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(1*S*,2*S*,6*R*,7a*R*)-2-Benzyl-1,6-dihydroxyhexahydropyrrolizin-3-one

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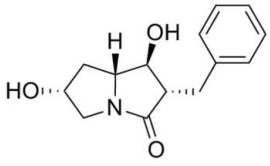
Key indicators: single-crystal X-ray study; T = 100 K; mean σ(C–C) = 0.002 Å; R factor = 0.026; wR factor = 0.078; data-to-parameter ratio = 13.3.

In the title compound, $C_{14}H_{17}NO_3$, the dihedral angles show that the H atoms at two stereocenters are in a *trans*-diaxial configuration. In the crystal, the molecules are linked by O—H···O hydrogen bonds. The absolute configuration of the molecule has been established on the basis of refinement of the Hooft and Flack parameters.

Related literature

For a synthetic sequence for the preparation of the title compound, see: de Luna Freire *et al.* (2011). For the use of this type of compounds as LFA-1 (Lymphocyte Function-Associated Antigen-1) inhibitors, see: Baumann (2007). For a related structure, see: Newton *et al.* (2004).



Experimental

Crystal data

$C_{14}H_{17}NO_3$
M_r = 247.29
Orthorhombic, $P2_12_12_1$
a = 6.6241 (3) Å
b = 13.6873 (6) Å
c = 13.9726 (6) Å
V = 1266.84 (10) Å^3
Z = 4
Cu $K\alpha$ radiation
μ = 0.74 mm^{-1}
T = 100 K
0.17 × 0.15 × 0.12 mm

Data collection

Bruker Kappa APEXII DUO diffractometer
26923 measured reflections
2295 independent reflections
2290 reflections with $I > 2\sigma(I)$
R_{int} = 0.026

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.026
$wR(F^2)$ = 0.078
S = 1.15
2295 reflections
172 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.17 e Å^{-3}
$\Delta\rho_{min}$ = −0.16 e Å^{-3}
Absolute structure: Flack (1983) and Hooft *et al.* (2008) [Hooft parameter = 0.00(2), (943 Bijvoet pairs)]
Flack parameter: 0.00 (16)

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

D—H···A	D—H	H···A	D···A	D—H···A
O3—H3A···O2^{i}	0.93 (2)	1.73 (2)	2.6395 (12)	164 (2)
O1—H1A···O3^{ii}	0.86 (2)	1.93 (2)	2.7716 (13)	167 (19)

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

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: *SHELXL97* (Sheldrick, 2008); molecular graphics: *WinGX* (Farrugia, 1999) and *PLATON* (Spek, 2009); software used to prepare material for publication: *publCIF* (Westrip, 2010) and *PLATON*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: PV2494).

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supplementary materials

Acta Cryst. (2012). E**68**, o587 [doi:10.1107/S1600536812002334]

(1*S*,2*S*,6*R*,7a*R*)-2-Benzyl-1,6-dihydroxyhexahydropyrrolizin-3-one

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Comment

The title compound can be used as a prototype for the development of new inhibitors of LFA-1 (lymphocyte function-associated antigen 1) with potential application as anti-inflammatory agents (Baumann, 2007). The title compound is a new asymmetric benzyl-pyrrolizidinone which has been synthesized in our laboratory and its crystal structure is presented in this article.

The title compound (Fig. 1) has four stereocenters and was prepared from a Morita-Baylis-Hillman adduct. The dihedral angles H3—C3—C4—H4 = -158° and H4—C4—C5—H5 = 163° show that H atoms 3, 4 and 5 at the two new stereocenters are in a *trans*-diaxial configuration. These values agree with the coupling constant values obtained for these H atoms in the ^{1}H NMR analysis. The crystal structure is stabilized by intermolecular hydrogen bonds (Tab. 1 & Fig. 2).

Experimental

The title compound was prepared using a synthetic sequence described in the literature (de Luna Freire *et al.*, 2011) and purified by flash silica gel column chromatography (CH_2Cl_2:MeOH – solvent gradient: 0:100 to 97:03) to afford 0.06 g (as a white solid) in 97% yield. It was then recrystallized using the liquid-vapor saturation method, dissolved in ethanol and crystallized with a vapor pressure of a second less polar liquid (ethyl ether), in a closed camera, providing the slow formation of crystals.

Refinement

The H-atoms bonded to C-atoms were included in the refinements at geometrically idealized positions with C—H = 0.95, 0.99 and 1.00 Å, for aryl, methylene and methyne H-atoms, respectively, with and U_{iso}(H) = 1.2 times U_{eq}(C). The H-atoms bonded to O atoms were allowed to refine freely. The Flack parameter was *x*=0.00 (16) (Flack, 1983). Further analysis of the absolute structure was performed using likelihood methods (Hooft *et al.*, 2008) with *PLATON* (Spek, 2009). A total of 943 Bijvoet pairs were included in the calculations. The resulting value of the Hooft parameter was *y* = 0.00 (2), with a probability for an inverted structure smaller than $1x10^{-100}$. These results indicated that the absolute structure has been correctly assigned.

Computing details

Data collection: *APEX2* (Bruker, 2010); cell refinement: *SAINT* (Bruker, 2010); data reduction: *SAINT* (Bruker, 2010); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *WinGX* (Farrugia, 1999) and *PLATON* (Spek, 2009); software used to prepare material for publication: *publCIF* (Westrip, 2010) and *PLATON* (Spek, 2009).

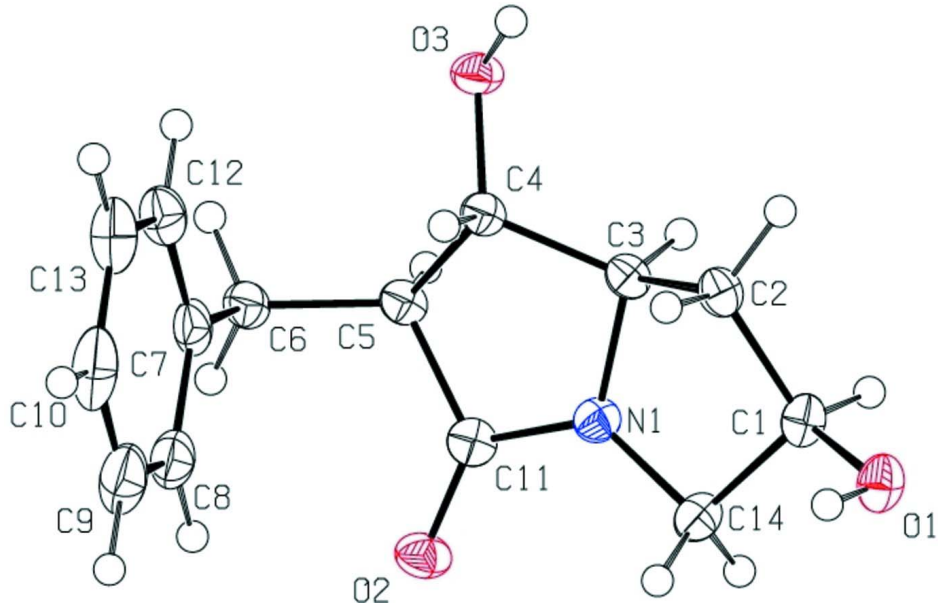

Figure 1

Molecular view of the title compound showing the atom-labeling scheme. Displacement ellipsoids are drawn at the 50% probability level. H atoms are presented as a small spheres of arbitrary radius.

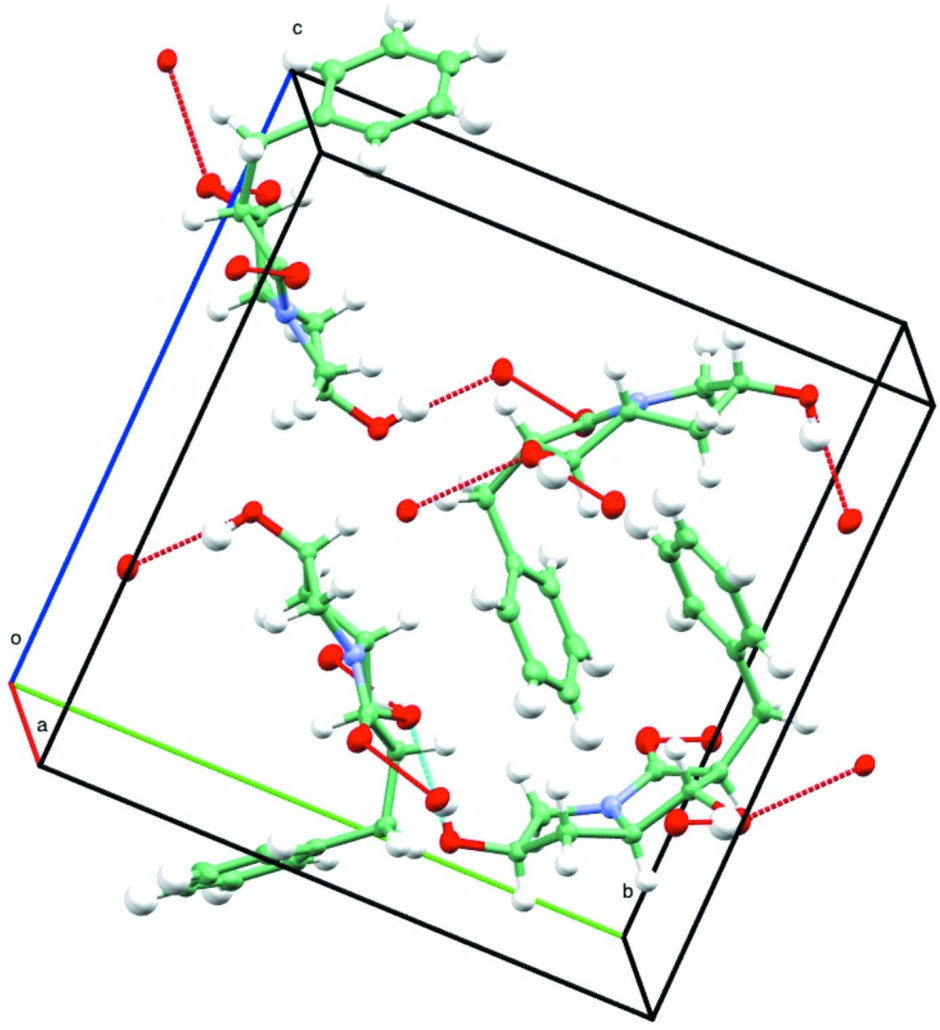

Figure 2

A unit cell packing diagram of the title compound showing hydrogen bonds as dashed lines.

(1*S*,2*S*,6*R*,7a*R*)-2-Benzyl-1,6- dihydroxyhexahydropyrrolizin-3-one

Crystal data

$C_{14}H_{17}NO_3$
$M_r = 247.29$
Orthorhombic, $P2_12_12_1$
$a = 6.6241$ (3) Å
$b = 13.6873$ (6) Å
$c = 13.9726$ (6) Å
$V = 1266.84$ (10) Å^3
$Z = 4$
$F(000) = 528$
$D_x = 1.297$ Mg m^{-3}
Cu $K\alpha$ radiation, $\lambda = 1.54178$ Å
Cell parameters from 2295 reflections
$\theta = 4.5$–$69.5°$
$\mu = 0.74$ mm^{-1}
$T = 100$ K
Rectangular, colourless
0.17 × 0.15 × 0.12 mm

Data collection

Bruker Kappa APEXII DUO diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
Bruker APEX CCD area–detector scans
26923 measured reflections
2295 independent reflections
2290 reflections with $I > 2\sigma(I)$

R_{int} = 0.026
θ_{max} = 69.5°, θ_{min} = 4.5°
h = −7→7
k = −15→16
l = −16→16

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.026
$wR(F^2)$ = 0.078
S = 1.15
2295 reflections
172 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
$w = 1/[\sigma^2(F_o^2) + (0.0491P)^2 + 0.1808P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.17 e Å^{-3}
$\Delta\rho_{min}$ = −0.16 e Å^{-3}
Extinction correction: *SHELXL97* (Sheldrick, 2008), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.0086 (8)
Absolute structure: Flack (1983) and Hooft *et al.* (2008) [Hooft parameter = 0.00(2), (943 Bijvoet pairs)]′
Flack parameter: 0.00 (16)

Special details

Experimental. $[\alpha]_D^{20}$ + 51 (c 1, MeOH); *M*. p. 135–136° C; IR (KBr, ν_{max}): 3404, 3232, 2987, 2936, 2897, 2871, 1670, 1447, 1416, 1375, 1300, 1263, 1222,1175, 1121 cm-1; 1H NMR (500 MHz, CD_3OD) δ 1.55 (dddd, J = 13.4, 5.3, 4.0, 1.0 Hz, 1H, H-2 A); 2.25 (ddd, J = 13.4, 8.0, 5.4 Hz, 1H, H-2B); 2.93 (m, 2H, H-8, H-5); 3.02 (m, J = 7.5, 1.8 Hz, 1H, H-6); 3.08 (ddd, J = 12.0, 4.9, 1.3 Hz, 1H, H-14 A); 3.52 (dd, J = 12.0, 2.4 Hz, 1H, H-14B); 3.64 (m, $J_{H3,H4}$ = 7.0, J = 8.0, 5.3 Hz, 1H, H-3); 3.88 (dd,$J_{H4,H5}$ = 9.4, $J_{H3,H4}$ = 7.0 Hz, 1H, H-4); 4.41 (m, J = 5.1, 4.0, 3.0 Hz, 1H, H-1); 7.15 (m, 1H, H—Ar); 7.23 (m, 2H, H—Ar); 7.29 (m, 2H, H—Ar); ^{13}C NMR (62.5 MHz, $(CD_3)_2CO$) δ 34.4, 38.6, 52.3, 54.0, 65.6, 72.4, 80.6, 126.5, 128.7, 130.3, 141.0, 175.6; HRMS (ESI-TOF) Calcd. for $C_{14}H_{18}NO_3$ $[M + H]^+$ 248.1287. Found 248.1286.

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	0.38278 (15)	0.66691 (6)	0.06882 (7)	0.0253 (2)
O2	−0.07013 (13)	0.91601 (7)	0.27608 (7)	0.0264 (2)
O3	0.61142 (13)	1.03167 (6)	0.28357 (7)	0.0238 (2)
N1	0.21491 (16)	0.88149 (7)	0.19167 (7)	0.0204 (3)
C1	0.3664 (2)	0.76715 (9)	0.09195 (9)	0.0217 (3)
H1	0.3789	0.8046	0.0308	0.026*
C2	0.52450 (19)	0.80862 (9)	0.16169 (9)	0.0216 (3)
H2A	0.5399	0.7667	0.2189	0.026*
H2B	0.6575	0.8168	0.1304	0.026*
C3	0.42952 (18)	0.90724 (9)	0.18696 (9)	0.0193 (3)
H3	0.4529	0.9549	0.1338	0.023*
C4	0.46008 (18)	0.95809 (9)	0.28423 (9)	0.0192 (3)
H4	0.4913	0.9084	0.3346	0.023*

C5	0.25242 (19)	1.00357 (9)	0.30318 (9)	0.0200 (3)
H5	0.2442	1.0650	0.2646	0.024*
C6	0.20002 (19)	1.03016 (9)	0.40713 (9)	0.0235 (3)
H6A	0.2759	1.0898	0.4249	0.028*
H6B	0.0544	1.0461	0.4105	0.028*
C7	0.2453 (2)	0.95143 (9)	0.48012 (9)	0.0256 (3)
C8	0.1026 (3)	0.87967 (10)	0.50128 (10)	0.0335 (3)
H8	−0.0244	0.8801	0.4697	0.040*
C9	0.1451 (3)	0.80737 (11)	0.56844 (10)	0.0428 (4)
H9	0.0466	0.7591	0.5826	0.051*
C10	0.3288 (3)	0.80545 (11)	0.61435 (10)	0.0421 (4)
H10	0.3573	0.7556	0.6597	0.050*
C11	0.10960 (19)	0.92968 (8)	0.25733 (9)	0.0210 (3)
C12	0.4306 (2)	0.94914 (10)	0.52735 (9)	0.0288 (3)
H12	0.5291	0.9976	0.5137	0.035*
C13	0.4730 (3)	0.87670 (11)	0.59435 (10)	0.0372 (4)
H13	0.5996	0.8759	0.6263	0.045*
C14	0.1616 (2)	0.79469 (9)	0.13686 (9)	0.0235 (3)
H14A	0.0595	0.8096	0.0872	0.028*
H14B	0.1101	0.7420	0.1788	0.028*
H3A	0.737 (4)	1.0009 (16)	0.2841 (14)	0.050 (5)*
H1A	0.379 (3)	0.6326 (15)	0.1204 (16)	0.043 (5)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0350 (5)	0.0173 (4)	0.0237 (4)	0.0039 (4)	0.0009 (4)	−0.0011 (3)
O2	0.0173 (4)	0.0252 (4)	0.0368 (5)	0.0013 (4)	0.0018 (4)	−0.0020 (4)
O3	0.0171 (4)	0.0209 (4)	0.0334 (5)	−0.0010 (4)	0.0019 (4)	−0.0044 (4)
N1	0.0183 (5)	0.0200 (5)	0.0229 (5)	0.0017 (4)	−0.0018 (4)	−0.0007 (4)
C1	0.0283 (7)	0.0173 (6)	0.0194 (6)	0.0033 (5)	−0.0009 (5)	0.0005 (5)
C2	0.0212 (6)	0.0210 (6)	0.0226 (6)	0.0038 (5)	0.0022 (5)	−0.0001 (5)
C3	0.0181 (6)	0.0189 (5)	0.0209 (6)	0.0020 (5)	0.0013 (4)	0.0018 (5)
C4	0.0185 (6)	0.0166 (5)	0.0226 (6)	0.0008 (4)	0.0018 (4)	−0.0003 (5)
C5	0.0188 (6)	0.0162 (5)	0.0251 (6)	0.0022 (4)	0.0024 (5)	0.0008 (5)
C6	0.0225 (6)	0.0196 (6)	0.0283 (6)	0.0001 (5)	0.0056 (5)	−0.0049 (5)
C7	0.0351 (7)	0.0200 (6)	0.0217 (6)	0.0004 (5)	0.0089 (5)	−0.0059 (5)
C8	0.0473 (9)	0.0292 (7)	0.0242 (6)	−0.0096 (6)	0.0082 (6)	−0.0073 (5)
C9	0.0763 (13)	0.0275 (7)	0.0245 (7)	−0.0166 (8)	0.0113 (8)	−0.0040 (6)
C10	0.0797 (13)	0.0242 (7)	0.0223 (7)	0.0054 (8)	0.0092 (8)	−0.0007 (5)
C11	0.0203 (6)	0.0182 (5)	0.0246 (6)	0.0039 (5)	−0.0010 (5)	0.0023 (5)
C12	0.0342 (7)	0.0259 (6)	0.0263 (6)	0.0032 (6)	0.0067 (5)	−0.0017 (5)
C13	0.0522 (10)	0.0351 (8)	0.0244 (7)	0.0117 (7)	0.0047 (7)	−0.0030 (6)
C14	0.0238 (6)	0.0227 (6)	0.0240 (6)	0.0009 (5)	−0.0033 (5)	−0.0035 (5)

Geometric parameters (Å, º)

O1—C1	1.4138 (15)	C5—C6	1.5371 (16)
O1—H1A	0.86 (2)	C5—H5	1.0000
O2—C11	1.2333 (16)	C6—C7	1.5138 (18)

O3—C4	1.4210 (14)	C6—H6A	0.9900
O3—H3A	0.93 (2)	C6—H6B	0.9900
N1—C11	1.3279 (17)	C7—C12	1.394 (2)
N1—C14	1.4571 (16)	C7—C8	1.395 (2)
N1—C3	1.4661 (16)	C8—C9	1.392 (2)
C1—C2	1.5390 (18)	C8—H8	0.9500
C1—C14	1.5417 (18)	C9—C10	1.376 (3)
C1—H1	1.0000	C9—H9	0.9500
C2—C3	1.5306 (16)	C10—C13	1.393 (3)
C2—H2A	0.9900	C10—H10	0.9500
C2—H2B	0.9900	C12—C13	1.392 (2)
C3—C4	1.5403 (16)	C12—H12	0.9500
C3—H3	1.0000	C13—H13	0.9500
C4—C5	1.5328 (17)	C14—H14A	0.9900
C4—H4	1.0000	C14—H14B	0.9900
C5—C11	1.5258 (17)		

C1—O1—H1A	109.6 (14)	C6—C5—H5	107.3
C4—O3—H3A	107.9 (14)	C7—C6—C5	115.04 (10)
C11—N1—C14	129.85 (11)	C7—C6—H6A	108.5
C11—N1—C3	114.90 (10)	C5—C6—H6A	108.5
C14—N1—C3	114.06 (10)	C7—C6—H6B	108.5
O1—C1—C2	116.78 (11)	C5—C6—H6B	108.5
O1—C1—C14	113.45 (11)	H6A—C6—H6B	107.5
C2—C1—C14	104.54 (10)	C12—C7—C8	118.72 (13)
O1—C1—H1	107.2	C12—C7—C6	120.64 (12)
C2—C1—H1	107.2	C8—C7—C6	120.64 (14)
C14—C1—H1	107.2	C9—C8—C7	120.42 (16)
C3—C2—C1	101.05 (10)	C9—C8—H8	119.8
C3—C2—H2A	111.6	C7—C8—H8	119.8
C1—C2—H2A	111.6	C10—C9—C8	120.42 (15)
C3—C2—H2B	111.6	C10—C9—H9	119.8
C1—C2—H2B	111.6	C8—C9—H9	119.8
H2A—C2—H2B	109.4	C9—C10—C13	119.97 (15)
N1—C3—C2	101.36 (10)	C9—C10—H10	120.0
N1—C3—C4	101.33 (9)	C13—C10—H10	120.0
C2—C3—C4	123.23 (10)	O2—C11—N1	125.35 (12)
N1—C3—H3	109.9	O2—C11—C5	127.58 (11)
C2—C3—H3	109.9	N1—C11—C5	107.07 (11)
C4—C3—H3	109.9	C13—C12—C7	120.81 (14)
O3—C4—C5	110.27 (9)	C13—C12—H12	119.6
O3—C4—C3	114.03 (10)	C7—C12—H12	119.6
C5—C4—C3	102.59 (10)	C12—C13—C10	119.65 (16)
O3—C4—H4	109.9	C12—C13—H13	120.2
C5—C4—H4	109.9	C10—C13—H13	120.2
C3—C4—H4	109.9	N1—C14—C1	101.53 (10)
C11—C5—C4	102.40 (9)	N1—C14—H14A	111.5
C11—C5—C6	114.43 (10)	C1—C14—H14A	111.5
C4—C5—C6	117.51 (10)	N1—C14—H14B	111.5

C11—C5—H5	107.3	C1—C14—H14B	111.5
C4—C5—H5	107.3	H14A—C14—H14B	109.3

C(11)—N(1)—C(3)—C(2)	145.23 (10)	H(1)—C(1)—C(2)—C(3)	73
C(11)—N(1)—C(3)—C(4)	17.61 (13)	H(1)—C(1)—C(2)—H(2A)	−169
C(14)—N(1)—C(3)—C(2)	−23.51 (13)	H(1)—C(1)—C(2)—H(2B)	−46
C(14)—N(1)—C(3)—C(4)	−151.12 (10)	O(1)—C(1)—C(14)—H(14A)	−86
C(3)—N(1)—C(11)—O(2)	−176.49 (12)	O(1)—C(1)—C(14)—H(14B)	36
C(3)—N(1)—C(11)—C(5)	3.73 (13)	C(2)—C(1)—C(14)—H(14A)	145
C(14)—N(1)—C(11)—O(2)	−9.9 (2)	C(2)—C(1)—C(14)—H(14B)	−92
C(14)—N(1)—C(11)—C(5)	170.29 (11)	H(1)—C(1)—C(14)—N(1)	−87
C(3)—N(1)—C(14)—C(1)	−1.76 (13)	H(1)—C(1)—C(14)—H(14A)	32
C(11)—N(1)—C(14)—C(1)	−168.41 (12)	H(1)—C(1)—C(14)—H(14B)	154
O(1)—C(1)—C(2)—C(3)	−167.02 (10)	C(1)—C(2)—C(3)—H(3)	−78
C(14)—C(1)—C(2)—C(3)	−40.78 (12)	H(2A)—C(2)—C(3)—N(1)	−81
O(1)—C(1)—C(14)—N(1)	154.80 (10)	H(2A)—C(2)—C(3)—C(4)	31
C(2)—C(1)—C(14)—N(1)	26.51 (12)	H(2A)—C(2)—C(3)—H(3)	163
C(1)—C(2)—C(3)—N(1)	38.05 (11)	H(2B)—C(2)—C(3)—N(1)	157
C(1)—C(2)—C(3)—C(4)	149.83 (11)	H(2B)—C(2)—C(3)—C(4)	−91
N(1)—C(3)—C(4)—O(3)	−149.89 (9)	H(2B)—C(2)—C(3)—H(3)	41
N(1)—C(3)—C(4)—C(5)	−30.67 (11)	N(1)—C(3)—C(4)—H(4)	86
C(2)—C(3)—C(4)—O(3)	98.31 (13)	C(2)—C(3)—C(4)—H(4)	−26
C(2)—C(3)—C(4)—C(5)	−142.47 (11)	H(3)—C(3)—C(4)—O(3)	−34
O(3)—C(4)—C(5)—C(6)	−78.74 (13)	H(3)—C(3)—C(4)—C(5)	86
O(3)—C(4)—C(5)—C(11)	154.95 (10)	H(3)—C(3)—C(4)—H(4)	−158
C(3)—C(4)—C(5)—C(6)	159.44 (10)	O(3)—C(4)—C(5)—H(5)	42
C(3)—C(4)—C(5)—C(11)	33.13 (12)	C(3)—C(4)—C(5)—H(5)	−80
C(4)—C(5)—C(6)—C(7)	−47.18 (15)	H(4)—C(4)—C(5)—C(6)	43
C(11)—C(5)—C(6)—C(7)	72.99 (14)	H(4)—C(4)—C(5)—C(11)	−84
C(4)—C(5)—C(11)—O(2)	156.61 (12)	H(4)—C(4)—C(5)—H(5)	163
C(4)—C(5)—C(11)—N(1)	−23.61 (12)	C(4)—C(5)—C(6)—H(6A)	75
C(6)—C(5)—C(11)—O(2)	28.33 (18)	C(4)—C(5)—C(6)—H(6B)	−169
C(6)—C(5)—C(11)—N(1)	−151.89 (10)	C(11)—C(5)—C(6)—H(6A)	−165
C(5)—C(6)—C(7)—C(8)	−87.85 (15)	C(11)—C(5)—C(6)—H(6B)	−49
C(5)—C(6)—C(7)—C(12)	92.02 (14)	H(5)—C(5)—C(6)—C(7)	−168
C(6)—C(7)—C(8)—C(9)	179.96 (13)	H(5)—C(5)—C(6)—H(6A)	−46
C(12)—C(7)—C(8)—C(9)	0.1 (2)	H(5)—C(5)—C(6)—H(6B)	70
C(6)—C(7)—C(12)—C(13)	−179.84 (12)	H(5)—C(5)—C(11)—O(2)	−91
C(8)—C(7)—C(12)—C(13)	0.0 (2)	H(5)—C(5)—C(11)—N(1)	89
C(7)—C(8)—C(9)—C(10)	−0.4 (2)	H(6A)—C(6)—C(7)—C(8)	150
C(8)—C(9)—C(10)—C(13)	0.6 (2)	H(6A)—C(6)—C(7)—C(12)	−30
C(9)—C(10)—C(13)—C(12)	−0.5 (2)	H(6B)—C(6)—C(7)—C(8)	34
C(7)—C(12)—C(13)—C(10)	0.2 (2)	H(6B)—C(6)—C(7)—C(12)	−146
H(1A)—O(1)—C(1)—C(2)	56.8 (14)	C(6)—C(7)—C(8)—H(8)	0
H(1A)—O(1)—C(1)—C(14)	−64.9 (14)	C(12)—C(7)—C(8)—H(8)	−180
H(1A)—O(1)—C(1)—H(1)	177	C(6)—C(7)—C(12)—H(12)	0
H(3A)—O(3)—C(4)—C(3)	−76.3 (13)	C(8)—C(7)—C(12)—H(12)	−180
H(3A)—O(3)—C(4)—C(5)	168.9 (13)	C(7)—C(8)—C(9)—H(9)	180
H(3A)—O(3)—C(4)—H(4)	48	H(8)—C(8)—C(9)—C(10)	180

C(11)—N(1)—C(3)—H(3)	−99	H(8)—C(8)—C(9)—H(9)	0
C(14)—N(1)—C(3)—H(3)	93	C(8)—C(9)—C(10)—H(10)	−179
C(3)—N(1)—C(14)—H(14A)	−121	H(9)—C(9)—C(10)—C(13)	−179
C(3)—N(1)—C(14)—H(14B)	117	H(9)—C(9)—C(10)—H(10)	1
C(11)—N(1)—C(14)—H(14A)	73	C(9)—C(10)—C(13)—H(13)	179
C(11)—N(1)—C(14)—H(14B)	−50	H(10)—C(10)—C(13)—C(12)	180
O(1)—C(1)—C(2)—H(2A)	−48	H(10)—C(10)—C(13)—H(13)	0
O(1)—C(1)—C(2)—H(2B)	74	C(7)—C(12)—C(13)—H(13)	−180
C(14)—C(1)—C(2)—H(2A)	78	H(12)—C(12)—C(13)—C(10)	−180
C(14)—C(1)—C(2)—H(2B)	−159	H(12)—C(12)—C(13)—H(13)	0

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O3—H3*A*···O2^{i}	0.93 (2)	1.73 (2)	2.6395 (12)	164 (2)
O1—H1*A*···O3^{ii}	0.86 (2)	1.93 (2)	2.7716 (13)	167 (19)

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