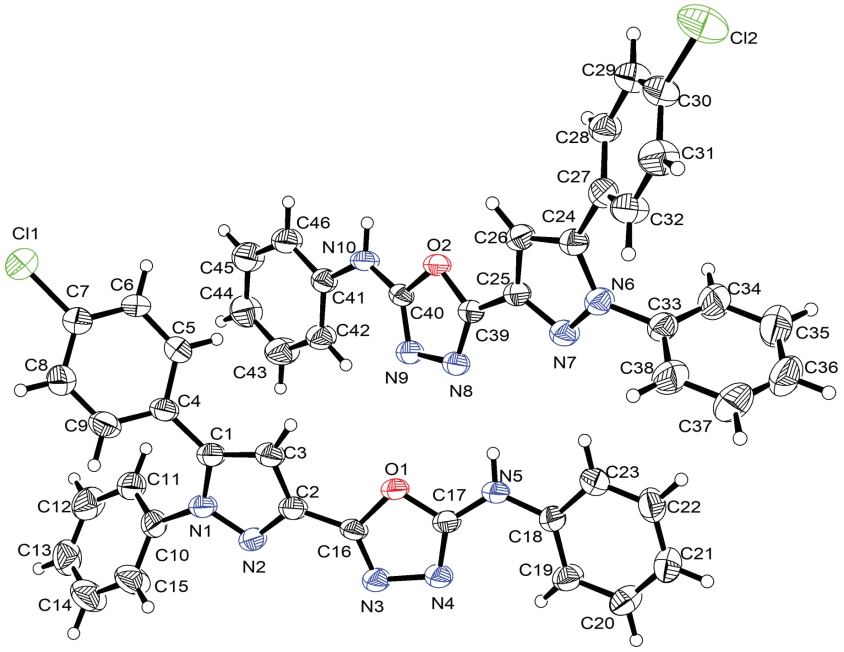
Mohammad Hayal Alotaibi*, Hanan A. Mohamed, Bakr F. Abdel-Wahab, Amany S. Hegazy, Benson M. Kariuki and Gamal A. El-Hiti*

Crystal structure of 5-(5-(4-chlorophenyl)-1-phenyl-1*H*-pyrazol-3-yl)-*N*-phenyl-2-amine, $C_{23}H_{16}ClN_5O$



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Abstract

$C_{23}H_{16}ClN_5O$, monoclinic, $P2_1/c$ (no. 14), $a = 10.0503(6)$ Å, $b = 17.5265(9)$ Å, $c = 22.8763(16)$ Å, $\beta = 91.554(6)°$, $V = 4028.1(4)$ Å^3, $Z = 8$, $R_{gt}(F) = 0.0748$, $wR_{ref}(F^2) = 0.2274$, $T = 296(2)$ K.

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Table 1: Data collection and handling.

Crystal:	Colourless needle
Size:	0.58 × 0.07 × 0.02 mm
Wavelength:	Mo $K\alpha$ radiation (0.71073 Å)
μ:	0.22 mm^{-1}
Diffractometer, scan mode:	SuperNova, ω
θ_{max}, completeness:	29.9°, >99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	41529, 10169, 0.077
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 4311
$N(param)_{refined}$:	541
Programs:	CrysAlis^{PRO} [1], SHELX [2, 3], WinGX/ORTEP [4]

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

***Corresponding authors: Mohammad Hayal Alotaibi,** National Center for Petrochemicals Technology, King Abdulaziz City for Science and Technology, P.O. Box 6086, Riyadh 11442, Saudi Arabia, e-mail: mhhalotaibi@kacst.edu.sa; and **Gamal A. El-Hiti,** Department of Optometry, College of Applied Medical Sciences, King Saud University, P.O. Box 10219, Riyadh 11433, Saudi Arabia, e-mail: gelhiti@ksu.edu.sa
Hanan A. Mohamed and Bakr F. Abdel-Wahab: Department of Chemistry, College of Science and Humanities, Shaqra University, Duwadimi, Saudi Arabia; and Applied Organic Chemistry Department, National Research Centre, Dokki, Giza, Egypt
Amany S. Hegazy and Benson M. Kariuki: School of Chemistry, Cardiff University, Main Building, Park Place, Cardiff CF10 3AT, UK

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

Atom	x	y	z	U_{iso}*/U_{eq}
C1	0.3890(3)	0.07595(17)	0.33409(14)	0.0516(8)
C2	0.2807(3)	0.16753(16)	0.37830(15)	0.0533(8)
C3	0.3989(3)	0.15107(17)	0.34998(15)	0.0576(9)
H3	0.469334	0.184128	0.343358	0.069*
C4	0.4836(3)	0.02830(16)	0.30237(14)	0.0505(8)
C5	0.6183(3)	0.03274(17)	0.31636(15)	0.0603(9)
H5	0.646981	0.066288	0.345563	0.072*
C6	0.7099(3)	−0.01036(17)	0.28876(14)	0.0564(8)
H6	0.799750	−0.006717	0.299160	0.068*
C7	0.6675(4)	−0.05933(18)	0.24531(15)	0.0589(9)
C8	0.5356(4)	−0.0643(2)	0.22852(15)	0.0673(10)
H8	0.508679	−0.096643	0.198204	0.081*
C9	0.4436(4)	−0.02083(19)	0.25713(14)	0.0626(9)
H9	0.354015	−0.024200	0.246218	0.075*
C10	0.2165(3)	−0.02487(16)	0.35582(14)	0.0538(8)
C11	0.2939(4)	−0.08255(17)	0.37802(15)	0.0640(9)
H11	0.381462	−0.073152	0.390111	0.077*
C12	0.2412(5)	−0.15462(19)	0.38234(18)	0.0784(12)
H12	0.293765	−0.194014	0.397436	0.094*
C13	0.1129(5)	−0.1692(2)	0.36478(18)	0.0812(12)
H13	0.077768	−0.218024	0.367998	0.097*
C14	0.0370(5)	−0.1110(2)	0.3424(2)	0.0926(14)
H14	−0.050415	−0.120629	0.330133	0.111*
C15	0.0875(4)	−0.03850(19)	0.33775(19)	0.0822(12)
H15	0.034962	0.000806	0.322538	0.099*
C16	0.2424(3)	0.24025(15)	0.40315(14)	0.0495(8)
C17	0.2760(3)	0.35460(16)	0.43067(14)	0.0503(8)
C18	0.3036(3)	0.49227(15)	0.44615(13)	0.0490(8)
C19	0.1756(3)	0.50888(17)	0.46167(15)	0.0580(9)
H19	0.114822	0.469972	0.468144	0.070*
C20	0.1384(4)	0.58543(18)	0.46756(14)	0.0639(9)
H20	0.051930	0.597136	0.477916	0.077*
C21	0.2266(4)	0.64335(18)	0.45840(15)	0.0634(10)
H21	0.200325	0.693980	0.461928	0.076*
C22	0.3542(4)	0.62560(17)	0.44396(15)	0.0653(10)
H22	0.415008	0.664674	0.438103	0.078*
C23	0.3941(4)	0.55106(17)	0.43797(14)	0.0603(9)
H23	0.481363	0.539981	0.428466	0.072*
C24	0.8949(3)	0.56743(16)	0.33028(13)	0.0498(8)
C25	0.7852(3)	0.47294(15)	0.37025(13)	0.0467(7)
C26	0.9011(3)	0.49075(16)	0.34127(14)	0.0547(8)
H26	0.968906	0.457402	0.331405	0.066*
C27	0.9924(3)	0.61734(15)	0.30187(13)	0.0507(8)
C28	1.1260(3)	0.60727(16)	0.31558(14)	0.0545(8)
H28	1.151888	0.569497	0.342073	0.065*
C29	1.2208(4)	0.65193(18)	0.29083(14)	0.0606(9)
H29	1.310318	0.644402	0.300424	0.073*
C30	1.1833(4)	0.70797(18)	0.25173(14)	0.0572(9)
C31	1.0515(4)	0.71792(19)	0.23610(15)	0.0686(10)
H31	1.026625	0.754689	0.208604	0.082*
C32	0.9560(4)	0.67289(18)	0.26153(15)	0.0626(9)
H32	0.866609	0.679994	0.251428	0.075*
C33	0.7327(3)	0.66869(16)	0.36177(14)	0.0538(8)
C34	0.7923(4)	0.70961(19)	0.40582(18)	0.0785(12)
H34	0.860665	0.688312	0.428654	0.094*
C35	0.7496(5)	0.7833(2)	0.4160(2)	0.1021(16)
H35	0.789406	0.812020	0.445878	0.123*
C36	0.6488(5)	0.8138(2)	0.3822(2)	0.0916(14)
H36	0.620941	0.863560	0.388822	0.110*
C37	0.5894(5)	0.7721(2)	0.3392(2)	0.0909(14)
H37	0.520318	0.793112	0.316618	0.109*
C38	0.6310(4)	0.6986(2)	0.32887(18)	0.0773(11)
H38	0.589699	0.669661	0.299537	0.093*
C39	0.7431(3)	0.39879(15)	0.39055(13)	0.0464(7)
C40	0.7719(3)	0.28299(14)	0.41504(13)	0.0432(7)
C41	0.7971(3)	0.14847(15)	0.44222(13)	0.0469(7)
C42	0.6676(4)	0.13631(17)	0.45812(15)	0.0613(9)
H42	0.607118	0.176465	0.457657	0.074*
C43	0.6283(4)	0.06473(19)	0.47464(17)	0.0757(11)
H43	0.540876	0.056686	0.485467	0.091*
C44	0.7171(4)	0.00425(19)	0.47540(17)	0.0766(11)
H44	0.689683	−0.044051	0.486847	0.092*
C45	0.8442(4)	0.01608(19)	0.45932(18)	0.0752(11)
H45	0.904277	−0.024277	0.459469	0.090*
C46	0.8848(4)	0.08827(17)	0.44264(16)	0.0658(10)
H46	0.972141	0.096049	0.431630	0.079*
N1	0.2686(3)	0.05133(13)	0.35342(11)	0.0522(7)
N2	0.2005(3)	0.10730(13)	0.38100(12)	0.0570(7)
N3	0.1313(3)	0.26436(13)	0.42108(13)	0.0614(8)
N4	0.1515(3)	0.34066(13)	0.44056(13)	0.0591(7)
N5	0.3508(3)	0.41697(13)	0.43757(12)	0.0588(7)
H5A	0.435680	0.410948	0.436741	0.071*
N6	0.7773(3)	0.59160(13)	0.35255(11)	0.0523(7)
N7	0.7085(3)	0.53417(13)	0.37771(12)	0.0526(7)
N8	0.6283(3)	0.37335(12)	0.40348(11)	0.0492(6)
N9	0.6462(3)	0.29614(12)	0.42000(11)	0.0501(6)
N10	0.8453(3)	0.22012(12)	0.42502(11)	0.0518(7)
H10	0.929649	0.224014	0.420462	0.062*
O1	0.3407(2)	0.29346(10)	0.40777(10)	0.0539(6)
O2	0.84051(19)	0.34453(10)	0.39614(9)	0.0472(5)
Cl1	0.78374(11)	−0.11647(6)	0.21089(5)	0.0868(4)
Cl2	1.30314(11)	0.76686(6)	0.22326(4)	0.0885(4)

Source of material

The title compound was synthesized from treatment of 2-(5-(4-chlorophenyl)-1-phenyl-1*H*-pyrazole-3-carbonyl)-*N*-phenylhydrazinecarbothioamide with anhydrous sodium acetate in boiling ethanol for 5 h. The solid obtained on cooling the reaction mixture was filtered, washed with ethanol, dried and crystallized from dimethylformamide to give colorless crystals (63%) of the title compound, Mp. 238−240 °C.

Experimental details

H atoms were positioned geometrically and refined using a riding model. U_{iso}(H) for aromatic and N—H hydrogens were set to 1.2 times U_{eq} for the atoms they are bonded to. Aromatic C—H bonds were fixed at 0.93 Å and N—H at 0.86 Å. The weakly diffracting crystal yielded a *wR*2 factor slightly larger than 20%. Nevertheless the data weren't cut, as the strongest of them may provide some information.

Comment

Heterocycles containing the 1,3,4-oxadiazol-2-amine moiety show different biological activities and act as anticancer and antiproliferative agents [5–8]. Also, the pyrazole ring system is the core of a number of drugs such as celecoxib, fezolamide and difenamizole [9–11].

The asymmetric unit comprises two independent molecules (see the figure). The molecule consists of five rings: phenyl (R1), oxadiazolyl (R2), pyraxolyl (R3), chlorophenyl (R4) and a second phenyl (R5) ring linked to R3. The twist angles between the planes of pairs of linked rings R1–R2, R2–R3, R3–R4, R3–R5 are 17.54(11), 11.94(17), 43.54(12) and 52.49(10)° respectively for the first molecule (C1—C23) and 6.81(12), 18.80(13), 43.10(11) and 7844(11)° respectively for the second molecule (C24—C46). Weak intramolecular contacts of C—H···N type involving phenyl and oxadiazolyl rings occur. $R^2_2(9)$ rings due to N—H···N and C—H···N interactions between neighbouring molecules lead to the formation of ribbons parallel to [100].

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