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(3*S*,4*S*)-1-Benzhydryl-4-(cyclopropylcarbonyl)-3-[(1*R*)-1-hydroxyethyl]azetidin-2-one

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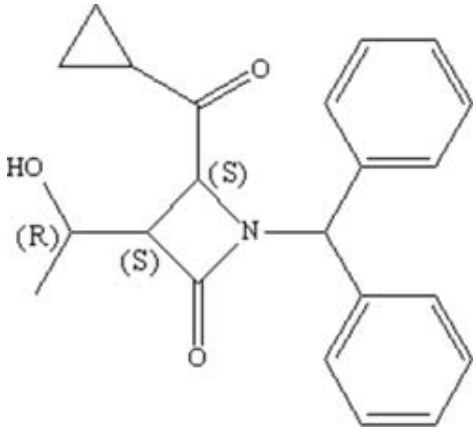
Key indicators: single-crystal X-ray study; T = 153 K; mean σ(C–C) = 0.007 Å; R factor = 0.046; wR factor = 0.157; data-to-parameter ratio = 8.5.

The title compound, $C_{22}H_{23}NO_3$, was obtained by the reaction of (2*R*,3*R*)-*N*-benzhydryl-*N*-(2-cyclopropyl-2-oxoethyl)-2,3-epoxybutyramide with Lithium bis(trimethylsilyl)amide in tetrahydrofuran solution. The absolute configuration has been assigned by reference to an unchanging chiral centre in the synthetic procedure. The molecular packing is stabilized by intermolecular O—H···O and C—H···O hydrogen bonds.

Related literature

For general background, see: Walsh (2000). For related structures see: Tinant *et al.* (2003); Wang *et al.* (2006). For related literature, see: Laurent *et al.* (2004).



Experimental

Crystal data

$C_{22}H_{23}NO_3$
M_r = 349.41
Monoclinic, $C2$
a = 18.8279 (13) Å
b = 6.1645 (4) Å
c = 16.3539 (10) Å
β = 99.529 (6)°
V = 1871.9 (2) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.08 mm^{-1}
T = 153 (2) K
0.42 × 0.40 × 0.35 mm

Data collection

Bruker APEX area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2002)
T_{min} = 0.966, T_{max} = 0.972
6131 measured reflections
2007 independent reflections
1312 reflections with $I > 2\sigma(I)$
R_{int} = 0.034

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.046
$wR(F^2)$ = 0.157
S = 1.07
2007 reflections
237 parameters
1 restraint
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.17 e Å^{-3}
$\Delta\rho_{min}$ = −0.19 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O3—H3A···O1^{i}	0.82	2.04	2.783 (4)	150
C18—H18···O2^{ii}	0.93	2.29	3.199 (6)	165

Symmetry codes: (i) $-x+\frac{3}{2}, y+\frac{1}{2}, -z$; (ii) $x, y+1, z$.

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT* (Bruker, 2001); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *SHELXL97*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: DN2179).

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supplementary materials

Acta Cryst. (2007). E**63**, o3220 [**doi:10.1107/S1600536807028139**]

(3*S*,4*S*)-1-Benzhydryl-4-(cyclopropylcarbonyl)-3-[(1*R*)-1-hydroxyethyl]azetidin-2-one

X.-F. Chen, L.-J. Wang, Y.-X. Gao, L.-R. Jin and J.-F. Zheng

Comment

In the course of the synthesis of acetoxyazetidinone, which is used as a key structure for the preparation of carbapenems, which are a growing class of β-lactam antibiotics (Walsh, 2000), the title compound, (I), was prepared and obtained as single crystals suitable for X-ray structural analysis.

The molecular structure of (I) is shown in Fig. 1. The correct enantiomer has been assigned by reference to an unchanging chiral center (C8) in the synthetic procedure (Tinant *et al.*, 2003). Bond lengths and angles in (I) are in agreement with values reported for a similar compound (Wang *et al.*, 2006). The dihedral angle between the C11-phenyl and C17-phenyl planes is 74.8 (2)°. Molecules are linked into helicoidal chains running along the *b* axis, through O—H···O hydrogen bonds. The chains are further connected through weak C—H···O intermolecular hydrogen bonds to build up a three dimensionnal network (table 1).

Experimental

To a precooled solution of anhydrous HMDS (0.387 g, 2.4 mmol) in anhydrous THF (3.5 ml) at 0°C, was added dropwise a 2.5 *M* solution of n-BuLi (0.96 ml, 2.4 mmol) in hexane. After stired 0.5 h, the fresh prepared LiHMDS-THF solution was dropwised at 0°C under argon to a solution of (2*R*,3*R*)-*N*-Benzhydryl-*N*-(2-cyclopropyl-2-oxoethyl)-2,3-epoxybutyramide (0.699 g, 2 mmol) in dry THF (20 ml). The cooling bath was removed and the mixture warmed to 20°C for 3 h. The reaction was quenched with a 1 *M* solution of HCl (2 ml). After dilution with ethyl acetate (10 ml), the solution was washed with $NaHCO_3$ solution (5%, 3×10 ml), brine (2×10 ml), dried over anhydrous $MgSO_4$, concentrated under vacuum and the crude product was purified by column chromatography (petroleum ether/ethyl acetate, 10:1 *v*/*v*) to give the titled compound as a white solid with 80% yield (Laurent *et al.*, 2004). Single crystals of (I) were developed in ethyl acetate solution by slow evaporation.

Refinement

All H atoms attached to C atoms and O atom were fixed geometrically and treated as riding with C—H = 0.93 Å ($C_{aromatic}$), 0.97 Å ($C_{methylene}$), 0.96Å (C_{methyl}), 0.98Å ($C_{methine}$) and O—H =0.82 Å with $U_{iso}(H) = 1.2U_{eq}(C_{aromatic}, C_{methylene}, C_{methine}$ and O) and with $U_{iso}(H) = 1.5U_{eq}(C_{methyl})$.

In the absence of significant anomalous scattering, the absolute configuration could not be reliably determined by X-ray analyses and then the Friedel pairs were merged and any references to the Flack parameter were removed.

Figures

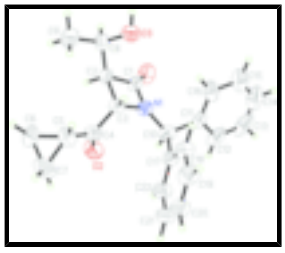


Fig. 1. The molecular structure of (I) showing the atom-numbering scheme. Ellipsoids are drawn at the 30% probability level. H atoms are represented as small spheres of arbitrary radii.

(3*S*,4*S*)-1-Benzhydryl-4-(cyclopropylcarbonyl)- 3-[(1*R*)-1-hydroxyethyl]azetidin-2-one

Crystal data

$C_{22}H_{23}NO_3$	$Z = 4$
$M_r = 349.41$	$F_{000} = 744$
Monoclinic, *C*2	$D_x = 1.240$ Mg m^{-3}
Hall symbol: C 2y	Mo *K*α radiation λ = 0.71073 Å
$a = 18.8279$ (13) Å	μ = 0.08 mm^{-1}
$b = 6.1645$ (4) Å	$T = 153$ (2) K
$c = 16.3539$ (10) Å	Chunk, colourless
β = 99.529 (6)º	0.42 × 0.40 × 0.35 mm
$V = 1871.9$ (2) Å^3	

Data collection

Bruker APEX area-detector diffractometer	2007 independent reflections
Radiation source: fine-focus sealed tube	1312 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{int} = 0.034$
$T = 153$(2) K	$\theta_{max} = 26.0$º
φ and ω scans	$\theta_{min} = 2.3$º
Absorption correction: multi-scan (SADABS; Sheldrick, 2002)	$h = -23 \rightarrow 19$
$T_{min} = 0.966$, $T_{max} = 0.972$	$k = -7 \rightarrow 7$
6131 measured reflections	$l = -20 \rightarrow 20$

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)] = 0.046$	$w = 1/[\sigma^2(F_o^2) + (0.0938P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.157$	$(\Delta/\sigma)_{max} = 0.001$
$S = 1.07$	$\Delta\rho_{max} = 0.17$ e Å^{-3}

2007 reflections
237 parameters
1 restraint
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map

$\Delta\rho_{min} = -0.19$ e Å^{-3}
Extinction correction: none

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 $F^2 > 2\text{sigma}(F^2)$ 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}
C1	0.7696 (2)	0.1736 (7)	0.1309 (2)	0.0406 (10)
C2	0.6968 (2)	0.1326 (7)	0.0804 (2)	0.0410 (10)
H2	0.6929	−0.0194	0.0626	0.049*
C3	0.6685 (2)	0.1585 (7)	0.1658 (2)	0.0395 (10)
H3	0.6373	0.2849	0.1676	0.047*
C4	0.6360 (2)	−0.0499 (8)	0.1902 (3)	0.0467 (11)
C5	0.5585 (2)	−0.0728 (8)	0.1700 (3)	0.0520 (12)
H5	0.5304	0.0612	0.1600	0.062*
C6	0.5307 (2)	−0.2690 (9)	0.1179 (3)	0.0591 (13)
H6A	0.4877	−0.2505	0.0767	0.071*
H6B	0.5659	−0.3690	0.1024	0.071*
C7	0.5227 (3)	−0.2602 (10)	0.2050 (4)	0.0776 (17)
H7A	0.5527	−0.3558	0.2432	0.093*
H7B	0.4746	−0.2374	0.2175	0.093*
C8	0.6701 (2)	0.2772 (7)	0.0089 (3)	0.0434 (10)
H8	0.7022	0.2608	−0.0322	0.052*
C9	0.5949 (2)	0.2184 (11)	−0.0319 (3)	0.0699 (15)
H9A	0.5633	0.2224	0.0085	0.105*
H9B	0.5949	0.0750	−0.0549	0.105*
H9C	0.5786	0.3202	−0.0754	0.105*
C10	0.7803 (2)	0.1968 (8)	0.2885 (2)	0.0463 (10)
H10	0.8022	0.0532	0.2991	0.056*
C11	0.8416 (2)	0.3641 (8)	0.3034 (2)	0.0436 (11)
C12	0.9019 (2)	0.3201 (12)	0.3639 (3)	0.0648 (15)
H12	0.9053	0.1918	0.3943	0.078*
C13	0.9561 (3)	0.4749 (16)	0.3768 (3)	0.084 (2)
H13	0.9957	0.4504	0.4180	0.101*

C14	0.9539 (3)	0.6563 (14)	0.3328 (4)	0.082 (2)
H14	0.9922	0.7533	0.3423	0.099*
C15	0.8956 (3)	0.7012 (10)	0.2738 (4)	0.0690 (15)
H15	0.8941	0.8296	0.2437	0.083*
C16	0.8382 (3)	0.5553 (8)	0.2583 (3)	0.0571 (13)
H16	0.7983	0.5859	0.2184	0.069*
C17	0.7270 (2)	0.2264 (8)	0.3487 (2)	0.0449 (11)
C18	0.6888 (3)	0.4147 (8)	0.3508 (3)	0.0513 (12)
H18	0.6943	0.5261	0.3140	0.062*
C19	0.6418 (3)	0.4411 (11)	0.4073 (3)	0.0654 (15)
H19	0.6155	0.5685	0.4080	0.079*
C20	0.6348 (3)	0.2774 (13)	0.4620 (3)	0.078 (2)
H20	0.6033	0.2938	0.4998	0.094*
C21	0.6738 (3)	0.0894 (11)	0.4616 (3)	0.0720 (16)
H21	0.6694	−0.0199	0.4996	0.086*
C22	0.7194 (3)	0.0628 (9)	0.4050 (3)	0.0631 (14)
H22	0.7453	−0.0654	0.4044	0.076*
N1	0.74373 (17)	0.1984 (6)	0.20291 (19)	0.0427 (8)
O1	0.83222 (16)	0.1818 (6)	0.11784 (18)	0.0582 (9)
O2	0.67671 (18)	−0.1934 (6)	0.2210 (2)	0.0668 (10)
O3	0.67549 (19)	0.4940 (5)	0.03812 (18)	0.0557 (9)
H3A	0.6622	0.5771	−0.0006	0.084*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.036 (2)	0.050 (3)	0.0348 (18)	0.0004 (19)	0.0028 (16)	−0.004 (2)
C2	0.039 (2)	0.048 (3)	0.036 (2)	−0.0072 (19)	0.0073 (17)	−0.0043 (19)
C3	0.035 (2)	0.045 (3)	0.040 (2)	0.0032 (18)	0.0087 (16)	0.0036 (19)
C4	0.047 (2)	0.052 (3)	0.044 (2)	0.003 (2)	0.0146 (19)	0.006 (2)
C5	0.041 (2)	0.049 (3)	0.069 (3)	0.004 (2)	0.016 (2)	−0.004 (2)
C6	0.040 (2)	0.073 (4)	0.059 (3)	0.000 (2)	−0.006 (2)	−0.012 (3)
C7	0.072 (4)	0.085 (4)	0.087 (4)	−0.020 (3)	0.043 (3)	0.000 (4)
C8	0.040 (2)	0.048 (3)	0.044 (2)	−0.0073 (19)	0.0147 (18)	−0.010 (2)
C9	0.050 (3)	0.088 (4)	0.067 (3)	−0.010 (3)	−0.006 (2)	0.010 (3)
C10	0.049 (2)	0.049 (3)	0.041 (2)	0.011 (2)	0.0063 (17)	0.000 (2)
C11	0.035 (2)	0.060 (3)	0.034 (2)	0.003 (2)	0.0004 (16)	−0.013 (2)
C12	0.044 (3)	0.109 (4)	0.039 (2)	0.004 (3)	0.0000 (19)	−0.011 (3)
C13	0.045 (3)	0.153 (7)	0.051 (3)	−0.007 (4)	−0.003 (2)	−0.033 (4)
C14	0.060 (4)	0.107 (5)	0.083 (4)	−0.023 (4)	0.023 (3)	−0.042 (4)
C15	0.060 (3)	0.063 (3)	0.088 (3)	−0.010 (3)	0.020 (3)	−0.027 (3)
C16	0.042 (3)	0.057 (3)	0.074 (3)	−0.006 (2)	0.014 (2)	−0.017 (3)
C17	0.045 (2)	0.051 (3)	0.038 (2)	−0.001 (2)	0.0043 (16)	0.004 (2)
C18	0.054 (3)	0.058 (3)	0.043 (2)	0.008 (2)	0.011 (2)	0.008 (2)
C19	0.053 (3)	0.090 (4)	0.057 (3)	0.002 (3)	0.018 (2)	−0.004 (3)
C20	0.074 (4)	0.117 (6)	0.049 (3)	−0.033 (4)	0.024 (3)	−0.006 (3)
C21	0.090 (4)	0.079 (4)	0.047 (3)	−0.027 (4)	0.010 (3)	0.018 (3)
C22	0.074 (3)	0.059 (3)	0.055 (3)	−0.004 (3)	0.005 (3)	0.021 (3)

N1	0.0403 (19)	0.050 (2)	0.0386 (16)	−0.0009 (17)	0.0100 (14)	−0.0008 (18)
O1	0.0389 (17)	0.078 (2)	0.0610 (17)	−0.0016 (16)	0.0181 (13)	−0.0173 (19)
O2	0.052 (2)	0.060 (2)	0.080 (2)	0.0008 (18)	−0.0124 (16)	0.024 (2)
O3	0.073 (2)	0.049 (2)	0.0468 (17)	−0.0001 (17)	0.0157 (16)	0.0040 (16)

Geometric parameters (Å, °)

C1—O1	1.234 (5)	C10—C17	1.530 (6)
C1—N1	1.355 (5)	C10—C11	1.536 (6)
C1—C2	1.500 (6)	C10—H10	0.9800
C2—C8	1.490 (6)	C11—C16	1.386 (7)
C2—C3	1.582 (5)	C11—C12	1.403 (6)
C2—H2	0.9800	C12—C13	1.388 (9)
C3—N1	1.466 (5)	C12—H12	0.9300
C3—C4	1.506 (6)	C13—C14	1.327 (11)
C3—H3	0.9800	C13—H13	0.9300
C4—O2	1.223 (6)	C14—C15	1.365 (9)
C4—C5	1.449 (6)	C14—H14	0.9300
C5—C7	1.497 (7)	C15—C16	1.397 (7)
C5—C6	1.521 (7)	C15—H15	0.9300
C5—H5	0.9800	C16—H16	0.9300
C6—C7	1.457 (7)	C17—C18	1.369 (7)
C6—H6A	0.9700	C17—C22	1.389 (6)
C6—H6B	0.9700	C18—C19	1.391 (6)
C7—H7A	0.9700	C18—H18	0.9300
C7—H7B	0.9700	C19—C20	1.369 (9)
C8—O3	1.417 (6)	C19—H19	0.9300
C8—C9	1.507 (6)	C20—C21	1.373 (10)
C8—H8	0.9800	C20—H20	0.9300
C9—H9A	0.9600	C21—C22	1.371 (8)
C9—H9B	0.9600	C21—H21	0.9300
C9—H9C	0.9600	C22—H22	0.9300
C10—N1	1.454 (5)	O3—H3A	0.8200
O1—C1—N1	129.6 (4)	H9B—C9—H9C	109.5
O1—C1—C2	136.4 (3)	N1—C10—C17	111.2 (3)
N1—C1—C2	94.0 (3)	N1—C10—C11	111.8 (3)
C8—C2—C1	119.3 (3)	C17—C10—C11	111.9 (3)
C8—C2—C3	120.9 (3)	N1—C10—H10	107.2
C1—C2—C3	84.7 (3)	C17—C10—H10	107.2
C8—C2—H2	109.9	C11—C10—H10	107.2
C1—C2—H2	109.9	C16—C11—C12	120.0 (5)
C3—C2—H2	109.9	C16—C11—C10	121.3 (4)
N1—C3—C4	116.2 (3)	C12—C11—C10	118.7 (5)
N1—C3—C2	86.5 (3)	C13—C12—C11	117.6 (6)
C4—C3—C2	111.0 (3)	C13—C12—H12	121.2
N1—C3—H3	113.4	C11—C12—H12	121.2
C4—C3—H3	113.4	C14—C13—C12	122.7 (6)
C2—C3—H3	113.4	C14—C13—H13	118.6
O2—C4—C5	124.2 (4)	C12—C13—H13	118.6

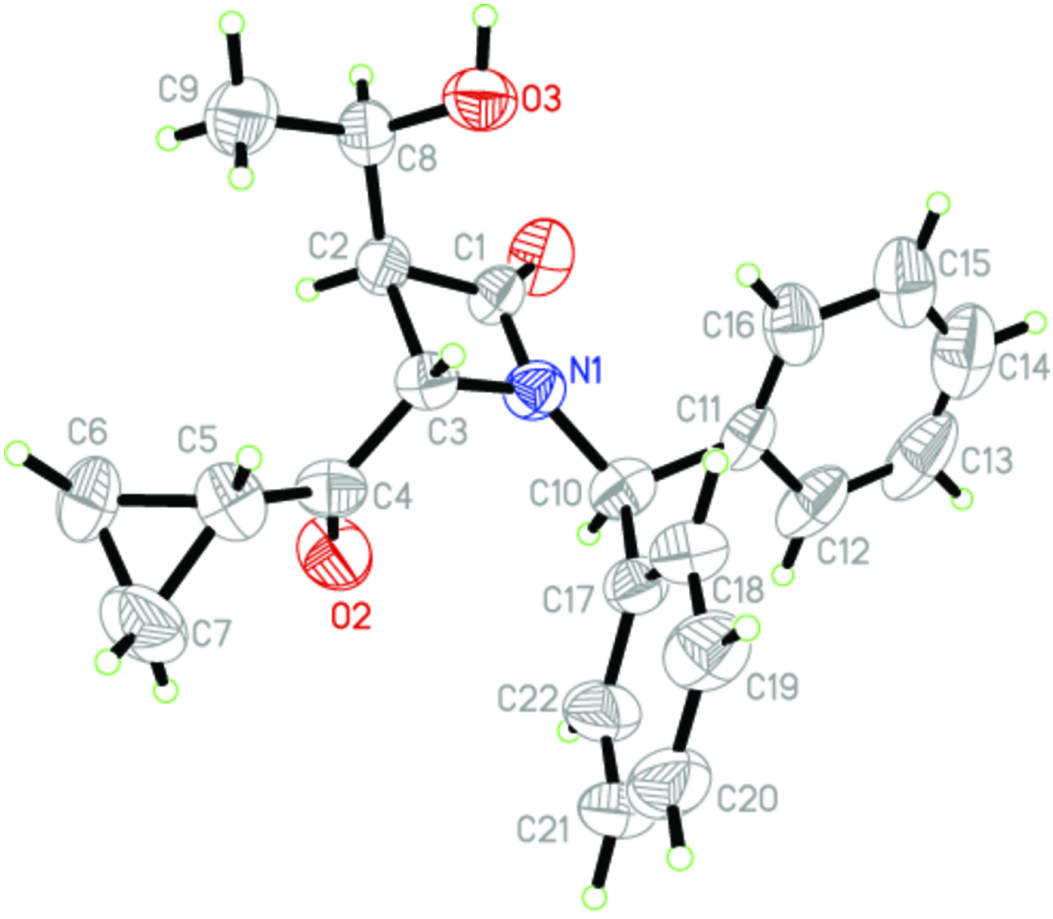
O2—C4—C3	118.1 (4)	C13—C14—C15	120.3 (6)
C5—C4—C3	117.5 (4)	C13—C14—H14	119.9
C4—C5—C7	119.2 (5)	C15—C14—H14	119.9
C4—C5—C6	116.4 (4)	C14—C15—C16	120.3 (6)
C7—C5—C6	57.7 (4)	C14—C15—H15	119.8
C4—C5—H5	116.8	C16—C15—H15	119.8
C7—C5—H5	116.8	C11—C16—C15	119.0 (5)
C6—C5—H5	116.8	C11—C16—H16	120.5
C7—C6—C5	60.3 (3)	C15—C16—H16	120.5
C7—C6—H6A	117.7	C18—C17—C22	119.2 (4)
C5—C6—H6A	117.7	C18—C17—C10	121.4 (4)
C7—C6—H6B	117.7	C22—C17—C10	119.4 (4)
C5—C6—H6B	117.7	C17—C18—C19	120.6 (5)
H6A—C6—H6B	114.9	C17—C18—H18	119.7
C6—C7—C5	62.0 (3)	C19—C18—H18	119.7
C6—C7—H7A	117.6	C20—C19—C18	119.3 (6)
C5—C7—H7A	117.6	C20—C19—H19	120.3
C6—C7—H7B	117.6	C18—C19—H19	120.3
C5—C7—H7B	117.6	C19—C20—C21	120.6 (5)
H7A—C7—H7B	114.7	C19—C20—H20	119.7
O3—C8—C2	107.7 (3)	C21—C20—H20	119.7
O3—C8—C9	112.7 (4)	C22—C21—C20	120.0 (5)
C2—C8—C9	111.9 (4)	C22—C21—H21	120.0
O3—C8—H8	108.1	C20—C21—H21	120.0
C2—C8—H8	108.1	C21—C22—C17	120.3 (5)
C9—C8—H8	108.1	C21—C22—H22	119.8
C8—C9—H9A	109.5	C17—C22—H22	119.8
C8—C9—H9B	109.5	C1—N1—C10	130.9 (3)
H9A—C9—H9B	109.5	C1—N1—C3	94.7 (3)
C8—C9—H9C	109.5	C10—N1—C3	132.0 (3)
H9A—C9—H9C	109.5	C8—O3—H3A	109.5

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O3—H3A···O1^{i}	0.82	2.04	2.783 (4)	150
C18—H18···O2^{ii}	0.93	2.29	3.199 (6)	165

Symmetry codes: (i) −*x*+3/2, *y*+1/2, −*z*; (ii) *x*, *y*+1, *z*.

Fig. 1


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