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3-Cyclopropyl-1-(4-methylphenylsulfonyl)piperidine-3,5-diol

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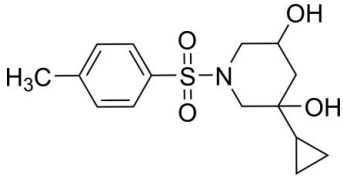
Key indicators: single-crystal X-ray study; T = 173 K; mean σ(C–C) = 0.005 Å; R factor = 0.069; wR factor = 0.128; data-to-parameter ratio = 14.0.

In the title compound, $C_{15}H_{21}NO_4S$, both hydroxy groups on the piperidine ring are located in axial positions, whereas the tosyl group and the cyclopropane ring are in equatorial positions. An intramolecular O—H···O hydrogen bond occurs. In the crystal, molecules form inversion dimers *via* pairs of O—H···O hydrogen bonds, generating cyclic $R_4^4(8)$ motifs, as noted previously in related diols.

Related literature

Azacyclohexanediol (piperidinediol) derivatives are widely found in natural products and are often incorporated into drugs, see: Nagahama *et al.* (2003); Fukushima *et al.* (2001). For related structures, see: Hidekazu *et al.* (2005); Karin *et al.* (2006). Similar hydrogen bonding has been seen in related diols, see: Ferguson *et al.* (1993).



Experimental

Crystal data

$C_{15}H_{21}NO_4S$
M_r = 311.39
Monoclinic, $P2_1/n$
a = 11.583 (5) Å
b = 5.598 (2) Å
c = 24.009 (9) Å
β = 102.905 (7)°
V = 1517.5 (10) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.23 mm^{-1}
T = 173 K
0.24 × 0.11 × 0.06 mm

Data collection

Rigaku MM007-HF CCD (Saturn 724+) diffractometer
Absorption correction: numerical (*CrystalClear*; Rigaku, 2002)
T_{min} = 0.947, T_{max} = 0.986
9795 measured reflections
2676 independent reflections
2431 reflections with $I > 2\sigma(I)$
R_{int} = 0.053

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.069
$wR(F^2)$ = 0.128
S = 1.23
2676 reflections
191 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.42 e Å^{-3}
$\Delta\rho_{min}$ = −0.32 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O3—H3B···O4	0.84	2.08	2.804 (3)	145
O4—H4···O3^{i}	0.84	2.01	2.837 (3)	167

Symmetry code: (i) $-x+1, -y+1, -z$.

Data collection: *CrystalClear* (Rigaku, 2007); cell refinement: *CrystalClear*; data reduction: *CrystalClear*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *DIAMOND* (Brandenburg, 1999); software used to prepare material for publication: *SHELXL97*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: GG2064).

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supporting information

Acta Cryst. (2012). E**68**, o231 [doi:10.1107/S1600536811054420]

3-Cyclopropyl-1-(4-methylphenylsulfonyl)piperidine-3,5-diol

Yi Wang and Yong-Yue Lin

S1. Comment

Azacyclohexanediol (piperidinediol) derivatives are widely found in natural products and often incorporated into drugs (Nagahama *et al.*, 2003). We report here the crystal structure of the title compound, a 1,3-piperidinediol derivative.

Bond lengths and angles were normal. The distance of N1—C8 and N1—C12 are 1.471 (4) and 1.478 (4) Å, respectively, which are in good agreement with normal N—C bond lengths [reported values range from 1.33 to 1.52 Å]. As shown as in Fig. 1, the two hydroxy groups in piperidine ring adopt a *cis* conformation. The twist angles of hydroxy groups and the piperidine are 112.0 (3) and 110.3 (3), respectively. Thus, the orientation of both hydroxy groups are axial in reference to the piperidine ring. A comparison with analogous 1,3-cyclohexanediol systems, shows that our observed system is in good agreement with similar 1,3-cyclohexanediols (Hidekazu *et al.*, 2005; Karin *et al.*, 2006). The cyclopropane C13—C15 planes is nearly coplanar with the azacyclohexane ring. A centrosymmetric dimer is generated by intermolecular O—H—O hydrogen bonding, and this has been seen previously in related diols (Ferguson *et al.*, 1993), as shown in Fig. 2.

S2. Experimental

A mixture of *N*-(2-cyclopropylallyl)-4-methyl-N–(2-oxoethyl)benzenesulfonamide (1 mmol), ferric chloride hexahydrate (0.1mmol) was stirred in dichloromethane (5 ml) for 24 h. The mixture was concentrated and the residue was purified by flash column chromatography with silica gel to afford the title products (yield 55%). Single crystals suitable for X-ray diffraction were grown by slow diffusion of ether into a solution of the compound in dichloromethane.

S3. Refinement

All H atoms were placed in calculated positions, with C—H = 0.93—0.98 Å, and refined using a riding model, with U_{iso}(H) = 1.2U_{eq}(C).

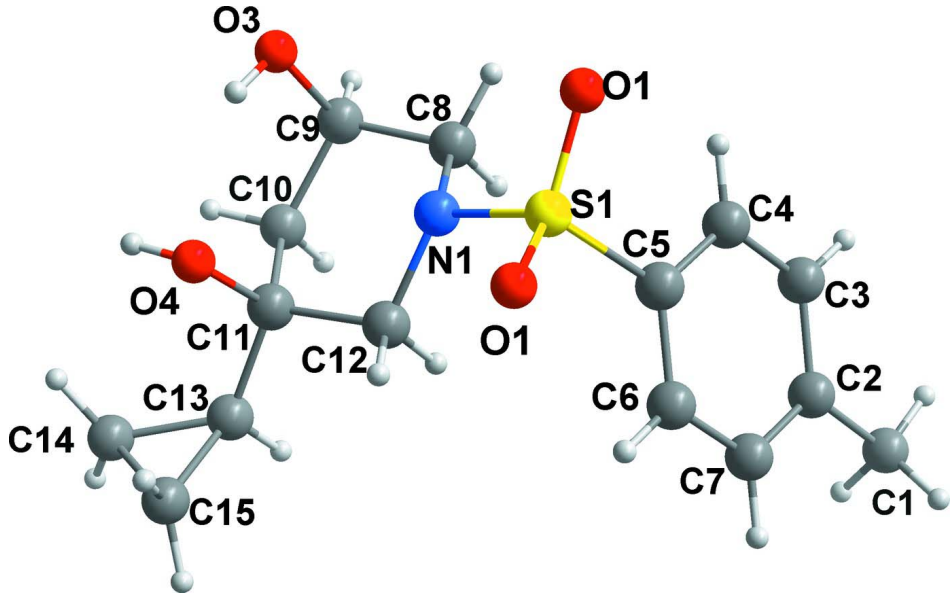

Figure 1

The molecular structure of the title compound, with atom labels and 30% probability displacement ellipsoids for non-H atoms.

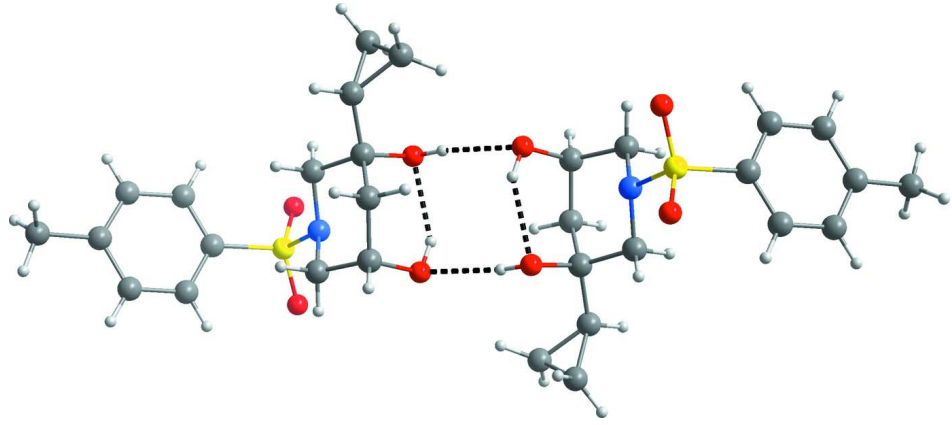

Figure 2

The dimeric unit showing the hydrogen bonding as dashed lines.

3-Cyclopropyl-1-(4-methylphenylsulfonyl)piperidine-3,5-diol

Crystal data

$C_{15}H_{21}NO_4S$
$M_r = 311.39$
Monoclinic, $P2_1/n$
Hall symbol: -p 2yn
$a = 11.583$ (5) Å
$b = 5.598$ (2) Å
$c = 24.009$ (9) Å
$\beta = 102.905$ (7)°
$V = 1517.5$ (10) Å^3
$Z = 4$

$F(000) = 664$
$D_x = 1.363$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 345 reflections
$\theta = 2.2$–27.5°
$\mu = 0.23$ mm^{-1}
$T = 173$ K
Plate, colorless
0.24 × 0.11 × 0.06 mm

Data collection

Rigaku MM007-HF CCD (Saturn 724+) diffractometer
Radiation source: rotating anode
Confocal monochromator
ω scans at fixed $\chi = 45°$
Absorption correction: numerical (*CrystalClear*; Rigaku, 2002)
$T_{min} = 0.947$, $T_{max} = 0.986$
9795 measured reflections
2676 independent reflections
2431 reflections with $I > 2\sigma(I)$
$R_{int} = 0.053$
$\theta_{max} = 25.0°$, $\theta_{min} = 1.7°$
$h = -13 \rightarrow 13$
$k = -6 \rightarrow 6$
$l = -28 \rightarrow 25$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.069$
$wR(F^2) = 0.128$
$S = 1.23$
2676 reflections
191 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
$w = 1/[\sigma^2(F_o^2) + (0.0211P)^2 + 2.2306P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.42$ e Å^{-3}
$\Delta\rho_{min} = -0.32$ e Å^{-3}

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
S1	0.48729 (7)	0.71212 (14)	0.21603 (3)	0.0254 (2)
O1	0.3693 (2)	0.8072 (4)	0.20391 (9)	0.0309 (6)
O2	0.5867 (2)	0.8711 (4)	0.22413 (9)	0.0333 (6)
O3	0.6103 (2)	0.5720 (4)	0.06890 (9)	0.0323 (6)
H3B	0.5388	0.5980	0.0535	0.049*
O4	0.3664 (2)	0.4775 (4)	0.04568 (9)	0.0293 (5)
H4	0.3722	0.4391	0.0126	0.044*
N1	0.4962 (2)	0.5392 (5)	0.16189 (10)	0.0239 (6)
C1	0.5488 (4)	0.0129 (7)	0.41161 (16)	0.0499 (11)
H1A	0.5012	0.0572	0.4390	0.075*
H1B	0.6325	0.0049	0.4312	0.075*
H1C	0.5231	−0.1434	0.3950	0.075*
C2	0.5327 (3)	0.1979 (6)	0.36466 (13)	0.0336 (8)
C3	0.6307 (3)	0.3092 (6)	0.35169 (13)	0.0314 (8)
H3A	0.7078	0.2742	0.3736	0.038*
C4	0.6166 (3)	0.4712 (6)	0.30688 (13)	0.0291 (8)
H4A	0.6839	0.5447	0.2976	0.035*
C5	0.5040 (3)	0.5246 (6)	0.27584 (12)	0.0242 (7)
C6	0.4056 (3)	0.4195 (6)	0.28916 (14)	0.0331 (8)
H6A	0.3282	0.4584	0.2682	0.040*
C7	0.4216 (3)	0.2568 (7)	0.33361 (14)	0.0390 (9)
H7A	0.3543	0.1840	0.3429	0.047*
C8	0.6136 (3)	0.4325 (6)	0.16365 (13)	0.0284 (8)
H8A	0.6255	0.2908	0.1890	0.034*
H8B	0.6768	0.5498	0.1788	0.034*

C9	0.6192 (3)	0.3603 (6)	0.10331 (13)	0.0296 (8)
H9A	0.6973	0.2823	0.1043	0.036*
C10	0.5208 (3)	0.1832 (6)	0.07993 (14)	0.0298 (8)
H10A	0.5367	0.0323	0.1018	0.036*
H10B	0.5213	0.1474	0.0396	0.036*
C11	0.3986 (3)	0.2754 (6)	0.08316 (13)	0.0262 (7)
C12	0.4001 (3)	0.3648 (6)	0.14376 (12)	0.0255 (7)
H12A	0.3231	0.4402	0.1445	0.031*
H12B	0.4118	0.2281	0.1706	0.031*
C13	0.3070 (3)	0.0790 (6)	0.06840 (13)	0.0308 (8)
H13A	0.3219	−0.0615	0.0948	0.037*
C14	0.2511 (3)	0.0174 (7)	0.00777 (14)	0.0370 (9)
H14A	0.2363	−0.1534	−0.0019	0.044*
H14B	0.2723	0.1158	−0.0227	0.044*
C15	0.1790 (3)	0.1328 (7)	0.04522 (15)	0.0415 (9)
H15A	0.1560	0.3021	0.0377	0.050*
H15B	0.1199	0.0330	0.0585	0.050*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0328 (5)	0.0215 (4)	0.0217 (4)	0.0001 (4)	0.0057 (3)	−0.0003 (3)
O1	0.0350 (14)	0.0271 (13)	0.0300 (12)	0.0092 (11)	0.0062 (10)	−0.0013 (10)
O2	0.0402 (15)	0.0251 (12)	0.0354 (13)	−0.0088 (11)	0.0105 (11)	−0.0012 (10)
O3	0.0342 (14)	0.0349 (14)	0.0274 (12)	0.0003 (11)	0.0057 (10)	0.0043 (10)
O4	0.0428 (14)	0.0237 (12)	0.0214 (11)	0.0061 (11)	0.0074 (10)	0.0032 (9)
N1	0.0276 (15)	0.0247 (15)	0.0201 (13)	0.0006 (12)	0.0066 (11)	0.0001 (11)
C1	0.058 (3)	0.050 (3)	0.040 (2)	0.000 (2)	0.0071 (19)	0.0173 (19)
C2	0.039 (2)	0.038 (2)	0.0236 (17)	0.0013 (18)	0.0070 (15)	0.0045 (16)
C3	0.0287 (19)	0.040 (2)	0.0234 (16)	0.0039 (16)	0.0011 (14)	0.0009 (15)
C4	0.0287 (19)	0.0338 (19)	0.0257 (17)	−0.0059 (16)	0.0083 (14)	−0.0033 (15)
C5	0.0292 (18)	0.0250 (17)	0.0182 (15)	−0.0005 (15)	0.0050 (13)	−0.0019 (13)
C6	0.0257 (19)	0.044 (2)	0.0289 (18)	−0.0042 (17)	0.0041 (14)	0.0028 (16)
C7	0.032 (2)	0.051 (2)	0.0355 (19)	−0.0083 (19)	0.0124 (16)	0.0103 (18)
C8	0.0316 (19)	0.0290 (19)	0.0243 (16)	0.0032 (15)	0.0058 (14)	0.0022 (15)
C9	0.034 (2)	0.0289 (19)	0.0269 (17)	0.0089 (15)	0.0084 (15)	0.0039 (14)
C10	0.040 (2)	0.0239 (18)	0.0265 (17)	0.0053 (15)	0.0102 (15)	−0.0025 (14)
C11	0.0379 (19)	0.0191 (17)	0.0216 (16)	0.0025 (15)	0.0064 (14)	0.0030 (13)
C12	0.0289 (19)	0.0237 (17)	0.0238 (16)	0.0004 (14)	0.0056 (14)	0.0006 (13)
C13	0.042 (2)	0.0240 (18)	0.0244 (17)	−0.0001 (16)	0.0030 (15)	0.0018 (14)
C14	0.045 (2)	0.032 (2)	0.0307 (19)	0.0002 (18)	0.0025 (16)	−0.0053 (16)
C15	0.038 (2)	0.041 (2)	0.046 (2)	−0.0009 (18)	0.0112 (18)	−0.0024 (18)

Geometric parameters (Å, º)

S1—O2	1.434 (2)	C6—H6A	0.9500
S1—O1	1.435 (2)	C7—H7A	0.9500
S1—N1	1.642 (3)	C8—C9	1.519 (4)

S1—C5	1.754 (3)	C8—H8A	0.9900
O3—C9	1.435 (4)	C8—H8B	0.9900
O3—H3B	0.8400	C9—C10	1.520 (5)
O4—C11	1.442 (4)	C9—H9A	1.0000
O4—H4	0.8401	C10—C11	1.526 (4)
N1—C12	1.471 (4)	C10—H10A	0.9900
N1—C8	1.478 (4)	C10—H10B	0.9900
C1—C2	1.511 (5)	C11—C13	1.514 (5)
C1—H1A	0.9800	C11—C12	1.535 (4)
C1—H1B	0.9800	C12—H12A	0.9900
C1—H1C	0.9800	C12—H12B	0.9900
C2—C7	1.375 (5)	C13—C15	1.493 (5)
C2—C3	1.389 (5)	C13—C14	1.495 (4)
C3—C4	1.389 (4)	C13—H13A	1.0000
C3—H3A	0.9500	C14—C15	1.502 (5)
C4—C5	1.383 (4)	C14—H14A	0.9900
C4—H4A	0.9500	C14—H14B	0.9900
C5—C6	1.382 (4)	C15—H15A	0.9900
C6—C7	1.384 (5)	C15—H15B	0.9900
O2—S1—O1	119.80 (14)	O3—C9—C10	112.0 (3)
O2—S1—N1	106.43 (13)	C8—C9—C10	109.8 (3)
O1—S1—N1	106.46 (13)	O3—C9—H9A	108.9
O2—S1—C5	108.78 (15)	C8—C9—H9A	108.9
O1—S1—C5	108.30 (14)	C10—C9—H9A	108.9
N1—S1—C5	106.28 (14)	C9—C10—C11	112.8 (3)
C9—O3—H3B	109.5	C9—C10—H10A	109.0
C11—O4—H4	109.0	C11—C10—H10A	109.0
C12—N1—C8	111.8 (2)	C9—C10—H10B	109.0
C12—N1—S1	116.65 (19)	C11—C10—H10B	109.0
C8—N1—S1	115.8 (2)	H10A—C10—H10B	107.8
C2—C1—H1A	109.5	O4—C11—C13	110.7 (3)
C2—C1—H1B	109.5	O4—C11—C10	110.3 (2)
H1A—C1—H1B	109.5	C13—C11—C10	110.6 (3)
C2—C1—H1C	109.5	O4—C11—C12	106.4 (2)
H1A—C1—H1C	109.5	C13—C11—C12	108.5 (3)
H1B—C1—H1C	109.5	C10—C11—C12	110.1 (3)
C7—C2—C3	118.9 (3)	N1—C12—C11	110.2 (2)
C7—C2—C1	120.9 (3)	N1—C12—H12A	109.6
C3—C2—C1	120.2 (3)	C11—C12—H12A	109.6
C4—C3—C2	120.4 (3)	N1—C12—H12B	109.6
C4—C3—H3A	119.8	C11—C12—H12B	109.6
C2—C3—H3A	119.8	H12A—C12—H12B	108.1
C5—C4—C3	119.5 (3)	C15—C13—C14	60.4 (2)
C5—C4—H4A	120.3	C15—C13—C11	121.7 (3)
C3—C4—H4A	120.3	C14—C13—C11	121.6 (3)
C6—C5—C4	120.7 (3)	C15—C13—H13A	114.2
C6—C5—S1	119.9 (2)	C14—C13—H13A	114.2

C4—C5—S1	119.3 (2)	C11—C13—H13A	114.2
C5—C6—C7	118.9 (3)	C13—C14—C15	59.8 (2)
C5—C6—H6A	120.6	C13—C14—H14A	117.8
C7—C6—H6A	120.6	C15—C14—H14A	117.8
C2—C7—C6	121.6 (3)	C13—C14—H14B	117.8
C2—C7—H7A	119.2	C15—C14—H14B	117.8
C6—C7—H7A	119.2	H14A—C14—H14B	114.9
N1—C8—C9	108.4 (3)	C13—C15—C14	59.9 (2)
N1—C8—H8A	110.0	C13—C15—H15A	117.8
C9—C8—H8A	110.0	C14—C15—H15A	117.8
N1—C8—H8B	110.0	C13—C15—H15B	117.8
C9—C8—H8B	110.0	C14—C15—H15B	117.8
H8A—C8—H8B	108.4	H15A—C15—H15B	114.9
O3—C9—C8	108.4 (3)		

O2—S1—N1—C12	−177.7 (2)	C12—N1—C8—C9	64.1 (3)
O1—S1—N1—C12	−48.9 (2)	S1—N1—C8—C9	−159.1 (2)
C5—S1—N1—C12	66.4 (2)	N1—C8—C9—O3	63.8 (3)
O2—S1—N1—C8	47.6 (2)	N1—C8—C9—C10	−58.8 (3)
O1—S1—N1—C8	176.4 (2)	O3—C9—C10—C11	−66.3 (3)
C5—S1—N1—C8	−68.3 (2)	C8—C9—C10—C11	54.2 (4)
C7—C2—C3—C4	−2.1 (5)	C9—C10—C11—O4	66.2 (3)
C1—C2—C3—C4	177.1 (3)	C9—C10—C11—C13	−170.9 (3)
C2—C3—C4—C5	1.2 (5)	C9—C10—C11—C12	−50.9 (3)
C3—C4—C5—C6	0.3 (5)	C8—N1—C12—C11	−61.5 (3)
C3—C4—C5—S1	−175.3 (2)	S1—N1—C12—C11	162.1 (2)
O2—S1—C5—C6	156.2 (3)	O4—C11—C12—N1	−66.4 (3)
O1—S1—C5—C6	24.5 (3)	C13—C11—C12—N1	174.4 (3)
N1—S1—C5—C6	−89.5 (3)	C10—C11—C12—N1	53.2 (3)
O2—S1—C5—C4	−28.1 (3)	O4—C11—C13—C15	−32.1 (4)
O1—S1—C5—C4	−159.8 (2)	C10—C11—C13—C15	−154.7 (3)
N1—S1—C5—C4	86.1 (3)	C12—C11—C13—C15	84.4 (4)
C4—C5—C6—C7	−1.0 (5)	O4—C11—C13—C14	40.3 (4)
S1—C5—C6—C7	174.6 (3)	C10—C11—C13—C14	−82.3 (4)
C3—C2—C7—C6	1.5 (6)	C12—C11—C13—C14	156.8 (3)
C1—C2—C7—C6	−177.7 (3)	C11—C13—C14—C15	−111.1 (4)
C5—C6—C7—C2	0.1 (5)	C11—C13—C15—C14	110.9 (3)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O3—H3*B*···O4	0.84	2.08	2.804 (3)	145
O4—H4···O3^{i}	0.84	2.01	2.837 (3)	167

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