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1,5-Dihydroxy-1-(3-nitrophenyl)pentan-3-one

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Key indicators

Single-crystal X-ray study
T = 153 K
Mean σ(C–C) = 0.002 Å
R factor = 0.027
wR factor = 0.073
Data-to-parameter ratio = 9.3

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

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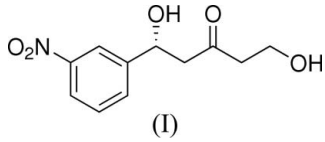
Intermolecular O—H···O and weak C—H···O hydrogen bonding helps to stabilize the crystal structure of the title compound, $C_{11}H_{13}NO_5$.

Comment

Optically active polyhydroxy compounds are very useful intermediates in asymmetric organic syntheses. We have recently reported the crystal structures of some reagents for the synthesis of polyhydroxy compounds (He *et al.*, 2006). The title compound, (I), is an important synthetic building block for optically active 1,3,5-triols (Chen *et al.*, 1987). We report here the crystal structure of (I).



(I)

The molecular structure of (I) is shown in Fig. 1. Bond lengths and angles in (I) are normal. The O1-hydroxy group forms bifurcated hydrogen bonds (Table 1). Intermolecular O—H···O and weak C—H···O hydrogen bonding helps to stabilize the crystal structure.

Experimental

To a solution of 3-nitrobenzaldehyde (75.5 mg, 0.5 mmol) and (*S*)-*N*-phenylpyrrolidine-2-carboxamide (19 mg, 20 mmol) in a mixture of water (1 ml) and THF (1 ml) was added 4-hydroxybutan-2-one (0.5 ml). The mixture was stirred at 273 K for 5 d. It was then treated with saturated ammonium chloride solution. The aqueous layer was extracted with ethyl acetate and dried over anhydrous magnesium sulfate. The solvent was removed and the residue was purified by column chromatography on silica gel (eluant: hexane–ethyl acetate 1:1). Colourless single crystals of (I) were obtained by recrystallization from an ethanol solution.

Crystal data

$C_{11}H_{13}NO_5$
M_r = 239.22
Orthorhombic, $P2_12_12_1$
a = 5.0521 (1) Å
b = 6.9787 (2) Å
c = 31.1510 (6) Å
V = 1098.29 (4) Å^3
Z = 4
D_x = 1.447 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 0.12 mm^{-1}
T = 153 (2) K
Block, colourless
0.24 × 0.23 × 0.15 mm

Data collection

Rigaku R-AXIS RAPID diffractometer
ω scans
Absorption correction: none
10797 measured reflections
1513 independent reflections
1467 reflections with $I > 2\sigma(I)$
R_{int} = 0.016
θ_{max} = 27.5°

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0486P)^2 + 0.259P]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)] = 0.027$	$(\Delta/\sigma)_{max} = 0.001$
$wR(F^2) = 0.073$	$\Delta\rho_{max} = 0.22$ e Å^{-3}
$S = 1.00$	$\Delta\rho_{min} = -0.16$ e Å^{-3}
1513 reflections	Extinction correction: *SHELXL97*
163 parameters	Extinction coefficient: 0.025 (4)
H atoms treated by a mixture of independent and constrained refinement	

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

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1*O*···O2	0.88 (2)	2.12 (2)	2.7977 (15)	133.6 (19)
O1—H1*O*···O3^{i}	0.88 (2)	2.33 (2)	2.9577 (15)	128.5 (18)
O3—H3*O*···O3^{ii}	0.86 (3)	1.95 (3)	2.8036 (9)	169 (3)
C8—H8*B*···O2^{iii}	0.99	2.33	3.2664 (18)	158
C10—H10*A*···O1^{iv}	0.99	2.36	3.1949 (17)	142

Symmetry codes: (i) $x, y-1, z$; (ii) $x-\frac{1}{2}, -y+\frac{5}{2}, -z+1$; (iii) $x+1, y, z$; (iv) $x, y+1, z$.

Carbon-bound H atom were positioned geometrically (C—H = 0.95–1.00 Å) and allowed to ride on the parent C atoms, with U_{iso}(H) = 1.2U_{eq}(C). Hydroxy H atoms were located in a difference Fourier map and refined isotropically. In the absence of significant anomalous scattering effects, Friedel pairs were merged; the absolute configuration of (I) was not determined, and is assigned here arbitrarily.

Data collection: *RAPID-AUTO* (Rigaku/MSC, 2004); cell refinement: *RAPID-AUTO*; data reduction: *RAPID-AUTO*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1997); software used to prepare material for publication: *SHELXTL*.

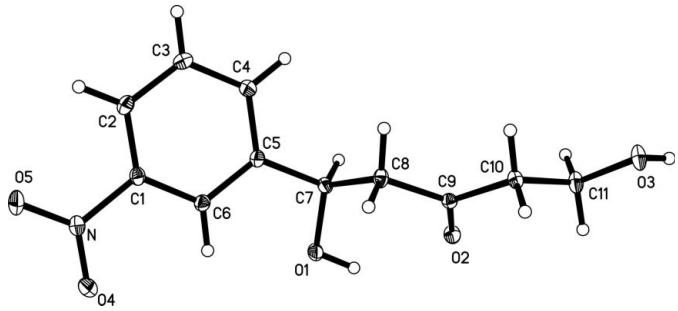


Figure 1
The molecular structure of (I) with 30% probability displacement ellipsoids (arbitrary spheres for H atoms).

The diffraction data were collected at the Centre for Testing and Analysis, Chengdu Branch, Chinese Academy of Sciences. We acknowledge financial support from China West Normal University (No. 05B022).

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