organic compounds

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

Jihong Li, Wenhai Wang, Jingbo Lan* and Jingsong You

Key Laboratory of Green Chemistry and Technology of the Ministry of Education, College of Chemistry, Sichuan University, Chengdu 610064, People's Republic of China
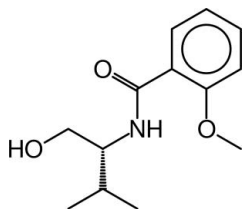
Correspondence e-mail: jingbolan@scu.edu.cn

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

$C_{13}H_{19}NO_3$	a = 9.015 (4) Å
M_r = 237.29	b = 10.386 (4) Å
Orthorhombic, $P2_12_12_1$	c = 14.005 (4) Å

V = 1311.3 (9) Å^3	μ = 0.09 mm^{-1}
Z = 4	T = 291 (2) K
Mo $K\alpha$ radiation	0.50 × 0.44 × 0.40 mm

Data collection

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

Refinement

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

Burnett, M. N. & Johnson, C. K. (1996). *ORTEPIII*. Report ORNL-6895. Oak Ridge National Laboratory, Tennessee, USA.

Gabe, E. J., Le Page, Y., Charland, J.-P., Lee, F. L. & White, P. S. (1989). *J. Appl. Cryst.* **22**, 384–387.

Gabe, E. J. & White, P. S. (1993). *DIFRAC*. American Crystallographic Association, Pittsburgh Meeting. Abstract PA104.

Ma, K. & You, J. (2007). *Chem. Eur. J.* **13**, 1863–1871.

Rechavi, D. & Lemaire, M. (2002). *Chem. Rev.* **102**, 3467–3494.

Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.

supplementary materials

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

J. Li, W. Wang, J. Lan and J. You

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.

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%).

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.

Figures

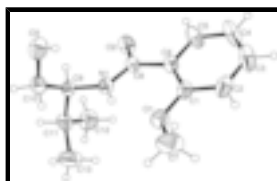Fig. 1. The molecular structure of (I), showing 30% probability displacement ellipsoids and the atomic numbering.

supplementary materials

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

Crystal data

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

Data collection

Enraf–Nonius CAD-4 diffractometer	$R_{int} = 0.010$
Radiation source: fine-focus sealed tube	$\theta_{max} = 25.5°$
Monochromator: graphite	$\theta_{min} = 2.4°$
$T = 291(2)$ K	$h = -3 \rightarrow 10$
$\omega/2\theta$ scans	$k = -3 \rightarrow 12$
Absorption correction: none	$l = -5 \rightarrow 16$
1457 measured reflections	3 standard reflections
1397 independent reflections	every 120 reflections
848 reflections with $I > 2\sigma(I)$	intensity decay: 0.4%

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)] = 0.045$	$w = 1/[\sigma^2(F_o^2) + (0.0778P)^2 + 0.0096P]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.136$	$(\Delta/\sigma)_{max} < 0.001$
$S = 1.02$	$\Delta\rho_{max} = 0.21$ e Å^{-3}
1397 reflections	$\Delta\rho_{min} = -0.14$ e Å^{-3}
164 parameters	Extinction correction: SHELXL97 (Sheldrick, 2008), Fc*=kFc[1+0.001xFc$^2\lambda^3$/sin(2θ)]$^{-1/4}$
Primary atom site location: structure-invariant direct methods	Extinction coefficient: 0.069 (8)
Secondary atom site location: difference Fourier map	

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 (Å^2)

	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, °)

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, °)

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 codes: (i) $x-1/2$, $-y+1/2$, $-z$.

Fig. 1