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The crystal structure of (*Z*)-6-(((5-chloro-2-hydroxyphenyl)amino)methylene)-4-nitrocyclohexa, $C_{13}H_9ClN_2O_4$

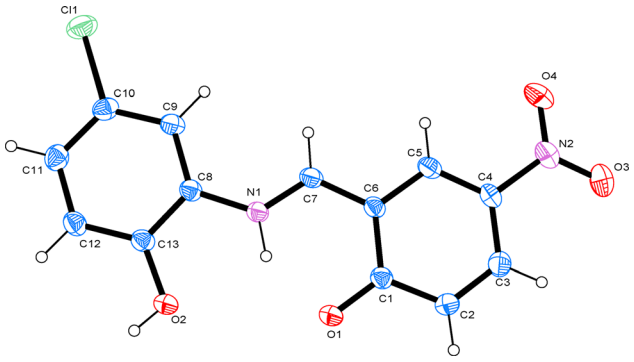

Table 1: Data collection and handling.

Crystal:	Yellow block
Size:	0.20 × 0.20 × 0.20 mm
Wavelength:	Mo *K*α radiation (0.71073 Å)
μ:	0.5 mm^{-1}
Diffractometer, scan mode:	Bruker SMART, φ and ω-scans
θ_{max}, completeness:	50°, >99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	28278, 7220, 0.055
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 4975
$N(param)_{refined}$:	582
Programs:	Bruker programs [1], SHELX [2, 3], WinGX [4]

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Abstract

$C_{13}H_9ClN_2O_4$, monoclinic, $P2_1/n$ (no. 14), a = 11.1882(11) Å, b = 8.0672(8) Å, c = 14.0813(15) Å, β = 94.084(2)°, V = 1267.7(2) $Å^3$, Z = 4, $R_{gt}(F)$ = 0.0427, $wR_{ref}(F^2)$ = 0.1104, T = 296(2) K.

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The molecular structure is shown in the figure. Table 1 contains crystallographic data and Table 2 contains the list of the atoms including atomic coordinates and displacement parameters.

Source of material

All chemicals were purchased from commercial sources and used as received without further purification. The 5-nitrosalicylaldehyde (10 mmol, 1.67 g) and 2-amino-4-chlorophenol (10 mmol, 1.44 g) were dissolved in MeOH (20 mL). The mixture was refluxed for 6 h, and then the precipitate was collected by filtration. The solid was filtered out and the title product was obtained by recrystallization from methanol.

Experimental details

All H-atoms bonded to C atoms were placed geometrically and refined using a riding model with common isotropic displacement factors U_{iso}(H) = 1.2 or 1.5 U_{eq}(parent C-atom).

Comment

Schiff bases have found widespread use in many disciplines. In the field of coordination chemistry, Schiff bases are important organic ligands for example [5, 6]. Because they contain the C=N group, the N atom contains lone electron pairs, and all kinds of functional atomic groups can be introduced around the C=N group, they are widely studied in the fields of chemistry and biology. Salicylidene-*o*-aminophenol are known to be a class of versatile Schiff bases ligands, capable of generating different molecular architectures and coordination polyhedral [7].

Single-crystal structure analysis revealed that the title compound crystallized in the monoclinic space group

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Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2).

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
Cl1	0.96355 (6)	0.19505 (8)	1.14732 (4)	0.0698 (2)
C1	0.65361 (17)	1.0021 (2)	0.92808 (13)	0.0412 (4)
C2	0.60136 (18)	1.1632 (2)	0.91906 (14)	0.0478 (5)
H2	0.577525	1.203771	0.858853	0.057*
C3	0.58588 (18)	1.2578 (3)	0.99644 (14)	0.0491 (5)
H3	0.552099	1.362764	0.988995	0.059*
C4	0.62043 (18)	1.1986 (2)	1.08769 (14)	0.0454 (5)
C5	0.67090 (18)	1.0452 (2)	1.10125 (13)	0.0455 (5)
H5	0.693287	1.007998	1.162452	0.055*
C6	0.68862 (17)	0.9448 (2)	1.02266 (12)	0.0391 (4)
C7	0.74210 (18)	0.7868 (2)	1.03703 (13)	0.0432 (5)
H7	0.764423	0.752649	1.098832	0.052*
C8	0.81586 (17)	0.5305 (2)	0.96850 (12)	0.0397 (4)
C9	0.85907 (18)	0.4513 (2)	1.05135 (13)	0.0436 (5)
H9	0.852714	0.500656	1.110466	0.052*
C10	0.91148 (18)	0.2982 (2)	1.04413 (14)	0.0469 (5)
C11	0.92281 (19)	0.2250 (3)	0.95649 (15)	0.0505 (5)
H11	0.95948	0.121933	0.952987	0.061*
C12	0.88005 (18)	0.3040 (2)	0.87451 (15)	0.0472 (5)
H12	0.887869	0.254449	0.815636	0.057*
C13	0.82525 (18)	0.4576 (2)	0.87963 (13)	0.0428 (5)
O1	0.66873 (13)	0.91151 (17)	0.85556 (9)	0.0534 (4)
O2	0.77827 (15)	0.54304 (17)	0.80354 (9)	0.0569 (4)
H2A	0.797015	0.497992	0.754551	0.085*
O3	0.5662 (2)	1.4437 (2)	1.15486 (14)	0.1115 (8)
O4	0.63459 (18)	1.2510 (2)	1.24904 (12)	0.0885 (6)
N1	0.76101 (15)	0.68826 (19)	0.96691 (11)	0.0410 (4)
N2	0.60616 (19)	1.3044 (3)	1.16911 (14)	0.0664 (6)
H1	0.7356 (18)	0.733 (3)	0.9099 (15)	0.064 (7)

*: U_{iso}.

$P2_1/n$. The title molecule exists as a zwitterion (ylide). The ORTEP diagram is presented in the figure. Bond lengths and angles are all in the expected ranges. The title molecule exhibits an *E* configuration. The bond length of C7=N1 is 1.297 Å, which is similar to those reported in the literature [8–10]. The bond length of C7–C6 is 1.416 Å, shorter than normal C–C single bonds (1.54 Å). The bond length of C10–Cl1 is 1.739 Å, respectively, which is similar to those reported in the literature [11].

Intramolecular and intermolecular hydrogen bonding exist in the crystal structure of the title compound. The oxygen atom N1 provides one intramolecular hydrogen bond to O1 (N1···O1 = 2.557(2) Å). The carbon atom C7 provides one weak intermolecular hydrogen bond to O4′ of another molecule (C7···O4′ = 3.236(3) Å; ′ = $-x + 3/2$, $y - 1/2$, $-z + 5/2$). The oxygen atom O2 provides one classical intermolecular hydrogen bond to O1″ of another molecule (O2···O1″ = 2.586(2) Å; ″ = $-x + 3/2$, $y - 1/2$, $-z + 3/2$).

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Conflict of interest statement: The authors declare no conflicts of interest regarding this article.

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