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Structure of *o*-(Salicylideneamino)phenol Hydrochloride

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Abstract

The cation $[C_{13}H_{12}NO_2]^+$ [(2-hydroxyphenyl)(salicylidene)ammonium] is nearly planar with an angle of 7.3 (1)° between planes C2–C7 and C10–C15 of the phenyl rings. The molecules are linked together by O1—HO1···Cl···HO16—O16 planar hydrogen bonds between the hydroxyl groups.

Comment

The fluorescence and physicochemical properties of chelates of this compound and its derivatives have been reported (Holzbecher, 1953; Babko, Volkova & Getman, 1967; Yamamoto, 1991). This is the first X-ray study of one of these compounds.

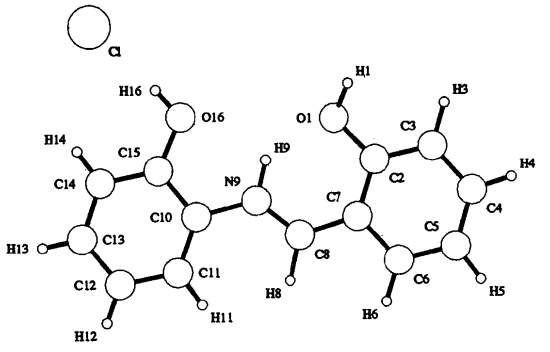

Fig. 1. View of the molecule with atom numbering.

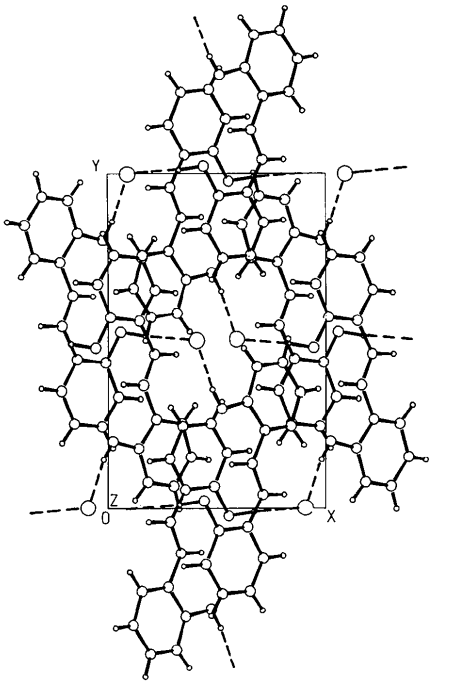

Fig. 2. Packing scheme. Hydrogen bonds are designated by dashed lines.

Experimental

Crystal data

$C_{13}H_{12}NO_2^+.Cl^-$
M_r = 249.70
Monoclinic
$P2_1/n$
a = 8.449 (1) Å
b = 12.660 (2) Å
c = 11.250 (3) Å
β = 94.05 (2)°
V = 1200.3 (4) Å^3
Z = 4
D_x = 1.38 Mg m^{-3}
Mo $K\alpha$ radiation
λ = 0.71073 Å
Cell parameters from 20 reflections
θ = 18–19°
μ = 0.30 mm^{-1}
T = 295 K
Plate
0.30 × 0.30 × 0.05 mm
Olive green
Crystal source: recrystallization from 95% methanol

Data collection

CAD-4 diffractometer
$\omega/2\theta$ scans
Absorption correction: none
3512 measured reflections
1618 independent reflections
1230 observed reflections [$I > 1.96\sigma(I)$]
R_{int} = 0.029
θ_{max} = 23°
$h = -9 \rightarrow 9$
$k = 0 \rightarrow 13$
$l = -12 \rightarrow 12$
2 standard reflections
frequency: 180 min
intensity variation: −0.7%

Refinement

Refinement on F	Unit weights applied
Final $R = 0.033$	$(\Delta/\sigma)_{max} = 0.001$
$wR = 0.033$	$\Delta\rho_{max} = 0.23$ e Å^{-3}
$S = 0.67$	$\Delta\rho_{min} = -0.16$ e Å^{-3}
1230 reflections	Extinction correction: none
202 parameters	Atomic scattering factors from *SHELX*76
All H-atom parameters refined	

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

$U_{eq} = \frac{1}{3}[U_{22} + 1/\sin^2\beta(U_{11} + U_{33} + 2U_{13}\cos\beta)]$.

	x	y	z	U_{eq}
Cl	0.0915 (1)	0.9981 (1)	0.1893 (1)	0.0542 (2)
O1	0.0568 (3)	0.5231 (2)	0.2830 (2)	0.0546 (7)
O16	−0.0222 (3)	0.7998 (2)	0.3073 (2)	0.0569 (7)
N9	−0.1629 (3)	0.6327 (2)	0.3870 (2)	0.0434 (8)
C2	−0.0112 (3)	0.4334 (2)	0.3213 (2)	0.0425 (9)
C3	0.0471 (4)	0.3338 (3)	0.2957 (3)	0.0505 (10)
C4	−0.0275 (4)	0.2459 (3)	0.3379 (3)	0.0563 (11)
C5	−0.1564 (4)	0.2540 (3)	0.4047 (3)	0.0593 (12)
C6	−0.2160 (4)	0.3520 (3)	0.4299 (3)	0.0522 (11)
C7	−0.1439 (3)	0.4435 (2)	0.3874 (2)	0.0406 (9)
C8	−0.2129 (4)	0.5418 (2)	0.4188 (3)	0.0437 (9)
C10	−0.2210 (3)	0.7340 (2)	0.4219 (2)	0.0399 (8)
C11	−0.3412 (4)	0.7466 (3)	0.4960 (3)	0.0496 (10)
C12	−0.3831 (4)	0.8468 (3)	0.5310 (3)	0.0579 (11)
C13	−0.3028 (4)	0.9331 (3)	0.4899 (3)	0.0576 (11)
C14	−0.1836 (4)	0.9211 (2)	0.4147 (3)	0.0510 (10)
C15	−0.1407 (3)	0.8205 (2)	0.3787 (2)	0.0431 (9)

Table 2. *Geometric parameters* (Å, °)

O1—C2	1.356 (4)	C10—C11	1.368 (4)
O16—C15	1.353 (4)	C10—C15	1.393 (4)
N9—C8	1.286 (4)	C11—C12	1.382 (5)
N9—C10	1.438 (4)	C12—C13	1.382 (5)
C2—C3	1.391 (5)	C13—C14	1.369 (5)
C2—C7	1.394 (4)	C14—C15	1.392 (4)
C3—C4	1.380 (5)	Cl···H1^{i}	2.02 (3)
C4—C5	1.370 (5)	Cl···H16	2.18 (4)
C5—C6	1.376 (5)	O1—H1	1.00 (3)
C6—C7	1.409 (5)	O16—H16	0.86 (4)
C7—C8	1.429 (4)		
O1—C2—C7	117.8 (2)	C2—C7—C6	119.3 (3)
O1—C2—C3	122.0 (3)	C6—C7—C8	116.0 (3)
O16—C15—C14	124.7 (2)	C2—C7—C8	124.7 (2)
O16—C15—C10	117.0 (2)	C11—C10—C15	121.4 (3)
N9—C10—C15	114.9 (2)	C10—C11—C12	119.8 (3)
N9—C10—C11	123.6 (3)	C11—C12—C13	119.3 (3)
C8—N9—C10	126.6 (3)	C12—C13—C14	121.2 (3)
C7—C8—N9	124.2 (3)	C13—C14—C15	120.0 (3)
C3—C2—C7	120.2 (3)	C10—C15—C14	118.3 (2)
C2—C3—C4	118.9 (3)	H1^{i}···Cl···H16	106.1 (14)
C3—C4—C5	121.9 (4)	Cl···H1^{i}—O1^{i}	162 (2)
C4—C5—C6	119.8 (3)	Cl···H16—O16	171 (4)
C5—C6—C7	119.9 (3)		

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

Equimolar amounts of salicylaldehyde and *o*-aminophenol in ethanol were mixed as described by Haegele (1992) from which red needle crystals of *o*-(salicylideneamino)phenol were precipitated. Olive-green crystals of the hydrochloride were obtained by recrystallization from 95% methanol after acidifying with 10% hydrochloric acid.

The structure was solved by direct methods (*SHELXS*86; Sheldrick, 1986) and refined anisotropically by block-diagonal least squares in three blocks (*SHELX*76; Sheldrick, 1976). H atoms were located from a $\Delta\rho$ map and expected geometry. All H atoms were refined isotropically. Other computer programs used: *SDP-Plus* (B. A. Frenz & Associates, Inc., 1984); *PARST* (Nardelli, 1991).

Lists of structure factors, anisotropic thermal parameters, H-atom coordinates and bond distances and angles involving H atoms have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 71267 (9 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: LI1045]

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Structure of Diphenanthro[1,2-*b*;2′,1′-*d*]-furan at 191 K

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Abstract

Diphenanthro[1,2-*b*;2′,1′-*d*]furan crystallizes in space group *Pnma* (No. 62) with the mirror plane bisecting the molecule and passing through the furan O atom. Although exhibiting packing similar to that of dibenzofuran, this diphenanthrofuran showed no disorder of the sort found in that close molecular analog. The C atoms of the individual phenanthrene rings of the title compound have a mean deviation of 0.027 (21) Å from the best least-squares plane describing the rings. The configuration and conformation of the individual phenanthrene rings in this