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Key indicators

Single-crystal X-ray study  
T = 273 K  
Mean  $\sigma(\text{C}-\text{C}) = 0.003 \text{ \AA}$   
R factor = 0.034  
wR factor = 0.100  
Data-to-parameter ratio = 7.4

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

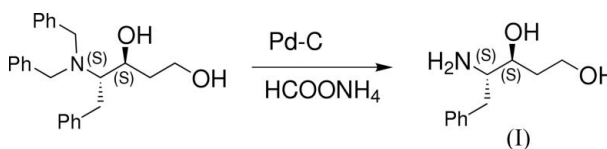
(3S,4S)-4-Amino-5-phenylpentane-1,3-diol

The title compound, C<sub>11</sub>H<sub>17</sub>NO<sub>2</sub>, was obtained by hydrogenolysis of (3S,4S)-4-(dibenzylamino)-5-phenylpentane-1,3-diol. In the crystal structure, intermolecular O—H···O, O—H···N and N—H···O hydrogen bonds link the molecules into a sheet parallel to the *ab* plane.

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Comment

The title compound, (I), is a key intermediate used to synthesize recycling chiral ligands for asymmetric catalysts such as pyridinyloxazolines. X-ray analysis of (I) reveals that the amino group is in a pyramidal configuration with the sum of the bond angles around N1 being 324°. Symmetry-related molecules are linked *via* O—H···O, O—H···N and N—H···O intermolecular hydrogen bonds into a sheet parallel to the *ab* plane.



Experimental

To a solution of (3S,4S)-4-(dibenzylamino)-5-phenylpentane-1,3-diol (1125 mg, 3 mmol) in methanol (20 ml), ammonium formate (1134 mg, 18 mmol) and palladium on carbon (10% Pd) (253 mg) were added and the mixture heated to 353 K. It was stirred for 3 h at 353 K (Stuk *et al.*, 1994). After completion of the reaction, the catalyst was filtered off, the filtrate was concentrated under reduced pressure to give the crude product. It was purified by column chromatography over silica gel (ethyl acetate–methanol, 20:1 *v/v*) to yield compound (I) (527 mg). Single crystals were obtained by recrystallization from a mixture of ethyl acetate and hexane.

Crystal data

C<sub>11</sub>H<sub>17</sub>NO<sub>2</sub>  
M<sub>r</sub> = 195.26  
Monoclinic, P2<sub>1</sub>  
a = 7.960 (3) Å  
b = 5.4531 (17) Å  
c = 12.142 (4) Å  
β = 95.379 (6)°  
V = 524.7 (3) Å<sup>3</sup>  
Z = 2

D<sub>x</sub> = 1.236 Mg m<sup>-3</sup>  
Mo Kα radiation  
Cell parameters from 2218 reflections  
θ = 2.6–28.5°  
μ = 0.09 mm<sup>-1</sup>  
T = 273 (2) K  
Block, colourless  
0.56 × 0.28 × 0.18 mm

Data collection

Bruker APEX area-detector diffractometer  
φ and ω scans  
Absorption correction: multi-scan (SADABS; Bruker, 2001)  
T<sub>min</sub> = 0.959, T<sub>max</sub> = 0.987  
2647 measured reflections

1019 independent reflections  
1006 reflections with I > 2σ(I)  
R<sub>int</sub> = 0.021  
θ<sub>max</sub> = 25.0°  
h = -9 → 8  
k = -6 → 6  
l = -14 → 13

## Refinement

Refinement on  $F^2$   
 $R[F^2 > 2\sigma(F^2)] = 0.034$   
 $wR(F^2) = 0.100$   
 $S = 0.98$   
 1019 reflections  
 137 parameters  
 All H-atom parameters refined

$$w = 1/[\sigma^2(F_o^2) + (0.0762P)^2 + 0.0605P]$$

where  $P = (F_o^2 + 2F_c^2)/3$   
 $(\Delta/\sigma)_{\max} = 0.001$   
 $\Delta\rho_{\max} = 0.11 \text{ e } \text{\AA}^{-3}$   
 $\Delta\rho_{\min} = -0.15 \text{ e } \text{\AA}^{-3}$

Table 1

Selected geometric parameters ( $\text{\AA}$ ,  $^\circ$ ).

N1—C4	1.465 (3)	C5—C6	1.500 (3)
O1—C1	1.406 (3)	C6—C7	1.370 (3)
O2—C3	1.416 (3)	C6—C11	1.375 (3)
C1—C2	1.496 (3)	C7—C8	1.374 (4)
C2—C3	1.508 (3)	C8—C9	1.369 (4)
C3—C4	1.523 (3)	C9—C10	1.361 (4)
C4—C5	1.523 (3)	C10—C11	1.372 (3)
O1—C1—C2	109.73 (17)	C7—C6—C11	118.5 (2)
C1—C2—C3	112.16 (19)	C7—C6—C5	121.6 (2)
O2—C3—C2	111.51 (18)	C11—C6—C5	119.9 (2)
O2—C3—C4	111.82 (18)	C6—C7—C8	120.8 (2)
C2—C3—C4	112.62 (19)	C9—C8—C7	119.9 (2)
N1—C4—C3	113.26 (16)	C10—C9—C8	119.9 (2)
N1—C4—C5	108.44 (16)	C9—C10—C11	120.0 (2)
C3—C4—C5	111.89 (19)	C10—C11—C6	120.8 (2)
C6—C5—C4	114.26 (17)		
C1—C2—C3—C4	164.94 (18)	C3—C4—C5—C6	62.5 (3)
C2—C3—C4—N1	59.3 (3)	C4—C5—C6—C7	-90.5 (3)
O2—C3—C4—C5	55.7 (2)	C4—C5—C6—C11	89.1 (2)
N1—C4—C5—C6	-171.8 (2)		

Table 2

Hydrogen-bond geometry ( $\text{\AA}$ ,  $^\circ$ ).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
O1—H1 $\cdots$ N1 <sup>i</sup>	0.82	1.96	2.763 (2)	167
O2—H2 $\cdots$ O1 <sup>ii</sup>	0.82	1.99	2.805 (2)	172
N1—H1N $\cdots$ O1 <sup>ii</sup>	0.87 (1)	2.43 (1)	3.288 (3)	168 (3)
N1—H2N $\cdots$ O1 <sup>iii</sup>	0.87 (1)	2.60 (1)	3.456 (3)	167 (3)

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

Amino H atoms were located in a difference map and were refined with an N—H distance restraint of 0.87 (1)  $\text{\AA}$ . All other H atoms were positioned geometrically (C—H = 0.93, 0.97 or 0.98  $\text{\AA}$  for phenyl, methylene or tertiary H atoms, respectively, and O—H = 0.82  $\text{\AA}$ ) and were included in the refinement in the riding-model approximation, with  $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{carrier atom})$ . In the absence of significant anomalous scattering effects, Friedel pairs were merged; the absolute configuration was assumed from the synthesis.

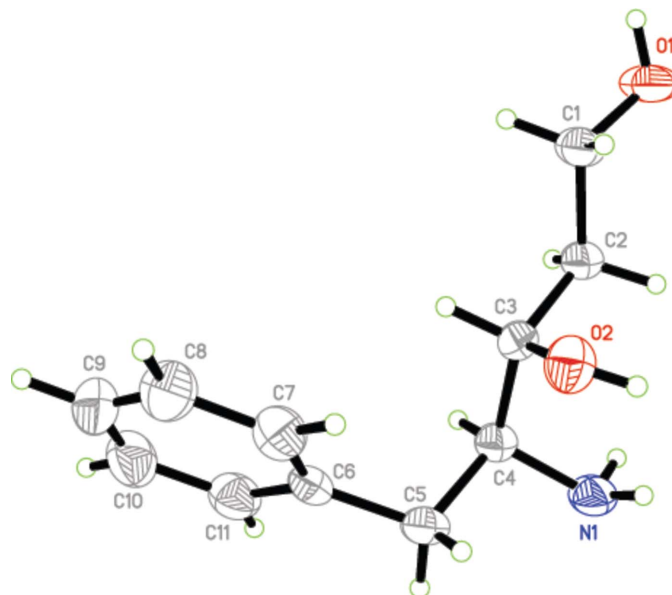


Figure 1

ORTEP III (Farrugia, 1997) plot of (I). Displacement ellipsoids are drawn at the 50% probability level. H atoms are drawn as spheres of arbitrary radii.

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 III (Farrugia, 1997); software used to prepare material for publication: SHELXL97.

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