Acta Cryst E CRYSTALLOGRAPHIC COMMUNICATIONS

OPEN ACCESS

ISSN 2056-9890

Crystal structure of 3-(3,4,5-trimethoxyphenyl)-1,2,3,4-tetrahydrocyclopenta[*b*]indole-2-carboxylic acid

Daniara C. Fernandes,[a] Deborah de Alencar Simoni,[b]* Manoel T. Rodrigues Jr,[a] Marilia S. Santos[a] and Fernando Coelho[a]

[a]Laboratory of Synthesis of Natural Products and Drugs, Institute of Chemistry, University of Campinas, PO Box 6154 – 13083-970, Campinas, SP, Brazil, and [b]Laboratory of Single Crystal X-Ray Diffraction, Institute of Chemistry, University of Campinas, PO Box 6154 – 13083-970, Campinas, SP, Brazil. *Correspondence e-mail: dsimoni@iqm.unicamp.br

Received 2 May 2015; accepted 5 May 2015

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

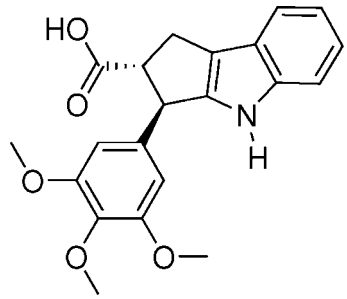
In the title compound, $C_{21}H_{21}NO_5$, obtained from a Morita–Baylis–Hillman adduct, the hydrogenated five-membered ring adopts a shallow envelope conformation, with the C atom bearing the carboxylic acid substituent deviating by 0.237 (1) Å from the mean plane of the other four atoms (r.m.s. deviation = 0.007 Å). The dihedral angle between the fused ring system (all atoms; r.m.s. deviation = 0.057 Å) and the pendant trimethoxy benzene ring is 66.65 (3)°. The C atoms of the *meta*-methoxy groups lie close to the plane of the benzene ring [deviations = 0.052 (1) and −0.083 (1) Å], whereas the C atom of the *para*-methoxy group is significantly displaced [deviation = −1.289 (1) Å]. In the crystal, carboxylic acid inversion dimers generate $R_2^2(8)$ loops. The dimers are connected by N—H···O hydrogen bonds, forming [011] chains. A C—H···O interaction is also observed.

Keywords: crystal stryctur; indole skeleton; Morita–Baylis–Hillman adduct; hydrogen bonding.

CCDC reference: 1063387

1. Related literature

For compounds presenting an indole skeleton unit and examples of them, see: Xu *et al.* (2012); Humphrey & Kuethe (2006). For methods of synthesis of indoles, see: Jordan *et al.* (2011); Humphrey & Kuethe (2006). For the use of Morita–Baylis–Hillman adducts as building blocks for organic synthesis, see: Basavaiah & Veeraraghavaiah (2012); Coelho *et al.* (2002).



2. Experimental

2.1. Crystal data

$C_{21}H_{21}NO_5$
M_r = 367.39
Triclinic, $P\overline{1}$
a = 7.203 (1) Å
b = 9.5844 (12) Å
c = 12.9957 (17) Å
α = 91.939 (5)°
β = 97.198 (6)°
γ = 91.716 (5)°
V = 889.1 (2) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.10 mm^{-1}
T = 100 K
0.34 × 0.17 × 0.13 mm

2.2. Data collection

Bruker APEX CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2010)
T_{min} = 0.967, T_{max} = 0.987
100846 measured reflections
7825 independent reflections
6558 reflections with $I > 2\sigma(I)$
R_{int} = 0.031

2.3. Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.038
$wR(F^2)$ = 0.119
S = 0.94
7825 reflections
248 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.58 e Å^{-3}
$\Delta\rho_{min}$ = −0.27 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O4—H4···O3^{i}	0.84	1.84	2.6748 (8)	176
N1—H1···O1^{ii}	0.88	2.20	2.9041 (8)	136
C12—H12B···O2^{iii}	0.98	2.62	3.3905 (11)	137

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

Data collection: *APEX2* (Bruker, 2010); cell refinement: *SAINT* (Bruker, 2010); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXLE* (Hübschle *et al.*, 2011); molecular graphics: *Mercury* (Macrae *et al.*, 2006); software used to prepare material for publication: *OLEX2* (Dolomanov *et al.*, 2003) and *publCIF* (Westrip, 2010).

Acknowledgements

The authors acknowledge the Fundação de Amparo à Pesquisa do Estado de São Paulo (FAPESP 2013/07600-3 and 09/51602-5) for financial support and the Conselho Nacional

de Desenvolvimento Científico e Tecnológico (CNPq) for a research fellowship. MTRJr and MSS thank CNPq and Fapesp for fellowships.

Supporting information for this paper is available from the IUCr electronic archives (Reference: HB7417).

References

Basavaiah, D. & Veeraraghavaiah, G. (2012). *Chem. Soc. Rev.* **41**, 68–78.
Bruker (2010). *APEX2*, *SAINT* and *SADABS*. Bruker AXS Inc., Madison, Wisconsin, USA.
Coelho, F., Almeida, W. P., Veronese, D., Mateus, C. R., Lopes, E. C. S., Rossi, R. C., Silveira, G. P. C. & Pavam, C. H. (2002). *Tetrahedron*, **58**, 7437–7447.
Dolomanov, O. V., Blake, A. J., Champness, N. R. & Schröder, M. (2003). *J. Appl. Cryst.* **36**, 1283–1284.
Hübschle, C. B., Sheldrick, G. M. & Dittrich, B. (2011). *J. Appl. Cryst.* **44**, 1281–1284.
Humphrey, G. R. & Kuethe, J. T. (2006). *Chem. Rev.* **106**, 2875–2911.
Jordan, J. A., Gribble, G. W. & Badenock, J. C. (2011). *Tetrahedron Lett.* **52**, 6772–6774.
Macrae, C. F., Edgington, P. R., McCabe, P., Pidcock, E., Shields, G. P., Taylor, R., Towler, M. & van de Streek, J. (2006). *J. Appl. Cryst.* **39**, 453–457.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Westrip, S. P. (2010). *J. Appl. Cryst.* **43**, 920–925.
Xu, B., Guo, Z. L., Jin, W. Y., Wang, Z. P., Peng, Y. G. & Guo, Q. X. (2012). *Angew. Chem. Int. Ed.* **51**, 1059–1062.

supporting information

Acta Cryst. (2015). E**71**, o395–o396 [doi:10.1107/S2056989015008786]

Crystal structure of 3-(3,4,5-trimethoxyphenyl)-1,2,3,4-tetrahydrocyclopenta[*b*]indole-2-carboxylic acid

Daniara Fernandes, Deborah de Alencar Simoni, Manoel T. Rodrigues, Marilia S. Santos and Fernando Coelho

S1. Introduction

Indole skeleton is an aromatic heterocycle possessing a benzene ring fused to a pyrrole ring which exhibits a wide range of biological and pharmacological activities. Compounds presenting this moiety have been successfully synthesized and used in medicinal chemistry (Xu *et al.*, 2012). In spite of the number of developed methods for the preparation of indoles (Xu *et al.*, 2012; Humphrey and Kuethe, 2006), our interest to use Morita–Baylis–Hillman adducts as building blocks for organic synthesis resulted in a successful stereoselective strategy to obtain compounds of this class.

S2. Experimental

S2.1. Synthesis and crystallization

\ The synthesis of 3-(3,4,5-trimethoxyphenyl)-1,2,3,4-tetrahydrocyclopenta[b]indole-2-\ carboxylic acid started with a mixture of 1 mmol of (±)-methyl 2-[hydroxy(3,4,5-trimethoxyphenyl)-methyl]acrylate (the Morita–Baylis–Hillman adduct), 1.2 mmol of indole and 1 mmol of 2-iodoxybenzoic acid, in acetonitrile (5 mL). This mixture was kept under reflux to give 1,3-dicarbonyl compound, which was further reduced by sodium tetrahydroborate, resulting in the corresponding *β*-hydroxy-carbonyl.

This was then treated with trifluoromethanesulfonic acid and submitted to basic hydrolysis. The cyclopenta[*b*]indole, obtained with excellent diastereoselectivity (>99:1) and overall yield of 70%, was purified by flash chromatography (hexane/ethyl acetate (60:40)). **3-(3,4,5-trimethoxyphenyl)-1,2,3,4-tetrahydrocyclopenta[b]indole-2-\ carboxylic acid** was dissolved in 10:1 (v/v) chloroform/methanol mixture and kept in the freezer to allow slowly formation of irregular colorless single crystals.

S2.2. Refinement

The positions of hydrogen atoms bound to carbon atoms were idealized and calculated by riding model, with C—H bond lengths of 0.95, 0.98 and 0.99 Å for phenyl, methyl and methylene, respectively. The isotropic displacement parameters values (Uiso(H)) were fixed at 1.5Ueq(C) for methyl H atoms and 1.2Ueq(C) for all other attached H atoms.

S3. Results and discussion

The molecules of the title compound present a trimethoxyphenyl ring bonded to a system of three rings, in which an indole skeleton unit is fused to a five-membered ring possessing a carboxylic unit (Fig. 1). It crystallized in $P\bar{1}$ space group and has a conformational structure determined by intra and intermolecular nonclassical (C—H—O) and intermolecular (O—H—O and N—H—O) bonding (Table 1, Fig. 2).

All of the rings in the structure are almost planar, with r.m.s. of 0.010, 0.066, 0.006 and 0.006 Å for trimethoxyphenyl, five-membered, pyrrole and benzene rings, respectively. The three rings fused system is essentially planar (r.m.s. deviation of 0.057 Å) and make a plane-plane angle of 113.35° with the trimethoxyphenyl ring.

With respect to the pyrrole ring, the benzene ring and the five-membered ring make dihedral angles C14—C13—N1—C5 = 176.67 (7)° and C3—C4—C16—C17 = -175.72 (8)°, respectively. The dihedral angle between the five-membered ring and its trimethoxyphenyl substituent (C5—C6—C7—C8) is -164.79 (16)°, while that between the five-membered ring and the carboxylic unit (C16—C17—C18—O4) is -56.30 (8)°, which is consistent with the expected *trans* relative configuration of this isomer.

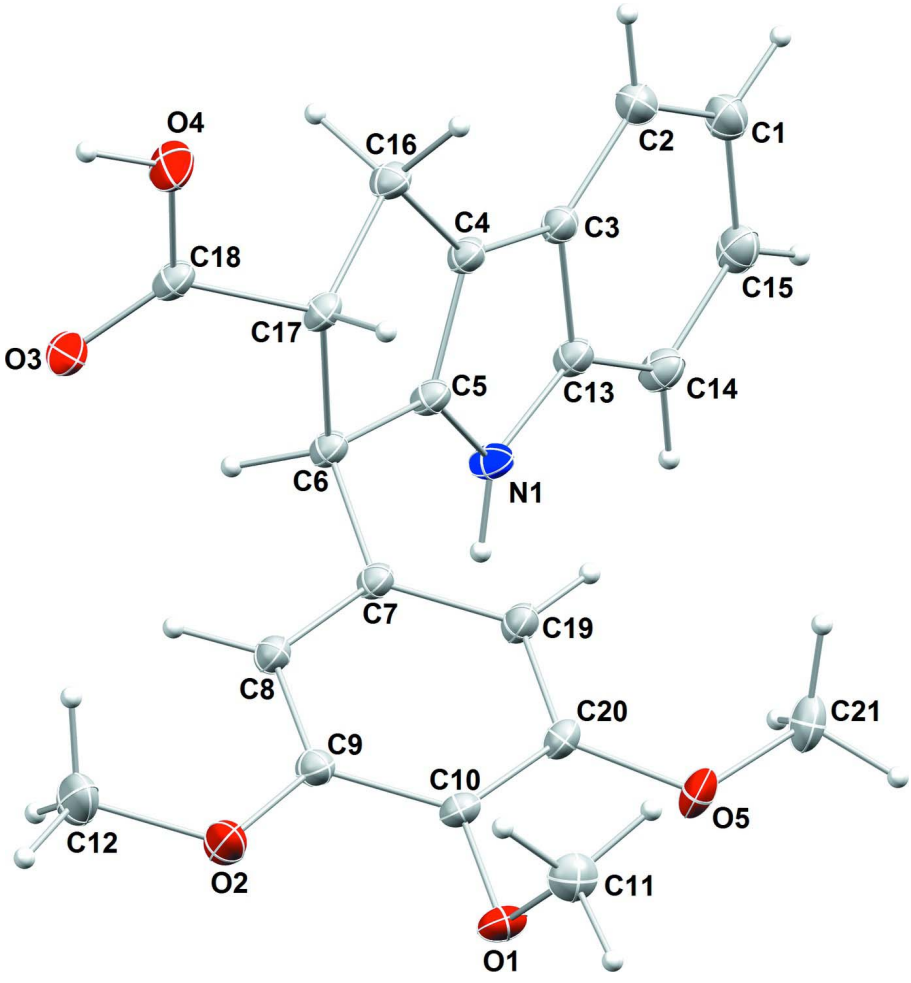


Figure 1

The molecular structure of the title compound with 50% probability displacement ellipsoids.

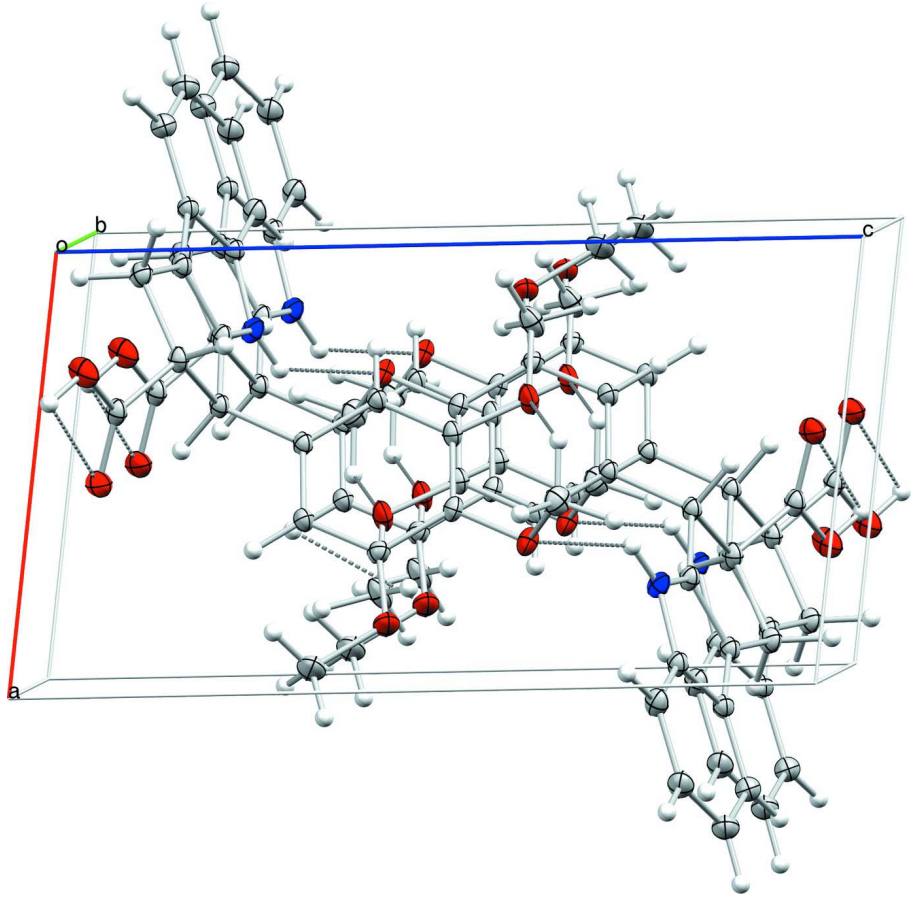


Figure 2

Crystal packing of the title compound, showing hydrogen-bonding interactions.

3-(3,4,5-Trimethoxyphenyl)-1,2,3,4-tetrahydrocyclopenta[*b*]indole-2-carboxylic acid

Crystal data

$C_{21}H_{21}NO_5$
$M_r = 367.39$
Triclinic, $P\overline{1}$
Hall symbol: -P 1
$a = 7.203$ (1) Å
$b = 9.5844$ (12) Å
$c = 12.9957$ (17) Å
$\alpha = 91.939$ (5)°
$\beta = 97.198$ (6)°
$\gamma = 91.716$ (5)°
$V = 889.1$ (2) Å^3
$Z = 2$
$F(000) = 388$
$D_x = 1.372$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 9708 reflections
$\theta = 2.6$–38°
$\mu = 0.10$ mm^{-1}
$T = 100$ K
Irregular, colourless
0.34 × 0.17 × 0.13 mm

Data collection

Bruker APEX CCD diffractometer
Radiation source: fine-focus sealed tube
Detector resolution: 8.3333 pixels mm^{-1}
phi and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2010)
$T_{min} = 0.967$, $T_{max} = 0.987$
100846 measured reflections
7825 independent reflections
6558 reflections with $I > 2\sigma(I)$

R_{int} = 0.031
θ_{max} = 35.0°, θ_{min} = 1.6°
h = −11→11
k = −15→15
l = −20→20

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.038
$wR(F^2)$ = 0.119
S = 0.94
7825 reflections
248 parameters
0 restraints
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.079P)^2 + 0.2107P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.58 e Å^{-3}
$\Delta\rho_{min}$ = −0.27 e Å^{-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.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	y	z	U_{iso}*/U_{eq}
O1	0.67935 (8)	0.20458 (6)	0.61432 (4)	0.01701 (10)
O2	0.85593 (8)	0.13066 (7)	0.45301 (4)	0.02026 (11)
O3	0.52886 (8)	0.14039 (6)	0.07684 (5)	0.02004 (11)
O4	0.27178 (9)	0.00009 (6)	0.05008 (5)	0.02346 (12)
H4	0.3380	−0.0455	0.0131	0.035*
O5	0.36545 (8)	0.34406 (6)	0.59020 (4)	0.01927 (11)
N1	0.20865 (9)	0.57757 (6)	0.22742 (5)	0.01503 (10)
H1	0.2993	0.6285	0.2636	0.018*
C1	−0.33636 (10)	0.65445 (9)	0.10853 (6)	0.01960 (13)
H1A	−0.4633	0.6671	0.0816	0.024*
C2	−0.25626 (10)	0.52708 (8)	0.09178 (6)	0.01735 (12)
H2	−0.3264	0.4538	0.0522	0.021*
C3	−0.07052 (9)	0.50802 (7)	0.13406 (5)	0.01394 (11)
C4	0.05337 (9)	0.39331 (7)	0.13915 (5)	0.01372 (11)
C5	0.21619 (9)	0.43919 (7)	0.19636 (5)	0.01331 (11)
C6	0.36676 (9)	0.33516 (7)	0.20740 (5)	0.01314 (11)
H6	0.4670	0.3624	0.1645	0.016*
C7	0.45411 (9)	0.30909 (7)	0.31710 (5)	0.01289 (11)
C8	0.62291 (9)	0.24015 (7)	0.33130 (5)	0.01407 (11)
H8	0.6873	0.2187	0.2736	0.017*
C9	0.69665 (9)	0.20293 (7)	0.43061 (5)	0.01429 (11)
C10	0.60455 (9)	0.23835 (7)	0.51579 (5)	0.01402 (11)
C11	0.58428 (11)	0.08561 (8)	0.65126 (6)	0.02034 (13)
H11A	0.4493	0.1005	0.6438	0.031*
H11B	0.6302	0.0737	0.7246	0.031*
H11C	0.6086	0.0016	0.6106	0.031*
C12	0.94999 (11)	0.08870 (10)	0.36769 (7)	0.02528 (16)

H12A	0.8643	0.0311	0.3179	0.038*
H12B	1.0587	0.0345	0.3927	0.038*
H12C	0.9918	0.1716	0.3337	0.038*
C13	0.03194 (10)	0.62141 (7)	0.19116 (5)	0.01434 (11)
C14	−0.04783 (11)	0.75053 (8)	0.20623 (6)	0.01750 (12)
H14	0.0223	0.8256	0.2437	0.021*
C15	−0.23267 (11)	0.76525 (8)	0.16470 (6)	0.01940 (13)
H15	−0.2905	0.8517	0.1743	0.023*
C16	0.06472 (10)	0.24706 (7)	0.09663 (5)	0.01538 (12)
H16A	0.0682	0.2448	0.0207	0.018*
H16B	−0.0415	0.1870	0.1125	0.018*
C17	0.25541 (9)	0.20188 (7)	0.15655 (5)	0.01396 (11)
H17	0.2239	0.1432	0.2146	0.017*
C18	0.36696 (10)	0.11304 (7)	0.09044 (5)	0.01515 (12)
C19	0.36205 (9)	0.34551 (7)	0.40179 (5)	0.01444 (11)
H19	0.2477	0.3931	0.3918	0.017*
C20	0.43904 (10)	0.31162 (7)	0.50143 (5)	0.01433 (11)
C21	0.19448 (12)	0.41674 (9)	0.58029 (6)	0.02137 (14)
H21A	0.2090	0.5011	0.5411	0.032*
H21B	0.1631	0.4429	0.6494	0.032*
H21C	0.0939	0.3561	0.5435	0.032*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0210 (2)	0.0173 (2)	0.0115 (2)	−0.00177 (18)	−0.00212 (16)	0.00000 (16)
O2	0.0164 (2)	0.0267 (3)	0.0175 (2)	0.00815 (19)	0.00017 (17)	−0.00044 (19)
O3	0.0183 (2)	0.0206 (3)	0.0210 (2)	0.00222 (19)	0.00369 (18)	−0.00790 (19)
O4	0.0243 (3)	0.0181 (3)	0.0283 (3)	−0.0019 (2)	0.0082 (2)	−0.0119 (2)
O5	0.0259 (3)	0.0208 (3)	0.0124 (2)	0.0063 (2)	0.00682 (18)	−0.00088 (18)
N1	0.0170 (2)	0.0125 (2)	0.0146 (2)	0.00230 (18)	−0.00148 (18)	−0.00317 (18)
C1	0.0167 (3)	0.0230 (3)	0.0192 (3)	0.0042 (2)	0.0019 (2)	0.0012 (2)
C2	0.0155 (3)	0.0197 (3)	0.0166 (3)	0.0007 (2)	0.0015 (2)	0.0002 (2)
C3	0.0151 (3)	0.0150 (3)	0.0116 (2)	0.0016 (2)	0.00173 (19)	−0.0009 (2)
C4	0.0155 (3)	0.0137 (3)	0.0118 (2)	0.0010 (2)	0.00152 (19)	−0.00208 (19)
C5	0.0160 (3)	0.0121 (3)	0.0115 (2)	0.0017 (2)	0.00085 (19)	−0.00173 (19)
C6	0.0153 (2)	0.0127 (3)	0.0113 (2)	0.0017 (2)	0.00156 (19)	−0.00173 (19)
C7	0.0147 (2)	0.0127 (3)	0.0111 (2)	0.0013 (2)	0.00167 (19)	−0.00187 (19)
C8	0.0146 (2)	0.0153 (3)	0.0123 (2)	0.0017 (2)	0.00190 (19)	−0.0019 (2)
C9	0.0136 (2)	0.0149 (3)	0.0139 (2)	0.0015 (2)	0.00027 (19)	−0.0019 (2)
C10	0.0163 (3)	0.0142 (3)	0.0109 (2)	−0.0002 (2)	−0.00008 (19)	−0.00137 (19)
C11	0.0230 (3)	0.0187 (3)	0.0195 (3)	0.0007 (2)	0.0022 (2)	0.0047 (2)
C12	0.0191 (3)	0.0344 (4)	0.0229 (3)	0.0105 (3)	0.0035 (3)	−0.0029 (3)
C13	0.0168 (3)	0.0141 (3)	0.0120 (2)	0.0029 (2)	0.00133 (19)	−0.00108 (19)
C14	0.0212 (3)	0.0152 (3)	0.0158 (3)	0.0046 (2)	0.0008 (2)	−0.0018 (2)
C15	0.0206 (3)	0.0199 (3)	0.0181 (3)	0.0071 (2)	0.0024 (2)	0.0002 (2)
C16	0.0169 (3)	0.0139 (3)	0.0148 (3)	0.0006 (2)	0.0008 (2)	−0.0035 (2)
C17	0.0176 (3)	0.0124 (3)	0.0119 (2)	0.0016 (2)	0.00258 (19)	−0.00245 (19)

C18	0.0195 (3)	0.0134 (3)	0.0124 (2)	0.0028 (2)	0.0016 (2)	−0.0023 (2)
C19	0.0163 (3)	0.0156 (3)	0.0117 (2)	0.0032 (2)	0.0025 (2)	−0.0011 (2)
C20	0.0179 (3)	0.0139 (3)	0.0115 (2)	0.0014 (2)	0.0033 (2)	−0.00188 (19)
C21	0.0257 (3)	0.0198 (3)	0.0208 (3)	0.0062 (3)	0.0110 (3)	−0.0001 (2)

Geometric parameters (Å, º)

O1—C10	1.3785 (8)	C7—C8	1.3961 (9)
O1—C11	1.4396 (10)	C8—C9	1.3949 (10)
O2—C9	1.3632 (9)	C8—H8	0.9500
O2—C12	1.4229 (10)	C9—C10	1.3974 (9)
O3—C18	1.2232 (9)	C10—C20	1.3973 (10)
O4—C18	1.3206 (9)	C11—H11A	0.9800
O4—H4	0.8400	C11—H11B	0.9800
O5—C20	1.3591 (8)	C11—H11C	0.9800
O5—C21	1.4269 (10)	C12—H12A	0.9800
N1—C5	1.3779 (9)	C12—H12B	0.9800
N1—C13	1.3834 (9)	C12—H12C	0.9800
N1—H1	0.8800	C13—C14	1.3980 (10)
C1—C2	1.3879 (11)	C14—C15	1.3857 (11)
C1—C15	1.4078 (11)	C14—H14	0.9500
C1—H1A	0.9500	C15—H15	0.9500
C2—C3	1.4009 (10)	C16—C17	1.5719 (10)
C2—H2	0.9500	C16—H16A	0.9900
C3—C13	1.4271 (10)	C16—H16B	0.9900
C3—C4	1.4347 (9)	C17—C18	1.5078 (9)
C4—C5	1.3601 (9)	C17—H17	1.0000
C4—C16	1.4975 (10)	C19—C20	1.3961 (9)
C5—C6	1.4921 (9)	C19—H19	0.9500
C6—C7	1.5164 (9)	C21—H21A	0.9800
C6—C17	1.5686 (10)	C21—H21B	0.9800
C6—H6	1.0000	C21—H21C	0.9800
C7—C19	1.3942 (9)		

C10—O1—C11	112.48 (6)	H11A—C11—H11C	109.5
C9—O2—C12	116.77 (6)	H11B—C11—H11C	109.5
C18—O4—H4	109.5	O2—C12—H12A	109.5
C20—O5—C21	117.21 (6)	O2—C12—H12B	109.5
C5—N1—C13	107.21 (6)	H12A—C12—H12B	109.5
C5—N1—H1	126.4	O2—C12—H12C	109.5
C13—N1—H1	126.4	H12A—C12—H12C	109.5
C2—C1—C15	121.12 (7)	H12B—C12—H12C	109.5
C2—C1—H1A	119.4	N1—C13—C14	129.41 (7)
C15—C1—H1A	119.4	N1—C13—C3	108.66 (6)
C1—C2—C3	119.10 (7)	C14—C13—C3	121.91 (6)
C1—C2—H2	120.5	C15—C14—C13	117.73 (7)
C3—C2—H2	120.5	C15—C14—H14	121.1
C2—C3—C13	118.90 (6)	C13—C14—H14	121.1

C2—C3—C4	135.29 (7)	C14—C15—C1	121.22 (7)
C13—C3—C4	105.75 (6)	C14—C15—H15	119.4
C5—C4—C3	107.07 (6)	C1—C15—H15	119.4
C5—C4—C16	112.09 (6)	C4—C16—C17	101.36 (5)
C3—C4—C16	140.65 (6)	C4—C16—H16A	111.5
C4—C5—N1	111.28 (6)	C17—C16—H16A	111.5
C4—C5—C6	115.13 (6)	C4—C16—H16B	111.5
N1—C5—C6	133.48 (6)	C17—C16—H16B	111.5
C5—C6—C7	116.44 (5)	H16A—C16—H16B	109.3
C5—C6—C17	100.20 (5)	C18—C17—C6	113.56 (6)
C7—C6—C17	111.15 (5)	C18—C17—C16	113.05 (5)
C5—C6—H6	109.5	C6—C17—C16	109.05 (5)
C7—C6—H6	109.5	C18—C17—H17	106.9
C17—C6—H6	109.5	C6—C17—H17	106.9
C19—C7—C8	120.56 (6)	C16—C17—H17	106.9
C19—C7—C6	120.63 (6)	O3—C18—O4	123.34 (6)
C8—C7—C6	118.70 (6)	O3—C18—C17	124.14 (6)
C9—C8—C7	119.72 (6)	O4—C18—C17	112.51 (6)
C9—C8—H8	120.1	C7—C19—C20	119.54 (6)
C7—C8—H8	120.1	C7—C19—H19	120.2
O2—C9—C8	124.76 (6)	C20—C19—H19	120.2
O2—C9—C10	115.23 (6)	O5—C20—C19	125.19 (6)
C8—C9—C10	120.01 (6)	O5—C20—C10	114.64 (6)
O1—C10—C20	119.89 (6)	C19—C20—C10	120.17 (6)
O1—C10—C9	120.14 (6)	O5—C21—H21A	109.5
C20—C10—C9	119.94 (6)	O5—C21—H21B	109.5
O1—C11—H11A	109.5	H21A—C21—H21B	109.5
O1—C11—H11B	109.5	O5—C21—H21C	109.5
H11A—C11—H11B	109.5	H21A—C21—H21C	109.5
O1—C11—H11C	109.5	H21B—C21—H21C	109.5

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O4—H4···O3^{i}	0.84	1.84	2.6748 (8)	176
N1—H1···O1^{ii}	0.88	2.20	2.9041 (8)	136
C12—H12*B*···O2^{iii}	0.98	2.62	3.3905 (11)	137

Symmetry codes: (i) −*x*+1, −*y*, −*z*; (ii) −*x*+1, −*y*+1, −*z*+1; (iii) −*x*+2, −*y*, −*z*+1.