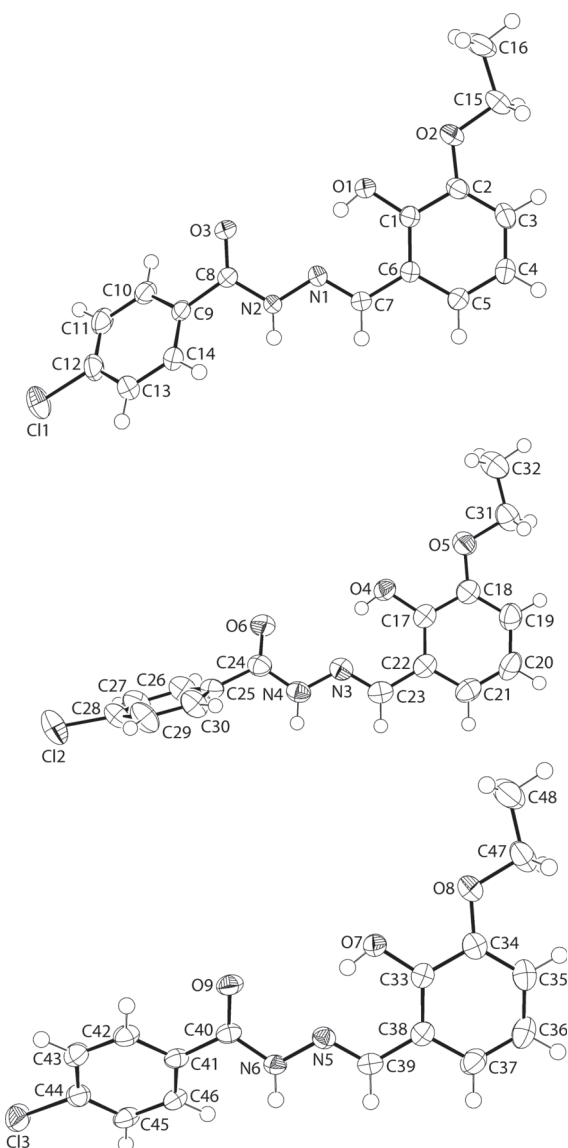


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# Crystal structure of 4-chloro-*N'*-[(1*E*)-(3-ethoxy-2-hydroxyphenyl)methylidene]benzohydrazide – a *Z'* = 3 structure, C<sub>16</sub>H<sub>15</sub>ClN<sub>2</sub>O<sub>3</sub>



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## Abstract

C<sub>16</sub>H<sub>15</sub>ClN<sub>2</sub>O<sub>3</sub>, orthorhombic, *Pbca* (no. 61),  $a = 18.7178(2)$  Å,  $b = 15.2296(2)$  Å,  $c = 32.0558(3)$  Å,  $V = 9137.97(18)$  Å<sup>3</sup>,  $Z = 24$ ,  $R_{gt}(F) = 0.0407$ ,  $wR_{ref}(F^2) = 0.1099$ ,  $T = 100(2)$  K.

CCDC no.: 1946616

The three molecules comprising the asymmetric unit are shown in the figure. Table 1 contains crystallographic data and Table 2 contains the list of the atoms including atomic coordinates and displacement parameters.

**Table 1:** Data collection and handling.

Crystal:	Yellow block
Size:	0.30 × 0.24 × 0.12 mm
Wavelength:	Cu <i>K</i> α radiation (1.54184 Å)
$\mu$ :	2.35 mm <sup>-1</sup>
Diffractometer, scan mode:	SuperNova, $\omega$
$\theta_{max}$ , completeness:	76.6°, >99%
$N(hkl)_{measured}$ , $N(hkl)_{unique}$ , $R_{int}$ :	31906, 9519, 0.034
Criterion for $I_{obs}$ , $N(hkl)_{gt}$ :	$I_{obs} > 2 \sigma(I_{obs})$ , 8480
$N(param)_{refined}$ :	616
Programs:	CrysAlis <sup>PRO</sup> [1], SHELX [2, 3], WinGX/ORTEP [4]

## Source of material

**General:** The melting point of the compound was measured on a Electrothermal digital melting point apparatus and was uncorrected. The elemental analysis was performed on a Perkin-Elmer EA2400 CHN analyser. The IR spectrum was recorded using a Perkin-Elmer RX1 spectrophotometer equipped as a Nujol mull in between KBr cell from 4000 to 400 cm<sup>-1</sup>. The <sup>1</sup>H and <sup>13</sup>C{<sup>1</sup>H} NMR spectra were recorded in CDCl<sub>3</sub> solution on a Bruker AVN FT-NMR 400 MHz NMR spectrometer with chemical shifts relative to tetramethylsilane.

**Synthesis:** 4-Chlorobenzhydrazide (Fluka, 0.85 g, 5.0 mmol) and 3-ethoxysalicylaldehyde (Aldrich, 0.83 g, 5.0 mmol) were dissolved in methanol (50 mL) and refluxed for 3 h. The mixture was filtered and allowed to stand at

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**Table 2:** Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å<sup>2</sup>).

Atom	x	y	z	$U_{iso}^*/U_{eq}$
Cl1	0.70677(3)	0.23421(4)	0.08440(2)	0.04725(13)
Cl2	1.02412(3)	0.40652(4)	0.45858(2)	0.04883(14)
Cl3	0.70514(2)	0.96072(3)	0.10114(2)	0.03421(11)
O1	0.51429(6)	0.28828(8)	0.38072(3)	0.0256(2)
H10	0.5356(11)	0.2820(15)	0.3580(4)	0.038*
O2	0.46888(6)	0.29100(8)	0.45669(3)	0.0294(2)
O3	0.52798(6)	0.26789(8)	0.26463(3)	0.0255(2)
O4	0.67005(6)	0.56582(8)	0.23750(3)	0.0264(2)
H40	0.6892(12)	0.5517(15)	0.2602(4)	0.040*
O5	0.59621(7)	0.59094(8)	0.16997(4)	0.0328(3)
O6	0.80607(7)	0.56269(10)	0.31806(4)	0.0396(3)
O7	0.67117(5)	0.64894(7)	0.37859(3)	0.0218(2)
H70	0.6634(12)	0.6760(13)	0.3561(4)	0.033*
O8	0.67684(6)	0.54346(8)	0.44266(3)	0.0283(2)
O9	0.71534(5)	0.78524(8)	0.29089(3)	0.0241(2)
N1	0.61641(6)	0.26216(8)	0.32920(4)	0.0198(2)
H2N	0.6870(5)	0.2555(13)	0.2839(6)	0.024*
N2	0.64083(6)	0.25484(9)	0.28917(4)	0.0206(2)
N3	0.67912(7)	0.49141(9)	0.30934(4)	0.0255(3)
N4	0.72222(8)	0.46748(9)	0.34182(4)	0.0262(3)
H4N	0.7052(11)	0.4381(13)	0.3631(5)	0.031*
N5	0.60035(7)	0.70692(8)	0.31556(4)	0.0198(2)
N6	0.60059(6)	0.75046(8)	0.27797(4)	0.0196(2)
H6N	0.5608(7)	0.7556(13)	0.2637(5)	0.023*
C1	0.56437(8)	0.27817(10)	0.41057(4)	0.0199(3)
C2	0.54069(8)	0.27973(10)	0.45242(5)	0.0218(3)
C3	0.58974(9)	0.26936(10)	0.48449(5)	0.0246(3)
H3	0.5742	0.2717	0.5127	0.030*
C4	0.66195(9)	0.25553(11)	0.47545(5)	0.0263(3)
H4	0.6950	0.2473	0.4976	0.032*
C5	0.68583(8)	0.25371(10)	0.43487(5)	0.0232(3)
H5	0.7351	0.2448	0.4291	0.028*
C6	0.63712(8)	0.26512(9)	0.40187(4)	0.0189(3)
C7	0.66222(7)	0.25704(10)	0.35905(4)	0.0195(3)
H7	0.7115	0.2482	0.3533	0.023*
C8	0.59214(8)	0.25925(10)	0.25817(4)	0.0196(3)
C9	0.62332(7)	0.25333(10)	0.21538(4)	0.0203(3)
C10	0.59394(8)	0.30620(11)	0.18417(5)	0.0252(3)
H10	0.5561	0.3456	0.1906	0.030*
C11	0.62024(9)	0.30093(12)	0.14376(5)	0.0296(3)
H11	0.6009	0.3368	0.1223	0.036*
C12	0.67504(9)	0.24271(12)	0.13525(5)	0.0296(3)
C13	0.70541(9)	0.19004(12)	0.16578(5)	0.0274(3)
H13	0.7432	0.1508	0.1592	0.033*
C14	0.67928(8)	0.19604(10)	0.20624(4)	0.0233(3)
H14	0.6996	0.1610	0.2277	0.028*
C15	0.43933(10)	0.29242(15)	0.49749(5)	0.0374(4)
H15A	0.4521	0.3479	0.5118	0.045*
H15B	0.4583	0.2428	0.5141	0.045*
C16	0.35951(10)	0.28498(14)	0.49350(6)	0.0378(4)
H16A	0.3417	0.3317	0.4752	0.057*
H16B	0.3375	0.2907	0.5211	0.057*
H16C	0.3472	0.2277	0.4816	0.057*
C17	0.60095(9)	0.53996(10)	0.23869(5)	0.0250(3)
C18	0.55938(9)	0.55638(11)	0.20300(5)	0.0284(3)

**Table 2 (continued)**

Atom	x	y	z	$U_{iso}^*/U_{eq}$
C19	0.48673(10)	0.53751(12)	0.20322(6)	0.0341(4)
H19	0.4583	0.5508	0.1795	0.041*
C20	0.45546(10)	0.49912(13)	0.23816(6)	0.0375(4)
H20	0.4055	0.4879	0.2384	0.045*
C21	0.49641(10)	0.47738(11)	0.27228(6)	0.0331(4)
H21	0.4750	0.4489	0.2955	0.040*
C22	0.56960(9)	0.49704(10)	0.27305(5)	0.0265(3)
C23	0.61245(9)	0.47228(10)	0.30918(5)	0.0267(3)
H23	0.5913	0.4426	0.3321	0.032*
C24	0.78782(9)	0.50665(11)	0.34305(5)	0.0277(3)
C25	0.83791(9)	0.47564(11)	0.37638(5)	0.0255(3)
C26	0.86842(11)	0.53822(11)	0.40257(5)	0.0337(4)
H26	0.8513	0.5969	0.4020	0.040*
C27	0.92309(11)	0.51612(12)	0.42936(5)	0.0346(4)
H27	0.9429	0.5585	0.4477	0.041*
C28	0.94831(10)	0.43126(12)	0.42892(5)	0.0326(4)
C29	0.91801(12)	0.36699(12)	0.40424(6)	0.0377(4)
H29	0.9354	0.3084	0.4051	0.045*
C30	0.86141(11)	0.38974(11)	0.37805(5)	0.0332(4)
H30	0.8390	0.3461	0.3614	0.040*
C31	0.56162(11)	0.58467(13)	0.13011(6)	0.0360(4)
H31A	0.5196	0.6241	0.1292	0.043*
H31B	0.5452	0.5238	0.1252	0.043*
C32	0.61505(12)	0.61098(15)	0.09745(6)	0.0431(4)
H32A	0.6308	0.6714	0.1026	0.065*
H32B	0.5929	0.6074	0.0698	0.065*
H32C	0.6563	0.5714	0.0986	0.065*
C33	0.60971(8)	0.60769(10)	0.38903(4)	0.0204(3)
C34	0.61165(8)	0.55055(10)	0.42375(5)	0.0237(3)
C35	0.55008(9)	0.50664(11)	0.43577(5)	0.0294(3)
H35	0.5513	0.4686	0.4593	0.035*
C36	0.48643(9)	0.51764(12)	0.41383(6)	0.0326(4)
H36	0.4446	0.4876	0.4226	0.039*
C37	0.48397(9)	0.57194(11)	0.37944(5)	0.0283(3)
H37	0.4405	0.5790	0.3645	0.034*
C38	0.54564(8)	0.61702(10)	0.36634(5)	0.0220(3)
C39	0.54352(8)	0.66727(10)	0.32763(5)	0.0217(3)
H39	0.5009	0.6706	0.3117	0.026*
C40	0.66375(7)	0.78660(9)	0.26694(4)	0.0194(3)
C41	0.66906(7)	0.82919(10)	0.22515(4)	0.0197(3)
C42	0.72202(8)	0.89326(10)	0.22056(5)	0.0230(3)
H42	0.7509	0.9087	0.2438	0.028*
C43	0.73302(8)	0.93446(11)	0.18268(5)	0.0252(3)
H43	0.7686	0.9785	0.1797	0.030*
C44	0.69057(8)	0.90969(11)	0.14913(5)	0.0241(3)
C45	0.63763(8)	0.84671(11)	0.15254(5)	0.0237(3)
H45	0.6091	0.8315	0.1291	0.028*
C46	0.62692(7)	0.80602(10)	0.19093(4)	0.0213(3)
H46	0.5909	0.7625	0.1938	0.026*
C47	0.68035(10)	0.48873(12)	0.47914(5)	0.0333(4)
H47A	0.6640	0.4286	0.4724	0.040*
H47B	0.6492	0.5127	0.5013	0.040*
C48	0.75665(11)	0.48689(15)	0.49370(6)	0.0435(5)
H48A	0.7867	0.4607	0.4720	0.065*
H48B	0.7601	0.4519	0.5193	0.065*
H48C	0.7728	0.5469	0.4993	0.065*

room temperature for 2 days whereupon yellow crystals were formed. The crystals were filtered, washed with methanol and air-dried. Yield: 1.35 g (84.7%). M.pt: 433–434 K. Calcd for C<sub>16</sub>H<sub>15</sub>ClN<sub>2</sub>O<sub>3</sub>: C 60.29; H 4.74; N 8.79%. Found: C 60.24; H 4.34; N, 8.78%. IR (cm<sup>-1</sup>): 3448 (br) ν(O–H), 3217 (s) ν(N–H), 1655 (s) ν(C=O), 1610 (s) ν(C=N). <sup>1</sup>H NMR (CDCl<sub>3</sub>, ppm): δ 1.39 (s, 3H, CH<sub>3</sub>), 3.98–4.08 (m, 2H, OCH<sub>2</sub>), 6.77–7.17 (m, 3H, Ar–H), 7.59–7.93 (m, 4H, Ph–H), 8.63 (s, 1H, HCN), 10.88 (s, 1H, N–H), 12.15 (s, 1H, O–H). <sup>13</sup>C{<sup>1</sup>H} NMR (CDCl<sub>3</sub>, ppm): δ 14.7 (CH<sub>3</sub>), 64.2 (CH<sub>2</sub>), 115.4, 118.9, 119.1, 121.0, 128.6, 129.5, 131.5, 136.8, 147.0, 147.5 (Ar–C).

### Experimental details

The C-bound H atoms were geometrically placed (C–H = 0.95–0.99 Å) and refined as riding with  $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$ . The O- and N-bound H-atoms were located in a difference Fourier map but were refined with distance restraints of O–H = 0.84 ± 0.01 Å and N–H = 0.88 ± 0.01 Å, respectively, and with  $U_{\text{iso}}(\text{H})$  set to 1.5 $U_{\text{eq}}(\text{O})$  and 1.2 $U_{\text{eq}}(\text{N})$ , respectively.

### Comment

Crystal structure determinations of neutral benzyltin compounds containing Schiff base ligands related to the title hydrazone molecule, 4-chloro-*N'*-[(1*E*)-(3-ethoxy-2-hydroxyphenyl)methylidene] benzohydrazide, a potentially dianionic tridentate ligand, have sometimes revealed unexpected synthetic outcomes. Thus, while the anticipated (2-FC<sub>6</sub>H<sub>4</sub>CH<sub>2</sub>)<sub>2</sub>Sn(L<sup>1</sup>) product was obtained from the reaction of (2-FC<sub>6</sub>H<sub>4</sub>CH<sub>2</sub>)<sub>2</sub>SnCl<sub>2</sub> and 4-chloro-*N'*-[(1*E*)-(5-chloro-2-hydroxyphenyl)methylidene] benzohydrazide (H<sub>2</sub>L<sup>1</sup>) [5], incompletely substituted species of composition (C<sub>6</sub>H<sub>5</sub>CH<sub>2</sub>)Sn(OHCH<sub>3</sub>)(L<sup>2</sup>)Cl [6] and (4-FC<sub>6</sub>H<sub>4</sub>CH<sub>2</sub>)Sn(OH<sub>2</sub>)(L<sup>3</sup>)Cl [7] were obtained when analogous reactions of the respective di-(substituted-benzyl)tin dichlorides with 4-chloro-*N'*-[(1*E*)-(2-hydroxyphenyl)methylidene] benzohydrazide (H<sub>2</sub>L<sup>2</sup>) and 1-hydroxy-*N'*-[(1*E*)-(2-hydroxy-4-methoxyphenyl)methylidene]naphthalene-2-carbohydrazide (H<sub>2</sub>L<sup>3</sup>), respectively, were conducted. Studies in this area are largely motivated by the biological activity, especially in the context of the quest for new anti-cancer drugs, of organotin derivatives of these molecules [8–10]. Complementing the above, are structural studies of the Schiff bases themselves [11, 12] and it was in this context that the title compound was studied crystallographically.

The crystallographic asymmetric unit of the title structure comprises three independent molecules as shown in the figure (70% displacement ellipsoids). Each independent molecule, hereafter designated as the Cl1-, Cl2- and Cl3-molecules, comprises a central chromophore defined by the C(=O)N(H)N=C atoms which is planar with r.m.s. deviations

of 0.0068 and 0.0171 Å for the Cl1 and Cl3 molecules, respectively. However, a difference in conformation for the Cl2-molecule is noted whereby a twist is evident as seen in the C23–N3–N4–C24 torsion angle of –166.67(14)°. The configuration about each of the imine-N1–C7 [1.2871(19) Å], N3–C23 [1.282(2) Å] and N5–C39 [1.283(2) Å] bonds is *E*. The dihedral angles between the central planes and appended hydroxy- and chloro-phenyl rings also reveal differences in conformation between the independent molecules, i.e. 4.49(3) & 38.97(7)°, 18.47(11) & 59.73(7)° and 9.83(10) and 25.05(7)°, for the Cl1–Cl3-molecules, respectively. The dihedral angles formed between the outer rings are 35.36(6), 77.69(6) and 31.3(7)°, respectively. Each molecule features an intramolecular hydroxy-O–H···N(imine) hydrogen bond [O1–H1o···N1: H1o···N1 = 1.798(19) Å, O1···N1 = 2.5572(16) Å with angle at H1o = 150.3(18) Å, O4–H4o···N3: H4o···N3 = 1.833(16) Å, O4···N3 = 2.5722(17) Å with angle at H4o = 146(2)° and O7–H7o···N5: H7o···N5 = 1.818(18) Å, O7···N5 = 2.5728(16) Å with angle at H7o = 148(2)°].

The crystal of the title compound features supramolecular, zig-zag chains along the *a*-axis direction sustained by amide-N–H···O(carbonyl) hydrogen bonds occurring between the Cl1- and Cl3-molecules [N2–H2n···O9<sup>i</sup>: H2n···O9<sup>i</sup> = 1.897(10) Å, N2···O9<sup>i</sup> = 2.7323(15) Å with angle at H2n = 157.8(18)° and N6–H6n···O3<sup>ii</sup>: H6n···O3<sup>ii</sup> = 1.903(14) Å, N6···O3<sup>ii</sup> = 2.7797(16) Å with angle at H6n = 177.1(15)° for symmetry operations (i) 3/2 – *x*, –1/2 + *y*, *z* and (ii) 1 – *x*, 1/2 + *y*, 1/2 – *z*]. Additional stability to the chains is afforded by π-stacking interactions between chlorophenyl rings [Cg(C9–C14)···Cg(C41–C46)<sup>i</sup> = 3.7110(9) Å with an angle of inclination = 2.52(8)°]. The amide-N4H atom of the Cl2-molecule does not participate in a hydrogen bond. The chains are linked into supramolecular layers in the *ab*-plane *via* a combination of parallel carbonyl-O···π(hydroxyphenyl) [C8–O3···Cg(C17–C22)<sup>iii</sup> = 3.9502(14) Å with angle at O3 = 98.81(9)° for (iii) 1 – *x*, –1/2 + *y*, 1/2 – *z*] and C–H···O interactions with the closest of these being of the type methylene-C–H···O(hydroxy) [C31–H31a···O1<sup>ii</sup>: H31a···O1<sup>ii</sup> = 2.60 Å, C31···O1<sup>ii</sup> = 3.429(2) Å with angle at H31a = 141°]. The links between layers along the *c*-axis to consolidate the three-dimensional molecular packing are of the type hydroxyphenyl- and methylene-C–H···Cl [C4–H4···Cl1<sup>iv</sup>: H4···Cl1<sup>iv</sup> = 2.81 Å, C4···Cl1<sup>iv</sup> = 3.5952(17) Å with angle at H4 = 141° and C15–H15b···Cl2<sup>v</sup>: H15b···Cl2<sup>v</sup> = 2.73 Å, C15···Cl2<sup>v</sup> 3.699(2) Å with angle at H15b = 166° for (iv) *x*, 1/2 – *y*, 1/2 + *z* and (v) –1/2 + *x*, 1/2 – *y*, 1 – *z*].

Finally, an analysis of the calculated Hirshfeld surfaces was conducted. This was accomplished employing Crystal Explorer 17 [13] and literature procedures [14], including the calculation of the full and decomposed two-dimensional

fingerprint plots. In particular, a recent study showed how such an analysis can be employed to differentiate between multiple molecules in the asymmetric unit [15]. The most prominent contacts on the Hirshfeld surface for each individual molecule are, not surprisingly, H···H contacts and the percentage contributions for the Cl1–Cl3-molecules, i.e. 38.5, 31.5 and 32.6%, differentiate between the Cl1-molecule on the one hand and the Cl2- and Cl3-molecules on the other. A similar differentiation is seen in the C···H/H···C contacts of 20.3, 26.7 and 25.6%, respectively. To a first approximation, the percentage contributions from the O···H/H···O [16.6, 17.0 and 17.5%] and Cl···H/H···Cl [15.6, 13.4 and 12.8%] contacts are about the same. The 3.6, 1.7 and 3.8% contributions from the C···C contacts and 1.1, 1.9 and 1.2% from the O···C/C···O contacts highlight small differences between the molecules.

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