Table 2. *Selected geometric parameters* (Å, °)

As—O1	1.654 (3)	N1—C3	1.448 (7)
As—O2	1.689 (4)	C1—C2	1.382 (7)
As—O3	1.705 (3)	C1—C6	1.379 (9)
As—C1	1.885 (5)	C2—C3	1.407 (8)
O4—N1	1.201 (7)	C3—C4	1.369 (9)
O5—N1	1.249 (7)	C4—C5	1.398 (8)
O6—C4	1.360 (7)	C3—C4	1.369 (8)
O1—As—O2	113.3 (2)	C2—C1—C6	120.9 (5)
O1—As—O3	109.6 (2)	C1—C2—C3	117.4 (5)
O1—As—C1	113.1 (2)	N1—C3—C2	115.3 (6)
O2—As—O3	104.5 (2)	N1—C3—C4	122.2 (5)
O2—As—C1	104.9 (2)	C2—C3—C4	122.5 (5)
O3—As—C1	111.1 (2)	O6—C4—C3	125.7 (5)
O4—N1—O5	123.3 (5)	O6—C4—C5	115.3 (6)
O4—N1—C3	120.8 (5)	C3—C4—C5	118.9 (5)
O5—N1—C3	115.9 (6)	C4—C5—C6	119.3 (6)
As—C1—C2	120.1 (4)	C1—C6—C5	121.0 (5)
As—C1—C6	119.0 (3)		

Anomalous-dispersion effects were included in F_c (Ibers & Hamilton, 1964).

Data collection: *MSC/AFC Diffractometer Control Software* (Molecular Structure Corporation, 1988); cell refinement: *MSC/AFC Diffractometer Control Software*; data reduction: *TEXSAN* (Molecular Structure Corporation, 1992); structure solution: *TEXSAN*; structure refinement: *DIRDIF* (Beurskens, 1984), *MITHRIL* (Gilmore, 1984); molecular graphics: *OR-TEP*II (Johnson, 1976).

References

Allen, F. H., Davies, J. E., Galloy, J. J., Johnson, O., Kennard, O., Mitchell, E. M., Mitchell, G. F., Smith, J. M. & Watson, D. G. (1991). *J. Chem. Inf. Comput. Sci.* **31**, 187–204.

Beurskens, P. T. (1984). *DIRDIF. Direct Methods for Difference Structures – an Automatic Procedure for Phase Extension and Refinement of Difference Structure Factors.* Technical Report 1984/1. Crystallography Laboratory, Toernooiveld, 6525 ED Nijmegen, The Netherlands.

Chatterjee, A. & Sen Gupta, S. P. (1977). *Acta Cryst.* B**33**, 3593–3595.

Cromer, D. T. & Waber, J. T. (1974). *International Tables for X-ray Crystallography*, Vol. IV, Table 2.2A. Birmingham: Kynoch Press. (Present distributor Kluwer Academic Publishers, Dordrecht.)

Dhubhghaill, O. M. N. & Sadler, P. J. (1991). *The Structure and Reactivity of Arsenic Compounds; Biological Activity and Drug Design. Structure and Bonding*, pp. 130–190. Berlin: Springer-Verlag.

Flack, H. D. & Vincent, M. G. (1980). *Acta Cryst.* A**36**, 495–496.

Gilmore, C. J. (1984). *J. Appl. Cryst.* **17**, 42–46.

Ibers, J. A. & Hamilton, W. C. (1964). *Acta Cryst.* **17**, 781–782.

Johnson, C. K. (1976). Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.

Molecular Structure Corporation (1988). *MSC/AFC Diffractometer Control Software*. MSC, 3200 Research Forest Drive, The Woodlands, TX 77381, USA.

Molecular Structure Corporation (1992). *TEXSAN. TEXRAY Single Crystal Structure Analysis Package*. MSC, 3200 Research Forest Drive, The Woodlands, TX 77381, USA.

Walker, N. & Stuart, D. (1983). *Acta Cryst.* A**39**, 158–166.

N-(2-Hydroxyphenyl)salicylaldimine

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Abstract

The title compound, 2-(salicylideneamino)phenol, $C_{13}H_{11}NO_2$, a potential tridentate ligand, has two crystallographically independent molecules in the asymmetric unit, one of which is disordered. Intramolecular hydrogen bonds occur between the pairs of atoms O(16) and N(8) [2.625 (7) Å], and O(1) and O(16) [3.571 (7) Å], the H atoms essentially being bonded to the O atoms.

Comment

Schiff base compounds have been used extensively as ligands in the field of coordination chemistry. Among these bases, salicylaldimines are of interest since intramolecular hydrogen bonding between O and N atoms plays an important role in the formation of metal complexes. Some analytical data are available on the O—H···N hydrogen-bond strength of 2-hydroxyaldimines (Freedman, 1961), but the situation for 2,2'-dihydroxyaldimines has not been explained. Recently, the hydrogen bonding in various Schiff bases was investigated by a potentiometric method (Gündüz, Gündüz, Kilic,

Atakol & Köseoglu, 1991). This suggested that intramolecular hydrogen bonding in the title compound, (I), occurs between two O atoms, leading to a nine-membered ring. However, the crystal structure determination indicates the existence of both O$\cdots$N and O$\cdots$O hydrogen bonds as follows: O(16)$\cdots$N(8) 2.625 (7) and O(1)$\cdots$O(16) 3.571 (7) Å, with the H atoms essentially being bonded to the O atoms.

(I)
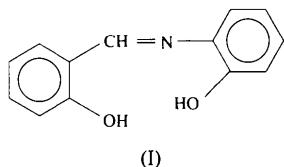
The two Schiff base moieties, O(1), C(2), C(3), C(4), C(5), C(6), C(7) and N(8), and N(8), C(9), C(10), C(11), C(12), C(13), C(14), C(15) and O(16) [both planar with a maximum deviation of 0.046 (7) Å], are inclined at an angle of 13.2 (7)° with respect to each other.
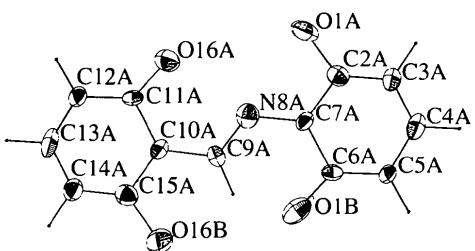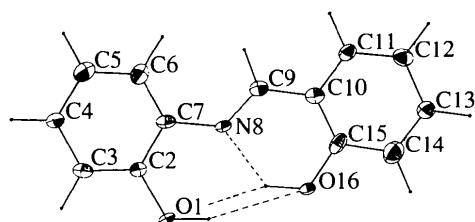

Fig. 1. The molecular structure of the title compound. Anisotropic displacement ellipsoids represent 50% probability boundaries and H atoms are presented as spheres of arbitrary radii.

Experimental

The title compound was obtained from the reaction of 2-aminophenol (0.01 mol) with a solution of salicylaldehyde (0.01 mol) in 50 ml of ethanol. The precipitate which separated from the solution was recrystallized for 5–6 d from 2 g of Schiff base in 25 ml of dioxane solution.

Crystal data

$C_{13}H_{11}NO_2$
$M_r = 213.24$

Mo $K\alpha$ radiation
$\lambda = 0.71069$ Å

Monoclinic
$C2/c$
$a = 38.480\,(10)$ Å
$b = 9.046\,(2)$ Å
$c = 12.344\,(1)$ Å
$\beta = 106.08\,(3)°$
$V = 4129\,(2)$ Å^3
$Z = 16$
$D_x = 1.372$ Mg m^{-3}

Cell parameters from 25 reflections
$\theta = 4.25{-}17.23°$
$\mu = 0.087$ mm^{-1}
$T = 300$ K
Prism
$1.0 \times 0.8 \times 0.3$ mm
Orange

Data collection

Enraf–Nonius CAD-4 diffractometer
$\omega{-}2\theta$ scans
Absorption correction:
  ψ scan (SDP; Frenz, 1985)
  $T_{min} = 0.704$, $T_{max} = 0.996$
5732 measured reflections
2497 independent reflections

1771 observed reflections
  [$F > 4\sigma(F)$]
$R_{int} = 0.044$
$\theta_{max} = 23°$
$h = -42 \rightarrow 42$
$k = 0 \rightarrow 9$
$l = -13 \rightarrow 13$
3 standard reflections
  frequency: 120 min
  intensity decay: 18.8%

Refinement

Refinement on F
$R = 0.09$
$wR = 0.09$
$S = 1.15$
1771 reflections
307 parameters
$w = 1/\sigma^2(F)$
$(\Delta/\sigma)_{max} = 0.276$
  [U_{22} for O(1A)]

$\Delta\rho_{max} = 0.315$ e Å^{-3}
$\Delta\rho_{min} = -0.449$ e Å^{-3}
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV, Table 2.3.1)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters* (Å^2)

$$U_{eq} = (1/3)\Sigma_i\Sigma_j U_{ij}a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	U_{eq}
O(1)	0.2252 (1)	0.1901 (5)	0.1782 (3)	0.040 (2)
C(2)	0.2046 (2)	0.2141 (7)	0.0658 (4)	0.033 (2)
C(3)	0.1761 (3)	0.3115 (8)	0.0452 (6)	0.048 (2)
C(4)	0.1561 (3)	0.3334 (9)	-0.0684 (5)	0.051 (2)
C(5)	0.1639 (3)	0.2492 (9)	-0.1536 (7)	0.073 (2)
C(6)	0.1920 (2)	0.1492 (8)	-0.1254 (6)	0.061 (2)
C(7)	0.2124 (2)	0.1340 (7)	-0.0193 (5)	0.038 (2)
N(8)	0.2410 (1)	0.0303 (5)	0.0149 (3)	0.032 (2)
C(9)	0.2575 (2)	-0.0435 (7)	-0.0484 (5)	0.040 (2)
C(10)	0.2856 (2)	-0.1445 (7)	-0.0095 (5)	0.040 (2)
C(11)	0.3009 (2)	-0.2120 (8)	-0.0847 (5)	0.044 (2)
C(12)	0.3301 (2)	-0.3055 (8)	-0.0518 (6)	0.053 (2)
C(13)	0.3437 (3)	-0.3352 (9)	0.0668 (6)	0.055 (2)
C(14)	0.3282 (2)	-0.2698 (9)	0.1416 (8)	0.073 (2)
C(15)	0.2989 (2)	-0.1769 (7)	0.1006 (5)	0.048 (2)
O(16)	0.2845 (1)	-0.1148 (4)	0.1826 (3)	0.039 (2)
O(1A)	0.0269 (3)	0.0668 (13)	0.3136 (8)	0.104 (2)
O(1B)	0.0350 (3)	0.1395 (11)	0.6841 (9)	0.098 (2)
C(2A)	0.0510 (3)	0.0390 (10)	0.4181 (6)	0.068 (2)
C(3A)	0.0768 (3)	-0.0644 (9)	0.4492 (8)	0.069 (2)
C(4A)	0.0933 (4)	-0.0891 (10)	0.5569 (8)	0.080 (2)
C(5A)	0.0823 (2)	-0.0211 (8)	0.6432 (6)	0.061 (2)
C(6A)	0.0543 (2)	0.0793 (8)	0.6175 (5)	0.058 (2)
C(7A)	0.0374 (2)	0.1139 (8)	0.4993 (6)	0.053 (2)
N(8A)	0.0084 (2)	0.2170 (8)	0.4639 (6)	0.083 (2)
C(9A)	-0.0059 (2)	0.2816 (8)	0.5355 (6)	0.060 (2)
C(10A)	-0.0352 (2)	0.3816 (8)	0.4976 (6)	0.055 (2)
C(11A)	-0.0522 (2)	0.4136 (9)	0.3792 (5)	0.062 (2)
C(12A)	-0.0803 (3)	0.5115 (9)	0.3541 (7)	0.074 (2)
C(13A)	-0.0917 (4)	0.5754 (11)	0.4391 (9)	0.091 (2)

$C_{13}H_{11}NO_2$

C(14A)	−0.0750 (3)	0.5548 (10)	0.5449 (7)	0.069 (2)
C(15A)	−0.0491 (3)	0.4543 (10)	0.5790 (7)	0.074 (2)
O(16A)	−0.0232 (3)	0.4345 (13)	0.6825 (8)	0.103 (2)
O(16B)	−0.0312 (3)	0.3642 (12)	0.3118 (9)	0.092 (2)

Table 2. *Selected geometric parameters* (Å, °)

O(1)—C(2)	1.411 (5)	O(1B)—C(6A)	1.363 (8)
C(7)—N(8)	1.418 (8)	C(7A)—N(8A)	1.428 (7)
N(8)—C(9)	1.317 (6)	N(8A)—C(9A)	1.301 (7)
C(9)—C(10)	1.395 (9)	C(9A)—C(10A)	1.419 (7)
O(16)—C(15)	1.399 (6)	O(16A)—C(15A)	1.397 (8)
O(1A)—C(2A)	1.388 (8)	O(16B)—C(11A)	1.385 (8)
C(7)—C(2)—O(1)	119.2 (5)	C(7A)—C(6A)—O(1B)	110.6 (8)
N(8)—C(7)—C(2)	115.8 (5)	N(8A)—C(7A)—C(2A)	120.3 (6)
N(8)—C(7)—C(6)	123.9 (6)	N(8A)—C(7A)—C(6A)	122.8 (7)
C(9)—N(8)—C(7)	128.4 (5)	C(9A)—N(8A)—C(7A)	121.8 (8)
C(10)—C(9)—N(8)	125.7 (6)	C(9A)—C(10A)—C(11A)	123.6 (7)
C(11)—C(10)—C(9)	119.7 (6)	C(9A)—C(10A)—C(15A)	118.8 (7)
C(15)—C(10)—C(9)	122.2 (6)	C(10A)—C(9A)—N(8A)	120.5 (8)
O(16)—C(15)—C(10)	121.8 (6)	O(16A)—C(15A)—C(10A)	105.8 (8)
C(7A)—C(2A)—O(1A)	106.6 (8)	O(16B)—C(11A)—C(10A)	111.3 (8)

Refinement was based on full-matrix least-squares methods with H atoms in calculated positions (C—H 1.08 Å), except for the H atoms at O(1) (0.821 Å) and O(16) (0.863 Å), which were taken from a difference Fourier map and whose coordinates were kept fixed during the refinement, together with those of the C(9) and C(9A) H atoms. The coordinates and isotropic temperature factors were kept fixed during refinement for all H atoms. The bond lengths C(15)—O(16), O(1)—C(2), N(8)—C(9), O(18)—C(6A), N(8A)—C(9A), O(1A)—C(2A), O(16B)—C(15A), C(11A)—O(16A), C(7A)—N(8A) and C(9A)—C(10A) were constrained during the refinement. The relatively high residuals can be attributed to the problem of modelling disorder in the second molecule coupled with radiation damage and weak diffraction (pairs of disordered atoms are labelled A and B)

Data collection: *Enraf–Nonius Structure Determination Package* (Frenz, 1985). Cell refinement: *Enraf–Nonius Structure Determination Package*. Data reduction: *Enraf–Nonius Structure Determination Package*. Program(s) used to solve structure: *SHELXS86* (Sheldrick, 1985). Program(s) used to refine structure: *SHELX76* (Sheldrick, 1976). Molecular graphics: *ORTEP*II (Johnson, 1976).

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: LI1119). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

References

Freedman, H. H. (1961). *J. Am. Chem. Soc.* **83**, 2900–2906.
Frenz, B. A. (1985). *Enraf–Nonius Structure Determination Package*. Enraf–Nonius, Delft, The Netherlands.
Gündüz, T., Gündüz, N., Kilic, E., Atakol, O. & Köseoglu, F. (1991). *Anal. Chim. Acta*, **249**, 427–431.
Johnson, C. K. (1976). *ORTEP*II. Report ORNL-5138. Oak Ridge National Laboratory, Tennesse, USA.
Sheldrick, G. M. (1976). *SHELX76. Program for Crystal Structure Determination*. Univ. of Cambridge, England.
Sheldrick, G. M. (1985). *SHELXS86. Program for the Solution of Crystal Structures*. Univ. of Göttingen, Germany.

Aminoethylammonium Tartrate

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Abstract

The structure of aminoethylammonium tartrate, C_2H_9-$N_2^+ \cdot C_4H_5O_6^-$, is ionic. Ethylenediamine forms a very stable salt with tartaric acid, similar to $C_2H_{10}N_2^{2+} \cdot$-$2HPO_4^{2-} \cdot 6H_2O$ [Averbuch-Pouchot, Durif & Guitel (1987). *Acta Cryst.* C43, 1896–1898] and $C_2H_{10}N_2^{2+} \cdot$-$2C_4H_5O_6^- \cdot 2H_2O$ [Perez, (1977). *Acta Cryst.* B33, 1083–1087]. The protonated ethylenediamine monocations are linked to the tartrate anions by strong N—H···O hydrogen bonds [N···O 2.921 (4), H···O 2.083 (4) Å, N—H···O 156.8 (3)°]. The bond lengths and angles are comparable with corresponding values observed in related molecules.

Comment

The cation and anion in the title salt, (I), do not display any unusual structural features, the C—C—C—C chain of the tartrate anion being antiperiplanar with a torsion angle value of 170.4 (3)°. The two halves of the tartrate anion, consisting of atoms C1, C2, O1, O2 and O3, and C3, C4, O4, O5 and O6, form individual planes, the dihedral angle between them being 32.16 (12)°. This dihedral angle is significantly smaller than that observed in other tartrates, *e.g.* 41° in manganese(II) L-tartrate (Soylu, 1985), 54.6° in D-tartaric acid (Okaya, Stemple & Kay, 1966), 57.3° in potassium hydrogen

(I)