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The crystal structure of 2-(1*H*-benzimidazol-2-yl)-3-bromo-5-chlorophenol, C₁₃H₈BrClN₂O

Table 1: Data collection and handling.

Crystal:	Yellow block
Size	0.20 × 0.16 × 0.12 mm
Wavelength:	Mo K α radiation (0.71073 Å)
μ :	3.66 mm ⁻¹
Diffractometer, scan mode:	Bruker D8/APEX2, ω
$\theta_{\max}$, completeness:	26.4°, >99%
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	12,189, 2447, 0.059
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 2001
$N(\text{param})_{\text{refined}}$:	164
Programs:	CrysAlis ^{PRO} [1], SHELX [2, 3], Diamond [4]

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Abstract

C₁₃H₈BrClN₂O, ($M = 323.57$ g/mol), monoclinic, $P2_1/c$ (no. 14), $a = 11.7584(5)$ Å, $b = 7.4336(3)$ Å, $c = 14.1134(6)$ Å, $\beta = 104.861(2)^\circ$, $V = 1192.35(9)$ Å³, $Z = 4$, $R_{\text{gt}}(F) = 0.0301$, $wR_{\text{ref}}(F^2) = 0.0794$, $T = 150.0$ K.

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Table 1 contains crystallographic data and Table 2 contains the list of the atoms including atomic coordinates and displacement parameters.

Source of material

3-Bromo-5-chlorosalicylaldehyde (0.224 g, 1 mmol) and 30 ml of anhydrous methanol were added in a wide-neck conical flask, monitored with a temperature probe and warmed up to 60 °C with stirring, then 1 mmol (0.108 g) of o-phenylenediamine was dropwisely added. Over 30 min the mixture gradually turned to light yellow, and refluxed for another 16 h. The obtained solution was then filtered and the filtrate was left to crystallise in an open vessel at room temperature. Colorless block crystals were isolated from the yellow mother liquor in the following 24 h.

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Experimental details

The structure was solved with the Olex2 program [2] as an interface together with the SHELXT and SHELXL programs [3, 4]. All H atoms were placed in geometrically idealized positions and refined using a riding model, with O–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 aryl.

Comment

Benzimidazoles are a class of heterocyclic compounds which are of significant interest [5–8]. Benzimidazoles species are also important intermediates in many organic synthesis and have a wide range of applications in synthetic chemistry. Therefore, the research on the synthesis and application of benzimidazoles have attracted significant attention in the past few decades [8, 12]. As a part of our current research interest in this work, we report a new hetero-halogenated benzimidazole compound.

X-ray diffraction analysis reveals that the title compound contains a planar benzimidazole molecule, as shown in the Figure. The central five non-hydrogen conjugated atoms [C(=O)C–C=N] constitute the chromophore. The dihedral angle between the benzimidazole and phenyl ring is 1.9°, indicating the co-planar nature of the whole molecule. In addition, the central chromophore and benzimidazole generates an intramolecular O–H...N hydrogen bond [O1–H1...N1: O1–H1 = 0.85(1) Å,

Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²).

Atom	x	y	z	<i>U</i> _{iso} */ <i>U</i> _{eq}
Br1	0.20010 (3)	0.01099 (4)	0.802903 (19)	0.04137 (11)
Cl1	0.37660 (6)	−0.12755 (11)	0.48547 (6)	0.04886 (19)
O1	−0.01818 (16)	−0.1434 (3)	0.66908 (14)	0.0414 (4)
H1	−0.0785	−0.1898	0.6320	0.062*
N1	−0.15012 (19)	−0.2828 (3)	0.51059 (16)	0.0365 (5)
N2	−0.08208 (19)	−0.3468 (3)	0.38078 (16)	0.0345 (5)
H2	−0.0324	−0.3572	0.3439	0.041*
C1	0.0568 (2)	−0.2082 (3)	0.52815 (19)	0.0331 (5)
C2	0.0699 (2)	−0.1444 (3)	0.62404 (19)	0.0348 (6)
C3	0.1802 (2)	−0.0775 (3)	0.67424 (18)	0.0342 (5)
C4	0.2745 (2)	−0.0738 (3)	0.6335 (2)	0.0365 (6)
H4	0.3488	−0.0296	0.6697	0.044*
C5	0.2588 (2)	−0.1362 (4)	0.5384 (2)	0.0368 (6)
C6	0.1514 (2)	−0.2015 (3)	0.4858 (2)	0.0350 (6)
H6	0.1419	−0.2422	0.4204	0.042*
C7	−0.0578 (2)	−0.2795 (3)	0.47372 (19)	0.0336 (5)
C8	−0.2002 (2)	−0.3961 (3)	0.35539 (19)	0.0349 (6)
C9	−0.2410 (2)	−0.3552 (3)	0.4375 (2)	0.0361 (6)
C10	−0.3593 (2)	−0.3852 (4)	0.4360 (2)	0.0438 (7)
H10	−0.3888	−0.3566	0.4908	0.053*
C11	−0.4307 (3)	−0.4578 (4)	0.3518 (2)	0.0479 (7)
H11	−0.5111	−0.4799	0.3486	0.057*
C12	−0.3876 (3)	−0.4999 (4)	0.2708 (2)	0.0471 (7)
H12	−0.4394	−0.5511	0.2143	0.057*
C13	−0.2718 (3)	−0.4693 (4)	0.2706 (2)	0.0425 (6)
H13	−0.2429	−0.4968	0.2154	0.051*

H1...N1 = 1.893(4) Å, O1...N1 = 2.589(8) Å with angle at H1 = 147.84°. Bond lengths and angles are in the expected ranges [9–11].

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

References

1. Oxford Diffraction Ltd. *CrysAlis^{PRO}*; Abingdon, Oxfordshire, England, 2006.
2. Sheldrick G. M. SHELXT - Integrated space-group and crystal-structure determination. *Acta Crystallogr.* 2015, *A71*, 3–8.
3. Sheldrick G. M. Crystal structure refinement with *SHELXL*. *Acta Crystallogr.* 2015, *C71*, 3–8.
4. Brandenburg K. *DIAMOND. Visual Crystal Structure Information System. Ver. 4.0*; Crystal Impact: Bonn, Germany, 2015.
5. Ravikant, Kumar R. Review on synthesis and application of Schiff base and its transition metal complexes. *Res. J. Chem. Environ. Sci.* 2014, *2*, 1–4.
6. Naeimi H., Safari J., Heidarneshad A. Synthesis of Schiff base ligands derived from condensation of salicylaldehyde derivatives and synthetic diamine. *Dyes Pigments* 2007, *73*, 251–253.
7. Yang X., Jones R. A., Wiester M. J., Oye M. M., Wong W.-K. Transformation of a luminescent benzimidazole-based Yb₃ cluster into a one-dimensional coordination polymer. *Cryst. Growth Des.* 2010, *10*, 970–976.
8. Wu Q., Pu Q., Wu Y., Shi H., He Y., Li J., Fan Q. POM-assisted synthesis of the first cyclohexanediamine-based Salen-type Mn III-dimer complexes. *J. Coord. Chem.* 2015, *68*, 1010–1020.
9. Takagi K., Yamada Y., Fukuda R., Ehara M., Takeuchi D. ESIPT emission behavior of methoxy-substituted 2-hydroxyphenyl-benzimidazole isomers. *New J. Chem.* 2018, *42*, 5923–5928.
10. Klammer B. E., Windorff C. J., Marsh M. L., Meeker D. S., Albrecht-Schmitt T. E. Schiff-base coordination complexes with plutonium (IV) and cerium (IV). *Chem. Commun.* 2018, *54*, 8634–8636.
11. Barwiolek M., Wojtczak A., Kozakiewicz A., Babinska M., Tafelska-Kaczmarek A., Larsen E., Szlyk E. The synthesis, characterization and fluorescence properties of new benzimidazole derivatives. *J. Lumin.* 2019, *211*, 88–95.
12. Odame F., Krause J., Hosten E. C., Betz R., Lobb K., Tshentu Z. R., Frost C. L. Synthesis, characterization and DPPH scavenging activity of some benzimidazole derivatives. *Bull. Chem. Soc. Ethiop.* 2018, *32*, 271–284.