Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

2-Amino-7,7-dimethyl-5-oxo-4-(*p*-tolyl)-5,6,7,8-tetrahydro-4*H*-chromene-3-carbonitrile

Sumati Anthal,[a] Goutam Brahmachari,[b] Sujay Laskar,[b] Bubun Banerjee,[b] Rajni Kant[a] and Vivek K. Gupta[a]*

[a]Post-Graduate Department of Physics & Electronics, University of Jammu, Jammu Tawi 180 006, India, and [b]Laboratory of Natural Products & Organic Synthesis, Department of Chemistry, Visva-Bharati University, Santiniketan 731 235, West Bengal, India
Correspondence e-mail: vivek_gupta2k2@hotmail.com

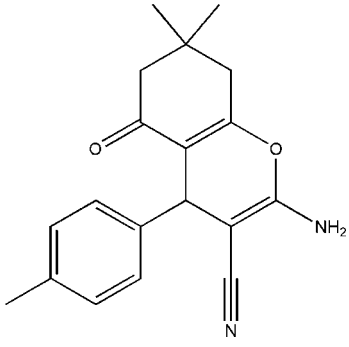
Received 20 July 2012; accepted 25 July 2012

Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.003 Å; R factor = 0.046; wR factor = 0.115; data-to-parameter ratio = 14.4.

In the title molecule, $C_{19}H_{20}N_2O_2$, the cyclohexene ring adopts a sofa conformation, while the pyran ring adopts a flattened boat conformation. In the crystal, molecules are linked by N—H···N and N—H···O hydrogen bonds, forming a two-dimensional network parallel to (010).

Related literature

For background to compounds containing the 4*H*-pyran unit, see: Brahmachari (2010); Hatakeyama *et al.* (1988). For the biological activity of compounds containing a tetrahydrobenzo[*b*]pyran ring system, see: Andreani & Lapi (1960); Bonsignore *et al.* (1993); Brahmachari (2011); Konkoy *et al.* (2001). For 2-amino-4*H*-pyrans as photoactive materials, see: Armetso *et al.* (1989). For the synthesis of related compounds, see: Jin *et al.* (2004); Balalaie *et al.* (2007). For related structures, see: Tu *et al.* (2001); Wang (2011). For ring conformations, see: Duax *et al.* (1975).



Experimental

Crystal data

$C_{19}H_{20}N_2O_2$
M_r = 308.37
Monoclinic, $P2_1/n$
a = 9.4622 (3) Å
b = 16.8820 (5) Å
c = 10.8301 (4) Å
β = 111.842 (4)°
V = 1605.82 (9) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.08 mm^{-1}
T = 293 K
0.30 × 0.20 × 0.20 mm

Data collection

Oxford Diffraction Xcalibur Sapphire3 diffractometer
Absorption correction: multi-scan (*CrysAlis PRO*; Oxford Diffraction, 2010)
T_{min} = 0.862, T_{max} = 1.000
18449 measured reflections
3149 independent reflections
2428 reflections with $I > 2\sigma(I)$
R_{int} = 0.037

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.046
$wR(F^2)$ = 0.115
S = 1.05
3149 reflections
219 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.20 e Å^{-3}
$\Delta\rho_{min}$ = −0.22 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N13—H131···O5^{i}	0.89 (2)	2.06 (2)	2.913 (2)	161 (2)
N13—H132···N15^{ii}	0.87 (2)	2.35 (2)	3.168 (2)	156 (2)

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

Data collection: *CrysAlis PRO* (Oxford Diffraction, 2010); cell refinement: *CrysAlis PRO*; data reduction: *CrysAlis PRO*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3* (Farrugia, 1997); software used to prepare material for publication: *PLATON* (Spek, 2009).

SL is grateful to the CSIR, New Delhi, for the award of a Senior Research Fellowship. BB is grateful to the UGC, New Delhi, for the awarding of a Junior Research Fellowship. RK acknowledges the Department of Science & Technology for the single-crystal X-ray diffractometer sanctioned as a National Facility under project No. SR/S2/CMP-47/2003.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: LH5503).

References

Andreani, L. L. & Lapi, E. (1960). *Boll. Chim. Farm.* **99**, 583–586.
Armetso, D., Horspool, W. M., Martin, N., Ramos, A. & Seoane, C. (1989). *J. Org. Chem.* **54**, 3069–3072.
Balalaie, S., Bararjanian, M. & Sheikh-Ahmadi, M. (2007). *Synth. Commun.* **37**, 1097–1108.
Bonsignore, L., Loy, G., Secci, D. & Calignano, A. (1993). *Eur. J. Med. Chem.* **28**, 517–520.
Brahmachari, G. (2010). In *Handbook of Pharmaceutical Natural Products*. Weinheim: Wiley-VCH Verlag GmbH & Co.
Brahmachari, G. (2011). *Asia Pac. Biotech News*, **15**, 21–26.
Duax, W. L. & Norton, D. A. (1975). *Atlas of Steroid Structures*, Vol. 1. New York: Plenum Press.

Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
Hatakeyama, S., Ochi, N., Numata, H. & Takano, S. (1988). *J. Chem. Soc. Chem. Commun.* pp. 1202–1204.
Jin, T. S., Wang, A. Q., Wang, X., Zhang, J. S. & Li, T. S. (2004). *Synlett*, **5**, 871–873.
Konkoy, C. S., Fick, D. B., Cai, S. X., Lan, N. C. & Keana, J. F. W. (2001). PCT Appl. WO 0075123; *Chem. Abstr.* (2000), **134**, 29313a.
Oxford Diffraction (2010). *CrysAlis PRO*. Oxford Diffraction Ltd, Yarnton, England.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Spek, A. L. (2009). *Acta Cryst.* D**65**, 148–155.
Tu, S.-J., Deng, X., Fang, Y.-Y., Guo, Y.-M., Du, M. & Liu, X.-H. (2001). *Acta Cryst.* E**57**, o358–o359.
Wang, X. (2011). *Acta Cryst.* E**67**, o832.

supporting information

Acta Cryst. (2012). E**68**, o2592–o2593 [https://doi.org/10.1107/S1600536812033570]

2-Amino-7,7-dimethyl-5-oxo-4-(*p*-tolyl)-5,6,7,8-tetrahydro-4*H*-chromene-3-carbonitrile

Sumati Anthal, Goutam Brahmachari, Sujay Laskar, Bubun Banerjee, Rajni Kant and Vivek K. Gupta

S1. Comment

4*H*-Pyran units constitute structural features of a broad range of bioactive natural products (Brahmachari, 2010; Hatakeyama *et al.*, 1988). The tetrahydrobenzo[*b*]pyran ring is of particular interest because compounds bearing this structural motif exhibit diverse biological activities such as spasmolytic, anticancer and anti-anaphylactin agents (Andreani *et al.*, 1960; Bonsignore *et al.*, 1993), anti-Alzheimer's disease (Brahmachari, 2011), anti-Huntington's disease, anti-Parkinson's disease and anti-HIV (Konkoy *et al.*, 2001). 2-Amino-4*H*-pyrans have also been found to be useful as photoactive materials (Armetso *et al.*, 1989). Hence, investigation of the structural features of biologically relevant tetrahydrobenzo[*b*]pyran derivatives is of both scientific and practical interest. In continuation of our efforts to develop useful synthetic protocols for biologically significant molecules, we herein report an efficient and environmentally benign synthesis and the crystal structure of the title compound. The bond lengths and angles of the title compound are normal and correspond to those observed in related structures (Tu *et al.*, 2001; Wang, 2011). The cyclohexene ring adopts a sofa conformation while the pyran ring adopts a flattened boat conformation with asymmetry parameters [ΔCs(C7) = 5.71] and [ΔCs (O1—C4) = 0.08; ΔCs (C2—C3) = 9.8)] respectively (Duax *et al.*, 1975). In the crystal structure, intermolecular N—H···N and N—H···O hydrogen bonds link the molecules into a two-dimensional network parallel to (010) (Fig.2).

S2. Experimental

The synthesis of the title compounds was carried out *via* one-pot multi-component reaction in aqueous ethanol using low-cost and environmentally benign sodium formate as catalyst at room temperature. An oven-dried screw cap test tube was charged with a magnetic stir bar, *p*-methylbenzaldehyde (0.12 g, 1 mmol), malononitrile (0.066 g, 1 mmol) and sodium formate (0.136 g, 20 mol %) in 5 ml aqueous ethanol. The reaction mixture was then started to stir vigorously and after 20 min 1 mmol of dimedone (0.14 g) was added, and continued to stir. After completion of the overall reaction (2 h) as monitored by TLC, a white solid was precipitated out, filtered off, and washed with aqueous ethanol. Recrystallization from ethanol afforded the title compound as white block-shaped crystals (252 mg, yield 82%) with the m.p. 492–494 K (lit. 492–495 K) (Balalaie *et al.*, 2007). Rf 0.82 (EtOAc). White solid; FT—IR (KBr) *v*max 3375, 3256, 3180, 2961, 2920, 2885, 2187, 1677, 1637, 1607, 1512, 1460, 1414, 1366, 1215, 1137, 1030, 825, 764, 565 cm-1; 1*H*-NMR (DMSO-d6, 400 MHz) & 13 C-NMR (DMSO-d6; 100 MHz) data are in excellent agreement with literature values (Jin *et al.*, 2004; Balalaie *et al.*, 2007); TOF-MS: calculated for $C_{19}H_{20}N_2O_2Na$ 331.1422 [*M* + Na]+; found 331.1426. For crystallization 60 mg of the compound was dissolved in 20 ml mixture of ethanol and water (5:1) and left for several days at ambient temperature which yielded white block-shaped crystals.

S3. Refinement

H131 and H132 attached to N13 were located in a difference map and refined isotropically. The remaining H atoms were positioned geometrically and were treated as riding on their parent C atoms, with C—H distances of 0.93–0.98 Å; and with U_{iso}(H) = 1.2U_{eq}(C), except for the methyl groups where U_{iso}(H) = 1.5U_{eq}(C).

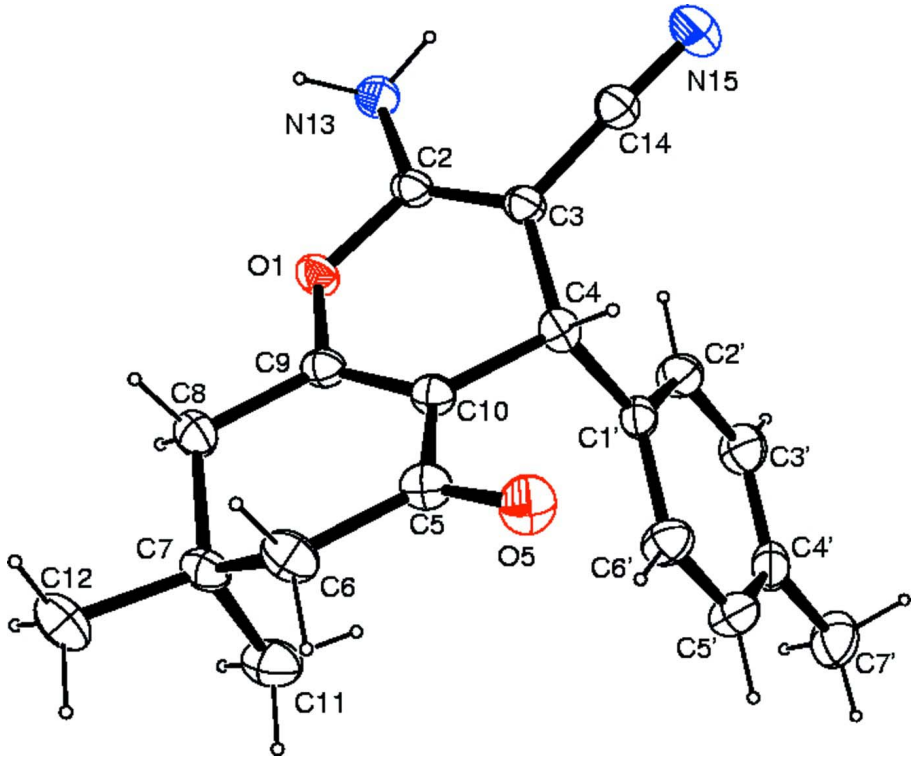


Figure 1

The molecular structure of the title compound with ellipsoids are drawn at the 40% probability level. H atoms are shown as small spheres of arbitrary radii.

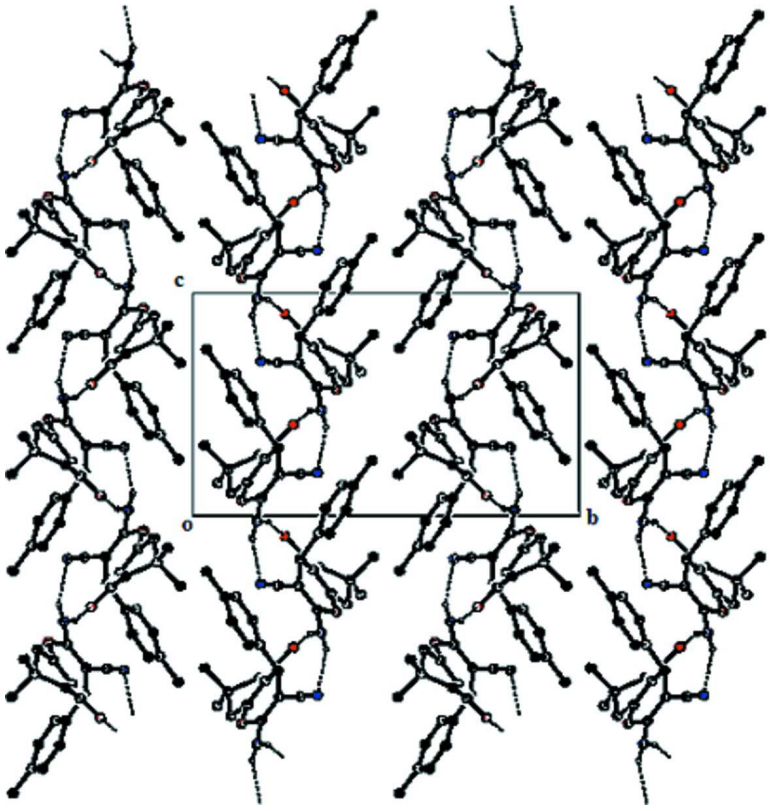


Figure 2

The packing arrangement of molecules viewed along the *a* axis. Hydrogen bonds are shown as dashed lines.

2-Amino-7,7-dimethyl-5-oxo-4-(*p*-tolyl)-5,6,7,8-tetrahydro- 4*H*-chromene-3-carbonitrile

Crystal data

$C_{19}H_{20}N_2O_2$
$M_r = 308.37$
Monoclinic, $P2_1/n$
Hall symbol: -P 2yn
$a = 9.4622$ (3) Å
$b = 16.8820$ (5) Å
$c = 10.8301$ (4) Å
$\beta = 111.842$ (4)°
$V = 1605.82$ (9) Å^3
$Z = 4$
$F(000) = 656$
$D_x = 1.276$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 7806 reflections
$\theta = 3.6$–29.1°
$\mu = 0.08$ mm^{-1}
$T = 293$ K
Block, white
0.30 × 0.20 × 0.20 mm

Data collection

Oxford Diffraction Xcalibur Sapphire3 diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
Detector resolution: 16.1049 pixels mm^{-1}
ω scans
Absorption correction: multi-scan (*CrysAlis PRO*; Oxford Diffraction, 2010)
$T_{min} = 0.862$, $T_{max} = 1.000$
18449 measured reflections
3149 independent reflections
2428 reflections with $I > 2\sigma(I)$
$R_{int} = 0.037$
$\theta_{max} = 26.0°$, $\theta_{min} = 3.6°$
$h = -11 \rightarrow 11$
$k = -20 \rightarrow 20$
$l = -13 \rightarrow 13$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.046$
$wR(F^2) = 0.115$
$S = 1.05$
3149 reflections
219 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 atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0501P)^2 + 0.4013P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.20$ e Å^{-3}
$\Delta\rho_{min} = -0.22$ e Å^{-3}

Special details

Experimental. Absorption correction: *CrysAlis PRO*, Oxford Diffraction Ltd., Version 1.171.34.40 (release 27–08-2010 CrysAlis171. NET) (compiled Aug 27 2010,11:50:40) Empirical absorption correction using spherical harmonics, implemented in SCALE3 ABSPACK scaling algorithm.

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}
O1	0.62616 (12)	0.12747 (6)	0.06792 (11)	0.0370 (3)
C1′	0.87094 (17)	0.17332 (9)	0.42111 (15)	0.0324 (4)
C2′	1.00784 (18)	0.14575 (11)	0.41998 (17)	0.0433 (4)
H2′	1.0343	0.1568	0.3472	0.052*
C3′	1.1067 (2)	0.10204 (11)	0.52474 (19)	0.0498 (5)
H3′	1.1978	0.0842	0.5206	0.060*
C4′	1.0732 (2)	0.08433 (10)	0.63476 (17)	0.0442 (4)
C5′	0.9371 (2)	0.11238 (11)	0.63673 (18)	0.0504 (5)
H5′	0.9118	0.1018	0.7103	0.060*
C6′	0.8370 (2)	0.15591 (11)	0.53232 (17)	0.0456 (4)
H6′	0.7459	0.1737	0.5367	0.055*
C7′	1.1810 (2)	0.03593 (13)	0.7475 (2)	0.0645 (6)
H7′1	1.1854	−0.0172	0.7175	0.097*
H7′2	1.1456	0.0350	0.8199	0.097*
H7′3	1.2807	0.0592	0.7770	0.097*
C2	0.74563 (17)	0.17699 (9)	0.07890 (15)	0.0324 (4)
C3	0.80680 (17)	0.22511 (9)	0.18489 (15)	0.0325 (4)
C4	0.75981 (17)	0.22126 (9)	0.30428 (15)	0.0325 (4)
H4	0.7564	0.2755	0.3353	0.039*
O5	0.53082 (14)	0.26138 (8)	0.40897 (12)	0.0488 (3)
C5	0.49500 (18)	0.21056 (10)	0.32139 (16)	0.0372 (4)

C6	0.33944 (19)	0.17324 (11)	0.27258 (19)	0.0455 (4)
H6A	0.2688	0.2083	0.2076	0.055*
H6B	0.3062	0.1686	0.3469	0.055*
C7	0.33113 (18)	0.09159 (10)	0.20960 (17)	0.0386 (4)
C8	0.39789 (18)	0.09955 (11)	0.10153 (17)	0.0406 (4)
H8A	0.4110	0.0472	0.0705	0.049*
H8B	0.3267	0.1284	0.0267	0.049*
C9	0.54697 (16)	0.14144 (9)	0.14978 (15)	0.0321 (4)
C10	0.60023 (17)	0.18777 (9)	0.25695 (15)	0.0312 (4)
C11	0.4198 (2)	0.03164 (12)	0.3151 (2)	0.0568 (5)
H11A	0.5238	0.0486	0.3562	0.085*
H11B	0.4164	−0.0192	0.2743	0.085*
H11C	0.3752	0.0277	0.3813	0.085*
C12	0.1651 (2)	0.06452 (13)	0.1450 (2)	0.0579 (5)
H12A	0.1612	0.0141	0.1030	0.087*
H12B	0.1084	0.1027	0.0797	0.087*
H12C	0.1218	0.0598	0.2120	0.087*
N13	0.78732 (18)	0.16693 (10)	−0.02573 (15)	0.0432 (4)
C14	0.92224 (19)	0.27927 (10)	0.18699 (16)	0.0396 (4)
N15	1.01708 (19)	0.32308 (10)	0.19332 (17)	0.0578 (5)
H131	0.864 (2)	0.1956 (12)	−0.0300 (19)	0.055 (6)*
H132	0.716 (2)	0.1541 (12)	−0.101 (2)	0.059 (6)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0355 (6)	0.0427 (6)	0.0391 (6)	−0.0084 (5)	0.0212 (5)	−0.0064 (5)
C1′	0.0317 (8)	0.0332 (8)	0.0311 (8)	−0.0050 (6)	0.0102 (6)	−0.0051 (6)
C2′	0.0344 (9)	0.0567 (11)	0.0404 (9)	0.0021 (8)	0.0159 (7)	0.0041 (8)
C3′	0.0340 (9)	0.0609 (12)	0.0502 (11)	0.0054 (8)	0.0108 (8)	0.0035 (9)
C4′	0.0430 (10)	0.0388 (10)	0.0396 (10)	−0.0060 (8)	0.0024 (8)	−0.0022 (7)
C5′	0.0597 (12)	0.0567 (12)	0.0361 (9)	−0.0019 (9)	0.0194 (9)	0.0032 (8)
C6′	0.0459 (10)	0.0552 (11)	0.0400 (10)	0.0052 (8)	0.0210 (8)	0.0014 (8)
C7′	0.0638 (13)	0.0584 (13)	0.0519 (12)	0.0001 (10)	−0.0010 (10)	0.0096 (10)
C2	0.0269 (7)	0.0373 (9)	0.0351 (8)	−0.0002 (6)	0.0139 (6)	0.0059 (7)
C3	0.0289 (8)	0.0349 (9)	0.0345 (8)	−0.0019 (6)	0.0127 (6)	0.0044 (6)
C4	0.0311 (8)	0.0311 (8)	0.0366 (8)	−0.0005 (6)	0.0141 (7)	−0.0034 (6)
O5	0.0463 (7)	0.0556 (8)	0.0513 (7)	0.0043 (6)	0.0260 (6)	−0.0126 (6)
C5	0.0367 (9)	0.0374 (9)	0.0412 (9)	0.0067 (7)	0.0188 (7)	0.0033 (7)
C6	0.0359 (9)	0.0487 (11)	0.0608 (11)	0.0036 (8)	0.0284 (8)	−0.0003 (8)
C7	0.0317 (8)	0.0417 (10)	0.0482 (10)	−0.0005 (7)	0.0215 (7)	0.0046 (7)
C8	0.0322 (8)	0.0479 (10)	0.0422 (9)	−0.0069 (7)	0.0146 (7)	−0.0028 (7)
C9	0.0289 (8)	0.0362 (9)	0.0343 (8)	0.0034 (6)	0.0156 (7)	0.0046 (7)
C10	0.0290 (8)	0.0327 (8)	0.0345 (8)	0.0027 (6)	0.0147 (6)	0.0018 (6)
C11	0.0588 (12)	0.0526 (12)	0.0643 (13)	0.0024 (9)	0.0290 (10)	0.0162 (10)
C12	0.0373 (10)	0.0653 (13)	0.0772 (14)	−0.0096 (9)	0.0283 (10)	−0.0017 (11)
N13	0.0352 (8)	0.0630 (10)	0.0359 (8)	−0.0091 (7)	0.0184 (7)	0.0004 (7)
C14	0.0355 (9)	0.0425 (10)	0.0399 (9)	−0.0034 (8)	0.0132 (7)	0.0035 (7)

N15	0.0501 (9)	0.0570 (10)	0.0648 (11)	−0.0187 (8)	0.0198 (8)	0.0028 (8)

Geometric parameters (Å, º)

O1—C2	1.3751 (18)	O5—C5	1.229 (2)
O1—C9	1.3774 (18)	C5—C10	1.464 (2)
C1′—C2′	1.381 (2)	C5—C6	1.505 (2)
C1′—C6′	1.389 (2)	C6—C7	1.527 (2)
C1′—C4	1.540 (2)	C6—H6A	0.9700
C2′—C3′	1.384 (2)	C6—H6B	0.9700
C2′—H2′	0.9300	C7—C11	1.523 (2)
C3′—C4′	1.375 (3)	C7—C8	1.529 (2)
C3′—H3′	0.9300	C7—C12	1.532 (2)
C4′—C5′	1.380 (3)	C8—C9	1.488 (2)
C4′—C7′	1.508 (2)	C8—H8A	0.9700
C5′—C6′	1.385 (3)	C8—H8B	0.9700
C5′—H5′	0.9300	C9—C10	1.333 (2)
C6′—H6′	0.9300	C11—H11A	0.9600
C7′—H7′1	0.9600	C11—H11B	0.9600
C7′—H7′2	0.9600	C11—H11C	0.9600
C7′—H7′3	0.9600	C12—H12A	0.9600
C2—N13	1.342 (2)	C12—H12B	0.9600
C2—C3	1.348 (2)	C12—H12C	0.9600
C3—C14	1.418 (2)	N13—H131	0.88 (2)
C3—C4	1.517 (2)	N13—H132	0.87 (2)
C4—C10	1.512 (2)	C14—N15	1.145 (2)
C4—H4	0.9800		

C2—O1—C9	117.83 (12)	C5—C6—C7	114.58 (13)
C2′—C1′—C6′	117.19 (15)	C5—C6—H6A	108.6
C2′—C1′—C4	121.74 (14)	C7—C6—H6A	108.6
C6′—C1′—C4	121.08 (14)	C5—C6—H6B	108.6
C1′—C2′—C3′	121.49 (17)	C7—C6—H6B	108.6
C1′—C2′—H2′	119.3	H6A—C6—H6B	107.6
C3′—C2′—H2′	119.3	C11—C7—C6	110.02 (15)
C4′—C3′—C2′	121.53 (17)	C11—C7—C8	111.30 (14)
C4′—C3′—H3′	119.2	C6—C7—C8	107.18 (14)
C2′—C3′—H3′	119.2	C11—C7—C12	109.22 (15)
C3′—C4′—C5′	117.14 (16)	C6—C7—C12	110.29 (14)
C3′—C4′—C7′	120.99 (18)	C8—C7—C12	108.80 (15)
C5′—C4′—C7′	121.87 (18)	C9—C8—C7	112.37 (14)
C4′—C5′—C6′	121.89 (17)	C9—C8—H8A	109.1
C4′—C5′—H5′	119.1	C7—C8—H8A	109.1
C6′—C5′—H5′	119.1	C9—C8—H8B	109.1
C5′—C6′—C1′	120.77 (17)	C7—C8—H8B	109.1
C5′—C6′—H6′	119.6	H8A—C8—H8B	107.9
C1′—C6′—H6′	119.6	C10—C9—O1	122.74 (14)
C4′—C7′—H7′1	109.5	C10—C9—C8	125.90 (14)

C4′—C7′—H7′2	109.5	O1—C9—C8	111.36 (13)
H7′1—C7′—H7′2	109.5	C9—C10—C5	117.73 (14)
C4′—C7′—H7′3	109.5	C9—C10—C4	121.39 (14)
H7′1—C7′—H7′3	109.5	C5—C10—C4	120.70 (14)
H7′2—C7′—H7′3	109.5	C7—C11—H11A	109.5
N13—C2—C3	128.83 (15)	C7—C11—H11B	109.5
N13—C2—O1	109.91 (14)	H11A—C11—H11B	109.5
C3—C2—O1	121.24 (14)	C7—C11—H11C	109.5
C2—C3—C14	119.09 (15)	H11A—C11—H11C	109.5
C2—C3—C4	122.04 (13)	H11B—C11—H11C	109.5
C14—C3—C4	118.83 (14)	C7—C12—H12A	109.5
C10—C4—C3	107.42 (12)	C7—C12—H12B	109.5
C10—C4—C1′	111.87 (12)	H12A—C12—H12B	109.5
C3—C4—C1′	113.31 (13)	C7—C12—H12C	109.5
C10—C4—H4	108.0	H12A—C12—H12C	109.5
C3—C4—H4	108.0	H12B—C12—H12C	109.5
C1′—C4—H4	108.0	C2—N13—H131	118.5 (13)
O5—C5—C10	120.66 (15)	C2—N13—H132	116.8 (14)
O5—C5—C6	121.07 (15)	H131—N13—H132	116.9 (19)
C10—C5—C6	118.19 (15)	N15—C14—C3	177.67 (18)

C6′—C1′—C2′—C3′	0.5 (3)	C10—C5—C6—C7	26.8 (2)
C4—C1′—C2′—C3′	−179.38 (16)	C5—C6—C7—C11	67.88 (19)
C1′—C2′—C3′—C4′	−0.2 (3)	C5—C6—C7—C8	−53.27 (19)
C2′—C3′—C4′—C5′	−0.4 (3)	C5—C6—C7—C12	−171.57 (15)
C2′—C3′—C4′—C7′	179.35 (17)	C11—C7—C8—C9	−71.62 (19)
C3′—C4′—C5′—C6′	0.6 (3)	C6—C7—C8—C9	48.72 (18)
C7′—C4′—C5′—C6′	−179.09 (18)	C12—C7—C8—C9	167.98 (15)
C4′—C5′—C6′—C1′	−0.3 (3)	C2—O1—C9—C10	−15.5 (2)
C2′—C1′—C6′—C5′	−0.2 (3)	C2—O1—C9—C8	163.73 (13)
C4—C1′—C6′—C5′	179.64 (15)	C7—C8—C9—C10	−19.0 (2)
C9—O1—C2—N13	−166.15 (13)	C7—C8—C9—O1	161.72 (13)
C9—O1—C2—C3	15.4 (2)	O1—C9—C10—C5	168.43 (13)
N13—C2—C3—C14	6.5 (3)	C8—C9—C10—C5	−10.7 (2)
O1—C2—C3—C14	−175.37 (14)	O1—C9—C10—C4	−6.7 (2)
N13—C2—C3—C4	−171.42 (16)	C8—C9—C10—C4	174.14 (15)
O1—C2—C3—C4	6.7 (2)	O5—C5—C10—C9	−170.15 (15)
C2—C3—C4—C10	−25.3 (2)	C6—C5—C10—C9	6.7 (2)
C14—C3—C4—C10	156.78 (14)	O5—C5—C10—C4	5.0 (2)
C2—C3—C4—C1′	98.75 (17)	C6—C5—C10—C4	−178.09 (14)
C14—C3—C4—C1′	−79.14 (18)	C3—C4—C10—C9	25.20 (19)
C2′—C1′—C4—C10	129.01 (16)	C1′—C4—C10—C9	−99.76 (17)
C6′—C1′—C4—C10	−50.87 (19)	C3—C4—C10—C5	−149.80 (14)
C2′—C1′—C4—C3	7.4 (2)	C1′—C4—C10—C5	85.24 (17)
C6′—C1′—C4—C3	−172.49 (15)	C2—C3—C14—N15	−157 (5)
O5—C5—C6—C7	−156.36 (16)	C4—C3—C14—N15	20 (5)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N13—H131···O5^{i}	0.89 (2)	2.06 (2)	2.913 (2)	161 (2)
N13—H132···N15^{ii}	0.87 (2)	2.35 (2)	3.168 (2)	156 (2)

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