

Acta Crystallographica Section E

## Structure Reports

Online

ISSN 1600-5368

## Bis{(E)-3-[(2-hydroxybenzylidene)-amino]propyl}ammonium chloride

Murad A. AlDamen\* and Salim F. Haddad

Department of Chemistry, The University of Jordan, Amman 11942, Jordan  
Correspondence e-mail: maldamen@ju.edu.jo

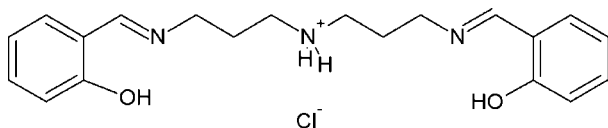
Received 6 September 2012; accepted 2 November 2012

Key indicators: single-crystal X-ray study;  $T = 100$  K; mean  $\sigma(\text{C}-\text{C}) = 0.005$  Å;  
R factor = 0.076; wR factor = 0.150; data-to-parameter ratio = 17.1.

The title salt,  $\text{C}_{20}\text{H}_{26}\text{N}_3\text{O}_2^+\cdot\text{Cl}^-$ , lies across a twofold crystallographic axis with the central N atom of the cation and the chloride anion sitting on this axis,  $Z' = 0.5$ . There is an intramolecular hydrogen bond between the hydroxy H atom and the imino N atom. The chloride anion and the cation are connected into chains along the  $a$  axis by an  $\text{N}-\text{H}\cdots\text{Cl}$  hydrogen bond. In the crystal, the chains are linked *via*  $\text{C}-\text{H}\cdots\text{Cl}$  interactions forming two-dimensional networks lying parallel to (101).

## Related literature

For applications of similar ligands, see: Taha *et al.* (2011a,b).  
For analogous structures, see: Ramazani *et al.* (2006); Cheng *et al.* (2009); Chen *et al.* (2011); Pavel *et al.* (2007).



## Experimental

## Crystal data

 $\text{C}_{20}\text{H}_{26}\text{N}_3\text{O}_2^+\cdot\text{Cl}^-$  $M_r = 375.88$ Orthorhombic,  $Pccn$  $a = 4.9848$  (3) Å $b = 38.714$  (2) Å $c = 10.3299$  (7) Å $V = 1993.5$  (2) Å<sup>3</sup> $Z = 4$ Mo  $K\alpha$  radiation $\mu = 0.21$  mm<sup>-1</sup> $T = 100$  K $0.52 \times 0.37 \times 0.04$  mm

## Data collection

Oxford Diffraction Xcalibur (Eos, Gemini ultra) diffractometer  
Absorption correction: analytical [*CrysAlis PRO* (Oxford Diffraction, 2009), based on expressions derived from Clark &

Reid (1995)]  
 $T_{\min} = 0.94$ ,  $T_{\max} = 0.992$   
8874 measured reflections  
2040 independent reflections  
1654 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.049$

## Refinement

 $R[F^2 > 2\sigma(F^2)] = 0.076$  $wR(F^2) = 0.150$  $S = 1.21$ 

2040 reflections

119 parameters

Only H-atom displacement parameters refined

 $\Delta\rho_{\max} = 0.39$  e Å<sup>-3</sup> $\Delta\rho_{\min} = -0.88$  e Å<sup>-3</sup>

Table 1

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$\text{N1}-\text{H1A}\cdots\text{Cl1}$	0.91	2.22	3.128 (2)	176
$\text{O1}-\text{H1}\cdots\text{N2}$	0.97	1.70	2.577 (4)	148
$\text{C1}-\text{H1C}\cdots\text{Cl1}^{\dagger}$	0.97	2.70	3.642 (4)	164

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

Data collection: *CrysAlis PRO* (Oxford Diffraction, 2009); cell refinement: *CrysAlis PRO*; data reduction: *CrysAlis PRO*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *OLEX2* (Dolomanov *et al.*, 2009); software used to prepare material for publication: *OLEX2*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: GO2068).

## References

- Chen, Y., Ge, Y., Zhou, W., Ye, L., Gu, Z., Ma, G., Li, W., Li, H. & Cai, Y. (2011). *Inorg. Chem. Commun.* **14**, 1228–1232.  
Cheng, K., Zheng, Q., Qian, Y., Shi, L., Zhao, J. & Zhu, H. (2009). *Bioorg. Med. Chem.* **17**, 7861–7871.  
Clark, R. C. & Reid, J. S. (1995). *Acta Cryst.* **A51**, 887–897.  
Dolomanov, O. V., Bourhis, L. J., Gildea, R. J., Howard, J. A. K. & Puschmann, H. (2009). *J. Appl. Cryst.* **42**, 339–341.  
Oxford Diffraction (2009). *CrysAlis PRO*. Oxford Diffraction Ltd, Yarnton, England.  
Pavel, K., Kamenicek, J., Petricek, V., Kurecka, A., Kalinska, B. & Mrozinski, J. (2007). *Polyhedron*, **26**, 535–542.  
Ramazani, A., Dolatyari, L., Morsali, A., Yilmaz, V. T. & Bueyuekguengoer, O. (2006). *J. Iran. Chem. Soc.*, **3**, 367–370.  
Sheldrick, G. M. (2008). *Acta Cryst.* **A64**, 112–122.  
Taha, Z. A., Ajlouni, A. M., Al-Hassan, K. A., Hijazi, A. K. & Faiq, A. B. (2011a). *Spectrochim. Acta Part A*, **81**, 317–323.  
Taha, Z. A., Ajlouni, A. M., Al Momani, W. & Al-Ghazawi, A. A. (2011b). *Spectrochim. Acta Part A*, **81**, 570–577.

## supporting information

*Acta Cryst.* (2012). E68, o3314 [doi:10.1107/S1600536812045424]

**Bis{(E)-3-[(2-hydroxybenzylidene)amino]propyl}ammonium chloride****Murad A. AlDamen and Salim F. Haddad****S1. Comment**

Tetradentate Schiff bases are flexible ligands and difficult to crystallize and most of the crystal structures published for these ligands are complexes with transition metals or actinides/actinides.

As far as we can determine there are no published structures for this type of ligand and published data for these types of ligand with antimicrobial or of interest because of their optical properties have been obtained from solid state techniques like NMR or elemental analysis Taha *et al.* (2011a); Taha *et al.* (2011b). We crystallized (I) as salt. (Fig. 1). The molecule sits across a two-fold crystallographic axis with the ammonium nitrogen atom, N1, and the Cl anion sitting on the two-fold axis.

The bond lengths of N1—C1 and N2—C3 are 1.501 (4) and 1.456 (4) respectively indicate that these are single bonds and the bond length of N2—C4 (1.273 (4) Å) shows that this is a double bond exhibits a double bond. The distances are in agreement with other salicylic and dipropylamino ligands and complexes found in literature (Ramazani *et al.* (2006), Cheng *et al.* (2009), Pavel *et al.* (2007) & Chen *et al.* (2011)).

There is an intramolecular hydrogen bond between the phenolic oxygen, O1 and N2, Table 1, Fig1 The N1—HH1A...Cl1 hydrogen bond connects the chloride ion to two adjacent ligands to form chains which run parallel to the *a* axis, Table 1 and Fig. 1. In addition there is a short contact between C1 and Cl1, Table 1. There are no C—H... $\pi$  interactions nor is there any  $\pi$ - $\pi$  stacking.

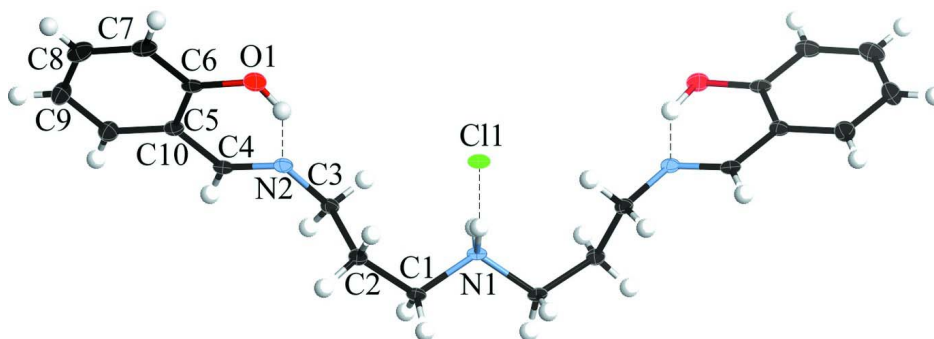
**S2. Experimental**

Facile condensation of 3,3 -Diaminodipropylamine and salicylaldehyde 1:2 molar ratio afforded a neutral condensate as following:

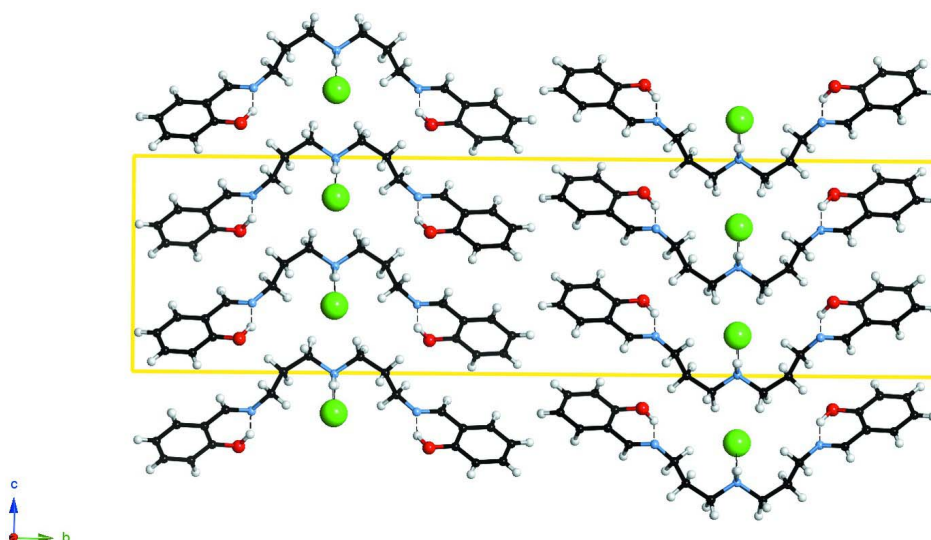
3,3 -Diaminodipropylamine (30 ml, 0.208 mol) was added dropwise to a solution of salicylaldehyde (50.7 g, 0.416 mol) in absolute ethanol (250 ml); the solution became instantly yellow. The mixture was heated for 1 h at 50°C. Evaporation of the solvent under reduced pressure afforded the desired compound in 95% yield as yellow oil. The obtained Schiff base was then mixed with cyanuric acid in ethanol with few drops of 1M HCl added. The mixture then stir heated (40°C) for 24 then filtered. After few days crystals suitable for X-ray diffraction were formed.

**S3. Refinement**

H atoms were treated as riding atoms with C—H(aromatic), 0.93Å and C—H(CH<sub>2</sub>), 0.97Å, with  $U_{iso} = 1.2U_{eq}(C)$ . The hydrogens attached N1 and O1 were located on a difference map and refined as riding atoms with  $U_{iso} = 1.2U_{eq}(N1)$  and  $U_{iso} = 1.5U_{eq}(O1)$ . The positions of these latter atoms was confirmed on a final difference map. The position of the Cl anion was chosen so that it formed a hydrogen bonded asymmetric unit.

**Figure 1**

Asymmetric units and the atom-numbering schemes for (I) with thermal displacement ellipsoids at 30% probability (except hydrogens).

**Figure 2**

View of the molecular packing diagram, view perpendicular to the *bc* plane.

### Bis{(E)-3-[(2-hydroxybenzylidene)amino]propyl}ammonium chloride

#### Crystal data

$C_{20}H_{26}N_3O_2^+ \cdot Cl^-$

$M_r = 375.88$

Orthorhombic, *Pccn*

$a = 4.9848$  (3) Å

$b = 38.714$  (2) Å

$c = 10.3299$  (7) Å

$V = 1993.5$  (2) Å<sup>3</sup>

$Z = 4$

$F(000) = 800$

$D_x = 1.252$  Mg m<sup>-3</sup>

Mo  $K\alpha$  radiation,  $\lambda = 0.7107$  Å

Cell parameters from 2556 reflections

$\theta = 3.7$ – $26.3^\circ$

$\mu = 0.21$  mm<sup>-1</sup>

$T = 100$  K

Prism, white

$0.52 \times 0.37 \times 0.04$  mm

#### Data collection

Oxford Diffraction Xcalibur (Eos, Gemini ultra)  
diffractometer

Radiation source: fine-focus sealed tube

Graphite monochromator

Detector resolution: 16.0122 pixels mm<sup>-1</sup>

$\omega$  scans

Absorption correction: analytical  
 [CrysAlis PRO (Oxford Diffraction, 2009),  
 based on expressions derived from Clark &  
 Reid (1995)]  
 $T_{\min} = 0.94$ ,  $T_{\max} = 0.992$   
 8874 measured reflections  
 2040 independent reflections

1654 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.049$   
 $\theta_{\max} = 26.4^\circ$ ,  $\theta_{\min} = 4.0^\circ$   
 $h = -6 \rightarrow 5$   
 $k = -48 \rightarrow 48$   
 $l = -12 \rightarrow 12$

*Refinement*

Refinement on  $F^2$   
 Least-squares matrix: full  
 $R[F^2 > 2\sigma(F^2)] = 0.076$   
 $wR(F^2) = 0.150$   
 $S = 1.21$   
 2040 reflections  
 119 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  
 Only H-atom displacement parameters refined  
 $w = 1/[\sigma^2(F_o^2) + (0.0141P)^2 + 6.1394P]$   
 where  $P = (F_o^2 + 2F_c^2)/3$   
 $(\Delta/\sigma)_{\max} < 0.001$   
 $\Delta\rho_{\max} = 0.39 \text{ e } \text{\AA}^{-3}$   
 $\Delta\rho_{\min} = -0.88 \text{ e } \text{\AA}^{-3}$

*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.

*Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters ( $\text{\AA}^2$ )*

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
O1	-0.1873 (5)	0.13196 (7)	0.8454 (3)	0.0340 (7)
H1	-0.0740	0.1460	0.7900	0.051*
N1	0.2500	0.2500	0.5049 (4)	0.0174 (8)
H1A	0.1010	0.2508	0.5560	0.021*
N2	0.1804 (6)	0.14754 (7)	0.6799 (3)	0.0229 (7)
C1	0.2328 (7)	0.21793 (8)	0.4236 (3)	0.0213 (7)
H1B	0.4094	0.2126	0.3897	0.026*
H1C	0.1150	0.2223	0.3507	0.026*
C2	0.1289 (7)	0.18696 (8)	0.4984 (3)	0.0230 (8)
H2A	0.0861	0.1688	0.4373	0.028*
H2B	-0.0364	0.1935	0.5414	0.028*
C3	0.3212 (7)	0.17249 (9)	0.5993 (3)	0.0236 (8)
H3A	0.4716	0.1614	0.5566	0.028*
H3B	0.3901	0.1911	0.6526	0.028*
C4	0.2480 (7)	0.11586 (8)	0.6762 (3)	0.0226 (7)
H4	0.3885	0.1092	0.6225	0.027*
C5	0.1112 (7)	0.08970 (9)	0.7539 (3)	0.0234 (8)
C6	-0.1012 (7)	0.09900 (9)	0.8369 (3)	0.0257 (8)
C7	-0.2244 (9)	0.07364 (10)	0.9119 (4)	0.0345 (9)
H7	-0.3635	0.0796	0.9676	0.041*
C8	-0.1417 (9)	0.03980 (11)	0.9042 (4)	0.0388 (10)
H8	-0.2260	0.0231	0.9547	0.047*

C9	0.0656 (9)	0.03018 (10)	0.8224 (4)	0.0394 (10)
H9	0.1205	0.0073	0.8177	0.047*
C10	0.1893 (8)	0.05520 (9)	0.7478 (4)	0.0325 (9)
H10	0.3278	0.0489	0.6924	0.039*
Cl1	-0.2500	0.2500	0.68790 (11)	0.0223 (3)

*Atomic displacement parameters (Å<sup>2</sup>)*

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
O1	0.0353 (16)	0.0380 (14)	0.0286 (15)	0.0007 (12)	0.0106 (13)	-0.0020 (12)
N1	0.0110 (18)	0.0282 (19)	0.0131 (19)	-0.0011 (17)	0.000	0.000
N2	0.0248 (16)	0.0259 (14)	0.0180 (15)	-0.0025 (12)	-0.0038 (13)	-0.0005 (12)
C1	0.0195 (16)	0.0289 (17)	0.0156 (16)	-0.0015 (15)	-0.0012 (14)	-0.0030 (13)
C2	0.0200 (18)	0.0259 (17)	0.0230 (19)	-0.0021 (14)	-0.0039 (15)	-0.0010 (14)
C3	0.0235 (19)	0.0263 (17)	0.0208 (18)	-0.0040 (14)	-0.0011 (15)	-0.0029 (14)
C4	0.0210 (17)	0.0310 (17)	0.0158 (16)	0.0000 (15)	0.0007 (15)	-0.0017 (14)
C5	0.0202 (18)	0.0298 (18)	0.0201 (18)	-0.0024 (14)	-0.0037 (15)	0.0004 (14)
C6	0.0252 (19)	0.0342 (19)	0.0178 (18)	-0.0038 (16)	-0.0034 (15)	-0.0008 (15)
C7	0.031 (2)	0.048 (2)	0.024 (2)	-0.0092 (19)	0.0037 (18)	0.0034 (17)
C8	0.037 (2)	0.045 (2)	0.035 (2)	-0.0136 (19)	-0.0037 (19)	0.0133 (19)
C9	0.045 (3)	0.030 (2)	0.043 (3)	0.0001 (18)	0.000 (2)	0.0099 (18)
C10	0.032 (2)	0.0319 (19)	0.033 (2)	0.0000 (17)	0.0020 (18)	0.0014 (17)
Cl1	0.0132 (5)	0.0368 (6)	0.0169 (5)	-0.0037 (5)	0.000	0.000

*Geometric parameters (Å, °)*

O1—C6	1.349 (4)	C3—H3B	0.9700
O1—H1	0.9705	C4—C5	1.461 (5)
N1—C1 <sup>i</sup>	1.501 (4)	C4—H4	0.9300
N1—C1	1.501 (4)	C5—C10	1.393 (5)
N1—H1A	0.9117	C5—C6	1.409 (5)
N2—C4	1.272 (4)	C6—C7	1.394 (5)
N2—C3	1.456 (4)	C7—C8	1.376 (6)
C1—C2	1.518 (4)	C7—H7	0.9300
C1—H1B	0.9700	C8—C9	1.386 (6)
C1—H1C	0.9700	C8—H8	0.9300
C2—C3	1.522 (5)	C9—C10	1.383 (5)
C2—H2A	0.9700	C9—H9	0.9300
C2—H2B	0.9700	C10—H10	0.9300
C3—H3A	0.9700		
C6—O1—H1	107.8	H3A—C3—H3B	108.2
C1 <sup>i</sup> —N1—C1	112.0 (3)	N2—C4—C5	121.8 (3)
C1 <sup>i</sup> —N1—H1A	110.0	N2—C4—H4	119.1
C1—N1—H1A	107.8	C5—C4—H4	119.1
C4—N2—C3	119.6 (3)	C10—C5—C6	118.8 (3)
N1—C1—C2	112.8 (3)	C10—C5—C4	120.6 (3)
N1—C1—H1B	109.0	C6—C5—C4	120.5 (3)

C2—C1—H1B	109.0	O1—C6—C7	119.3 (3)
N1—C1—H1C	109.0	O1—C6—C5	121.3 (3)
C2—C1—H1C	109.0	C7—C6—C5	119.3 (3)
H1B—C1—H1C	107.8	C8—C7—C6	120.4 (4)
C1—C2—C3	115.1 (3)	C8—C7—H7	119.8
C1—C2—H2A	108.5	C6—C7—H7	119.8
C3—C2—H2A	108.5	C7—C8—C9	121.0 (4)
C1—C2—H2B	108.5	C7—C8—H8	119.5
C3—C2—H2B	108.5	C9—C8—H8	119.5
H2A—C2—H2B	107.5	C10—C9—C8	119.0 (4)
N2—C3—C2	109.4 (3)	C10—C9—H9	120.5
N2—C3—H3A	109.8	C8—C9—H9	120.5
C2—C3—H3A	109.8	C9—C10—C5	121.5 (4)
N2—C3—H3B	109.8	C9—C10—H10	119.3
C2—C3—H3B	109.8	C5—C10—H10	119.3
C1 <sup>i</sup> —N1—C1—C2	-161.9 (3)	C10—C5—C6—C7	1.0 (5)
N1—C1—C2—C3	-70.5 (4)	C4—C5—C6—C7	-178.7 (3)
C4—N2—C3—C2	114.2 (3)	O1—C6—C7—C8	179.5 (4)
C1—C2—C3—N2	169.3 (3)	C5—C6—C7—C8	-0.6 (6)
C3—N2—C4—C5	-179.2 (3)	C6—C7—C8—C9	0.2 (6)
N2—C4—C5—C10	180.0 (3)	C7—C8—C9—C10	0.0 (6)
N2—C4—C5—C6	-0.4 (5)	C8—C9—C10—C5	0.4 (6)
C10—C5—C6—O1	-179.2 (3)	C6—C5—C10—C9	-0.8 (6)
C4—C5—C6—O1	1.2 (5)	C4—C5—C10—C9	178.8 (4)

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

*Hydrogen-bond geometry (Å, °)*

<i>D</i> —H... <i>A</i>	<i>D</i> —H	H... <i>A</i>	<i>D</i> ... <i>A</i>	<i>D</i> —H... <i>A</i>
N1—H1A...C11	0.91	2.22	3.128 (2)	176
O1—H1...N2	0.97	1.70	2.577 (4)	148
C1—H1C...C11 <sup>ii</sup>	0.97	2.70	3.642 (4)	164

Symmetry code: (ii)  $-x-1/2, y, z-1/2$ .