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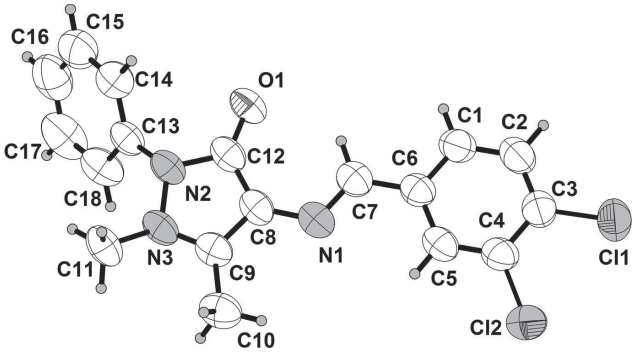
The crystal structure of 4-((3,4-dichlorobenzylidene)amino)-1,5-dimethyl-2-phenyl-1,2-dihydro-3*H*-pyrazol-3-one, $C_{18}H_{15}Cl_2N_3O$



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Abstract

$C_{18}H_{15}Cl_2N_3O$, monoclinic, $P2_1/c$ (no. 14), $a = 9.370(2)$ Å, $b = 28.367(9)$ Å, $c = 6.7161(16)$ Å, $\beta = 103.10(2)°$, $V = 1738.6(8)$ Å^3, $Z = 4$, $R_{gt}(F) = 0.0416$, $wR_{ref}(F^2) = 0.0821$, $T = 293(2)$ K.

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The asymmetric unit of the title crystal structure is shown in the figure (50% probability displacement ellipsoids). Tables 1 and 2 contain details on crystal structure and measurement conditions and a list of the atoms including atomic coordinates and displacement parameters.

Source of materials

Firstly 7.5 mL of a methanol solution of 4-amino-1,5-dimethyl-2-phenyl-1,2-dihydro-3*H*-pyrazol-3-one (0.203 g/1 mmol) was added to 7.5 mL of a methanol solution of 3,4-dichlorobenzaldehyde (0.175 g/1 mmol) in the presence of 1 drop of acetic acid as a catalyst. This mixture was refluxed for 3 h. The resultant solution was cooled to room temperature. The yellow solid of 4-((3,4-dichlorobenzylidene)amino)-1,5-dimethyl-2-phenyl-1,2-dihydro-3*H*-pyrazol-3-one formed, which was filtered and recrystallized in toluene (yield 95%: m.p. 513−515 K).

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Table 1: Crystal collection and handling.

Crystal:	Prism, colourless
Size:	0.490 × 0.267 × 0.080 mm
Wavelength:	Mo $K\alpha$ radiation ($\lambda = 0.71073$ Å)
μ:	0.383 mm^{-1}
Diffractometer, scan mode:	STOE IPDS 2, ω-scans
$2\theta_{max}$, completeness:	25.05°, >99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	12303, 3062, 0.0739
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\sigma(I_{obs})$, 1375
$N(param)_{refined}$:	219
Programs:	X-AREA [1], SHELX [2, 3]

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

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
C1	0.0213(3)	0.43107(11)	0.8578(4)	0.0814(8)
H1	0.013527	0.455320	0.947894	0.098
C2	−0.0368(3)	0.38753(12)	0.8830(5)	0.0847(9)
H2	−0.083974	0.382684	0.989151	0.102
C3	−0.0256(3)	0.35139(10)	0.7533(5)	0.0773(8)
C4	0.0458(3)	0.35887(11)	0.5957(4)	0.0777(8)
C5	0.1012(3)	0.40246(11)	0.5688(4)	0.0754(8)
H5	0.146190	0.407309	0.460513	0.090
C6	0.0917(3)	0.43938(10)	0.6992(4)	0.0693(7)
C7	0.1514(3)	0.48577(10)	0.6729(4)	0.0745(8)
H7	0.134965	0.510341	0.756653	0.089
C8	0.2854(3)	0.53837(11)	0.5248(4)	0.0674(7)
C9	0.3694(3)	0.54908(11)	0.3872(4)	0.0699(8)
C10	0.3939(3)	0.52027(10)	0.2130(4)	0.0891(9)
H10A	0.343233	0.534332	0.086650	0.134
H10B	0.357411	0.488938	0.222861	0.134
H10C	0.496887	0.518946	0.216929	0.134
C11	0.4671(3)	0.62404(10)	0.2747(4)	0.0892(9)
H11A	0.524675	0.607298	0.196372	0.134
H11B	0.522654	0.650126	0.343224	0.134
H11C	0.379247	0.635544	0.185078	0.134
C12	0.2855(3)	0.57881(10)	0.6526(4)	0.0684(8)
C13	0.4472(4)	0.64863(11)	0.7065(4)	0.0673(8)
C14	0.3691(4)	0.68343(12)	0.7745(5)	0.0814(9)
H14	0.267522	0.684105	0.731633	0.098
C15	0.4406(5)	0.71752(13)	0.9066(5)	0.0998(10)
H15	0.387014	0.740709	0.955091	0.120
C16	0.5905(6)	0.71725(15)	0.9664(5)	0.1142(13)
H16	0.638884	0.740430	1.054317	0.137
C17	0.6687(4)	0.68284(17)	0.8965(5)	0.1115(12)
H17	0.770418	0.682801	0.936235	0.134
C18	0.5973(4)	0.64800(13)	0.7669(5)	0.0913(10)
H18	0.650660	0.624341	0.721147	0.110

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Table 2 (continued)

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
N1	0.2262(2)	0.49390(8)	0.5377(3)	0.0728(6)
N2	0.3680(2)	0.61289(9)	0.5786(3)	0.0710(6)
N3	0.4287(2)	0.59230(9)	0.4260(3)	0.0737(6)
O1	0.2332(2)	0.58523(6)	0.8044(3)	0.0821(6)
Cl1	−0.09801(10)	0.29718(3)	0.79073(15)	0.1120(3)
Cl2	0.06154(11)	0.31356(3)	0.43009(15)	0.1208(4)

Experimental details

The structure was solved by Direct Methods and refined by full-matrix least-square techniques. All H atoms were positioned geometrically (C—H = 0.93–0.96 Å) and refined with a riding model.

Comment

Schiff bases have an important role in biological systems with several applications. They and their metal complexes also exhibited a broad range of biological activities, including antifungal, antibacterial, antimalarial, antiproliferative, anti-inflammatory, antiviral, anticancer and antipyretic properties. In addition to biological performances Schiff bases have a variety applications in inorganic (as the catalysts), analytical (as the analytical reagents), organic material chemistry (as the polymers) and industrial fields (as the dyes) [5, 6].

The asymmetric unit of the title compound, $C_{18}H_{15}Cl_2N_3O$, contains aromatic ring systems that are bridged with an imine group. The dichlorobenzyl and pyrazol groups adopt an *E*-configuration around the double bond of C=N. While all the aromatic rings in the molecular system are almost planar, the entire molecular system is not planar. The maximum deviation was seen at pyrazol ring for atom N2 with the value of −0.0426(2) Å.

The dihedral angle between the ring systems of C1—C6(I), C13—C18(II) and C8—C12(III) are (I)–(II) = 63.86(1)°, (I)–(III) = 7.51(1)° and (II)–(III) = 56.44(1)°, respectively. In the molecular structure, all the bond lengths and angles are in good agreement with the similar structure of 4-[(2,4-dichlorobenzylidene)amino]-1,5-dimethyl-2-phenyl-1,2-dihydropyrazol-3-one [7].

In the crystal, molecules are only linked through weak C—H···O type intermolecular hydrogen bonds. In these H bonds, O1 atom of the molecule accepts different H atoms, namely, C2—H2···O1 ($-x$, $-y+1$, $-z+2$) that generates the graph-set descriptor $R_2^2(18)$ [8] and C10—H10A···O1 (x, y, $z-1$). There is also C—H···O type intramolecular H bond, namely, C7—H7···O1 that generates the graph-set descriptor S(6) for the ring motif [8].

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