IUCrData

ISSN 2414-3146

N-(5-Chloro-2-hydroxyphenyl)-*N*′-(3-hydroxypropyl)oxalamide

Chang-Kai Wang, Kang Zheng, Yan-Tuan Li and Zhi-Yong Wu*

Key Laboratory of Marine Drugs, Ministry of Education of China, School of Medicine and Pharmacy, Ocean University of China, Qingdao 266003, People's Republic of China. *Correspondence e-mail: wuzy@ouc.edu.cn

Received 25 April 2016
Accepted 3 May 2016

Edited by J. Simpson, University of Otago, New Zealand

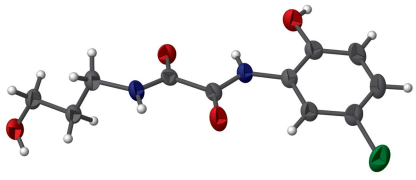
Keywords: crystal structure; oxamide compounds; hydrogen bonds.

CCDC reference: 1446193

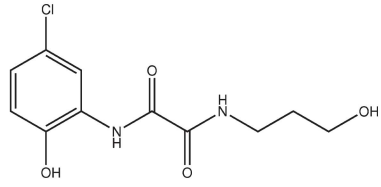
Structural data: full structural data are available from iucrdata.iucr.org

In the structure of the title *N*,*N*′-bis(substituted)oxamide compound, $C_{11}H_{13}ClN_2O_4$, the chlorohydroxyphenyl ring plane subtends an angle of 15.06 (13)° to the plane of the oxalamide unit. This in turn is inclined to the hydroxypropyl substituent by 78.03 (14)°. In the crystal, classical O—H···O and N—H···O hydrogen bonds give rise to a three-dimensional supramolecular structure.

3D view



Chemical scheme



Structure description

Oxamide complexes are of considerable current interest due to their DNA-binding properties and cytotoxic activity (Martínez-Martínez *et al.*, 1998; Li *et al.*, 2012; Yue *et al.*, 2012 and Zheng *et al.*, 2012). The title oxamide compound, *N*-(5-chloro-2-hydroxyphenyl)-*N*′-(3-hydroxypropyl)oxalamide (H_3chhpox), adopts a *transoid* conformation as expected (Fig. 1). The benzene ring substituent is almost coplanar with the oxamide group with a C7—N1—C1—C6 torsion angle of 11.8 (4)° while the other hydroxyphenyl substituent arm is almost orthogonal to this plane with a C8—N2—C9—C10 torsion angle of 92.4 (3)°.

In the crystal, layers are formed parallel to the *ac* plane through O—H···O hydrogen bonds (Fig. 2, Table 1). Inversion-related N—H···O hydrogen bonds between the oxamide groups connect the parallel layers into a three-dimensional supramolecular structure.

Synthesis and crystallization

The synthesis of the title compound (H_3chhpox) was achieved in two steps. The first was the preparation of *N*-(5-chloro-2-hydroxyphenyl)oxalamide (H_3chox) according to a reported method (Marmur, 1961). Next, H_3chox (5 mmol, 1.22 g) in 20 mL absolute

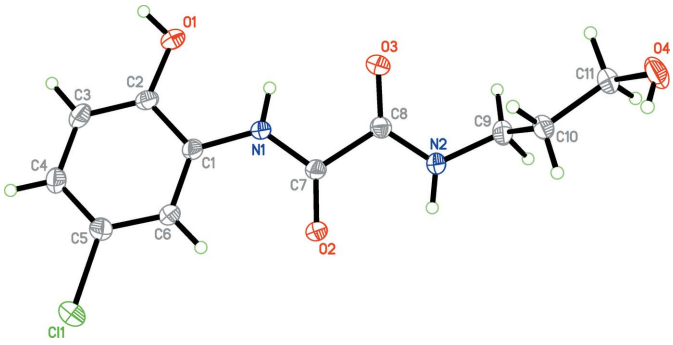


Figure 1
The molecular structure with displacement ellipsoids drawn at the 30% probability level.

ethanol was added dropwise to 20 mL of an absolute ethanol solution containing 3-amino-1-propanol (6 mmol, 0.76 mL) at 273 K. The resulting solution was stirred for 2 h, and H_3chhpox was precipitated as a white powder. It was then recrystallized from ethanol at 273 K and dried under vacuum. Well-shaped colorless single crystals were obtained by slow evaporation of an ethanol solution of the recrystallized product. Yield: 83%. Analysis calculated for $C_{11}H_{13}N_2O_4Cl$: C, 48.45; H, 4.81; N, 10.27%. Found: C, 48.96; H, 4.77; N, 10.65%.

Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2.

Acknowledgements

This project was supported by the National Natural Science Foundation of China (Nos. 51273184 and 81202399) and the Open Research Fund Program of the Key Laboratory of Marine Drugs (Ocean University of China), Ministry of Education [No. KLMD(OUC) 201401].

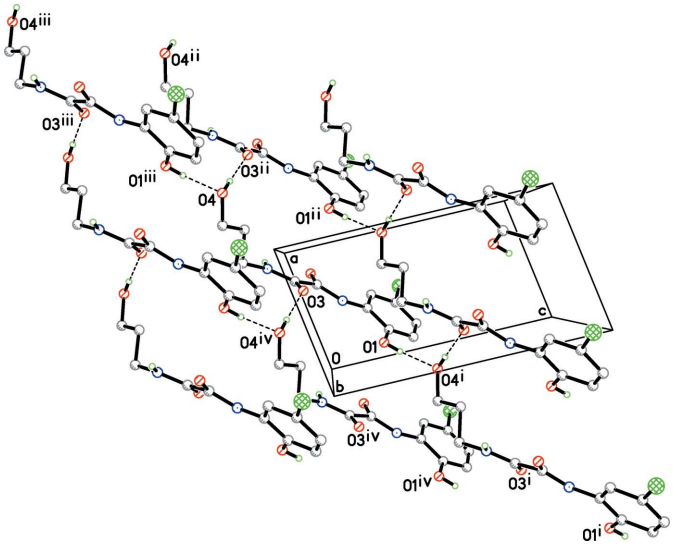


Figure 2
The two-dimensional hydrogen-bonding network parallel to (010), constructed by classical O—H···O interactions. [Symmetry codes: (i) $x-\frac{3}{2}, \frac{3}{2}-y, z+\frac{1}{2}$; (ii) $x+1, y, z$; (iii) $x+\frac{3}{2}, \frac{3}{2}-y, z-\frac{1}{2}$; (iv) $x-1, y, z$.]

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

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1A···O4^{i}	0.81 (2)	1.88 (2)	2.690 (2)	173 (3)
O4—H4A···O3^{ii}	0.81 (2)	1.98 (2)	2.794 (2)	178 (3)
N2—H2···O2^{iii}	0.88 (2)	2.12 (3)	2.916 (2)	150 (2)

Symmetry codes: (i) $x-\frac{3}{2}, -y+\frac{3}{2}, z+\frac{1}{2}$; (ii) $x+1, y, z$; (iii) $-x+2, -y+1, -z$.

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{11}H_{13}ClN_2O_4$
M_r	272.68
Crystal system, space group	Monoclinic, $P2_1/n$
Temperature (K)	296
a, b, c (Å)	6.1422 (14), 18.117 (4), 11.061 (2)
β (°)	98.896 (8)
V (Å^3)	1216.0 (5)
Z	4
Radiation type	Mo $K\alpha$
μ (mm^{-1})	0.32
Crystal size (mm)	0.49 × 0.16 × 0.03
Data collection	
Diffractometer	Bruker APEX area-detector
Absorption correction	Multi-scan (*SADABS*; Bruker, 2002)
T_{min}, T_{max}	0.697, 0.746
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	10616, 2779, 1672
R_{int}	0.054
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.651
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.047, 0.107, 1.00
No. of reflections	2779
No. of parameters	215
No. of restraints	2
H-atom treatment	All H-atom parameters refined
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.25, −0.20

Computer programs: *SMART* and *SAINT* (Bruker, 2002), *SHELXS97* and *XP* in *SHELXTL* (Sheldrick, 2008), *SHELXL2014* (Sheldrick, 2015), and *WinGX* (Farrugia, 2012).

References

Bruker (2002). *SMART*, *SAINT* and *SADABS*. Bruker AXS Inc., Madison, Wisconsin, USA.
Farrugia, L. J. (2012). *J. Appl. Cryst.* **45**, 849–854.
Li, X.-W., Tao, L., Li, Y.-T., Wu, Z.-Y. & Yan, C.-W. (2012). *Eur. J. Med. Chem.* **54**, 697–708.
Marmur, J. (1961). *J. Mol. Biol.* **3**, 208–218.
Martínez-Martínez, F. J., Padilla-Martínez, I. I., Brito, M. A., Geniz, E. D., Rojas, R. C., Saavedra, J. B. R., Höpfl, H., Tlahuextl, M. & Contreras, R. (1998). *J. Chem. Soc. Perkin Trans. 2*, pp. 401–406.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Sheldrick, G. M. (2015). *Acta Cryst.* C**71**, 3–8.
Yue, X.-T., Li, X.-W. & Wu, Z.-Y. (2012). *Acta Cryst.* E**68**, o8.
Zheng, Y.-J., Zheng, K., Wu, Z.-Y. & Li, Y. (2012). *Acta Cryst.* E**68**, o895.

full crystallographic data

IUCrData (2016). **1**, x160737 [doi:10.1107/S2414314616007379]

N-(5-Chloro-2-hydroxyphenyl)-*N*′-(3-hydroxypropyl)oxalamide

Chang-Kai Wang, Kang Zheng, Yan-Tuan Li and Zhi-Yong Wu

N-(5-Chloro-2-hydroxyphenyl)-*N*′-(3-hydroxypropyl)ethanediamide

Crystal data

$C_{11}H_{13}ClN_2O_4$
M_r = 272.68
Monoclinic, $P2_1/n$
a = 6.1422 (14) Å
b = 18.117 (4) Å
c = 11.061 (2) Å
β = 98.896 (8)°
V = 1216.0 (5) Å^3
Z = 4
$F(000)$ = 568
D_x = 1.490 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 2400 reflections
θ = 3.5–25.0°
μ = 0.32 mm^{-1}
T = 296 K
Prism, colorless
0.49 × 0.16 × 0.03 mm

Data collection

Bruker APEX area-detector diffractometer
Radiation source: fine-focus sealed tube
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2002)
T_{min} = 0.697, T_{max} = 0.746
10616 measured reflections
2779 independent reflections
1672 reflections with $I > 2\sigma(I)$
R_{int} = 0.054
θ_{max} = 27.5°, θ_{min} = 3.5°
h = −7→7
k = −23→23
l = −13→14

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.047
$wR(F^2)$ = 0.107
S = 1.00
2779 reflections
215 parameters
2 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: difference Fourier map
All H-atom parameters refined
$w = 1/[\sigma^2(F_o^2) + (0.0419P)^2 + 0.2977P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.25 e Å^{-3}
$\Delta\rho_{min}$ = −0.20 e Å^{-3}

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds 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 $F^2 > 2\sigma(F^2)$ 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 (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
Cl1	0.54932 (13)	0.33941 (3)	0.39890 (7)	0.0621 (3)
O1	0.2065 (3)	0.63169 (9)	0.24549 (16)	0.0484 (5)
O2	0.7936 (3)	0.50435 (8)	0.08106 (16)	0.0485 (5)
O3	0.6905 (2)	0.69246 (8)	0.03286 (14)	0.0400 (4)
O4	1.4561 (3)	0.80399 (10)	−0.10518 (19)	0.0523 (5)
N1	0.5547 (3)	0.58139 (10)	0.15803 (17)	0.0334 (5)
N2	0.8959 (3)	0.61585 (10)	−0.06633 (18)	0.0356 (5)
C1	0.4604 (3)	0.53575 (11)	0.23849 (19)	0.0312 (5)
C2	0.2779 (4)	0.56333 (12)	0.2848 (2)	0.0356 (5)
C3	0.1802 (4)	0.52106 (14)	0.3647 (2)	0.0449 (6)
C4	0.2617 (4)	0.45182 (13)	0.4005 (2)	0.0450 (6)
C5	0.4420 (4)	0.42605 (12)	0.3552 (2)	0.0390 (6)
C6	0.5429 (4)	0.46656 (12)	0.2747 (2)	0.0345 (5)
C7	0.7077 (3)	0.56439 (11)	0.0884 (2)	0.0310 (5)
C8	0.7663 (3)	0.63118 (11)	0.0148 (2)	0.0308 (5)
C9	0.9727 (4)	0.67265 (14)	−0.1426 (2)	0.0385 (6)
C10	1.1919 (4)	0.70438 (13)	−0.0862 (2)	0.0363 (6)
C11	1.2472 (4)	0.77284 (13)	−0.1527 (2)	0.0398 (6)
H1	0.507 (4)	0.6260 (14)	0.150 (2)	0.049 (7)*
H1A	0.122 (4)	0.6487 (14)	0.288 (2)	0.061 (9)*
H2	0.946 (4)	0.5705 (14)	−0.068 (2)	0.052 (8)*
H3	0.060 (4)	0.5401 (13)	0.394 (2)	0.048 (7)*
H4	0.191 (4)	0.4237 (12)	0.453 (2)	0.044 (7)*
H4A	1.527 (5)	0.7724 (14)	−0.065 (3)	0.086 (12)*
H6	0.663 (4)	0.4498 (12)	0.2439 (19)	0.038 (6)*
H9A	0.863 (4)	0.7103 (13)	−0.158 (2)	0.048 (7)*
H9B	0.985 (4)	0.6512 (13)	−0.223 (2)	0.051 (7)*
H10A	1.300 (4)	0.6690 (13)	−0.089 (2)	0.049 (7)*
H10B	1.191 (4)	0.7173 (12)	0.001 (2)	0.047 (7)*
H11A	1.134 (4)	0.8107 (13)	−0.142 (2)	0.051 (7)*
H11B	1.244 (4)	0.7627 (13)	−0.239 (2)	0.055 (8)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.0836 (6)	0.0395 (4)	0.0696 (5)	0.0142 (3)	0.0319 (4)	0.0192 (3)
O1	0.0533 (12)	0.0387 (9)	0.0596 (12)	0.0148 (8)	0.0290 (10)	0.0017 (8)
O2	0.0544 (11)	0.0292 (9)	0.0709 (12)	0.0096 (8)	0.0384 (10)	0.0073 (8)
O3	0.0459 (10)	0.0263 (8)	0.0499 (10)	0.0023 (7)	0.0141 (8)	0.0018 (7)
O4	0.0427 (11)	0.0400 (10)	0.0750 (14)	−0.0110 (9)	0.0117 (10)	0.0195 (10)

N1	0.0382 (11)	0.0235 (10)	0.0425 (12)	0.0032 (8)	0.0186 (9)	0.0008 (8)
N2	0.0357 (11)	0.0289 (10)	0.0461 (12)	−0.0019 (9)	0.0184 (10)	0.0019 (9)
C1	0.0342 (12)	0.0274 (11)	0.0339 (12)	−0.0022 (9)	0.0108 (10)	−0.0037 (9)
C2	0.0374 (13)	0.0289 (11)	0.0425 (14)	0.0035 (10)	0.0127 (11)	−0.0046 (10)
C3	0.0439 (15)	0.0473 (15)	0.0495 (16)	0.0031 (12)	0.0257 (13)	−0.0036 (12)
C4	0.0556 (17)	0.0402 (14)	0.0447 (15)	−0.0047 (12)	0.0253 (13)	0.0021 (12)
C5	0.0497 (15)	0.0310 (12)	0.0383 (14)	0.0001 (11)	0.0129 (12)	−0.0003 (10)
C6	0.0364 (14)	0.0310 (12)	0.0393 (13)	0.0033 (10)	0.0154 (11)	−0.0009 (10)
C7	0.0307 (12)	0.0257 (11)	0.0383 (13)	−0.0012 (10)	0.0105 (10)	−0.0026 (10)
C8	0.0272 (12)	0.0285 (11)	0.0366 (13)	−0.0026 (9)	0.0050 (10)	−0.0007 (10)
C9	0.0384 (15)	0.0399 (14)	0.0395 (15)	−0.0035 (12)	0.0129 (12)	0.0060 (12)
C10	0.0351 (14)	0.0324 (12)	0.0430 (15)	−0.0028 (11)	0.0105 (11)	0.0063 (11)
C11	0.0433 (15)	0.0352 (13)	0.0429 (16)	−0.0037 (12)	0.0131 (12)	0.0075 (12)

Geometric parameters (Å, º)

Cl1—C5	1.742 (2)	C3—C4	1.385 (3)
O1—C2	1.362 (3)	C3—H3	0.92 (2)
O1—H1A	0.813 (17)	C4—C5	1.366 (3)
O2—C7	1.217 (2)	C4—H4	0.93 (2)
O3—C8	1.232 (2)	C5—C6	1.374 (3)
O4—C11	1.426 (3)	C6—H6	0.91 (2)
O4—H4A	0.812 (17)	C7—C8	1.532 (3)
N1—C7	1.340 (3)	C9—C10	1.507 (3)
N1—C1	1.404 (3)	C9—H9A	0.96 (2)
N1—H1	0.86 (2)	C9—H9B	0.98 (2)
N2—C8	1.317 (3)	C10—C11	1.507 (3)
N2—C9	1.454 (3)	C10—H10A	0.93 (2)
N2—H2	0.88 (2)	C10—H10B	0.99 (2)
C1—C6	1.388 (3)	C11—H11A	1.00 (2)
C1—C2	1.395 (3)	C11—H11B	0.97 (2)
C2—C3	1.375 (3)		
C2—O1—H1A	111.1 (19)	C1—C6—H6	118.3 (14)
C11—O4—H4A	107 (2)	O2—C7—N1	126.43 (19)
C7—N1—C1	128.59 (18)	O2—C7—C8	122.01 (18)
C7—N1—H1	114.1 (16)	N1—C7—C8	111.56 (17)
C1—N1—H1	117.2 (16)	O3—C8—N2	125.7 (2)
C8—N2—C9	122.0 (2)	O3—C8—C7	119.94 (18)
C8—N2—H2	117.3 (16)	N2—C8—C7	114.32 (18)
C9—N2—H2	120.4 (16)	N2—C9—C10	112.3 (2)
C6—C1—C2	119.7 (2)	N2—C9—H9A	109.1 (14)
C6—C1—N1	123.15 (19)	C10—C9—H9A	111.4 (14)
C2—C1—N1	117.09 (18)	N2—C9—H9B	108.7 (14)
O1—C2—C3	124.1 (2)	C10—C9—H9B	109.7 (14)
O1—C2—C1	116.49 (19)	H9A—C9—H9B	106 (2)
C3—C2—C1	119.4 (2)	C11—C10—C9	111.5 (2)
C2—C3—C4	120.9 (2)	C11—C10—H10A	109.9 (14)

C2—C3—H3	118.0 (15)	C9—C10—H10A	108.7 (14)
C4—C3—H3	121.1 (15)	C11—C10—H10B	108.4 (13)
C5—C4—C3	118.8 (2)	C9—C10—H10B	110.5 (13)
C5—C4—H4	121.6 (14)	H10A—C10—H10B	108 (2)
C3—C4—H4	119.6 (14)	O4—C11—C10	113.8 (2)
C4—C5—C6	121.9 (2)	O4—C11—H11A	106.9 (13)
C4—C5—Cl1	119.99 (18)	C10—C11—H11A	107.2 (13)
C6—C5—Cl1	118.14 (17)	O4—C11—H11B	108.6 (14)
C5—C6—C1	119.2 (2)	C10—C11—H11B	110.7 (15)
C5—C6—H6	122.5 (14)	H11A—C11—H11B	109.4 (19)
C7—N1—C1—C6	11.8 (4)	C2—C1—C6—C5	0.5 (3)
C7—N1—C1—C2	−169.5 (2)	N1—C1—C6—C5	179.1 (2)
C6—C1—C2—O1	179.8 (2)	C1—N1—C7—O2	0.9 (4)
N1—C1—C2—O1	1.1 (3)	C1—N1—C7—C8	−179.4 (2)
C6—C1—C2—C3	−0.9 (3)	C9—N2—C8—O3	2.5 (4)
N1—C1—C2—C3	−179.5 (2)	C9—N2—C8—C7	−178.6 (2)
O1—C2—C3—C4	179.9 (2)	O2—C7—C8—O3	−173.5 (2)
C1—C2—C3—C4	0.5 (4)	N1—C7—C8—O3	6.7 (3)
C2—C3—C4—C5	0.2 (4)	O2—C7—C8—N2	7.5 (3)
C3—C4—C5—C6	−0.6 (4)	N1—C7—C8—N2	−172.22 (19)
C3—C4—C5—Cl1	179.7 (2)	C8—N2—C9—C10	92.4 (3)
C4—C5—C6—C1	0.3 (4)	N2—C9—C10—C11	−168.6 (2)
Cl1—C5—C6—C1	179.97 (18)	C9—C10—C11—O4	−178.3 (2)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1*A*···O4^{i}	0.81 (2)	1.88 (2)	2.690 (2)	173 (3)
O4—H4*A*···O3^{ii}	0.81 (2)	1.98 (2)	2.794 (2)	178 (3)
N2—H2···O2^{iii}	0.88 (2)	2.12 (3)	2.916 (2)	150 (2)

Symmetry codes: (i) $x-3/2, -y+3/2, z+1/2$; (ii) $x+1, y, z$; (iii) $-x+2, -y+1, -z$.