Xindi Xu, Hui Tian, Xia Wang, Man Wang and Qiong Wu*

Crystal structure of 4-bromo-\(N'\)-(3-chloro-2-hydroxyphenyl)methylidene]benzohydrazide, \(\text{C}_{14}\text{H}_{7}\text{Br}_{2}\text{N}_{2}\text{O}_{2}\)

Abstract

\(\text{C}_{14}\text{H}_{7}\text{Br}_{2}\text{N}_{2}\text{O}_{2}\) triclinic, \(P2_1/n\) (no. 14), \(a = 4.8308(2)\, \text{Å}\), \(b = 6.1438(2)\, \text{Å}\), \(c = 23.0836(9)\, \text{Å}\), \(\beta = 91.0784(14)^\circ\), \(V = 684.99(4)\, \text{Å}^3\), \(Z = 2\), \(R_{\text{wp}}(F) = 0.0477\), \(wR_{\text{wp}}(F^2) = 0.1108\), \(T = 150.0\, \text{K}\).

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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

The structure was solved with the Olex2 program [2] as an interface together with the SHELX and SHELXL programs [3, 4]. All H atoms were placed in geometrically

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Table 1: Data collection and handling.

| Crystal:          | Yellow needle            |
| Size:             | \(0.20 \times 0.20 \times 0.18\, \text{mm}\) |
| Wavelength:       | Mo K\(\alpha\) radiation (0.71073\, \text{Å}) |
| \(\mu\):          | 5.92 \text{mm}^{-1}     |
| Diffractometer, scan mode: | Bruker APEX-II, \(\varphi\) and \(\omega\) |
| \(R_{\text{wp}}\) completeness: | 26.4\%, >99% |
| \(N(hkl)_{\text{measured}}, N(hkl)_{\text{unique}}, R_{\text{int}}:\ | 7475, 1392, 0.032 |
| Criterion for \(I_{\text{obs}}\), \(N(hkl)_{\text{gt}}:\ | \(I_{\text{obs}} > 2 \sigma(I_{\text{obs}}), 1256\) |
| \(N(\text{param})_{\text{refined}}:\ | 101 |
| Programs:         | CrysAlis\textsuperscript{PRO} [1], Olex2 [2], SHELX [3, 4] |

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

<table>
<thead>
<tr>
<th>Atom</th>
<th>(x)</th>
<th>(y)</th>
<th>(z)</th>
<th>(U_{\text{iso}}^{*})/(U_{\text{eq}})</th>
</tr>
</thead>
<tbody>
<tr>
<td>Br1</td>
<td>0.19186 (15)</td>
<td>0.44226 (11)</td>
<td>0.29563 (3)</td>
<td>0.0497 (2)</td>
</tr>
<tr>
<td>O1(^*)</td>
<td>0.3352 (16)</td>
<td>-0.0667 (15)</td>
<td>0.4698 (3)</td>
<td>0.0398 (19)</td>
</tr>
<tr>
<td>H1(^*)</td>
<td>0.259304</td>
<td>-0.160050</td>
<td>0.491143</td>
<td>0.060*</td>
</tr>
<tr>
<td>O2(^*)</td>
<td>-0.3915 (17)</td>
<td>-0.3968 (13)</td>
<td>0.4479 (4)</td>
<td>0.042 (2)</td>
</tr>
<tr>
<td>N1</td>
<td>0.0380 (10)</td>
<td>-0.4121 (8)</td>
<td>0.4838 (2)</td>
<td>0.0395 (12)</td>
</tr>
<tr>
<td>C2</td>
<td>0.1643 (12)</td>
<td>-0.0134 (10)</td>
<td>0.4274 (3)</td>
<td>0.0401 (14)</td>
</tr>
<tr>
<td>C6</td>
<td>-0.2070 (12)</td>
<td>-0.1013 (10)</td>
<td>0.3593 (3)</td>
<td>0.0428 (15)</td>
</tr>
<tr>
<td>H6</td>
<td>-0.357737</td>
<td>-0.191717</td>
<td>0.347766</td>
<td>0.051*</td>
</tr>
<tr>
<td>C1</td>
<td>-0.0621 (12)</td>
<td>-0.1478 (10)</td>
<td>0.4104 (3)</td>
<td>0.0369 (13)</td>
</tr>
<tr>
<td>C4</td>
<td>0.0867 (12)</td>
<td>0.2048 (10)</td>
<td>0.3430 (3)</td>
<td>0.0378 (13)</td>
</tr>
<tr>
<td>C7</td>
<td>-0.1447 (13)</td>
<td>-0.3393 (11)</td>
<td>0.4451 (3)</td>
<td>0.0439 (15)</td>
</tr>
<tr>
<td>C5</td>
<td>-0.1371 (12)</td>
<td>0.0729 (11)</td>
<td>0.3250 (3)</td>
<td>0.0418 (14)</td>
</tr>
<tr>
<td>H5</td>
<td>-0.237255</td>
<td>0.103173</td>
<td>0.290092</td>
<td>0.050*</td>
</tr>
<tr>
<td>C3</td>
<td>0.2328 (12)</td>
<td>0.1653 (10)</td>
<td>0.3933 (3)</td>
<td>0.0390 (13)</td>
</tr>
<tr>
<td>H3</td>
<td>0.379983</td>
<td>0.259160</td>
<td>0.404846</td>
<td>0.047*</td>
</tr>
</tbody>
</table>

*Occupancy: 0.5.
idealized positions and refined using a riding model, with 
\( O-\text{H} = 0.84 \) (phenolic hydroxyl), 0.95 Å (benzene), and 
with \( U_{\text{iso}}(\text{H}) = 1.2 \ U_{\text{eq}}(\text{C}) \) for H atoms on phenolic hydroxyl 
and benzene.

**Experimental details**

4-Bromo-2-hydroxybenzaldehyde (0.201 g, 1.0 mmol) 
was dissolved in 30 mL methanol forming a colorless 
solution. Additionally 20 mL methanol containing 0.215 g 
4-bromobenzohydrazide (1 mmol) was added to above 
solution. The obtained mixture stirred for 12 h at room 
temperature. Then the yellow filtrate was sealed in a 
beaker with cling film and kept undisturbed at room 
temperature. The yellow square crystals of the title com-
pound were obtained after 1 week.

**Comment**

Hydrazones characterized by the \(-\text{NHN=CH}\)-group repre-
sent an important class of compounds in the field of 
medical science and ion recognition [5, 6]. As a part of our 
current research interest in exploring the relationship be-
tween molecular structure and physicochemical properties 
of halogenated Schiff-base compounds [7, 8], in this work, 
we report a new bromohydrazone.

X-ray diffraction analysis reveals that the compound 
contains a planar hydrazone molecule, as shown in 
the figure. The five non-hydrogen conjugated atoms 
\([\text{C(=O)}\text{N}=\text{N}=\text{C}]\) constitute the central chromophore and 
over all exhibit an E configuration. The dihedral angles 
formed between the central plane and the least-squares 
planes through the flanking phenyl ring is 17.92(15)°, 
indicating the twisted nature of the whole molecule. In 
addition, the central chromophore and bromo-phenol 
ring generates an intramolecular hydroxy \( \text{N} \cdot \text{H} \cdots \text{O} \) 
hydrogen bond. [O1–H1⋯N1: O1–H1 = 0.84(1) Å, H1⋯N1 = d 
1.888(5) Å, O1⋯N1 = 2.5813(10) Å with angle at H1 = 138.9]. 
Bond lengths and angles are in the expected ranges [9].

**Author contribution:** All the authors have accepted 
responsibility for the entire content of this submitted 
manuscript and approved submission.

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\text{bis-2- bromo-4-chlorophenolato-} \equiv \text{N,N',O,O'}\text{nickel(II),} 
\text{C}_{20}\text{H}_{16}\text{Br}_2\text{Cl}_3\text{N}_2\text{O}_2\). Z. Kristallogr. NCS 2020, 235, 863–864.
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