Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

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

Hao Zhang,[a] Fen-Er Chen[a]* and Min-Qin Chen[b]

[a]Department of Chemistry, Fudan Uniersity, Shanghai 200433, People's Republic of China, and [b]Research Center of Analysis and Measurement, Fudan Uniersity, Shanghai 200433, People's Republic of China
Correspondence e-mail: rfchen@fudan.edu.cn

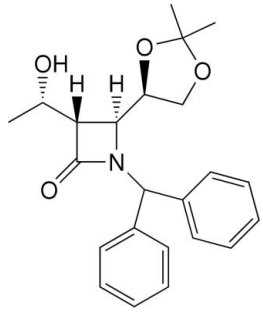
Received 29 May 2007; accepted 1 June 2007

Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.005 Å; R factor = 0.048; wR factor = 0.123; data-to-parameter ratio = 9.5.

Slow diffusion of hexane into an ethyl acetate solution of the title compound, $C_{23}H_{27}NO_4$, gave X-ray quality crystals. In the crystal structure, intermolecular O—H···O hydrogen bonds link molecules into one-dimensional chains. In addition, weak C—H···O hydrogen bonds help stabilize the crystal structure.

Related literature

For background information, see: Kawabata *et al.* (1988). For the synthetic procedure, see: Sarko *et al.* (1996).



Experimental

Crystal data

$C_{23}H_{27}NO_4$
M_r = 381.46
Monoclinic, $P2_1$
a = 10.720 (4) Å
b = 9.192 (4) Å
c = 10.819 (4) Å
β = 90.890 (5)°
V = 1065.9 (7) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.08 mm^{-1}
T = 293 (2) K
0.15 × 0.15 × 0.12 mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.988, T_{max} = 0.990
5246 measured reflections
2466 independent reflections
1973 reflections with $I > 2\sigma(I)$
R_{int} = 0.026

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.048
$wR(F^2)$ = 0.123
S = 1.02
2466 reflections
260 parameters
2 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.19 e Å^{-3}
$\Delta\rho_{min}$ = −0.13 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
C12—H12···O4	0.98	2.42	3.141 (3)	130
O2—H2*X*···O1^{i}	0.806 (19)	1.99 (2)	2.787 (4)	170 (5)
C7—H7···O3^{ii}	0.98	2.45	3.357 (4)	153

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

Data collection: *SMART* (Bruker, 2000); cell refinement: *SAINT* (Bruker, 2000); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 2000); software used to prepare material for publication: *SHELXTL*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: LH2404).

References

Bruker (2000). *SMART*, *SAINT* and *SHELXTL*. Bruker AXS Inc., Madison, Wisconsin, USA.
Kawabata, T., Kimura, Y., Ito, Y. & Terashima, S. (1988). *Tetrahedron*, **44**, 2149–2165.
Sarko, C. R., Collibee, S. E., Knorr, A. L. & DiMare, M. (1996). *J. Org. Chem.* **61**, 868–873.
Sheldrick, G. M. (1996). *SADABS*. University of Göttingen, Germany.
Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.

supplementary materials

Acta Cryst. (2007). E**63**, o3099 [**doi:10.1107/S160053680702692X**]

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

H. Zhang, F.-E. Chen and M.-Q. Chen

Comment

During our study on the synthesis of carbapenems, a class of antibiotics with pronounced broad-spectrum antibacterial activity, (Kawabata *et al.*, 1988), the title compound was produced and is a key intermediate in our newly designed synthetic route to carbapenems. The title compound was synthesized through the reduction of (3*S*,4*S*)-3-acetyl-1-benzhydryl-4-((*R*)-2,2-dimethyl- 1,3-dioxolan-4-yl)-2-azetidinone from *L*-ascorbic acid. The absolute configuration was difficult to determined by other analytical methods due to the free rotation of C3—C5 single bond. Here we report the crystal structure of the title compound.

Fig. 1 shows the molecular structrue of the title compound. The enantiomer was selected on the basis of the configuration of the starting material. All chiral carbon atoms are *S*-configuration except C7. The 4-membered ring is almost a planar with a dihedral of 1.2 (2)° for N1—C2—C3—C4, In the 5-membered ring, the dihedral of C9—O4—C7—C8 is 3.4 (4)°, and the dihedral angle of C9/O3/C8/C7 is −18.6 (5)°. The C9—C10 and C9—C11 bond lengths are shorter than the typical Csp^3—Csp^3 bond distance, possibly as a result of marginal disorder in this area.

Experimental

The literature procedure according to Sarko *et al.* (1996) was followed. To a cold (195k) solution of (3*S*,4*S*)-3-acetyl-1-benzhydryl −4-((*R*)-2,2-dimethyl-1,3-dioxolan-4-yl)-2-azetidinone (1.0 mmol) in 10 mL of CH_2Cl_2, was added $TiCl_4$ (110µl, 1.0 mmol) with vigrous stirring under Argon atomsphere. After 10 min, pyr·BH_3 (140µl, 1.0 mmol) was slowly added. The reaction was quenched with 1 N HCl (2 ml) after 15 min, and the reaction mixture was warmed to r.t., the organic layer was separated and washed with brine, dried over anhyd. $MgSO_4$, and concentrated *in vacuo*, Chromatography gave the title compound 28 mg (73%), m.p. 415 K. Full spectroscopic and physical characterization will be reported elsewhere.

Refinement

In the absence of significant anomalous dispersion effects Fridel pairs were merged. All H atoms were placed in geometrically idealized position and constrained to ride on their parent atoms, with C—H distances in the range 0.93–0.98 Å and with U_{iso}(H) = 1.5U_{eq}(C) for methyl H atoms and 1.2U_{eq}(C) for other H atoms. Methyl groups were allowed to rotate freely about the C—C bond. The H atom bonded to O was isotropically refined with a distance restraint of O—H = 0.80 (6) Å. Atoms C10/C11/O3/O4 have larger than normal anistropic displacement parameters and this may be due to marginal disorder in this part of the molecule. This was not modelled.

Figures

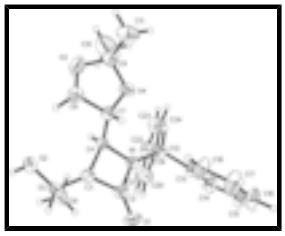


Fig. 1. The molecular structure drawing for (I) showing 30% probability of displacement ellipsoids and the atom-numbering scheme.

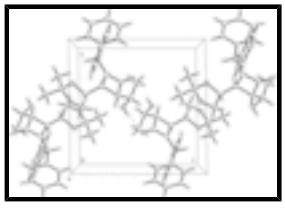


Fig. 2. The packing of the title compound, viewed along the *b* axis. Hydrogen bonds shown as dashed lines.

(3*S*,4*S*)-1-Benzhydryl-4-[(*R*)-2,2-dimethyl-1,3- dioxolan-4-yl]-3-[(*S*)-1-hydroxyethyl]-2-azetidinone

Crystal data

$C_{23}H_{27}NO_4$	F_{000} = 408
M_r = 381.46	D_x = 1.188 Mg m^{-3}
Monoclinic, $P2_1$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: P 2yb	Cell parameters from 769 reflections
a = 10.720 (4) Å	θ = 2.7–23.4º
b = 9.192 (4) Å	μ = 0.08 mm^{-1}
c = 10.819 (4) Å	*T* = 293 (2) K
β = 90.890 (5)º	Block, colorless
V = 1065.9 (7) Å^3	0.15 × 0.15 × 0.12 mm
Z = 2	

Data collection

Bruker SMART APEX CCD area-detector diffractometer	2466 independent reflections
Radiation source: fine-focus sealed tube	1973 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.026
T = 293(2) K	θ_{max} = 27.1º
φ and ω scans	θ_{min} = 1.9º
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	*h* = −11→13
T_{min} = 0.988, T_{max} = 0.990	*k* = −8→11
5246 measured reflections	*l* = −13→13

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H atoms treated by a mixture of independent and constrained refinement
$R[F^2 > 2\sigma(F^2)] = 0.048$	$w = 1/[\sigma^2(F_o^2) + (0.0578P)^2 + 0.1569P]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.123$	$(\Delta/\sigma)_{max} < 0.001$
$S = 1.02$	$\Delta\rho_{max} = 0.19$ e Å^{-3}
2466 reflections	$\Delta\rho_{min} = -0.13$ e Å^{-3}
260 parameters	Extinction correction: none
2 restraints	
Primary atom site location: structure-invariant direct methods	
Secondary atom site location: difference Fourier map	

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 $F^2 > 2$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	0.2959 (2)	0.5939 (3)	0.5357 (2)	0.0796 (8)
O2	0.5052 (3)	0.1621 (3)	0.6167 (2)	0.0893 (9)
H2X	0.566 (3)	0.135 (6)	0.580 (4)	0.111 (17)*
O3	0.4631 (3)	0.2094 (4)	1.0163 (3)	0.1206 (14)
O4	0.3240 (2)	0.3847 (4)	0.98493 (18)	0.0871 (9)
N1	0.25812 (18)	0.4805 (3)	0.72615 (19)	0.0455 (5)
C2	0.3178 (2)	0.5096 (3)	0.6201 (3)	0.0535 (7)
C3	0.4185 (2)	0.3955 (3)	0.6467 (2)	0.0508 (7)
H3	0.4985	0.4420	0.6668	0.061*
C4	0.3440 (2)	0.3646 (3)	0.7658 (2)	0.0425 (5)
H4	0.3043	0.2685	0.7643	0.051*
C5	0.4357 (3)	0.2721 (4)	0.5551 (3)	0.0673 (9)
H5	0.4845	0.3082	0.4857	0.081*
C6	0.3138 (4)	0.2112 (6)	0.5050 (4)	0.0938 (14)
H6A	0.3305	0.1295	0.4525	0.141*

H6B	0.2707	0.2849	0.4583	0.141*
H6C	0.2629	0.1805	0.5725	0.141*
C7	0.4106 (3)	0.3944 (4)	0.8868 (2)	0.0559 (7)
H7	0.4479	0.4917	0.8853	0.067*
C8	0.5090 (3)	0.2823 (5)	0.9209 (3)	0.0751 (11)
H8A	0.5238	0.2174	0.8520	0.090*
H8B	0.5869	0.3295	0.9440	0.090*
C9	0.3664 (3)	0.2851 (4)	1.0748 (3)	0.0676 (8)
C10	0.2653 (6)	0.1807 (9)	1.1042 (6)	0.163 (3)
H10A	0.1959	0.2325	1.1380	0.244*
H10B	0.2957	0.1110	1.1635	0.244*
H10C	0.2389	0.1312	1.0302	0.244*
C11	0.4124 (6)	0.3664 (8)	1.1846 (4)	0.127 (2)
H11A	0.4768	0.4328	1.1604	0.190*
H11B	0.4455	0.2993	1.2447	0.190*
H11C	0.3448	0.4200	1.2199	0.190*
C12	0.1470 (2)	0.5359 (3)	0.7899 (2)	0.0450 (6)
H12	0.1652	0.5270	0.8786	0.054*
C13	0.1287 (3)	0.6983 (3)	0.7648 (3)	0.0496 (6)
C14	0.0195 (3)	0.7568 (4)	0.7190 (3)	0.0618 (8)
H14	−0.0455	0.6958	0.6947	0.074*
C15	0.0056 (3)	0.9070 (4)	0.7088 (4)	0.0747 (10)
H15	−0.0689	0.9453	0.6779	0.090*
C16	0.0989 (4)	0.9976 (4)	0.7433 (4)	0.0877 (12)
H16	0.0888	1.0979	0.7371	0.105*
C17	0.2092 (4)	0.9395 (4)	0.7879 (5)	0.1029 (16)
H17	0.2743	1.0009	0.8113	0.123*
C18	0.2237 (3)	0.7912 (4)	0.7980 (4)	0.0837 (12)
H18	0.2988	0.7535	0.8277	0.100*
C19	0.0345 (2)	0.4398 (3)	0.7639 (2)	0.0453 (6)
C20	−0.0062 (3)	0.4086 (4)	0.6451 (3)	0.0656 (8)
H20	0.0368	0.4464	0.5784	0.079*
C21	−0.1091 (3)	0.3227 (5)	0.6239 (4)	0.0798 (11)
H21	−0.1352	0.3028	0.5433	0.096*
C22	−0.1727 (3)	0.2669 (5)	0.7205 (4)	0.0815 (11)
H22	−0.2433	0.2106	0.7053	0.098*
C23	−0.1347 (3)	0.2920 (5)	0.8398 (4)	0.0797 (10)
H23	−0.1772	0.2504	0.9053	0.096*
C24	−0.0301 (3)	0.3819 (4)	0.8620 (3)	0.0610 (7)
H24	−0.0045	0.4023	0.9427	0.073*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0720 (14)	0.0947 (19)	0.0724 (15)	0.0133 (14)	0.0149 (11)	0.0440 (15)
O2	0.0973 (19)	0.100 (2)	0.0714 (15)	0.0469 (17)	0.0206 (14)	0.0062 (15)
O3	0.136 (3)	0.104 (2)	0.124 (2)	0.064 (2)	0.066 (2)	0.058 (2)
O4	0.0758 (14)	0.140 (3)	0.0460 (11)	0.0508 (17)	0.0084 (10)	0.0085 (14)

N1	0.0405 (10)	0.0474 (13)	0.0487 (11)	0.0027 (10)	0.0049 (8)	0.0084 (10)
C2	0.0450 (13)	0.0583 (18)	0.0573 (16)	0.0002 (13)	0.0058 (11)	0.0156 (15)
C3	0.0385 (12)	0.0630 (18)	0.0512 (14)	0.0037 (12)	0.0094 (10)	0.0127 (14)
C4	0.0426 (12)	0.0394 (13)	0.0457 (13)	0.0022 (11)	0.0069 (10)	0.0031 (11)
C5	0.0659 (17)	0.089 (2)	0.0475 (15)	0.0158 (19)	0.0178 (13)	0.0044 (17)
C6	0.093 (3)	0.118 (4)	0.070 (2)	0.008 (3)	0.001 (2)	−0.030 (2)
C7	0.0565 (15)	0.0640 (19)	0.0471 (14)	0.0067 (14)	0.0006 (11)	−0.0025 (14)
C8	0.0577 (17)	0.112 (3)	0.0558 (17)	0.029 (2)	−0.0008 (14)	0.009 (2)
C9	0.0714 (18)	0.070 (2)	0.0620 (18)	0.0051 (18)	0.0129 (15)	0.0057 (17)
C10	0.177 (6)	0.143 (6)	0.172 (6)	−0.071 (5)	0.089 (5)	−0.030 (5)
C11	0.157 (4)	0.152 (5)	0.070 (2)	0.012 (5)	−0.035 (3)	−0.009 (3)
C12	0.0435 (13)	0.0476 (15)	0.0440 (13)	0.0034 (11)	0.0046 (10)	0.0008 (11)
C13	0.0460 (14)	0.0447 (15)	0.0583 (16)	0.0007 (12)	0.0049 (12)	−0.0006 (13)
C14	0.0498 (16)	0.0489 (17)	0.087 (2)	0.0037 (13)	0.0058 (15)	−0.0006 (16)
C15	0.0621 (18)	0.054 (2)	0.108 (3)	0.0180 (16)	0.0096 (18)	0.0102 (19)
C16	0.085 (3)	0.0405 (17)	0.138 (4)	0.0032 (18)	0.012 (2)	0.003 (2)
C17	0.085 (3)	0.051 (2)	0.172 (5)	−0.012 (2)	−0.022 (3)	−0.009 (3)
C18	0.069 (2)	0.050 (2)	0.131 (3)	−0.0026 (17)	−0.026 (2)	0.001 (2)
C19	0.0368 (12)	0.0401 (14)	0.0592 (15)	0.0045 (10)	0.0031 (11)	−0.0009 (12)
C20	0.0621 (17)	0.068 (2)	0.0663 (18)	−0.0026 (16)	0.0014 (14)	−0.0035 (17)
C21	0.067 (2)	0.079 (3)	0.093 (2)	−0.0069 (19)	−0.0119 (18)	−0.021 (2)
C22	0.0520 (17)	0.068 (2)	0.124 (3)	−0.0136 (18)	0.0027 (19)	−0.013 (2)
C23	0.0549 (17)	0.077 (2)	0.108 (3)	−0.0097 (18)	0.0213 (18)	0.005 (2)
C24	0.0544 (15)	0.0589 (18)	0.0700 (17)	0.0027 (15)	0.0117 (13)	0.0000 (16)

Geometric parameters (Å, °)

O1—C2	1.218 (4)	C10—H10C	0.9599
O2—C5	1.416 (4)	C11—H11A	0.9599
O2—H2X	0.806 (19)	C11—H11B	0.9599
O3—C8	1.331 (4)	C11—H11C	0.9599
O3—C9	1.408 (4)	C12—C19	1.518 (4)
O4—C9	1.405 (4)	C12—C13	1.529 (4)
O4—C7	1.424 (4)	C12—H12	0.9800
N1—C2	1.349 (3)	C13—C18	1.373 (5)
N1—C4	1.468 (3)	C13—C14	1.374 (4)
N1—C12	1.476 (3)	C14—C15	1.393 (5)
C2—C3	1.529 (4)	C14—H14	0.9300
C3—C5	1.519 (5)	C15—C16	1.349 (5)
C3—C4	1.553 (3)	C15—H15	0.9300
C3—H3	0.9800	C16—C17	1.378 (6)
C4—C7	1.506 (4)	C16—H16	0.9300
C4—H4	0.9800	C17—C18	1.376 (6)
C5—C6	1.515 (5)	C17—H17	0.9300
C5—H5	0.9800	C18—H18	0.9300
C6—H6A	0.9599	C19—C20	1.381 (4)
C6—H6B	0.9599	C19—C24	1.383 (4)
C6—H6C	0.9599	C20—C21	1.373 (5)
C7—C8	1.517 (4)	C20—H20	0.9300

C7—H7	0.9800	C21—C22	1.357 (6)
C8—H8A	0.9700	C21—H21	0.9300
C8—H8B	0.9700	C22—C23	1.367 (5)
C9—C11	1.482 (6)	C22—H22	0.9300
C9—C10	1.486 (6)	C23—C24	1.411 (5)
C10—H10A	0.9599	C23—H23	0.9300
C10—H10B	0.9599	C24—H24	0.9300
C5—O2—H2X	114 (4)	H10A—C10—H10B	109.5
C8—O3—C9	112.5 (3)	C9—C10—H10C	109.5
C9—O4—C7	110.5 (2)	H10A—C10—H10C	109.5
C2—N1—C4	95.11 (19)	H10B—C10—H10C	109.5
C2—N1—C12	136.5 (2)	C9—C11—H11A	109.5
C4—N1—C12	128.4 (2)	C9—C11—H11B	109.5
O1—C2—N1	132.5 (3)	H11A—C11—H11B	109.5
O1—C2—C3	134.8 (3)	C9—C11—H11C	109.5
N1—C2—C3	92.7 (2)	H11A—C11—H11C	109.5
C5—C3—C2	118.8 (2)	H11B—C11—H11C	109.5
C5—C3—C4	118.3 (3)	N1—C12—C19	110.9 (2)
C2—C3—C4	84.89 (18)	N1—C12—C13	110.9 (2)
C5—C3—H3	110.8	C19—C12—C13	115.8 (2)
C2—C3—H3	110.8	N1—C12—H12	106.2
C4—C3—H3	110.8	C19—C12—H12	106.2
N1—C4—C7	114.1 (2)	C13—C12—H12	106.2
N1—C4—C3	87.29 (18)	C18—C13—C14	118.3 (3)
C7—C4—C3	116.4 (2)	C18—C13—C12	117.9 (3)
N1—C4—H4	112.3	C14—C13—C12	123.6 (3)
C7—C4—H4	112.3	C13—C14—C15	120.4 (3)
C3—C4—H4	112.3	C13—C14—H14	119.8
O2—C5—C6	110.5 (4)	C15—C14—H14	119.8
O2—C5—C3	107.1 (2)	C16—C15—C14	120.8 (3)
C6—C5—C3	113.4 (3)	C16—C15—H15	119.6
O2—C5—H5	108.6	C14—C15—H15	119.6
C6—C5—H5	108.6	C15—C16—C17	119.1 (3)
C3—C5—H5	108.6	C15—C16—H16	120.5
C5—C6—H6A	109.5	C17—C16—H16	120.5
C5—C6—H6B	109.5	C18—C17—C16	120.5 (4)
H6A—C6—H6B	109.5	C18—C17—H17	119.8
C5—C6—H6C	109.5	C16—C17—H17	119.8
H6A—C6—H6C	109.5	C13—C18—C17	120.9 (4)
H6B—C6—H6C	109.5	C13—C18—H18	119.6
O4—C7—C4	109.3 (2)	C17—C18—H18	119.6
O4—C7—C8	103.6 (2)	C20—C19—C24	118.7 (3)
C4—C7—C8	114.0 (3)	C20—C19—C12	122.1 (3)
O4—C7—H7	109.9	C24—C19—C12	119.2 (2)
C4—C7—H7	109.9	C21—C20—C19	121.1 (3)
C8—C7—H7	109.9	C21—C20—H20	119.5
O3—C8—C7	105.5 (2)	C19—C20—H20	119.5
O3—C8—H8A	110.6	C22—C21—C20	120.1 (3)
C7—C8—H8A	110.6	C22—C21—H21	120.0

O3—C8—H8B	110.6	C20—C21—H21	120.0
C7—C8—H8B	110.6	C21—C22—C23	121.1 (3)
H8A—C8—H8B	108.8	C21—C22—H22	119.4
O4—C9—O3	104.1 (2)	C23—C22—H22	119.4
O4—C9—C11	109.0 (4)	C22—C23—C24	118.9 (3)
O3—C9—C11	111.8 (4)	C22—C23—H23	120.5
O4—C9—C10	109.9 (4)	C24—C23—H23	120.5
O3—C9—C10	108.8 (4)	C19—C24—C23	120.0 (3)
C11—C9—C10	112.9 (4)	C19—C24—H24	120.0
C9—C10—H10A	109.5	C23—C24—H24	120.0
C9—C10—H10B	109.5		
C4—N1—C2—O1	177.6 (4)	C8—O3—C9—O4	20.7 (5)
C12—N1—C2—O1	−2.4 (6)	C8—O3—C9—C11	−96.9 (5)
C4—N1—C2—C3	−1.3 (2)	C8—O3—C9—C10	137.8 (5)
C12—N1—C2—C3	178.8 (3)	C2—N1—C12—C19	97.6 (4)
O1—C2—C3—C5	−58.0 (5)	C4—N1—C12—C19	−82.4 (3)
N1—C2—C3—C5	120.8 (3)	C2—N1—C12—C13	−32.6 (4)
O1—C2—C3—C4	−177.6 (4)	C4—N1—C12—C13	147.4 (3)
N1—C2—C3—C4	1.2 (2)	N1—C12—C13—C18	−58.6 (4)
C2—N1—C4—C7	119.1 (2)	C19—C12—C13—C18	173.9 (3)
C12—N1—C4—C7	−61.0 (3)	N1—C12—C13—C14	126.2 (3)
C2—N1—C4—C3	1.2 (2)	C19—C12—C13—C14	−1.4 (4)
C12—N1—C4—C3	−178.8 (3)	C18—C13—C14—C15	−1.0 (5)
C5—C3—C4—N1	−121.2 (2)	C12—C13—C14—C15	174.2 (3)
C2—C3—C4—N1	−1.1 (2)	C13—C14—C15—C16	0.2 (6)
C5—C3—C4—C7	123.1 (3)	C14—C15—C16—C17	0.6 (7)
C2—C3—C4—C7	−116.8 (3)	C15—C16—C17—C18	−0.5 (8)
C2—C3—C5—O2	−163.1 (3)	C14—C13—C18—C17	1.2 (7)
C4—C3—C5—O2	−62.6 (3)	C12—C13—C18—C17	−174.3 (5)
C2—C3—C5—C6	−40.9 (4)	C16—C17—C18—C13	−0.4 (9)
C4—C3—C5—C6	59.5 (4)	N1—C12—C19—C20	−55.3 (3)
C9—O4—C7—C4	125.3 (3)	C13—C12—C19—C20	72.3 (3)
C9—O4—C7—C8	3.4 (4)	N1—C12—C19—C24	125.1 (3)
N1—C4—C7—O4	70.7 (3)	C13—C12—C19—C24	−107.4 (3)
C3—C4—C7—O4	170.2 (3)	C24—C19—C20—C21	0.4 (5)
N1—C4—C7—C8	−173.9 (2)	C12—C19—C20—C21	−179.2 (3)
C3—C4—C7—C8	−74.5 (3)	C19—C20—C21—C22	0.0 (6)
C9—O3—C8—C7	−18.6 (5)	C20—C21—C22—C23	−1.4 (6)
O4—C7—C8—O3	9.0 (4)	C21—C22—C23—C24	2.3 (6)
C4—C7—C8—O3	−109.7 (4)	C20—C19—C24—C23	0.5 (5)
C7—O4—C9—O3	−13.8 (4)	C12—C19—C24—C23	−179.8 (3)
C7—O4—C9—C11	105.7 (4)	C22—C23—C24—C19	−1.8 (5)
C7—O4—C9—C10	−130.1 (4)		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C12—H12···O4	0.98	2.42	3.141 (3)	130
O2—H2X···O1^{i}	0.806 (19)	1.99 (2)	2.787 (4)	170 (5)

C7—H7···O3^{ii}	0.98	2.45	3.357 (4)	153

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

Fig. 1

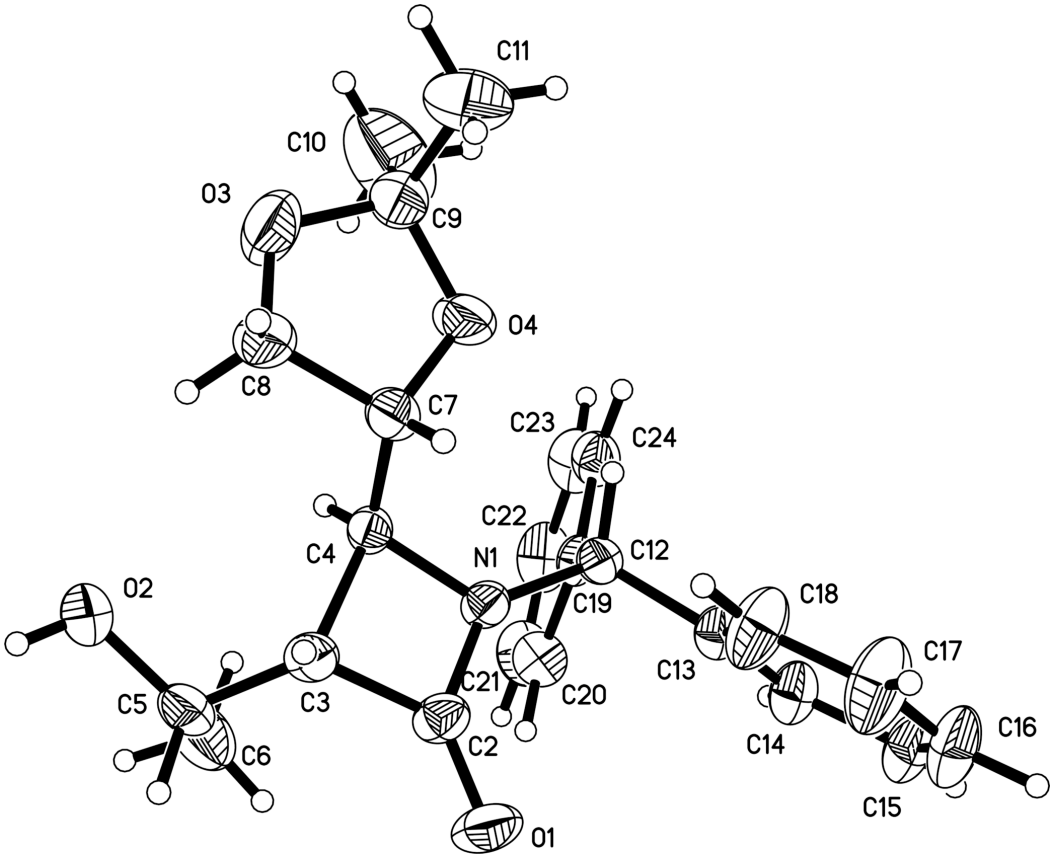


Fig. 2