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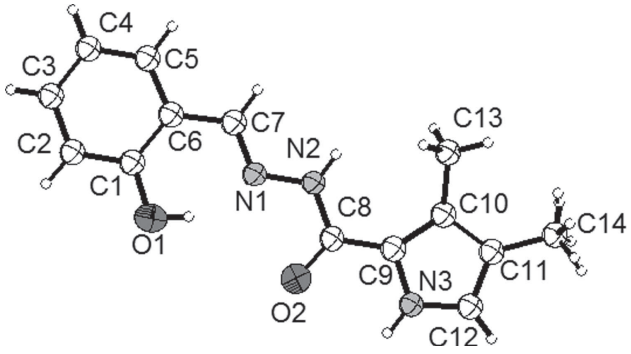
# Crystal structure of N′-(2-hydroxybenzylidene)-3,4-dimethyl-1H-pyrrole-2-carbohydrazide, $C_{14}H_{15}N_3O_2$

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## Abstract

$C_{14}H_{15}N_3O_2$, monoclnic, $P2_1/c$ (no. 14), $a = 8.2642(4)$ Å, $b = 6.0481(3)$ Å, $c = 25.6897(11)$ Å, $\beta = 95.319(3)°$, $V = 1278.51(10)$ Å$^3$, $Z = 4$, $R_{gt}(F) = 0.0584$, $wR_{ref}(F^2) = 0.1931$, $T = 296$ K.

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The crystal structure is shown in the figure. Tables 1–3 contain details of the measurement method and a list of the atoms including atomic coordinates and displacement parameters.

## Source of material

The title compound was synthesized by condensation of equimolar of 3,4-dimethyl-1H-pyrrole-2-carbohydrazide [1] and salicylaldehyde in ethanol solution under refluxing. The crystal suitble for single X-ray diffraction was obtained by evaporating the corresponding reaction solutions at room temperature.

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

| | |
|---|---|
| Crystal: | Colorless, block, size 0.10×0.12×0.15 mm |
| Wavelength: | Mo $K_\alpha$ radiation (0.71073 Å) |
| $\mu$: | 0.92 cm$^{-1}$ |
| Diffractometer, scan mode: | Bruker APEX-II CCD, $\varphi$ and $\omega$ scans |
| $2\theta_{max}$: | 55.84° |
| $N(hkl)_{measured}$, $N(hkl)_{unique}$: | 11424, 3048 |
| Criterion for $I_{obs}$, $N(hkl)_{gt}$: | $I_{obs} > 2\,\sigma(I_{obs})$, 1508 |
| $N(param)_{refined}$: | 176 |
| Programs: | SHELX [4] |

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

| Atom | Site | x | y | z | $U_{iso}$ |
|---|---|---|---|---|---|
| H(2) | 4e | 0.3718 | 0.4565 | 0.1033 | 0.064 |
| H(3) | 4e | 0.3751 | 0.8733 | −0.0349 | 0.063 |
| H(7) | 4e | 0.5182 | 0.3866 | 0.1839 | 0.061 |
| H(5) | 4e | 0.6538 | 0.3432 | 0.2719 | 0.075 |
| H(12) | 4e | 0.2245 | 0.6981 | −0.1070 | 0.066 |
| H(2A) | 4e | 0.8904 | 1.0321 | 0.2675 | 0.078 |
| H(3A) | 4e | 0.9433 | 0.8078 | 0.3390 | 0.092 |
| H(13A) | 4e | 0.2778 | 0.1686 | 0.0440 | 0.094 |
| H(13B) | 4e | 0.1008 | 0.1669 | 0.0161 | 0.094 |
| H(13C) | 4e | 0.1432 | 0.3177 | 0.0651 | 0.094 |
| H(4) | 4e | 0.8220 | 0.4638 | 0.3420 | 0.090 |
| H(14A)* | 4e | 0.0461 | 0.1719 | −0.0645 | 0.098 |
| H(14B)* | 4e | 0.1337 | 0.2358 | −0.1139 | 0.098 |
| H(14C)* | 4e | −0.0233 | 0.3629 | −0.1013 | 0.098 |
| H(1) | 4e | 0.638 | 0.897 | 0.1619 | 0.098 |
| H(14D)* | 4e | 0.129 | 0.152 | −0.0834 | 0.011 |
| H(14E)* | 4e | 0.061 | 0.339 | −0.127 | 0.031 |
| H(14F)* | 4e | −0.049 | 0.285 | −0.076 | 0.038 |

*disordered methyl group, occupancy factor: 0.5

## Experimental details

All the H atoms were located in the difference electron density map. The H atoms were situated into the idealized positions with the carrier atom-H distances = 0.93 Å for aryl, 0.97 for methylene, 0.96 Å for the methyl and 0.86 Å for the secondary

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**Table 3:** Atomic displacement parameters (Å$^2$).

| Atom | Site | *x* | *y* | *z* | $U_{11}$ | $U_{22}$ | $U_{33}$ | $U_{12}$ | $U_{13}$ | $U_{23}$ |
|---|---|---|---|---|---|---|---|---|---|---|
| N(2) | 4*e* | 0.4233(2) | 0.5798(3) | 0.10235(6) | 0.055(1) | 0.056(1) | 0.047(1) | −0.0043(8) | −0.0031(8) | 0.0048(9) |
| O(2) | 4*e* | 0.4804(2) | 0.8916(3) | 0.06004(5) | 0.0688(9) | 0.057(1) | 0.0575(9) | −0.0044(8) | −0.0023(7) | 0.0036(8) |
| C(9) | 4*e* | 0.3212(2) | 0.6268(4) | 0.01203(7) | 0.046(1) | 0.052(1) | 0.043(1) | 0.008(1) | 0.0013(9) | 0.000(1) |
| N(3) | 4*e* | 0.3251(2) | 0.7494(3) | −0.03270(6) | 0.0524(9) | 0.054(1) | 0.0519(9) | 0.0012(8) | 0.0022(7) | 0.0007(9) |
| O(1) | 4*e* | 0.7054(2) | 0.9665(3) | 0.18554(6) | 0.089(1) | 0.054(1) | 0.064(1) | −0.0094(8) | 0.0009(8) | 0.0087(8) |
| N(1) | 4*e* | 0.5199(2) | 0.6561(3) | 0.14436(6) | 0.055(1) | 0.054(1) | 0.0427(9) | 0.0047(9) | 0.0003(8) | 0.0002(9) |
| C(7) | 4*e* | 0.5582(2) | 0.5302(3) | 0.18335(7) | 0.052(1) | 0.048(1) | 0.053(1) | 0.000(1) | 0.005(1) | 0.001(1) |
| C(8) | 4*e* | 0.4123(2) | 0.7094(4) | 0.05852(7) | 0.044(1) | 0.052(1) | 0.051(1) | 0.009(1) | 0.0054(9) | 0.000(1) |
| C(1) | 4*e* | 0.7354(2) | 0.8252(4) | 0.22626(7) | 0.056(1) | 0.052(1) | 0.050(1) | 0.003(1) | 0.0063(9) | 0.004(1) |
| C(11) | 4*e* | 0.1764(2) | 0.4551(4) | −0.05459(8) | 0.045(1) | 0.055(1) | 0.056(1) | 0.009(1) | 0.0007(9) | −0.010(1) |
| C(6) | 4*e* | 0.6649(2) | 0.6137(3) | 0.22684(7) | 0.048(1) | 0.048(1) | 0.044(1) | 0.004(1) | 0.0039(9) | 0.003(1) |
| C(10) | 4*e* | 0.2273(2) | 0.4409(3) | −0.00058(7) | 0.046(1) | 0.049(1) | 0.051(1) | 0.012(1) | 0.0037(9) | −0.003(1) |
| C(5) | 4*e* | 0.7003(2) | 0.4830(4) | 0.27087(8) | 0.067(1) | 0.062(2) | 0.056(1) | 0.001(1) | −0.003(1) | 0.012(1) |
| C(12) | 4*e* | 0.2390(2) | 0.6471(4) | −0.07269(7) | 0.054(1) | 0.066(2) | 0.044(1) | 0.007(1) | −0.0018(9) | −0.005(1) |
| C(2) | 4*e* | 0.8410(2) | 0.8941(4) | 0.26830(8) | 0.064(1) | 0.060(2) | 0.070(2) | −0.008(1) | −0.001(1) | −0.011(1) |
| C(3) | 4*e* | 0.8729(3) | 0.7597(5) | 0.31098(9) | 0.071(2) | 0.094(2) | 0.062(2) | 0.004(2) | −0.013(1) | −0.012(2) |
| C(13) | 4*e* | 0.1834(2) | 0.2571(4) | 0.03429(8) | 0.066(1) | 0.051(2) | 0.071(1) | 0.004(1) | 0.005(1) | 0.001(1) |
| C(4) | 4*e* | 0.8015(3) | 0.5534(5) | 0.31275(9) | 0.082(2) | 0.083(2) | 0.056(1) | 0.006(2) | −0.009(1) | 0.016(1) |
| C(14) | 4*e* | 0.0741(3) | 0.2919(4) | −0.08641(9) | 0.067(2) | 0.059(2) | 0.067(2) | 0.009(1) | −0.005(1) | −0.012(1) |

amine hydrogens. The $U_{iso}$ values were constrained to be $1.5U_{eq}$ of the carrier atom for the methyl H atoms and $1.2U_{eq}$ for the remaining H atoms. The hydrogen atoms of the methyl group at C14 are disordered.

## Discussion

In our previous papers, we have reported a number of Schiff-bases bearing pyrrole units [1–3]. Particularly, the zinc(II), copper(II), nickel(II) complexes of Schiff-base ligand derived from 3,4-dimethyl-1H-pyrrole-2-carbohydrazide and 1-phenyl-3-methyl-4-benzoyl-2-pyrazolin-5-one [1] have certain antioxdative activity. Herein, the title compound was synthesized by condensation of 3,4-dimethyl-1H-pyrrole-2-carbohydrazide and salicylaldehyde in this work.

In the title compound (Figure), the imine C=N double bond has an *E* configuration. The dihedral angle between pyrrole (N3/C9—C12, r.m.s. deviation 0.0014 Å) and hydroxyphenyl moiety (C1—C6, r.m.s. deviation 0.0078 Å) in the hydrazone molecule is 13.07 (3)°. In the crystal, the molecules are linked into a centrosymmetric dimer by two intermolecular N—H···O hydrogen bonds, forming a $R_2^2(10)$ ring motif. An intramolecular O—H···N hydrogen bond is also present.

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