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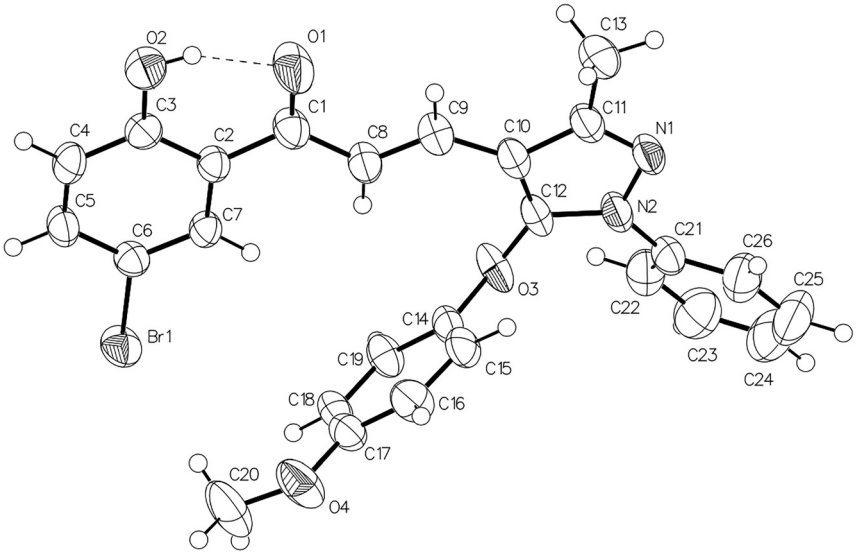
Crystal structure of (*E*)-1-(5-bromo-2-hydroxyphenyl)-3-(5-(4-methoxyphenoxy)-3-methyl-1-phenyl-1*H*-pyrazol-4-yl)prop-2-en-1-one, $C_{26}H_{21}BrN_2O_4$



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Abstract

$C_{26}H_{21}BrN_2O_4$, triclinic, $P\bar{1}$ (no. 2), a = 9.061(3) Å, b = 11.640(3) Å, c = 12.751(3) Å, α = 65.985(7)°, β = 87.683(8)°, γ = 72.332(12)°, V = 1165.1(5) Å^3, Z = 2, $R_{gt}(F)$ = 0.0351, $wR_{ref}(F^2)$ = 0.0873, T = 296(2) 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.

Table 1: Data collection and handling.

Crystal:	Yellow plate
Size:	0.27 × 0.26 × 0.07 mm
Wavelength:	Mo *K*α radiation (0.71073 Å)
μ:	1.80 mm^{-1}
Diffractometer, scan mode:	PHOTON III M14, φ and ω
θ_{max}, completeness:	28.2°, >99 %
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	35180, 5741, 0.034
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\sigma(I_{obs})$, 4809
$N(param)_{refined}$:	301
Programs:	Bruker,[1] SHELX,[2,3] Olex2[4]

1 Source of material

Aqueous KOH solution (8 ml, 40 %) was added to an equimolar amount of 5-chloro-3-methyl-1-phenyl-1H-pyrazole-4-carboxaldehyde (10 mmol, 2.2 g) and 4-methoxyphenol (10 mmol, 1.24 g) dissolved in 30 ml of dimethylforamide, and the reaction mixture was refluxed 1 h. After cooling the reaction mixture, it was stirred at room temperature overnight. Ice water was added to the mixture and acidified

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

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
O1	0.3467 (2)	0.3140 (2)	0.6147 (2)	0.0865 (7)
C1	0.4788 (2)	0.3256 (2)	0.6099 (2)	0.0474 (5)
C2	0.5934 (2)	0.25934 (18)	0.55031 (16)	0.0368 (4)
C3	0.5530 (2)	0.17987 (19)	0.50480 (18)	0.0405 (4)
C4	0.6585 (3)	0.1178 (2)	0.44685 (19)	0.0473 (5)
H4	0.6310	0.0648	0.4178	0.057*
C5	0.8023 (2)	0.13411 (19)	0.43238 (18)	0.0439 (4)
H5	0.8723	0.0930	0.3932	0.053*
C6	0.8429 (2)	0.21282 (18)	0.47673 (16)	0.0372 (4)
C7	0.7421 (2)	0.27380 (18)	0.53503 (16)	0.0367 (4)
H7	0.7723	0.3251	0.5647	0.044*
O2	0.41309 (18)	0.16097 (17)	0.51522 (17)	0.0592 (4)
H2	0.3617	0.1996	0.5524	0.089*
Br1	1.04244 (2)	0.23495 (2)	0.45403 (2)	0.05277 (9)
C8	0.5185 (2)	0.4044 (2)	0.66162 (18)	0.0438 (4)
H8	0.6144	0.4191	0.6518	0.053*
C9	0.4185 (3)	0.4562 (2)	0.72327 (18)	0.0450 (4)
H9	0.3260	0.4357	0.7325	0.054*
C10	0.4354 (2)	0.53917 (19)	0.77694 (17)	0.0420 (4)
C11	0.3215 (2)	0.5963 (2)	0.83723 (17)	0.0445 (5)
N1	0.3659 (2)	0.67564 (18)	0.86999 (15)	0.0472 (4)
N2	0.5117 (2)	0.67133 (16)	0.83302 (14)	0.0440 (4)
C12	0.5548 (2)	0.59001 (18)	0.77816 (17)	0.0421 (4)
C13	0.1663 (3)	0.5766 (3)	0.8634 (2)	0.0610 (6)
H13A	0.1807	0.4874	0.9188	0.092*
H13B	0.1107	0.5923	0.7939	0.092*
H13C	0.1079	0.6374	0.8945	0.092*
O3	0.69404 (18)	0.57521 (13)	0.73363 (14)	0.0516 (4)
C14	0.8012 (2)	0.44604 (18)	0.77316 (17)	0.0385 (4)
C15	0.7964 (2)	0.3468 (2)	0.87783 (17)	0.0478 (5)
H15	0.7236	0.3636	0.9276	0.057*
C16	0.9017 (3)	0.2218 (2)	0.90783 (19)	0.0545 (5)
H16	0.8985	0.1532	0.9777	0.065*
C17	1.0123 (2)	0.1978 (2)	0.83436 (18)	0.0451 (5)
C18	1.0187 (2)	0.2997 (2)	0.73174 (18)	0.0433 (4)
H18	1.0948	0.2846	0.6835	0.052*
C19	0.9115 (2)	0.42468 (19)	0.70047 (18)	0.0432 (4)
H19	0.9144	0.4936	0.6307	0.052*
O4	1.1096 (2)	0.07001 (16)	0.87145 (15)	0.0694 (5)
C20	1.2096 (4)	0.0358 (3)	0.7944 (3)	0.0857 (10)
H20A	1.1501	0.0596	0.7239	0.129*
H20B	1.2622	−0.0580	0.8282	0.129*
H20C	1.2848	0.0823	0.7785	0.129*
C21	0.5908 (3)	0.7521 (2)	0.85127 (18)	0.0471 (5)
C22	0.6713 (3)	0.8181 (2)	0.7668 (2)	0.0580 (6)
H22	0.6773	0.8092	0.6974	0.070*
C23	0.7435 (4)	0.8980 (3)	0.7860 (3)	0.0742 (8)
H23	0.7994	0.9421	0.7296	0.089*
C24	0.7324 (4)	0.9122 (3)	0.8882 (3)	0.0847 (9)
H24	0.7806	0.9661	0.9008	0.102*
C25	0.6505 (4)	0.8469 (3)	0.9714 (3)	0.0859 (10)
H25	0.6431	0.8572	1.0402	0.103*
C26	0.5785 (3)	0.7660 (3)	0.9543 (2)	0.0640 (6)
H26	0.5230	0.7219	1.0109	0.077*

with 6N HCl (pH = 3–4). The resulting precipitate was filtered, washed with ethanol to give 5-(4-methoxyphenoxy)-3-methyl-1-phenyl-1H-pyrazole-4-carbaldehyde (1.07 g, 33 %) compound. To a solution of aldehyde compound obtained above (1 mmol, 0.308 mg) in 20 ml ethanol, was added 1-(4-bromo-2-hydroxyphenyl)ethanone (1 mmol, 0.215 g) and stirred to furnish clear solution. To the reaction mixture a catalytic amount of piperidine was given and stirred at room temperature overnight to give precipitate. The resulting precipitate was filtered and washed with ethanol. The crude solid recrystallized from ethanol solution to form crystals suitable for X-ray diffraction.

2 Experimental details

Data collections and reduction were carried out using the Bruker software APEX2 and SAINT including SADABS.[1] Hydrogen atoms were placed in their geometrically idealized positions and constrained to ride on their parent atoms with C–H = 0.96 Å (methyl), U_{iso} (H) = 1.5 U_{eq} (C), C–H = 0.94 Å (aromatic), U_{iso} (H) = 1.2 U_{eq} (C), O–H = 0.83 Å (hydroxyl), U_{iso} (H) = 1.5 U_{eq} (O).

3 Comment

Pyrazole is a five-membered conjugated aromatic compound that exhibits a wide range of biological activities, including anticancer, antibiotic, antifungal, anti-inflammatory, and antiviral properties.[5] Expanding the widespread use of pyrazole, a huge variety of synthesis methods have been reported over the years.[6,7] The presence of an aryl group in the pyrazole increases the pharmacological properties to some extent.[8,9] Therefore, the title pyrazole compound having a poly-aromatic substituent was synthesized and its crystal structure was identified. The molecular structure of the title compound is shown in the Figure. The central pyrazole ring (C12/C10/C11/N1/N2) connects three benzene rings (C2–C7, C14–C19 and C21–C26). The C21–C26 benzene ring is directly connected to N2 of the pyrazole ring. However, the C14–C19 benzene ring and C2–C7 benzene ring are connected to C12 and C10 of the pyrazole ring through O3 oxygen and C1–C3 enone system, respectively. The *trans* configuration of the C8 db C9 double bond in the enone group is confirmed by the dihedral angle of C1–C8 db C9–C10 of 177.8 (2)°.

The dihedral angle between the pyrazole ring and the C2–C7 benzene ring is 4.05(2)°, indicating that the rings are close to coplanar. On the other hand, the pyrazole ring forms dihedral angles of 70.7(1)° with the C14–C19 benzene ring and 39.1(2)° with the C21–C26 benzene ring, respectively, indicating that these rings are highly twisted with each other. The hydroxyl group attached to the C2–C7 benzene ring forms an intramolecular O2–H2···O1 hydrogen bond. In the crystal, pairs of C15–H15–N1 hydrogen bonds generate inversion dimers with graph-set notation R^2_2 (14) and the dimers are linked by pairwise C4–H4–O2 hydrogen bonds.

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