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rac-(*S*)-2-(1*H*-Imidazol-1-yl)-3-methylbutan-1-ol

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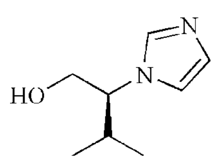
Key indicators: single-crystal X-ray study; T = 292 K; mean σ(C–C) = 0.004 Å; R factor = 0.067; wR factor = 0.197; data-to-parameter ratio = 15.7.

In the crystal structure of the title compound, $C_8H_{14}N_2O$, intermolecular O—H···N hydrogen bonds link molecules related by translation along the *a* axis into chains. Weak intermolecular C—H···O hydrogen bonds and C—H···π interactions enhance the crystal packing stability.

Related literature

For useful applications of imidazole derivatives, see Lu *et al.* (2006); Zou *et al.* (2006). For details of the synthesis, see Bao *et al.* (2003); Guo *et al.* (2006).



Experimental

Crystal data

$C_8H_{14}N_2O$
M_r = 154.21
Monoclinic, $P2_1/n$
a = 7.356 (4) Å
b = 7.212 (3) Å
c = 16.549 (5) Å
β = 90.54 (3)°
V = 877.9 (7) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.08 mm^{-1}
T = 292 K
0.58 × 0.54 × 0.42 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
Absorption correction: none
1931 measured reflections
1630 independent reflections
965 reflections with $I > 2\sigma(I)$
R_{int} = 0.004
3 standard reflections every 120 reflections intensity decay: 0.3%

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.067
$wR(F^2)$ = 0.197
S = 1.18
1630 reflections
104 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.31 e Å^{-3}
$\Delta\rho_{min}$ = −0.24 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1···N2^{i}	0.82	1.93	2.751 (3)	176
C1—H1A···O1^{ii}	0.93	2.43	3.353 (4)	173
C4—H4···Cg^{ii}	0.98	2.86	3.716 (4)	146

Symmetry codes: (i) $x-1, y, z$; (ii) $-x+\frac{3}{2}, y-\frac{1}{2}, -z+\frac{1}{2}$. Cg is the centroid of the C1–C3/N1/N2 ring.

Data collection: *DIFRAC* (Gabe & White, 1993); cell refinement: *DIFRAC*; data reduction: *NRCVAX* (Gabe *et al.*, 1989); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEPIII* (Burnett & Johnson, 1996); software used to prepare material for publication: *SHELXL97* and *PLATON* (Spek, 2009).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: CV2517).

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rac-(*S*)-2-(1*H*-Imidazol-1-yl)-3-methylbutan-1-ol

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S1. Comment

Imidazole is important for biological systems, and its derivatives have attracted widespread interest due to their further expanded application in perfume chemistry and in the construction of some interesting metal–organic frameworks (Lu *et al.* 2006; Zou *et al.* 2006). Here, we report the crystal structure of the title compound, (I), which is a basic unit of constructing chiral receptors and could be applied for the preparation of perfume.

As shown in Fig. 1, there is a chiral center at C4 derived from the source *L*-valine. In the crystal, intermolecular O—H···N hydrogen bonds (Table 1) link the molecules related by translation along axis *a* into chains. Weak intermolecular C—H···O hydrogen bonds and C—H···*π* interactions (Table 1) enhance the crystal packing stability.

S2. Experimental

The title compound was prepared according to the literature (Guo *et al.* 2006). Starting from *L*-valine, methyl 2-(1*H*-imidazol-1-yl)-3-methylbutanoate was easily prepared according to literature procedure (Bao *et al.* 2003). Following, $NaBH_4$ (1.52 g, 40 mmol) was added to methyl 2-(1*H*-imidazol-1-yl)-3-methylbutanoate (1.82 g, 10.0 mmol) in ethanol (50 ml) at 273 K during 30 min. The mixture was stirred at 333 K for another 20 h and then evaporated under vacuum. The residue was diluted with 50 ml saturated K_2CO_3 and extracted with 30 ml ethyl acetate. The organic layer was dried over anhydrous Na_2SO_4 and concentrated under reduced pressure. The resulting residue was purified by column chromatography on silica gel eluting with CH_2Cl_2/CH_3OH (20/1, v/v). Then, colourless crystals suitable for X-ray analysis can be obtained by recrystallization of the compound from ethyl acetate.

S3. Refinement

All H atoms were positioned geometrically and refined in the riding model approximation, with C—H = 0.93–0.98 Å and O—H = 0.82 Å, and $U_{iso}(H)$ = 1.2–1.5 U_{eq} of the parent atom.

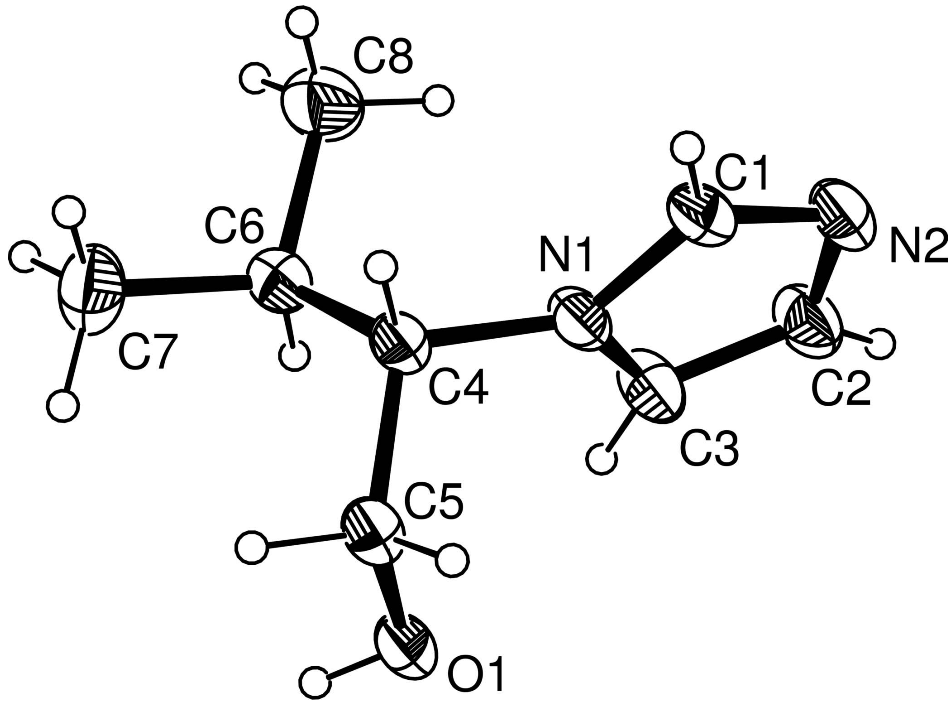

Figure 1

The molecular structure of (I), showing 30% probability displacement ellipsoids and the atomic numbering.

rac-(*S*)-2-(1*H*-Imidazol-1-yl)-3-methylbutan-1-ol

Crystal data

$C_8H_{14}N_2O$
M_r = 154.21
Monoclinic, $P2_1/n$
Hall symbol: -P 2yn
a = 7.356 (4) Å
b = 7.212 (3) Å
c = 16.549 (5) Å
β = 90.54 (3)°
V = 877.9 (7) Å^3
Z = 4
F(000) = 336
D_x = 1.167 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 21 reflections
θ = 4.6–7.4°
μ = 0.08 mm^{-1}
T = 292 K
Block, colourless
0.58 × 0.54 × 0.42 mm

Data collection

Enraf–Nonius CAD-4
diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
$\omega/2\theta$ scans
1931 measured reflections
1630 independent reflections
965 reflections with $I > 2\sigma(I)$
R_{int} = 0.004
θ_{max} = 25.5°, θ_{min} = 2.5°
h = −8→8
k = 0→8
l = −7→20
3 standard reflections every 120 reflections
intensity decay: 0.3%

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.067$
$wR(F^2) = 0.197$
$S = 1.18$
1630 reflections
104 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0941P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.31$ e Å^{-3}
$\Delta\rho_{min} = -0.24$ e Å^{-3}
Extinction correction: *SHELXS97* (Sheldrick, 2008), $Fc^* = kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.046 (11)

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}
O1	0.4065 (2)	0.1870 (3)	0.31165 (14)	0.0557 (7)
H1	0.3011	0.1697	0.3255	0.084*
N1	0.7629 (3)	0.0340 (3)	0.34211 (14)	0.0459 (7)
N2	1.0479 (3)	0.1263 (4)	0.34916 (16)	0.0598 (8)
C4	0.6043 (3)	−0.0816 (4)	0.32307 (17)	0.0456 (8)
H4	0.6473	−0.1848	0.2898	0.055*
C5	0.4713 (4)	0.0284 (4)	0.27157 (18)	0.0480 (8)
H5A	0.5306	0.0660	0.2221	0.058*
H5B	0.3692	−0.0503	0.2570	0.058*
C1	0.9353 (4)	0.0047 (4)	0.31778 (19)	0.0514 (8)
H1A	0.9698	−0.0901	0.2830	0.062*
C3	0.7676 (4)	0.1855 (4)	0.39170 (18)	0.0559 (8)
H3	0.6696	0.2406	0.4174	0.067*
C6	0.5261 (4)	−0.1657 (4)	0.39954 (16)	0.0487 (8)
H6	0.4769	−0.0643	0.4322	0.058*
C2	0.9438 (4)	0.2392 (4)	0.3958 (2)	0.0597 (9)
H2	0.9875	0.3387	0.4260	0.072*
C7	0.3712 (5)	−0.2990 (5)	0.3805 (2)	0.0693 (10)
H7A	0.4164	−0.4013	0.3495	0.104*
H7B	0.2786	−0.2355	0.3500	0.104*
H7C	0.3208	−0.3444	0.4300	0.104*
C8	0.6711 (5)	−0.2629 (5)	0.4495 (2)	0.0761 (11)
H8A	0.6199	−0.3035	0.4996	0.114*

H8B	0.7693	−0.1786	0.4602	0.114*
H8C	0.7157	−0.3681	0.4202	0.114*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0302 (11)	0.0505 (12)	0.0864 (15)	0.0012 (9)	−0.0025 (10)	−0.0011 (11)
N1	0.0288 (13)	0.0493 (13)	0.0595 (15)	0.0029 (11)	−0.0026 (10)	−0.0055 (11)
N2	0.0307 (14)	0.0628 (16)	0.086 (2)	−0.0005 (12)	−0.0039 (13)	−0.0031 (14)
C4	0.0325 (15)	0.0424 (15)	0.0617 (18)	−0.0002 (12)	−0.0060 (13)	−0.0027 (14)
C5	0.0355 (16)	0.0499 (16)	0.0586 (17)	−0.0024 (13)	−0.0038 (13)	−0.0008 (14)
C1	0.0334 (16)	0.0518 (17)	0.069 (2)	0.0086 (14)	0.0035 (14)	−0.0014 (14)
C3	0.0353 (16)	0.0655 (19)	0.0669 (19)	0.0006 (15)	−0.0020 (14)	−0.0129 (16)
C6	0.0407 (16)	0.0520 (17)	0.0532 (17)	0.0002 (14)	−0.0043 (13)	0.0040 (14)
C2	0.0405 (17)	0.0628 (19)	0.075 (2)	−0.0027 (15)	−0.0125 (15)	−0.0133 (16)
C7	0.066 (2)	0.071 (2)	0.071 (2)	−0.0228 (19)	0.0010 (17)	0.0087 (18)
C8	0.063 (2)	0.085 (2)	0.081 (2)	0.0034 (19)	−0.0120 (19)	0.025 (2)

Geometric parameters (Å, º)

O1—C5	1.408 (3)	C3—C2	1.354 (4)
O1—H1	0.8200	C3—H3	0.9300
N1—C1	1.351 (4)	C6—C8	1.515 (4)
N1—C3	1.367 (3)	C6—C7	1.522 (4)
N1—C4	1.466 (3)	C6—H6	0.9800
N2—C1	1.310 (3)	C2—H2	0.9300
N2—C2	1.362 (4)	C7—H7A	0.9600
C4—C5	1.516 (3)	C7—H7B	0.9600
C4—C6	1.521 (4)	C7—H7C	0.9600
C4—H4	0.9800	C8—H8A	0.9600
C5—H5A	0.9700	C8—H8B	0.9600
C5—H5B	0.9700	C8—H8C	0.9600
C1—H1A	0.9300		
C5—O1—H1	109.5	N1—C3—H3	126.9
C1—N1—C3	106.6 (2)	C8—C6—C4	111.6 (2)
C1—N1—C4	126.5 (2)	C8—C6—C7	110.0 (3)
C3—N1—C4	126.8 (2)	C4—C6—C7	111.6 (2)
C1—N2—C2	105.6 (2)	C8—C6—H6	107.8
N1—C4—C5	109.4 (2)	C4—C6—H6	107.8
N1—C4—C6	110.8 (2)	C7—C6—H6	107.8
C5—C4—C6	115.4 (2)	C3—C2—N2	110.1 (3)
N1—C4—H4	107.0	C3—C2—H2	125.0
C5—C4—H4	107.0	N2—C2—H2	125.0
C6—C4—H4	107.0	C6—C7—H7A	109.5
O1—C5—C4	112.3 (2)	C6—C7—H7B	109.5
O1—C5—H5A	109.1	H7A—C7—H7B	109.5
C4—C5—H5A	109.1	C6—C7—H7C	109.5

O1—C5—H5B	109.1	H7A—C7—H7C	109.5
C4—C5—H5B	109.1	H7B—C7—H7C	109.5
H5A—C5—H5B	107.9	C6—C8—H8A	109.5
N2—C1—N1	111.6 (3)	C6—C8—H8B	109.5
N2—C1—H1A	124.2	H8A—C8—H8B	109.5
N1—C1—H1A	124.2	C6—C8—H8C	109.5
C2—C3—N1	106.1 (3)	H8A—C8—H8C	109.5
C2—C3—H3	126.9	H8B—C8—H8C	109.5
C1—N1—C4—C5	−115.0 (3)	C1—N1—C3—C2	−0.8 (3)
C3—N1—C4—C5	69.3 (3)	C4—N1—C3—C2	175.7 (2)
C1—N1—C4—C6	116.8 (3)	N1—C4—C6—C8	−51.7 (3)
C3—N1—C4—C6	−59.0 (3)	C5—C4—C6—C8	−176.6 (2)
N1—C4—C5—O1	−61.5 (3)	N1—C4—C6—C7	−175.2 (2)
C6—C4—C5—O1	64.2 (3)	C5—C4—C6—C7	59.9 (3)
C2—N2—C1—N1	0.0 (3)	N1—C3—C2—N2	0.8 (4)
C3—N1—C1—N2	0.5 (3)	C1—N2—C2—C3	−0.5 (4)
C4—N1—C1—N2	−175.9 (2)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1···N2^{i}	0.82	1.93	2.751 (3)	176
C1—H1*A*···O1^{ii}	0.93	2.43	3.353 (4)	173
C4—H4···*Cg*^{ii}	0.98	2.86	3.716 (4)	146

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