Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

(*S*)-*N*-(1-Hydroxymethyl-2-methylpropyl)-2-methoxybenzamide

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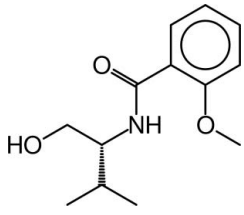
Received 8 March 2008; accepted 13 April 2008

Key indicators: single-crystal X-ray study; T = 291 K; mean σ(C–C) = 0.006 Å; R factor = 0.046; wR factor = 0.136; data-to-parameter ratio = 8.5.

The title compound, $C_{13}H_{19}NO_3$, is an important synthetic intermediate. Weak O—H···O and N—H···O hydrogen bonds enhance the stability of the crystal structure.

Related literature

For related literature, see: Ma & You (2007); Rechavi & Lemaire (2002).



Experimental

Crystal data

$C_{13}H_{19}NO_3$
M_r = 237.29
Orthorhombic, $P2_12_12_1$
a = 9.015 (4) Å
b = 10.386 (4) Å
c = 14.005 (4) Å
V = 1311.3 (9) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.09 mm^{-1}
T = 291 (2) K
0.50 × 0.44 × 0.40 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
Absorption correction: none
1457 measured reflections
1397 independent reflections
848 reflections with $I > 2\sigma(I)$
R_{int} = 0.010
3 standard reflections every 120 reflections
intensity decay: 0.4%

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.045
$wR(F^2)$ = 0.136
S = 1.02
1397 reflections
164 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.21 e Å^{-3}
$\Delta\rho_{min}$ = −0.14 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O3—H3···O2^{i}	0.82	2.00	2.806 (4)	170
N1—H1N1···O1	0.86	1.96	2.656 (4)	137

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

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*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: ER2052).

References

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Gabe, E. J., Le Page, Y., Charland, J.-P., Lee, F. L. & White, P. S. (1989). *J. Appl. Cryst.* **22**, 384–387.
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Rechavi, D. & Lemaire, M. (2002). *Chem. Rev.* **102**, 3467–3494.
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supporting information

Acta Cryst. (2008). E**64**, o1052 [doi:10.1107/S160053680801009X]

(*S*)-*N*-(1-Hydroxymethyl-2-methylpropyl)-2-methoxybenzamide

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S1. Comment

Oxazoline ligands have been proved to be a class of chiral ligands, being capable of forming a broad variety of metal complexes that are capable of catalyzing a great number of reactions with excellent enantioselectivity (Rechavi & Lemaire, 2002). It is believed that the oxazoline ring can be modified structurally by replacing the O atom with a substituted N atom, leading to new types of imidazoline ligands (Ma & You, 2007). However, all those ligands can prepared by this compound as an intermediate. Herein, we report the synthesis and structure of the title compound (I).

As shown in Fig. 1, there is a chiral center at C9 derived from *L*-valinol. The C—N bond lengths are 1.318 (4) Å and 1.463 (4) Å, and the C8—N1—C9 angle is 125.3 (3) °. A combination of O—H···O and N—H···O hydrogen bonds interactions provide packing forces in the crystal structure of the title compound.

S2. Experimental

NaH (8.7 g, 60%, 0.216 mol) was added portionwise to a stirred solution of *L*-valinol (22.1 g, 0.215 mol) in dry THF (120 ml). The mixture was stirred at ambient temperature for 1 h. To this solution was added 2-Methoxy-benzoic acid methyl ester (17.8 g, 0.107 mol) dissolved in THF (50 ml). The mixture was refluxed for 12 h under nitrogen, quenched with H_2O (10 ml) and concentrated by evaporation of the solvent. The residue was dissolved in CH_2Cl_2 (100 ml), washed with H_2O, brine, and dried over $MgSO_4$. And then removal of the solvent *in vacuo* gave a white solid, which was recrystallized from ethyl acetate and petroleum ether to afford the title compound as white crystals (22.8 g, 90%).

S3. Refinement

H atoms were positioned geometrically and refined in the riding model approximation with O—H = 0.82 Å, N—H = 0.86 Å, and C—H = 0.93, 0.96, 0.97 or 0.98 Å. The U_{iso}(H) = 1.5 U_{eq}(C) for the CH_3 while it was set to 1.2 U_{eq}(C,*N*,*O*) for all other H atoms. Due to abscence of significant anomalous dispersion effects, the reflection data were merged.

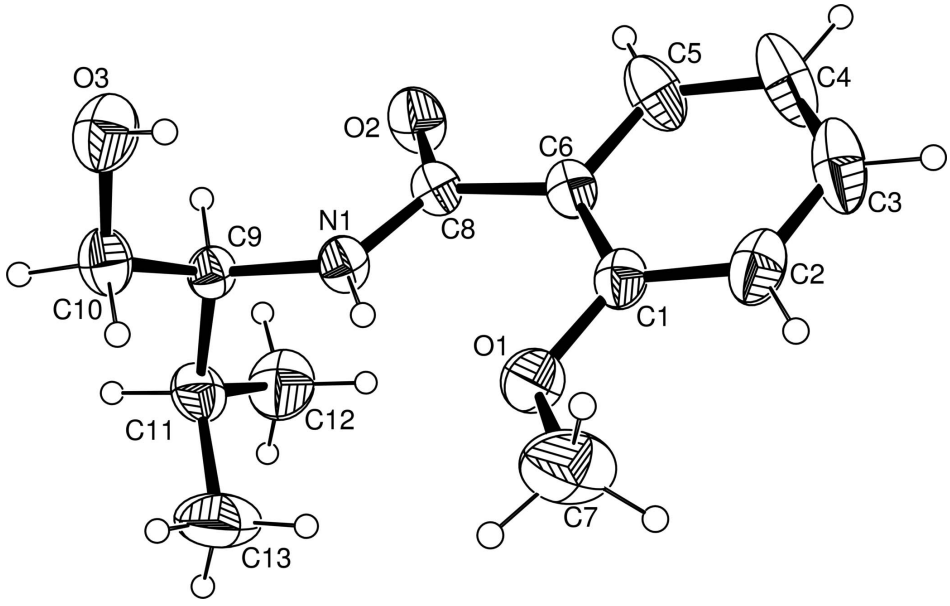

Figure 1

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

(*S*)-*N*-(1-Hydroxymethyl-2-methylpropyl)-2-methoxybenzamide

Crystal data

$C_{13}H_{19}NO_3$
$M_r = 237.29$
Orthorhombic, $P2_12_12_1$
Hall symbol: P 2ac 2ab
$a = 9.015$ (4) Å
$b = 10.386$ (4) Å
$c = 14.005$ (4) Å
$V = 1311.3$ (9) Å^3
$Z = 4$
$F(000) = 512$
$D_x = 1.202$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 25 reflections
$\theta = 4.5$–$6.7°$
$\mu = 0.09$ mm^{-1}
$T = 291$ K
Block, colourless
0.50 × 0.44 × 0.40 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
$\omega/2\theta$ scans
1457 measured reflections
1397 independent reflections
848 reflections with $I > 2\sigma(I)$
$R_{int} = 0.010$
$\theta_{max} = 25.5°$, $\theta_{min} = 2.4°$
$h = -3\rightarrow10$
$k = -3\rightarrow12$
$l = -5\rightarrow16$
3 standard reflections every 120 reflections
intensity decay: 0.4%

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.045$
$wR(F^2) = 0.136$
$S = 1.02$
1397 reflections
164 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

$w = 1/[\sigma^2(F_o^2) + (0.0778P)^2 + 0.0096P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.21$ e Å^{-3}
$\Delta\rho_{min} = -0.14$ e Å^{-3}
Extinction correction: *SHELXL97* (Sheldrick, 2008), $Fc^* = kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.069 (8)

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	0.1432 (3)	0.3872 (3)	0.16126 (19)	0.0666 (8)
O2	0.5192 (3)	0.3286 (3)	−0.00264 (18)	0.0703 (8)
O3	0.2632 (4)	0.0046 (3)	−0.0111 (2)	0.0810 (10)
H3	0.1945	0.0562	−0.0140	0.097*
N1	0.3536 (3)	0.2340 (2)	0.0926 (2)	0.0487 (8)
H1N1	0.2711	0.2433	0.1227	0.058*
C1	0.1842 (4)	0.4786 (3)	0.0963 (3)	0.0514 (9)
C2	0.1074 (5)	0.5935 (4)	0.0851 (3)	0.0709 (12)
H2	0.0240	0.6099	0.1223	0.085*
C3	0.1535 (6)	0.6826 (4)	0.0201 (4)	0.0878 (16)
H3A	0.1009	0.7591	0.0133	0.105*
C4	0.2760 (6)	0.6610 (4)	−0.0355 (4)	0.0929 (18)
H4	0.3078	0.7226	−0.0791	0.112*
C5	0.3519 (5)	0.5457 (4)	−0.0255 (3)	0.0745 (13)
H5	0.4337	0.5300	−0.0642	0.089*
C6	0.3096 (4)	0.4533 (3)	0.0402 (3)	0.0489 (9)
C7	0.0018 (6)	0.3961 (7)	0.2048 (3)	0.109 (2)
H7A	−0.0038	0.4739	0.2416	0.163*
H7B	−0.0130	0.3233	0.2460	0.163*
H7C	−0.0735	0.3970	0.1564	0.163*
C8	0.4020 (4)	0.3328 (3)	0.0425 (2)	0.0459 (9)
C9	0.4288 (4)	0.1097 (3)	0.1012 (2)	0.0456 (8)
H9	0.5050	0.1060	0.0514	0.055*
C10	0.3202 (5)	0.0025 (3)	0.0824 (3)	0.0609 (10)
H10A	0.3691	−0.0794	0.0932	0.073*
H10B	0.2387	0.0091	0.1273	0.073*
C11	0.5075 (5)	0.0978 (4)	0.1981 (3)	0.0632 (11)
H11	0.5515	0.0115	0.2002	0.076*
C12	0.6339 (6)	0.1925 (5)	0.2074 (4)	0.0939 (16)
H12A	0.5957	0.2787	0.2045	0.141*
H12B	0.7031	0.1794	0.1562	0.141*

H12C	0.6832	0.1796	0.2674	0.141*
C13	0.4068 (6)	0.1084 (6)	0.2833 (3)	0.107 (2)
H13A	0.4630	0.0946	0.3406	0.160*
H13B	0.3299	0.0447	0.2788	0.160*
H13C	0.3631	0.1926	0.2849	0.160*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0560 (16)	0.0735 (18)	0.0704 (16)	0.0177 (16)	0.0102 (13)	0.0020 (15)
O2	0.0553 (16)	0.0652 (18)	0.0903 (18)	−0.0022 (15)	0.0249 (16)	0.0137 (16)
O3	0.072 (2)	0.071 (2)	0.099 (2)	0.0007 (16)	−0.0180 (18)	−0.0113 (17)
N1	0.0369 (15)	0.0481 (16)	0.0610 (17)	0.0057 (15)	0.0078 (14)	0.0039 (14)
C1	0.050 (2)	0.046 (2)	0.058 (2)	−0.0001 (18)	−0.0117 (19)	−0.0054 (18)
C2	0.064 (3)	0.060 (3)	0.089 (3)	0.015 (2)	−0.016 (2)	−0.019 (2)
C3	0.070 (3)	0.048 (2)	0.146 (4)	0.002 (2)	−0.043 (3)	0.008 (3)
C4	0.067 (3)	0.059 (3)	0.152 (5)	−0.011 (3)	−0.030 (3)	0.047 (3)
C5	0.053 (2)	0.066 (2)	0.104 (3)	−0.010 (2)	−0.011 (2)	0.031 (3)
C6	0.044 (2)	0.0442 (18)	0.059 (2)	−0.0051 (17)	−0.0149 (17)	0.0013 (17)
C7	0.077 (3)	0.156 (6)	0.094 (3)	0.036 (4)	0.030 (3)	0.020 (4)
C8	0.037 (2)	0.046 (2)	0.054 (2)	−0.0041 (17)	−0.0023 (16)	0.0044 (18)
C9	0.0387 (18)	0.0447 (19)	0.0534 (19)	0.0069 (17)	0.0040 (15)	−0.0004 (17)
C10	0.053 (2)	0.050 (2)	0.080 (3)	0.0044 (19)	0.000 (2)	0.004 (2)
C11	0.062 (3)	0.061 (3)	0.067 (2)	0.016 (2)	−0.009 (2)	0.011 (2)
C12	0.096 (3)	0.093 (3)	0.093 (3)	0.000 (3)	−0.037 (3)	−0.011 (3)
C13	0.120 (4)	0.146 (5)	0.054 (2)	0.031 (5)	0.004 (3)	0.016 (3)

Geometric parameters (Å, º)

O1—C1	1.365 (4)	C6—C8	1.504 (5)
O1—C7	1.416 (5)	C7—H7A	0.9600
O2—C8	1.232 (4)	C7—H7B	0.9600
O3—C10	1.406 (5)	C7—H7C	0.9600
O3—H3	0.8200	C9—C10	1.506 (5)
N1—C8	1.318 (4)	C9—C11	1.537 (5)
N1—C9	1.463 (4)	C9—H9	0.9800
N1—H1N1	0.8600	C10—H10A	0.9700
C1—C2	1.389 (5)	C10—H10B	0.9700
C1—C6	1.402 (5)	C11—C13	1.503 (6)
C2—C3	1.363 (6)	C11—C12	1.511 (6)
C2—H2	0.9300	C11—H11	0.9800
C3—C4	1.369 (7)	C12—H12A	0.9600
C3—H3A	0.9300	C12—H12B	0.9600
C4—C5	1.386 (6)	C12—H12C	0.9600
C4—H4	0.9300	C13—H13A	0.9600
C5—C6	1.383 (5)	C13—H13B	0.9600
C5—H5	0.9300	C13—H13C	0.9600

C1—O1—C7	119.1 (4)	N1—C8—C6	118.4 (3)
C10—O3—H3	109.5	N1—C9—C10	109.7 (3)
C8—N1—C9	125.3 (3)	N1—C9—C11	111.0 (3)
C8—N1—H1N1	117.4	C10—C9—C11	113.3 (3)
C9—N1—H1N1	117.4	N1—C9—H9	107.5
O1—C1—C2	122.5 (4)	C10—C9—H9	107.5
O1—C1—C6	117.5 (3)	C11—C9—H9	107.5
C2—C1—C6	120.0 (4)	O3—C10—C9	112.9 (3)
C3—C2—C1	120.4 (4)	O3—C10—H10A	109.0
C3—C2—H2	119.8	C9—C10—H10A	109.0
C1—C2—H2	119.8	O3—C10—H10B	109.0
C2—C3—C4	121.0 (4)	C9—C10—H10B	109.0
C2—C3—H3A	119.5	H10A—C10—H10B	107.8
C4—C3—H3A	119.5	C13—C11—C12	109.8 (4)
C3—C4—C5	118.8 (4)	C13—C11—C9	114.6 (3)
C3—C4—H4	120.6	C12—C11—C9	111.8 (3)
C5—C4—H4	120.6	C13—C11—H11	106.7
C6—C5—C4	122.0 (5)	C12—C11—H11	106.7
C6—C5—H5	119.0	C9—C11—H11	106.7
C4—C5—H5	119.0	C11—C12—H12A	109.5
C5—C6—C1	117.7 (4)	C11—C12—H12B	109.5
C5—C6—C8	116.0 (3)	H12A—C12—H12B	109.5
C1—C6—C8	126.2 (3)	C11—C12—H12C	109.5
O1—C7—H7A	109.5	H12A—C12—H12C	109.5
O1—C7—H7B	109.5	H12B—C12—H12C	109.5
H7A—C7—H7B	109.5	C11—C13—H13A	109.5
O1—C7—H7C	109.5	C11—C13—H13B	109.5
H7A—C7—H7C	109.5	H13A—C13—H13B	109.5
H7B—C7—H7C	109.5	C11—C13—H13C	109.5
O2—C8—N1	122.0 (3)	H13A—C13—H13C	109.5
O2—C8—C6	119.6 (3)	H13B—C13—H13C	109.5

C7—O1—C1—C2	13.4 (5)	C9—N1—C8—C6	179.2 (3)
C7—O1—C1—C6	−167.0 (4)	C5—C6—C8—O2	9.9 (5)
O1—C1—C2—C3	179.3 (3)	C1—C6—C8—O2	−171.7 (3)
C6—C1—C2—C3	−0.3 (6)	C5—C6—C8—N1	−169.6 (3)
C1—C2—C3—C4	−0.0 (6)	C1—C6—C8—N1	8.8 (5)
C2—C3—C4—C5	0.9 (7)	C8—N1—C9—C10	−130.9 (4)
C3—C4—C5—C6	−1.5 (7)	C8—N1—C9—C11	103.2 (4)
C4—C5—C6—C1	1.2 (6)	N1—C9—C10—O3	63.2 (4)
C4—C5—C6—C8	179.7 (4)	C11—C9—C10—O3	−172.2 (3)
O1—C1—C6—C5	−179.8 (3)	N1—C9—C11—C13	59.7 (4)
C2—C1—C6—C5	−0.2 (5)	C10—C9—C11—C13	−64.2 (5)
O1—C1—C6—C8	1.8 (5)	N1—C9—C11—C12	−66.1 (4)
C2—C1—C6—C8	−178.6 (3)	C10—C9—C11—C12	170.0 (3)
C9—N1—C8—O2	−0.3 (5)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O3—H3···O2^{i}	0.82	2.00	2.806 (4)	170
N1—H1*N*1···O1	0.86	1.96	2.656 (4)	137

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