) Å | $\mu = 0.10$ mm$^{-1}$ |
| $\alpha = 109.77$ (3)° | $T = 294$ K |
| $\beta = 95.42$ (1)° | Block, colourless |
| $\gamma = 104.41$ (2)° | 0.26 × 0.24 × 0.21 mm |
| $V = 1017.4$ (4) Å$^3$ | |

*Data collection*

| | |
|---|---|
| Nonius MACH3 diffractometer | 2653 reflections with $I > 2\sigma(I)$ |
| Radiation source: fine-focus sealed tube | $R_{int} = 0.013$ |
| graphite | $\theta_{max} = 25.0°$, $\theta_{min} = 2.1°$ |
| ω–2θ scans | $h = -1\rightarrow8$ |
| Absorption correction: ψ scan (North *et al.*, 1968) | $k = -12\rightarrow12$ |
| $T_{min} = 0.976$, $T_{max} = 0.980$ | $l = -17\rightarrow17$ |
| 4471 measured reflections | 3 standard reflections every 60 min |
| 3574 independent reflections | intensity decay: none |

*Refinement*

| | |
|---|---|
| Refinement on $F^2$ | Secondary atom site location: difference Fourier map |
| Least-squares matrix: full | Hydrogen site location: inferred from neighbouring sites |
| $R[F^2 > 2\sigma(F^2)] = 0.043$ | H atoms treated by a mixture of independent and constrained refinement |
| $wR(F^2) = 0.129$ | $w = 1/[\sigma^2(F_o^2) + (0.0609P)^2 + 0.3979P]$ where $P = (F_o^2 + 2F_c^2)/3$ |
| $S = 1.03$ | $(\Delta/\sigma)_{max} < 0.001$ |
| 3574 reflections | $\Delta\rho_{max} = 0.23$ e Å$^{-3}$ |

| | |
|---|---|
| 268 parameters | $\Delta\rho_{min} = -0.18$ e Å$^{-3}$ |
| 0 restraints | Extinction correction: *SHELXL97* (Sheldrick, 2008), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$ |
| Primary atom site location: structure-invariant direct methods | Extinction coefficient: 0.013 (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 (Å$^2$)*

| | *x* | *y* | *z* | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| C22 | 0.4600 (4) | 0.2952 (4) | 0.0056 (2) | 0.0841 (9) |
| H22A | 0.4922 | 0.3586 | −0.0293 | 0.101* |
| H22B | 0.3534 | 0.2153 | −0.0339 | 0.101* |
| H22C | 0.5642 | 0.2613 | 0.0178 | 0.101* |
| H1N | −0.286 (4) | −0.135 (3) | 0.2897 (18) | 0.061 (7)* |
| O4 | 0.68460 (19) | 0.35699 (15) | 0.22606 (11) | 0.0519 (4) |
| O3 | 0.36747 (19) | 0.26111 (15) | 0.50993 (10) | 0.0523 (4) |
| N1 | −0.1712 (2) | −0.09684 (18) | 0.30488 (13) | 0.0405 (4) |
| O1 | 0.41288 (19) | −0.16053 (16) | 0.25103 (12) | 0.0588 (4) |
| C6 | 0.1238 (2) | −0.11864 (19) | 0.27165 (13) | 0.0323 (4) |
| C1 | −0.0644 (2) | −0.17812 (19) | 0.25700 (13) | 0.0347 (4) |
| C8 | 0.0925 (2) | 0.09809 (19) | 0.40306 (13) | 0.0346 (4) |
| C9 | −0.0944 (2) | 0.0320 (2) | 0.38398 (14) | 0.0356 (4) |
| C15 | 0.4536 (2) | 0.20151 (19) | 0.27900 (14) | 0.0371 (4) |
| H15 | 0.5452 | 0.1991 | 0.3256 | 0.044* |
| O2 | 0.0990 (2) | 0.30115 (19) | 0.54511 (14) | 0.0765 (6) |
| C5 | 0.2404 (3) | −0.2085 (2) | 0.23209 (15) | 0.0407 (5) |
| C16 | 0.5036 (3) | 0.28065 (19) | 0.22121 (15) | 0.0388 (4) |
| O6 | 0.4166 (2) | 0.36880 (18) | 0.09651 (13) | 0.0659 (5) |
| C19 | 0.1316 (3) | 0.1295 (2) | 0.19881 (14) | 0.0393 (4) |
| H19 | 0.0068 | 0.0788 | 0.1914 | 0.047* |
| C7 | 0.2165 (2) | 0.03861 (18) | 0.33288 (13) | 0.0330 (4) |
| H7 | 0.3332 | 0.0471 | 0.3740 | 0.040* |
| C14 | 0.2679 (2) | 0.12545 (18) | 0.26814 (13) | 0.0332 (4) |
| O5 | 0.0588 (2) | 0.21801 (19) | 0.06860 (13) | 0.0656 (5) |
| C3 | −0.0564 (3) | −0.3963 (2) | 0.12134 (17) | 0.0557 (6) |
| H3A | −0.0602 | −0.3578 | 0.0694 | 0.067* |
| H3B | −0.1143 | −0.4991 | 0.0910 | 0.067* |

| | | | | |
|---|---|---|---|---|
| C18 | 0.1810 (3) | 0.2088 (2) | 0.14062 (15) | 0.0430 (5) |
| C17 | 0.3684 (3) | 0.2858 (2) | 0.15175 (15) | 0.0438 (5) |
| C11 | 0.1788 (3) | 0.2281 (2) | 0.49145 (15) | 0.0446 (5) |
| C2 | −0.1646 (3) | −0.3315 (2) | 0.19699 (16) | 0.0448 (5) |
| H2A | −0.2869 | −0.3398 | 0.1631 | 0.054* |
| H2B | −0.1832 | −0.3840 | 0.2408 | 0.054* |
| C10 | −0.2342 (3) | 0.0813 (2) | 0.44419 (16) | 0.0479 (5) |
| H10A | −0.1986 | 0.0885 | 0.5112 | 0.058* |
| H10B | −0.3565 | 0.0135 | 0.4152 | 0.058* |
| H10C | −0.2366 | 0.1733 | 0.4445 | 0.058* |
| C4 | 0.1457 (3) | −0.3643 (2) | 0.16960 (18) | 0.0539 (6) |
| H4A | 0.1496 | −0.4198 | 0.2111 | 0.065* |
| H4B | 0.2156 | −0.3952 | 0.1179 | 0.065* |
| C20 | −0.1304 (3) | 0.1336 (3) | 0.04654 (18) | 0.0630 (7) |
| H20A | −0.1993 | 0.1503 | −0.0050 | 0.076* |
| H20B | −0.1835 | 0.1588 | 0.1051 | 0.076* |
| H20C | −0.1369 | 0.0342 | 0.0244 | 0.076* |
| C21 | 0.8277 (3) | 0.3578 (3) | 0.29756 (19) | 0.0573 (6) |
| H21A | 0.9466 | 0.4146 | 0.2935 | 0.069* |
| H21B | 0.8305 | 0.2616 | 0.2845 | 0.069* |
| H21C | 0.8025 | 0.3978 | 0.3630 | 0.069* |
| C13 | 0.6642 (3) | 0.4322 (3) | 0.5930 (2) | 0.0768 (8) |
| H13A | 0.7287 | 0.5156 | 0.6512 | 0.092* |
| H13B | 0.6768 | 0.4544 | 0.5347 | 0.092* |
| H13C | 0.7174 | 0.3564 | 0.5904 | 0.092* |
| C12 | 0.4662 (3) | 0.3867 (3) | 0.59723 (18) | 0.0667 (7) |
| H12A | 0.4530 | 0.3652 | 0.6565 | 0.080* |
| H12B | 0.4124 | 0.4635 | 0.6007 | 0.080* |

*Atomic displacement parameters (Å$^2$)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| C22 | 0.0715 (18) | 0.133 (3) | 0.080 (2) | 0.0367 (18) | 0.0272 (15) | 0.072 (2) |
| O4 | 0.0339 (8) | 0.0517 (9) | 0.0690 (10) | 0.0004 (6) | 0.0116 (7) | 0.0298 (8) |
| O3 | 0.0343 (8) | 0.0524 (9) | 0.0480 (8) | 0.0054 (6) | 0.0031 (6) | −0.0016 (7) |
| N1 | 0.0211 (8) | 0.0445 (9) | 0.0519 (10) | 0.0076 (7) | 0.0058 (7) | 0.0150 (8) |
| O1 | 0.0272 (8) | 0.0560 (9) | 0.0804 (11) | 0.0141 (7) | 0.0118 (7) | 0.0085 (8) |
| C6 | 0.0257 (9) | 0.0357 (10) | 0.0354 (9) | 0.0083 (7) | 0.0064 (7) | 0.0137 (8) |
| C1 | 0.0276 (9) | 0.0381 (10) | 0.0394 (10) | 0.0079 (8) | 0.0057 (8) | 0.0172 (8) |
| C8 | 0.0294 (9) | 0.0378 (10) | 0.0375 (10) | 0.0117 (8) | 0.0073 (8) | 0.0139 (8) |
| C9 | 0.0310 (10) | 0.0411 (10) | 0.0407 (10) | 0.0154 (8) | 0.0094 (8) | 0.0189 (8) |
| C15 | 0.0280 (9) | 0.0374 (10) | 0.0428 (11) | 0.0072 (8) | 0.0046 (8) | 0.0139 (8) |
| O2 | 0.0504 (10) | 0.0693 (11) | 0.0758 (12) | 0.0162 (8) | 0.0164 (9) | −0.0140 (9) |
| C5 | 0.0321 (10) | 0.0433 (11) | 0.0455 (11) | 0.0111 (8) | 0.0085 (8) | 0.0152 (9) |
| C16 | 0.0322 (10) | 0.0323 (10) | 0.0487 (11) | 0.0065 (8) | 0.0120 (8) | 0.0128 (8) |
| O6 | 0.0674 (11) | 0.0684 (11) | 0.0800 (12) | 0.0172 (9) | 0.0201 (9) | 0.0508 (10) |
| C19 | 0.0279 (9) | 0.0405 (10) | 0.0452 (11) | 0.0065 (8) | 0.0061 (8) | 0.0138 (9) |
| C7 | 0.0230 (8) | 0.0367 (10) | 0.0379 (10) | 0.0082 (7) | 0.0054 (7) | 0.0129 (8) |

| | | | | | | |
|---|---|---|---|---|---|---|
| C14 | 0.0278 (9) | 0.0311 (9) | 0.0370 (10) | 0.0084 (7) | 0.0077 (7) | 0.0082 (8) |
| O5 | 0.0493 (10) | 0.0834 (12) | 0.0730 (11) | 0.0173 (8) | −0.0018 (8) | 0.0458 (10) |
| C3 | 0.0474 (13) | 0.0447 (12) | 0.0554 (13) | 0.0020 (10) | 0.0087 (10) | 0.0035 (10) |
| C18 | 0.0408 (11) | 0.0454 (11) | 0.0438 (11) | 0.0160 (9) | 0.0042 (9) | 0.0167 (9) |
| C17 | 0.0456 (12) | 0.0406 (11) | 0.0496 (12) | 0.0129 (9) | 0.0128 (9) | 0.0215 (9) |
| C11 | 0.0380 (11) | 0.0462 (11) | 0.0463 (11) | 0.0114 (9) | 0.0111 (9) | 0.0133 (9) |
| C2 | 0.0322 (10) | 0.0419 (11) | 0.0521 (12) | 0.0030 (8) | 0.0034 (9) | 0.0145 (9) |
| C10 | 0.0340 (11) | 0.0593 (13) | 0.0570 (13) | 0.0217 (9) | 0.0163 (9) | 0.0223 (10) |
| C4 | 0.0470 (12) | 0.0435 (12) | 0.0636 (14) | 0.0163 (10) | 0.0146 (11) | 0.0080 (10) |
| C20 | 0.0466 (13) | 0.0887 (18) | 0.0515 (13) | 0.0309 (13) | 0.0002 (10) | 0.0186 (13) |
| C21 | 0.0307 (11) | 0.0586 (14) | 0.0789 (16) | 0.0055 (10) | 0.0112 (11) | 0.0268 (12) |
| C13 | 0.0482 (14) | 0.0747 (17) | 0.0697 (17) | 0.0042 (12) | −0.0008 (12) | −0.0059 (14) |
| C12 | 0.0475 (13) | 0.0650 (15) | 0.0528 (14) | 0.0008 (11) | 0.0051 (11) | −0.0078 (12) |

*Geometric parameters (Å, °)*

| | | | |
|---|---|---|---|
| C22—O6 | 1.402 (3) | C19—H19 | 0.9300 |
| C22—H22A | 0.9600 | C7—C14 | 1.525 (2) |
| C22—H22B | 0.9600 | C7—H7 | 0.9800 |
| C22—H22C | 0.9600 | O5—C18 | 1.372 (2) |
| O4—C16 | 1.375 (2) | O5—C20 | 1.413 (3) |
| O4—C21 | 1.421 (3) | C3—C2 | 1.506 (3) |
| O3—C11 | 1.350 (2) | C3—C4 | 1.516 (3) |
| O3—C12 | 1.446 (3) | C3—H3A | 0.9700 |
| N1—C1 | 1.374 (2) | C3—H3B | 0.9700 |
| N1—C9 | 1.379 (3) | C18—C17 | 1.399 (3) |
| N1—H1N | 0.83 (3) | C2—H2A | 0.9700 |
| O1—C5 | 1.234 (2) | C2—H2B | 0.9700 |
| C6—C1 | 1.358 (2) | C10—H10A | 0.9600 |
| C6—C5 | 1.453 (3) | C10—H10B | 0.9600 |
| C6—C7 | 1.512 (3) | C10—H10C | 0.9600 |
| C1—C2 | 1.486 (3) | C4—H4A | 0.9700 |
| C8—C9 | 1.355 (2) | C4—H4B | 0.9700 |
| C8—C11 | 1.461 (3) | C20—H20A | 0.9600 |
| C8—C7 | 1.528 (2) | C20—H20B | 0.9600 |
| C9—C10 | 1.508 (3) | C20—H20C | 0.9600 |
| C15—C16 | 1.379 (3) | C21—H21A | 0.9600 |
| C15—C14 | 1.386 (2) | C21—H21B | 0.9600 |
| C15—H15 | 0.9300 | C21—H21C | 0.9600 |
| O2—C11 | 1.209 (2) | C13—C12 | 1.458 (3) |
| C5—C4 | 1.505 (3) | C13—H13A | 0.9600 |
| C16—C17 | 1.389 (3) | C13—H13B | 0.9600 |
| O6—C17 | 1.376 (2) | C13—H13C | 0.9600 |
| C19—C18 | 1.384 (3) | C12—H12A | 0.9700 |
| C19—C14 | 1.387 (3) | C12—H12B | 0.9700 |
| O6—C22—H22A | 109.5 | O5—C18—C19 | 125.06 (18) |
| O6—C22—H22B | 109.5 | O5—C18—C17 | 114.73 (18) |
| H22A—C22—H22B | 109.5 | C19—C18—C17 | 120.20 (18) |
| O6—C22—H22C | 109.5 | O6—C17—C16 | 120.62 (18) |

| | | | |
|---|---|---|---|
| H22A—C22—H22C | 109.5 | O6—C17—C18 | 120.21 (18) |
| H22B—C22—H22C | 109.5 | C16—C17—C18 | 119.13 (18) |
| C16—O4—C21 | 117.61 (16) | O2—C11—O3 | 121.13 (19) |
| C11—O3—C12 | 116.21 (17) | O2—C11—C8 | 126.93 (19) |
| C1—N1—C9 | 122.70 (16) | O3—C11—C8 | 111.93 (16) |
| C1—N1—H1N | 116.1 (17) | C1—C2—C3 | 111.44 (16) |
| C9—N1—H1N | 120.6 (17) | C1—C2—H2A | 109.3 |
| C1—C6—C5 | 119.71 (16) | C3—C2—H2A | 109.3 |
| C1—C6—C7 | 121.31 (16) | C1—C2—H2B | 109.3 |
| C5—C6—C7 | 118.90 (15) | C3—C2—H2B | 109.3 |
| C6—C1—N1 | 119.52 (17) | H2A—C2—H2B | 108.0 |
| C6—C1—C2 | 124.04 (17) | C9—C10—H10A | 109.5 |
| N1—C1—C2 | 116.30 (16) | C9—C10—H10B | 109.5 |
| C9—C8—C11 | 120.34 (17) | H10A—C10—H10B | 109.5 |
| C9—C8—C7 | 120.86 (17) | C9—C10—H10C | 109.5 |
| C11—C8—C7 | 118.80 (16) | H10A—C10—H10C | 109.5 |
| C8—C9—N1 | 119.58 (17) | H10B—C10—H10C | 109.5 |
| C8—C9—C10 | 126.59 (18) | C5—C4—C3 | 113.84 (18) |
| N1—C9—C10 | 113.77 (16) | C5—C4—H4A | 108.8 |
| C16—C15—C14 | 120.44 (18) | C3—C4—H4A | 108.8 |
| C16—C15—H15 | 119.8 | C5—C4—H4B | 108.8 |
| C14—C15—H15 | 119.8 | C3—C4—H4B | 108.8 |
| O1—C5—C6 | 121.43 (18) | H4A—C4—H4B | 107.7 |
| O1—C5—C4 | 120.26 (18) | O5—C20—H20A | 109.5 |
| C6—C5—C4 | 118.29 (16) | O5—C20—H20B | 109.5 |
| O4—C16—C15 | 124.14 (18) | H20A—C20—H20B | 109.5 |
| O4—C16—C17 | 115.44 (17) | O5—C20—H20C | 109.5 |
| C15—C16—C17 | 120.42 (17) | H20A—C20—H20C | 109.5 |
| C17—O6—C22 | 113.52 (19) | H20B—C20—H20C | 109.5 |
| C18—C19—C14 | 120.14 (17) | O4—C21—H21A | 109.5 |
| C18—C19—H19 | 119.9 | O4—C21—H21B | 109.5 |
| C14—C19—H19 | 119.9 | H21A—C21—H21B | 109.5 |
| C6—C7—C14 | 112.18 (15) | O4—C21—H21C | 109.5 |
| C6—C7—C8 | 110.40 (14) | H21A—C21—H21C | 109.5 |
| C14—C7—C8 | 111.39 (14) | H21B—C21—H21C | 109.5 |
| C6—C7—H7 | 107.6 | C12—C13—H13A | 109.5 |
| C14—C7—H7 | 107.6 | C12—C13—H13B | 109.5 |
| C8—C7—H7 | 107.6 | H13A—C13—H13B | 109.5 |
| C15—C14—C19 | 119.66 (17) | C12—C13—H13C | 109.5 |
| C15—C14—C7 | 119.36 (16) | H13A—C13—H13C | 109.5 |
| C19—C14—C7 | 120.98 (16) | H13B—C13—H13C | 109.5 |
| C18—O5—C20 | 118.35 (18) | O3—C12—C13 | 110.0 (2) |
| C2—C3—C4 | 110.79 (18) | O3—C12—H12A | 109.7 |
| C2—C3—H3A | 109.5 | C13—C12—H12A | 109.7 |
| C4—C3—H3A | 109.5 | O3—C12—H12B | 109.7 |
| C2—C3—H3B | 109.5 | C13—C12—H12B | 109.7 |
| C4—C3—H3B | 109.5 | H12A—C12—H12B | 108.2 |
| H3A—C3—H3B | 108.1 | | |
| C5—C6—C1—N1 | 171.81 (16) | C6—C7—C14—C15 | −120.58 (18) |

| | | | |
|---|---|---|---|
| C7—C6—C1—N1 | −5.1 (3) | C8—C7—C14—C15 | 115.10 (18) |
| C5—C6—C1—C2 | −3.6 (3) | C6—C7—C14—C19 | 59.5 (2) |
| C7—C6—C1—C2 | 179.45 (17) | C8—C7—C14—C19 | −64.8 (2) |
| C9—N1—C1—C6 | −14.9 (3) | C20—O5—C18—C19 | −4.4 (3) |
| C9—N1—C1—C2 | 160.86 (18) | C20—O5—C18—C17 | 174.56 (19) |
| C11—C8—C9—N1 | −174.65 (17) | C14—C19—C18—O5 | 178.62 (19) |
| C7—C8—C9—N1 | 5.4 (3) | C14—C19—C18—C17 | −0.3 (3) |
| C11—C8—C9—C10 | 2.4 (3) | C22—O6—C17—C16 | 91.5 (3) |
| C7—C8—C9—C10 | −177.55 (17) | C22—O6—C17—C18 | −90.4 (3) |
| C1—N1—C9—C8 | 14.7 (3) | O4—C16—C17—O6 | −3.2 (3) |
| C1—N1—C9—C10 | −162.71 (17) | C15—C16—C17—O6 | 177.59 (18) |
| C1—C6—C5—O1 | −173.80 (19) | O4—C16—C17—C18 | 178.77 (17) |
| C7—C6—C5—O1 | 3.2 (3) | C15—C16—C17—C18 | −0.5 (3) |
| C1—C6—C5—C4 | 4.2 (3) | O5—C18—C17—O6 | 3.4 (3) |
| C7—C6—C5—C4 | −178.84 (18) | C19—C18—C17—O6 | −177.62 (18) |
| C21—O4—C16—C15 | −2.2 (3) | O5—C18—C17—C16 | −178.56 (18) |
| C21—O4—C16—C17 | 178.60 (18) | C19—C18—C17—C16 | 0.5 (3) |
| C14—C15—C16—O4 | −178.84 (17) | C12—O3—C11—O2 | 0.6 (3) |
| C14—C15—C16—C17 | 0.3 (3) | C12—O3—C11—C8 | −178.82 (19) |
| C1—C6—C7—C14 | −103.04 (19) | C9—C8—C11—O2 | −12.8 (3) |
| C5—C6—C7—C14 | 80.0 (2) | C7—C8—C11—O2 | 167.2 (2) |
| C1—C6—C7—C8 | 21.8 (2) | C9—C8—C11—O3 | 166.62 (17) |
| C5—C6—C7—C8 | −155.10 (16) | C7—C8—C11—O3 | −13.4 (2) |
| C9—C8—C7—C6 | −22.0 (2) | C6—C1—C2—C3 | −24.4 (3) |
| C11—C8—C7—C6 | 158.08 (16) | N1—C1—C2—C3 | 160.04 (18) |
| C9—C8—C7—C14 | 103.36 (19) | C4—C3—C2—C1 | 49.9 (3) |
| C11—C8—C7—C14 | −76.6 (2) | O1—C5—C4—C3 | −158.6 (2) |
| C16—C15—C14—C19 | −0.2 (3) | C6—C5—C4—C3 | 23.5 (3) |
| C16—C15—C14—C7 | 179.89 (16) | C2—C3—C4—C5 | −50.4 (3) |
| C18—C19—C14—C15 | 0.1 (3) | C11—O3—C12—C13 | −164.3 (2) |
| C18—C19—C14—C7 | −179.92 (17) | | |

*Hydrogen-bond geometry (Å, °)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| C7—H7···O3 | 0.98 | 2.36 | 2.720 (2) | 101 |
| N1—H1N···O1$^{i}$ | 0.83 (3) | 2.21 (3) | 2.995 (2) | 160 (3) |
| C2—H2B···O4$^{ii}$ | 0.97 | 2.55 | 3.340 (3) | 138 |
| C10—H10B···O1$^{i}$ | 0.96 | 2.59 | 3.429 (3) | 146 |

Symmetry codes: (i) *x*−1, *y*, *z*; (ii) *x*−1, *y*−1, *z*.

**Fig. 1**

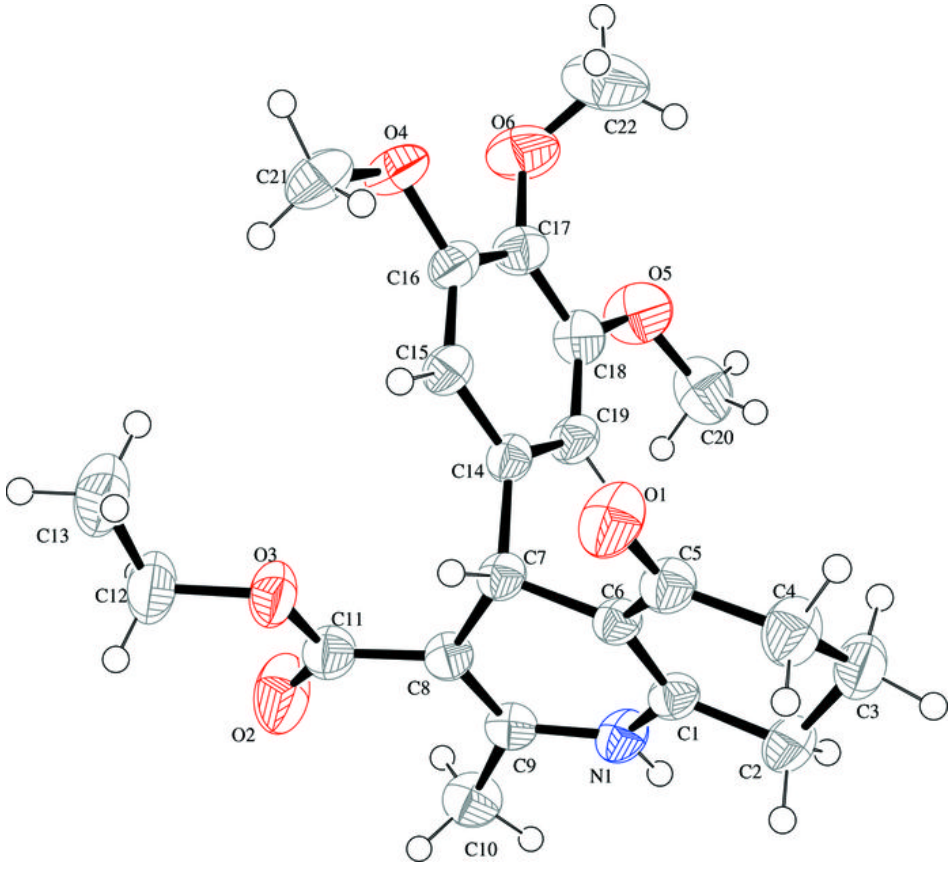
O4
O6
C22
C21
C17
C16
O5
C15
C18
C19
C20
C14
O1
C13
O3
C7
C5
C12
C6
C4
C11
C8
C3
O2
C1
C9
N1
C2
C10

**Fig. 2**