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Crystal structure of 4-bromo-2-(1*H*-pyrazol-3-yl)phenol, C₉H₇BrN₂O

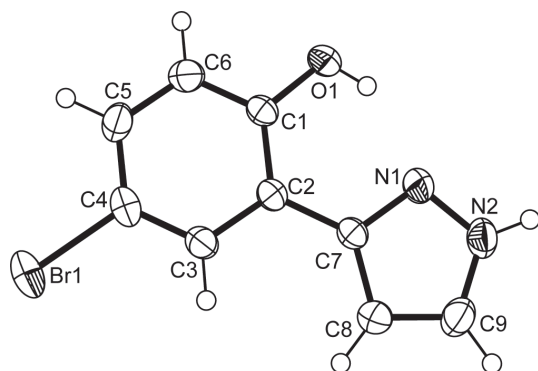


Table 1: Data collection and handling.

Crystal:	Colourless prism
Size:	0.20 × 0.13 × 0.10 mm
Wavelength:	Mo K α radiation (0.71073 Å)
μ :	46.1 cm ⁻¹
Diffractometer, scan mode:	Bruker APEX-II, φ and ω
2 $\theta_{\max}$, completeness:	60°, 96.9 %
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	12745, 2507, 0.029
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 1893
$N(\text{param})_{\text{refined}}$:	126
Programs:	Bruker programs [1], SHELX [2]

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Abstract

C₉H₇BrN₂O, triclinic, $C2/c$ (no. 15), $a = 16.255(3)$ Å, $b = 4.4119(9)$ Å, $c = 25.923(5)$ Å, $\beta = 107.99(3)^\circ$, $V = 1768.2(7)$ Å³, $Z = 8$, $R_{\text{gt}}(F) = 0.0450$, $wR_{\text{ref}}(F^2) = 0.0960$, $T = 150$ K.

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The asymmetric unit of the title crystal structure is shown in the figure. Tables 1 and 2 contain details of the measurement method and a list of the atoms including atomic coordinates and displacement parameters.

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

Atom	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
Br1	0.33374(3)	−0.11067(9)	0.46796(2)	0.05993(16)
N1	0.51366(15)	0.6170(5)	0.31190(9)	0.0341(5)
N2	0.58268(17)	0.8027(6)	0.32665(10)	0.0381(6)
O1	0.39242(14)	0.2440(5)	0.25796(7)	0.0352(4)
C1	0.38214(17)	0.1752(6)	0.30684(9)	0.0269(5)
C2	0.43628(16)	0.2911(6)	0.35612(9)	0.0265(5)
C3	0.42015(18)	0.2016(6)	0.40384(10)	0.0316(6)
H3	0.4552	0.2745	0.4370	0.038*
C4	0.35373(19)	0.0089(7)	0.40245(11)	0.0352(6)
C5	0.29958(19)	−0.1020(7)	0.35372(12)	0.0396(7)
H5	0.2538	−0.2302	0.3530	0.048*
C6	0.31504(19)	−0.0179(7)	0.30627(11)	0.0360(6)
H6	0.2796	−0.0926	0.2733	0.043*
C7	0.50711(17)	0.5008(6)	0.35784(9)	0.0266(5)
C8	0.5726(2)	0.6171(7)	0.40238(11)	0.0395(7)
H8	0.5824	0.5738	0.4389	0.047*
C9	0.6189(2)	0.8075(7)	0.38038(12)	0.0411(7)
H9	0.6670	0.9204	0.3994	0.049*
H2N	0.596(2)	0.902(7)	0.3031(14)	0.043(9)*
H10	0.430(3)	0.376(9)	0.2628(16)	0.063(13)*

Source of material

4-Bromo-2-(1*H*-pyrazol-3-yl)phenol was purchased from Sigma-Aldrich. The single crystals suitable for the X-ray diffraction experiment were obtained by recrystallization from methanol.

Experimental details

All hydrogen atoms were identified in a difference Fourier map. The aromatic hydrogen atoms were placed at geometrically idealized positions with C—H distances set to 0.93 Å and U_{iso} values set to $1.2U_{\text{eq}}$ of the parent C atom. Hydrogen atoms attached to the pyrazole N and phenol O atoms were refined isotropically.

Comment

Pyrazole and its derivatives represent one of the most important classes of organic heterocyclic compounds, with an unusual wide range of biological activities. Many pyrazole derivatives have been reported to possess antimicrobial, anti-inflammatory, analgesic, anticancer activities as well as the fungicidal and pesticidal activities [3, 4]. In addition, due to significant complexation abilities, pyrazole based ligands found applications as extraction reagents for metal ions [5]. Different pyrazole based ligands are used in the synthesis of complexes with the pronounced magnetic properties and also as building blocks of metal organic frameworks [6]. As part of our ongoing research on the synthesis, physico-chemical and structural properties of the pyrazole based coordination compounds [7, 8] herein we report the crystal structure of the title compound.

The title pyrazole derivative contains one independent molecule in the asymmetric unit. The bond lengths and angles of the phenyl-pyrazole core are consistent with those in similar molecules [9–23]. The dihedral angle between the pyrazole and phenyl ring is 8.6(2)° and reflects a nearly planar geometry of the molecule. The planar conformation is stabilized by the intramolecular hydrogen bond formed between the phenol hydroxy group and the pyrazole nitrogen [O1—H1...N1: O—H 0.83(4), H...N 1.87(4), O...N 2.615(3) Å, O—H...N = 148(4)°]. This intramolecular hydrogen bond is the main structural property of different 3{5}-(2'-hydroxyaryl)pyrazoles and it ensures the co-planarity of the phenyl-pyrazole fragment in the various intramolecular environments [9–23]. The presence of this intramolecular N—H...O hydrogen bond is related to the photostability of some hydroxyarylpyrazoles and their ability to act as UV stabilizers [9]. In the crystal packing, the strongest intermolecular hydrogen bond formed between the pyrazole N—H donor and the phenol O-acceptor [N2—H2n...O1ⁱ: N—H 0.85(4), N...O 3.051(3), H...O 2.22(4) Å, N—H...O 165(3)°, (i) $-x+1, +y+1, -z+1/2$], links the molecules along the *b* axis. This arrangement is supported by weak O—H...N interactions [O1—H1o...N1ⁱⁱ: O—H 0.83(4), O...N 3.165(3), H...N 2.61(5), O—H...N 126(4)°, (ii) $-x+1, +y, -z+1/2$] and π - π contacts between parallel arranged molecular planes (interplanar distance of 3.423 Å).

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