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(4*R*)-4-[(1*R*)-1,2-Dihydroxyethyl]-1-[(1*R*)-1-phenylethyl]pyrrolidin-2-one

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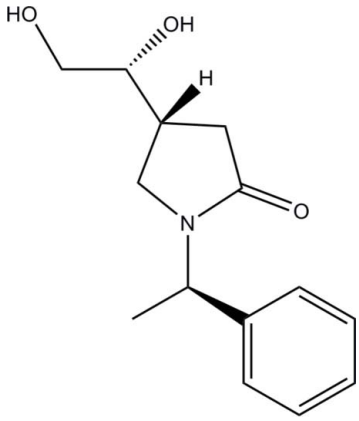
Key indicators: single-crystal X-ray study; T = 83 K; mean σ(C–C) = 0.005 Å; R factor = 0.045; wR factor = 0.099; data-to-parameter ratio = 8.8.

The title compound, $C_{14}H_{19}NO_3$, was obtained as one of the two isomers of a Sharpless asymmetric dihydroxylation reaction of (1*S*)-1-[(1*R*)-1-phenylethyl]-4-vinylpyrrolidin-2-one. The absolute stereochemistry of this isomer was determined from the known stereochemistry (*R*) at the bridge C atom between the phenyl and pyrrolidine rings. The molecules form one-dimensional tapes along the *b* axis *via* hydrogen bonding between the carbonyl O atom and the alcohol groups of neighbouring molecules. These assemble into sheets *via* interdigitative stacking of the phenyl rings and C—H···O interactions.

Related literature

For related literature see: Fava *et al.* (1999).



Experimental

Crystal data

$C_{14}H_{19}NO_3$
M_r = 249.30
Monoclinic, $P2_1$
a = 6.1953 (1) Å
b = 8.2895 (2) Å
c = 13.2737 (1) Å
β = 103.353 (2)°
V = 663.25 (2) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.09 mm^{-1}
T = 83 (2) K
0.28 × 0.18 × 0.10 mm

Data collection

Siemens SMART APEX CCD diffractometer
Absorption correction: none
4016 measured reflections
1461 independent reflections
1271 reflections with $I > 2\sigma(I)$
R_{int} = 0.040

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.045
$wR(F^2)$ = 0.099
S = 1.01
1461 reflections
166 parameters
1 restraint
H-atom parameters constrained
$\Delta\rho_{max}$ = 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
O3—H3···O1^{i}	0.82	1.96	2.743 (3)	158
O2—H2···O1^{i}	0.82	1.93	2.738 (3)	170

Symmetry code: (i) $x, y+1, z$.

Data collection: *SMART* (Siemens, 1995); cell refinement: *SAINT* (Siemens, 1995); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEPIII* (Burnett & Johnson, 1996) and *Mercury* (Macrae *et al.*, 2006); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: AT2618).

References

Burnett, M. N. & Johnson, C. K. (1996). *ORTEPIII*. Report ORNL-6895. Oak Ridge National Laboratory, Tennessee, USA.
Farrugia, L. J. (1999). *J. Appl. Cryst.* **32**, 837–838.
Fava, C., Galeazzi, R., Mobbili, G. & Orena, M. (1999). *Heterocycles*, **51**, 2463–2470.
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.
Siemens (1995). *SMART* and *SAINT*. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.

supplementary materials

Acta Cryst. (2008). E**64**, o1851 [**doi:10.1107/S1600536808027293**]

(4*R*)-4-[(1*R*)-1,2-Dihydroxyethyl]-1-[(1*R*)-1-phenylethyl]pyrrolidin-2-one

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Comment

The title compound (I) was obtained as one of the two isomers of a Sharpless asymmetric dihydroxylation reaction of (1*S*)-1-((1*R*)-1-phenylethyl)-4-vinylpyrrolidin-2-one (Fava *et al.*1999). The major isomer from the reaction was recrystallized to give a pure sample for X-ray analysis. The molecular structure of (I) is shown in Fig. 1. The assignment of the absolute stereochemistry is based on the known stereochemistry of C7 (*R*). This leads to the absolute configuration at C10 and C13 as *R*.

The molecules of (I) in the crystal form one dimensional tapes along the *b* axis *via* hydrogen bonding between the carbonyl oxygen, O1 and the two alcohol moieties O2—H and O3—H. These assemble by interdigitative stacking of phenyl rings between tapes and further connection by C11—H11B···O3, C5—H5···O2 interactions between adjacent molecules to form sheets near the b-c plane, Fig. 2 (Table 1).

Experimental

(4*R*)-4-[(1*R*)-1,2-Dihydroxyethyl]-1-[(1*R*)-1-phenylethyl]-2-pyrrolidinone (I): AD-mix-β (1.40 g, 1 mmol, Aldrich Cat. No. 392766) was dissolved in *tert*-butanol (5 ml) and water (5 ml). Methanesulfonamide (98 mg, 1 mmol) was added and cooled to 273 K, (1*S*)-1-((1*R*)-1-phenylethyl)-4-vinylpyrrolidin-2-one1 (216 mg, 1 mmol) was added and the reaction stirred for 24 h. Na_2SO_3 (1.5 g, 11.9 mmol) was added and stirred for another 90 minutes. The reaction was extracted with dichloromethane (4x100 ml), the combined organic layers were washed with 2 N KOH solution, dried (Na_2SO_4) and concentrated under reduced pressure. The residue was purified by chromatography, eluting with methanol/ethylacetate (3:7) to give the two isomers (4*R*)-4-[(1*R*)-1,2-dihydroxyethyl]-1-[(1*R*)-1-phenylethyl]-2-pyrrolidinone and (4*R*)-4-[(1*S*)-1,2-dihydroxyethyl]-1-[(1*R*)-1-phenylethyl]-2-pyrrolidinone (169 mg, 68%) in a ratio of 2:1. The title compound (I) was then obtained by recrystallization from ethylacetate as clear crystals. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.36–7.24 (m, 5 H), 5.47 (q, J=7.1 Hz, 1 H), 3.69 (dd, J=10.7, 2.8 Hz, 1 H), 3.67–3.60 (m, 1 H), 3.46 (dd, J=10.7, 7.0 Hz, 1 H), 3.35 (dd, J=10.0, 7.0 Hz, 1 H), 3.13 (dd, J=10.0, 8.0 Hz, 1 H), 2.66 (br s, 1 H), 2.46 (dd, J=15.2, 8.1 Hz, 1 H), 2.40–2.29 (m, 1 H), 2.26 (dd, J=15.2, 8.2 Hz, 1 H), 2.17 (br s, 1 H), 1.53 (t, J=7.1 Hz, 3 H). LCMS (APCI$^+$) calcd for $C_{14}H_{19}NO_3$: 250 (MH^+), found 250 (100%).

Refinement

Hydrogen atoms were placed in calculated positions and refined using the riding model [O—H = 0.82 Å C—H = 0.93–0.97 Å], with U_{iso}(H) = 1.5 times U_{eq}(O) and U_{iso}(H) = 1.2 or 1.5 times U_{eq}(C)..

Figures

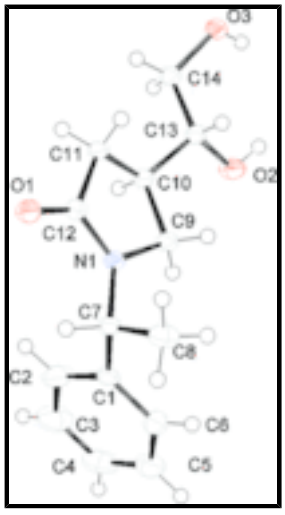

Fig. 1. Structure of (I) showing 50% probability displacement ellipsoids for non-hydrogen atoms and hydrogen atoms as arbitary spheres.

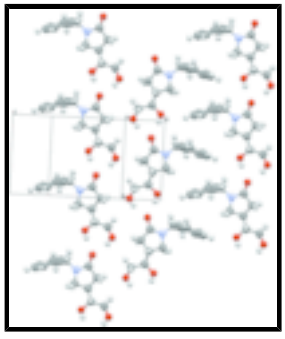

Fig. 2. Illustration of the arrangement of (I) into sheets.

(4*R*)-4-[(1*R*)-1,2-Dihydroxyethyl]-1-[(1*R*)-1- phenylethyl]pyrrolidin-2-one

Crystal data

$C_{14}H_{19}NO_3$	F_{000} = 268
M_r = 249.30	D_x = 1.248 Mg m^{-3}
Monoclinic, $P2_1$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: P 2yb	Cell parameters from 2597 reflections
a = 6.1953 (1) Å	θ = 1.6–26.4º
b = 8.2895 (2) Å	μ = 0.09 mm^{-1}
c = 13.2737 (1) Å	T = 83 (2) K
β = 103.353 (2)º	Plate, colourless
V = 663.25 (2) Å^3	0.28 × 0.18 × 0.10 mm
Z = 2	

Data collection

Siemens SMART APEX CCD diffractometer	1271 reflections with $I > 2\sigma(I)$

Radiation source: fine-focus sealed tube	$R_{int} = 0.040$
Monochromator: graphite	$\theta_{max} = 26.4°$
$T = 83(2)$ K	$\theta_{min} = 1.6°$
ω scans	$h = -7 \rightarrow 7$
Absorption correction: none	$k = -10 \rightarrow 10$
4016 measured reflections	$l = -7 \rightarrow 16$
1461 independent reflections	

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.045$	H-atom parameters constrained
$wR(F^2) = 0.099$	$w = 1/[\sigma^2(F_o^2) + (0.0168P)^2 + 0.6853P]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.01$	$(\Delta/\sigma)_{max} < 0.001$
1461 reflections	$\Delta\rho_{max} = 0.20$ e Å^{-3}
166 parameters	$\Delta\rho_{min} = -0.22$ e Å^{-3}
1 restraint	Extinction correction: none
Primary atom site location: structure-invariant direct methods	

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 $F^2 > \sigma(F^2)$ 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}
O3	0.1980 (4)	−0.0064 (3)	0.00048 (16)	0.0261 (5)
H3	0.2059	0.0736	−0.0348	0.039*
O1	0.3278 (5)	−0.7778 (3)	−0.12286 (19)	0.0277 (6)
O2	0.3068 (5)	−0.0804 (3)	−0.20720 (19)	0.0339 (6)
H2	0.3289	0.0108	−0.1832	0.051*
N1	0.4081 (4)	−0.5848 (3)	−0.2318 (2)	0.0200 (6)
C11	0.2531 (5)	−0.4926 (4)	−0.0993 (2)	0.0204 (6)
H11A	0.3554	−0.4685	−0.0339	0.024*
H11B	0.1070	−0.5120	−0.0869	0.024*
C7	0.5155 (5)	−0.6928 (4)	−0.2932 (2)	0.0216 (7)

H7	0.4748	−0.8031	−0.2784	0.026*
C13	0.3005 (6)	−0.1917 (4)	−0.1258 (3)	0.0231 (7)
H13	0.4473	−0.1964	−0.0784	0.028*
C9	0.4141 (6)	−0.4083 (4)	−0.2397 (3)	0.0231 (7)
H9A	0.3682	−0.3731	−0.3112	0.028*
H9B	0.5613	−0.3666	−0.2099	0.028*
C12	0.3308 (5)	−0.6349 (4)	−0.1506 (2)	0.0216 (7)
C1	0.4215 (5)	−0.6645 (4)	−0.4075 (2)	0.0222 (7)
C8	0.7661 (5)	−0.6810 (5)	−0.2569 (3)	0.0320 (8)
H8A	0.8137	−0.5754	−0.2721	0.048*
H8B	0.8341	−0.7605	−0.2922	0.048*
H8C	0.8089	−0.6998	−0.1837	0.048*
C10	0.2466 (6)	−0.3545 (4)	−0.1767 (3)	0.0217 (7)
H10	0.0983	−0.3499	−0.2228	0.026*
C6	0.5480 (6)	−0.6089 (4)	−0.4737 (2)	0.0297 (8)
H6	0.6968	−0.5842	−0.4474	0.036*
C14	0.1299 (6)	−0.1421 (4)	−0.0656 (2)	0.0236 (7)
H14A	−0.0084	−0.1162	−0.1142	0.028*
H14B	0.1024	−0.2326	−0.0240	0.028*
C4	0.2361 (7)	−0.6207 (6)	−0.6180 (3)	0.0433 (10)
H4	0.1743	−0.6056	−0.6882	0.052*
C2	0.2008 (6)	−0.6969 (6)	−0.4493 (3)	0.0395 (10)
H2A	0.1129	−0.7347	−0.4063	0.047*
C5	0.4561 (7)	−0.5893 (5)	−0.5789 (3)	0.0370 (9)
H5	0.5444	−0.5548	−0.6227	0.044*
C3	0.1060 (7)	−0.6746 (7)	−0.5538 (3)	0.0491 (12)
H3A	−0.0438	−0.6959	−0.5801	0.059*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O3	0.0388 (14)	0.0189 (12)	0.0245 (11)	−0.0048 (12)	0.0151 (10)	−0.0050 (11)
O1	0.0460 (16)	0.0153 (12)	0.0264 (13)	−0.0007 (11)	0.0178 (12)	0.0004 (10)
O2	0.0633 (18)	0.0161 (12)	0.0291 (13)	−0.0033 (14)	0.0247 (13)	−0.0008 (10)
N1	0.0294 (15)	0.0130 (13)	0.0208 (13)	−0.0012 (12)	0.0121 (11)	−0.0019 (11)
C11	0.0293 (16)	0.0170 (15)	0.0176 (14)	−0.0016 (15)	0.0111 (12)	−0.0007 (14)
C7	0.0299 (17)	0.0168 (17)	0.0202 (15)	0.0016 (15)	0.0103 (13)	−0.0010 (13)
C13	0.0341 (18)	0.0166 (16)	0.0219 (16)	−0.0032 (15)	0.0135 (14)	−0.0008 (14)
C9	0.035 (2)	0.0168 (16)	0.0211 (16)	−0.0030 (15)	0.0134 (14)	−0.0025 (14)
C12	0.0269 (17)	0.0193 (16)	0.0196 (15)	−0.0028 (14)	0.0071 (13)	−0.0005 (14)
C1	0.0292 (17)	0.0180 (17)	0.0203 (15)	0.0016 (14)	0.0073 (13)	−0.0056 (14)
C8	0.0330 (19)	0.038 (2)	0.0248 (17)	0.0042 (18)	0.0050 (14)	−0.0050 (16)
C10	0.0309 (19)	0.0178 (16)	0.0191 (16)	−0.0010 (15)	0.0112 (14)	−0.0004 (13)
C6	0.041 (2)	0.0249 (19)	0.0255 (17)	−0.0037 (17)	0.0125 (15)	0.0010 (16)
C14	0.0327 (18)	0.0182 (16)	0.0225 (15)	−0.0025 (14)	0.0116 (13)	−0.0022 (14)
C4	0.059 (3)	0.044 (3)	0.0239 (17)	0.014 (2)	0.0029 (17)	−0.0049 (18)
C2	0.034 (2)	0.060 (3)	0.0269 (18)	−0.003 (2)	0.0112 (15)	−0.011 (2)
C5	0.063 (3)	0.0251 (19)	0.0269 (18)	0.001 (2)	0.0178 (18)	0.0022 (16)

C3	0.035 (2)	0.075 (4)	0.034 (2)	0.002 (2)	0.0001 (17)	−0.017 (2)

Geometric parameters (Å, °)

O3—C14	1.429 (4)	C9—H9A	0.9700
O3—H3	0.8200	C9—H9B	0.9700
O1—C12	1.242 (4)	C1—C2	1.378 (5)
O2—C13	1.428 (4)	C1—C6	1.385 (4)
O2—H2	0.8200	C8—H8A	0.9600
N1—C12	1.342 (4)	C8—H8B	0.9600
N1—C9	1.468 (4)	C8—H8C	0.9600
N1—C7	1.469 (4)	C10—H10	0.9800
C11—C12	1.496 (5)	C6—C5	1.390 (5)
C11—C10	1.532 (4)	C6—H6	0.9300
C11—H11A	0.9700	C14—H14A	0.9700
C11—H11B	0.9700	C14—H14B	0.9700
C7—C1	1.513 (4)	C4—C5	1.367 (6)
C7—C8	1.519 (5)	C4—C3	1.376 (6)
C7—H7	0.9800	C4—H4	0.9300
C13—C10	1.511 (5)	C2—C3	1.388 (5)
C13—C14	1.521 (4)	C2—H2A	0.9300
C13—H13	0.9800	C5—H5	0.9300
C9—C10	1.542 (5)	C3—H3A	0.9300
C14—O3—H3	109.5	C6—C1—C7	123.0 (3)
C13—O2—H2	109.5	C7—C8—H8A	109.5
C12—N1—C9	112.7 (3)	C7—C8—H8B	109.5
C12—N1—C7	123.2 (3)	H8A—C8—H8B	109.5
C9—N1—C7	123.1 (3)	C7—C8—H8C	109.5
C12—C11—C10	104.2 (2)	H8A—C8—H8C	109.5
C12—C11—H11A	110.9	H8B—C8—H8C	109.5
C10—C11—H11A	110.9	C13—C10—C11	113.5 (3)
C12—C11—H11B	110.9	C13—C10—C9	113.2 (3)
C10—C11—H11B	110.9	C11—C10—C9	103.3 (3)
H11A—C11—H11B	108.9	C13—C10—H10	108.9
N1—C7—C1	110.1 (3)	C11—C10—H10	108.9
N1—C7—C8	110.3 (3)	C9—C10—H10	108.9
C1—C7—C8	115.9 (3)	C1—C6—C5	121.0 (3)
N1—C7—H7	106.7	C1—C6—H6	119.5
C1—C7—H7	106.7	C5—C6—H6	119.5
C8—C7—H7	106.7	O3—C14—C13	113.2 (3)
O2—C13—C10	106.3 (3)	O3—C14—H14A	108.9
O2—C13—C14	111.5 (3)	C13—C14—H14A	108.9
C10—C13—C14	111.6 (3)	O3—C14—H14B	108.9
O2—C13—H13	109.1	C13—C14—H14B	108.9
C10—C13—H13	109.1	H14A—C14—H14B	107.8
C14—C13—H13	109.1	C5—C4—C3	120.1 (4)
N1—C9—C10	102.6 (3)	C5—C4—H4	119.9
N1—C9—H9A	111.2	C3—C4—H4	119.9
C10—C9—H9A	111.2	C1—C2—C3	121.9 (4)

N1—C9—H9B	111.2	C1—C2—H2A	119.1
C10—C9—H9B	111.2	C3—C2—H2A	119.1
H9A—C9—H9B	109.2	C4—C5—C6	120.0 (3)
O1—C12—N1	124.5 (3)	C4—C5—H5	120.0
O1—C12—C11	126.1 (3)	C6—C5—H5	120.0
N1—C12—C11	109.5 (3)	C4—C3—C2	119.3 (4)
C2—C1—C6	117.6 (3)	C4—C3—H3A	120.4
C2—C1—C7	119.3 (3)	C2—C3—H3A	120.4

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O3—H3···O1^{i}	0.82	1.96	2.743 (3)	158
O2—H2···O1^{i}	0.82	1.93	2.738 (3)	170

Symmetry codes: (i) *x*, *y*+1, *z*.

Fig. 1

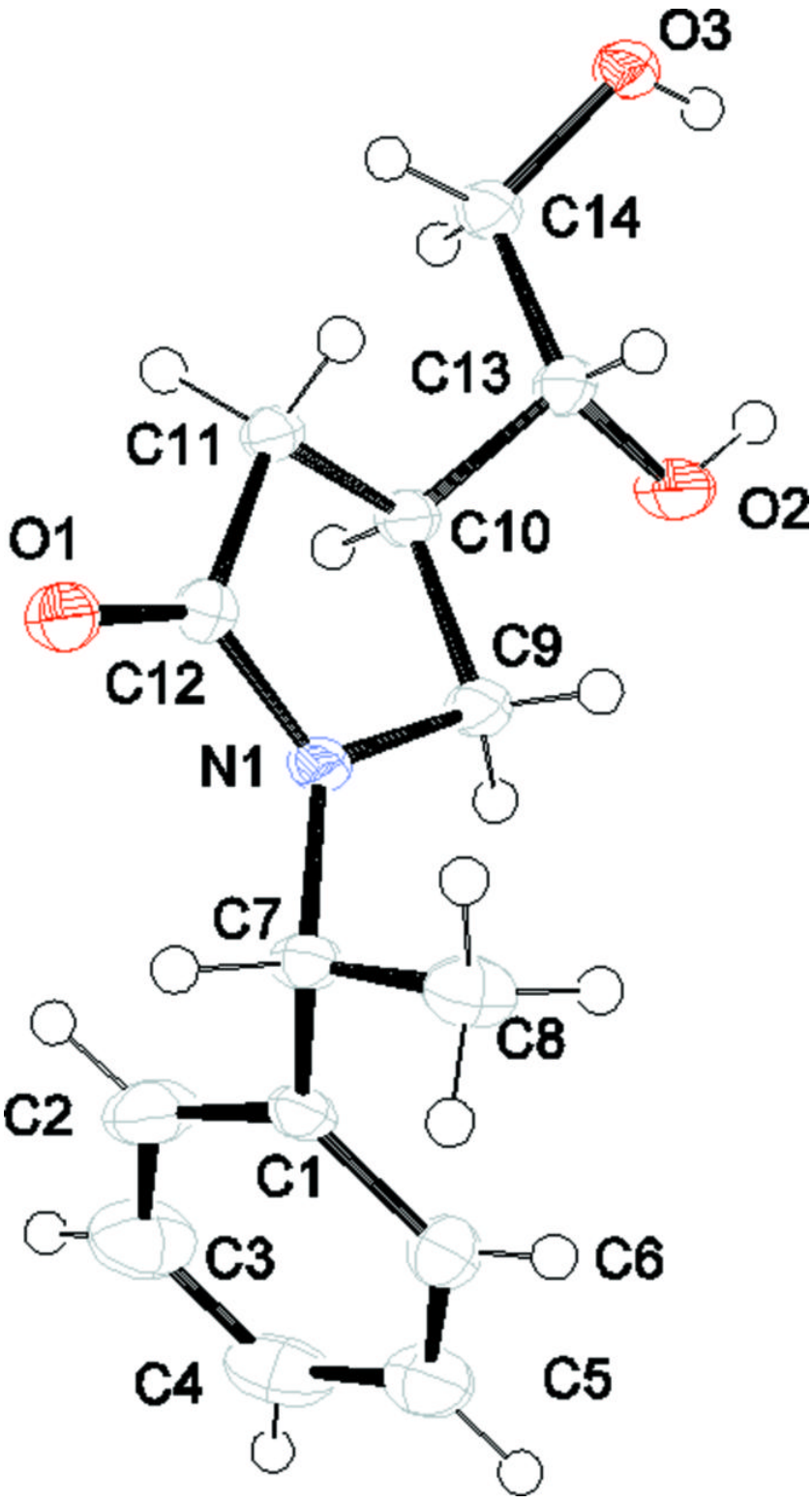

Fig. 2