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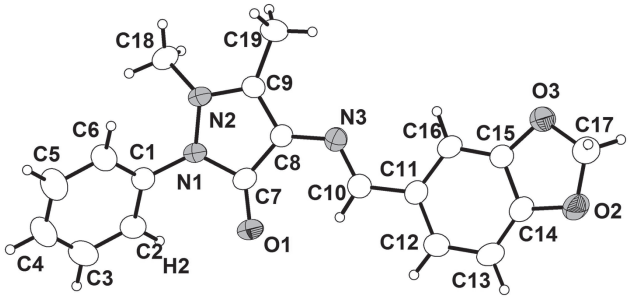
The crystal structure of 4-[(benzo[1,3]dioxol-5-ylmethylene)-amino]-1,5-dimethyl-2-phenyl-1,2-dihydro-pyrazol-3-one, $C_{19}H_{17}N_3O_3$



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Abstract

$C_{19}H_{17}N_3O_3$, monoclinic, $P2_1/a$ (no. 14), $a = 7.1173(4)$ Å, $b = 25.051(2)$ Å, $c = 9.3822(8)$ Å, $\beta = 100.484(6)°$, $V = 1644.9(2)$ Å^3, $Z = 4$, $R_{gt}(F) = 0.0571$, $wR_{ref}(F^2) = 0.1457$, $T = 296$ K.

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The 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.

Source of material

A mixture of 1,3-benzodioxole-5-carboxaldehyde (0.0033 mol, 0.5 g) and 4-amino-1,5-dimethyl-2-phenyl-1,2-dihydro-pyrazol-3-one (0.0033 mol, 0.67 g) in absolute ethanol

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Table 1: Data collection and handling.

Crystal:	Yellow blocks
	Size 0.44 × 0.27 × 0.11 mm
Wavelength:	Mo $K\alpha$ radiation (0.71073 Å)
μ:	0.9 cm^{-1}
Diffractometer, scan mode:	SuperNova, φ and ω
$2\theta_{max}$, completeness:	59°, 85.3%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	8775, 3897, 0.037
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 2358
$N(param)_{refined}$:	226
Programs:	SHELX [14], CrysAlis^{PRO} [15]

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

Atom	x	y	z	U_{iso}*/U_{eq}
C1	0.8310(3)	0.35371(8)	−0.0137(2)	0.0374(5)
C2	0.7184(3)	0.31565(9)	0.0363(3)	0.0459(6)
H2	0.7172	0.3127	0.1350	0.055*
C3	0.6075(4)	0.28200(10)	−0.0613(3)	0.0606(7)
H3	0.5299	0.2567	−0.0281	0.073*
C4	0.6110(4)	0.28558(11)	−0.2069(3)	0.0653(8)
H4	0.5379	0.2624	−0.2718	0.078*
C5	0.7225(4)	0.32344(11)	−0.2561(3)	0.0642(8)
H5	0.7245	0.3259	−0.3547	0.077*
C6	0.8324(3)	0.35812(9)	−0.1600(3)	0.0485(6)
H6	0.9065	0.3841	−0.1939	0.058*
C7	0.8580(3)	0.42317(9)	0.1772(2)	0.0359(5)
C8	0.9856(3)	0.46781(8)	0.2054(2)	0.0344(5)
C9	1.1320(3)	0.45994(8)	0.1306(2)	0.0345(5)
C10	0.8267(3)	0.51901(9)	0.3510(2)	0.0412(5)
H10	0.7369	0.4917	0.3440	0.049*
C11	0.8005(3)	0.56652(9)	0.4360(2)	0.0377(5)
C12	0.6316(3)	0.57225(9)	0.4884(3)	0.0456(6)
H12	0.5382	0.5460	0.4672	0.055*
C13	0.5974(3)	0.61591(9)	0.5715(3)	0.0476(6)
H13	0.4851	0.6191	0.6084	0.057*
C14	0.7364(3)	0.65355(9)	0.5961(3)	0.0423(5)
C15	0.9058(3)	0.64867(9)	0.5442(2)	0.0406(5)
C16	0.9432(3)	0.60544(9)	0.4663(2)	0.0410(5)
H16	1.0589	0.6019	0.4344	0.049*
C17	0.9063(4)	0.72766(10)	0.6527(3)	0.0603(7)

Table 2 (continued)

Atom	x	y	z	U_{iso}*/U_{eq}
H17A	0.8696	0.7576	0.5882	0.072*
H17B	0.9767	0.7412	0.7438	0.072*
C18	1.2537(3)	0.38073(10)	0.0136(3)	0.0502(6)
H18A	1.1983	0.3509	−0.0432	0.075*
H18B	1.3297	0.4012	−0.0413	0.075*
H18C	1.3329	0.3680	0.1008	0.075*
C19	1.3043(3)	0.49301(10)	0.1311(3)	0.0470(6)
H19A	1.3011	0.5233	0.1932	0.071*
H19B	1.4164	0.4722	0.1660	0.071*
H19C	1.3073	0.5050	0.0344	0.071*
N1	0.9411(2)	0.38897(7)	0.08763(19)	0.0376(4)
N2	1.1016(2)	0.41438(7)	0.0507(2)	0.0374(4)
N3	0.9689(2)	0.51374(7)	0.28597(19)	0.0380(4)
O1	0.7026(2)	0.41373(6)	0.21339(18)	0.0478(4)
O2	0.7409(2)	0.69968(7)	0.6766(2)	0.0596(5)
O3	1.0221(2)	0.69157(7)	0.5897(2)	0.0588(5)

(20 mL) was refluxed at 353 K for 2 h with continuous stirring, while a few drops of acetic acid were added as catalyst. The reaction progress was monitored by TLC. On completion, the solution was cooled followed by filtration. The precipitate obtained was purified by recrystallization from ethanol.

Experimental details

All hydrogen atoms were positioned geometrically C—H = 0.93 Å and refined as riding model with $U_{iso} = 1.2U_{eq}$(C). The C—H distances for methyl and methelene were set to 0.96 Å and 0.97 Å and were also refined as riding atoms with $U_{iso} = 1.5U_{eq}$(C) and $U_{iso} = 1.2U_{eq}$(C) respectively.

Discussion

Pyrazole chemistry is well being explored due to their fascinating applications in pharmaceutical [1] and other industries [2–4]. Demands of supramolecular chemistry and metal-organic framework (MOF) applications [5] in various industries encourage the synthesis of novel ligands which have capabilities for complexation [6] with various metals. In this regard, pyrazole based molecules gained reasonable attention. Pyrazoles have found their applications in biological systems as analgesic [7], anti-inflammatory [8], antifungal [9], anti-hyperglycemic [10], antimicrobial [11], antitumor [12], antiviral [13] agents.

The central pyrazole ring (N1/N2/C9/C8/C7) in the crystal structure of title compound is highly crowded. A phenyl ring is present on N_1, on the other hand N_2 and C_9 are flagged by methyl groups. C_7 is functionalised by a carbonyl group and benzo[1,3]dioxol-5-ylmethylene)-amino group is available at C_6. The pyrazole ring is almost planer with 0.0209 Å root mean square deviation of its fitted atoms, the most deviation was observed from N1 = −0.0392(1) Å and N2 = 0.0409(1) Å. C_{18} deviate more (r.m.s. −0.5556(4) Å) as compare to C_{19} (r.m.s. −0.1338(4) Å) to the plane produce through the fitted atoms of pyrazole ring. The ring system benzo[1,3]dioxole is not exactly planar as the dihedral angle between the benzene ring and dioxole ring is 2.14(2)°. The over all r.m.s. deviation of fitted atoms of benzo[1,3]dioxole is 0.0520 Å where the most deviations were observed from C_{17} −0.1204(2) Å and O_2 −0.0688(2) Å. The pyrazole ring is oriented with a dihedral angle of 46.20(1)° and 8.72(1)° with respect to phenyl (C_1-C_6) and benzo[1,3]dioxole ring systems. The phenyl ring is oriented with a dihedral angle of 53.91(1) with respect to benzo[1,3]dioxole. Non-classical intermolecular hydrogen bonding interaction may be present (H···O = 2.46 Å) which connect the molecules to dimers and generating an eighteen membered ring motif. These dimers are further connected through C—H···O (D(H···O) = 2.48 Å), interactions to give an infinite chain along *a*.

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