Acta Crystallographica Section E
Structure Reports
Online

ISSN 1600-5368

A perchlorate salt of 1-{[2-(2-hydroxyethylamino)ethylimino]methyl}naphthalen-2-ol

Hai-Liang Zhu[a]* and Zhong-Lu You[b]

[a]Department of Chemistry, Wuhan University of Science and Engineering, Wuhan 430073, People's Republic of China, and [b]Department of Chemistry, Liaoning Normal University, Dalian 116029, People's Republic of China

Correspondence e-mail:
Hailiang_zhu@163.com

Key indicators

Single-crystal X-ray study
T = 298 K
Mean σ(C–C) = 0.005 Å
Disorder in solvent or counterion
R factor = 0.053
wR factor = 0.145
Data-to-parameter ratio = 14.3

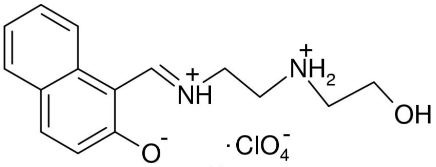
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Received 29 March 2005
Accepted 5 April 2005
Online 9 April 2005

In the title compound, 1-{[2-(2-hydroxyethylamino)ethylimino]methyl}naphthalen-2-ol perchlorate, $C_{15}H_{19}N_2O_2\cdot ClO_4$, the ions are linked through weak intermolecular N—H···O, O—H···O and C—H···O hydrogen bonds, forming columns along the *b* axis.

Comment

Schiff base compounds have been of great interest for many years. These compounds play an important role in the development of coordination chemistry related to catalysis and enzymatic reactions, magnetism, and molecular architectures. As an extension of our work on the structural characterization of Schiff base compounds, the crystal structure of the title compound, (I), is reported here.



(I)

In compound, (I), all bond lengths are within normal ranges (Allen *et al.*, 1987) (Fig. 1). As expected, the cation adopts a *trans* configuration with respect to the C11═N1 bond. In the crystal structure, the ions are linked through weak intermolecular N—H···O, O—H···O and C—H···O hydrogen bonds, forming columns along the *b* axis (Fig. 2 and Table 1).

Experimental

2-Hydroxy-1-naphthaldehyde (0.1 mmol, 8.6 mg) and *N*-(2-hydroxyethyl)ethylenediamine (0.1 mmol, 10.4 mg) were dissolved in ethanol

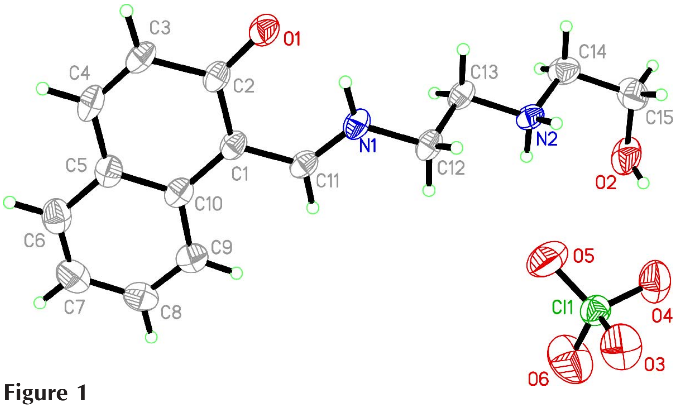


Figure 1
The structure of (I), showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 30% probability level. Only the major component of the disordered perchloate anion is shown.

(10 ml). The mixture was stirred for 15 min to give a clear yellow solution. To the solution was added an aqueous solution of $HClO_4$ (0.1 mmol, 70%), with stirring. The mixture was stirred at room temperature for another 15 min and filtered. The filtrate was kept in air for 3 d and yellow block-shaped crystals were formed on slow evaporation of the solvent.

Crystal data

$C_{15}H_{19}N_2O_2 \cdot ClO_4$
$M_r = 358.77$
Monoclinic, $P2_1$
$a = 9.254$ (2) Å
$b = 6.864$ (2) Å
$c = 13.174$ (3) Å
$\beta = 102.78$ (3)°
$V = 816.1$ (4) Å^3
$Z = 2$

$D_x = 1.460$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 2667 reflections
$\theta = 2.3$–23.7°
$\mu = 0.27$ mm^{-1}
$T = 298$ (2) K
Block, yellow
0.33 × 0.28 × 0.23 mm

Data collection

Bruker SMART CCD area-detector diffractometer
ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{\min} = 0.917$, $T_{\max} = 0.941$
8798 measured reflections

3692 independent reflections
2842 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.025$
$\theta_{\max} = 27.5°$
$h = -11 \rightarrow 11$
$k = -8 \rightarrow 8$
$l = -17 \rightarrow 16$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.053$
$wR(F^2) = 0.145$
$S = 1.04$
3692 reflections
259 parameters
H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0864P)^2 + 0.0182P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.001$
$\Delta\rho_{\max} = 0.50$ e Å^{-3}
$\Delta\rho_{\min} = -0.18$ e Å^{-3}
Extinction correction: none
Absolute structure: Flack (1983), with 1677 Friedel pairs
Flack parameter: 0.02 (9)

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···O2$^{\rm i}$	0.89 (1)	2.54 (3)	3.183 (4)	129 (3)
N1—H1···O1	0.89 (1)	1.83 (3)	2.572 (3)	139 (3)
O2—H2···O4	0.82	2.41	3.193 (6)	160
N2—H2B···O5	0.90	2.50	2.973 (6)	113
N2—H2B···O5′	0.90	2.44	3.104 (16)	131
N2—H2B···O1$^{\rm ii}$	0.90	1.98	2.731 (3)	140
N2—H2A···O4$'^{\rm iii}$	0.90	2.12	2.914 (18)	146
N2—H2A···O4$^{\rm iii}$	0.90	2.05	2.941 (5)	171
C6—H6···O6$^{\rm iv}$	0.93	2.57	3.302 (4)	136
C13—H13B···O2$^{\rm i}$	0.97	2.46	3.263 (4)	140

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

The O atoms of the perchlorate anion are disordered over two distinct sites with occupancies of 0.805 (6) and 0.195 (6). The Cl—O and O···O distances in both disordered components were restrained to be equal and also the U^{ij} components of the disordered O atoms were restrained to isotropic behaviour. Atom H1 was located in a difference Fourier map and refined isotropically, with the N—H distance restrained to 0.90 (1) Å. The other H atoms were placed in idealized positions and constrained to ride on their parent atoms, with C—H distances in the range 0.93–0.97 Å, N—H distances of 0.90 Å and an O—H distance of 0.82 Å, and with $U_{\rm iso}({\rm H}) = 1.2U_{\rm eq}$(C,N) or $1.5U_{\rm eq}$(O).

Data collection: *SMART* (Bruker, 1998); cell refinement: *SAINT* (Bruker, 1998); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997*a*); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997*a*); molecular graphics: *SHELXTL* (Sheldrick, 1997*b*); software used to prepare material for publication: *SHELXTL*.

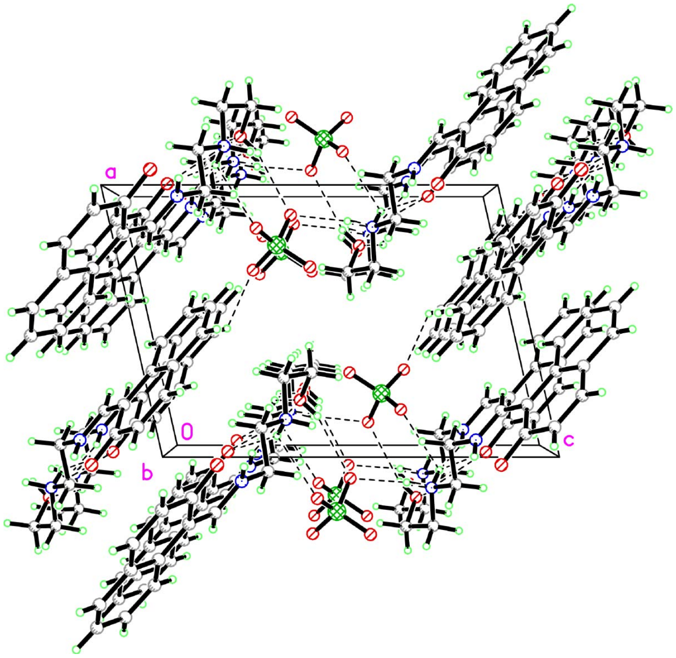


Figure 2
The crystal packing of (I), viewed along the b axis. Dashed lines indicate intermolecular hydrogen bonds.

This work was sponsored by the Scientific Research Foundation for Returned Overseas Chinese Scholars.

References

Allen, F. H., Kennard, O., Watson, D. G., Brammer, L., Orpen, A. G. & Taylor, R. (1987). *J. Chem. Soc. Perkin Trans.* 2, pp. S1–19.
Bruker (1998). *SMART* (Version 5.628) and *SAINT* (Version 6.02). Bruker AXS Inc., Madison, Wisconsin, USA.
Flack, H. D. (1983). *Acta Cryst.* A**39**, 876–881.
Sheldrick, G. M. (1996). *SADABS*. University of Göttingen, Germany.
Sheldrick, G. M. (1997*a*). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.
Sheldrick, G. M. (1997*b*). *SHELXTL*. Version. 5.10. Bruker AXS Inc., Madison, Wisconsin, USA.