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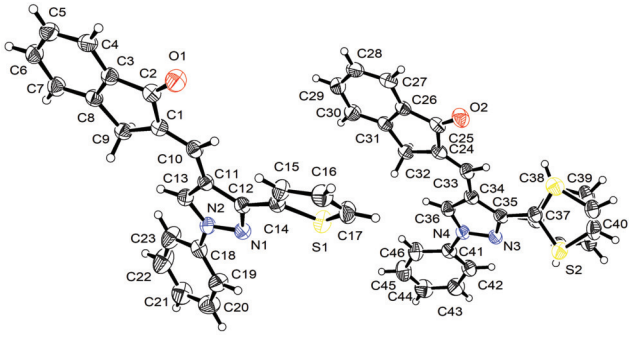
Synthesis and crystal structure of 2-((1-phenyl-3-(thiophen-2-yl)-1*H*-pyrazol-4-yl)methylene)-2,3-dihydro-1*H*-inden-1-one, $C_{23}H_{16}N_2OS$



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Abstract

$C_{23}H_{16}N_2OS$, orthorhombic, $Pna2_1$ (no. 33), $a = 19.4988(9)$ Å, $b = 19.5060(9)$ Å, $c = 9.2893(4)$ Å, $V = 3533.1(3)$ Å^3, $Z = 8$, $R_{gt}(F) = 0.0501$, $wR_{ref}(F^2) = 0.1269$, $T = 296(2)$ K.

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The asymmetric unit containing two crystallogaphically independent molecules is shown in the figure. Table 1 contains crystallographic data and Table 2 contains the list of the atoms including atomic coordinates and displacement parameters.

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Table 1: Data collection and handling.

Crystal:	Yellow plate
Size:	0.55 × 0.38 × 0.12 mm
Wavelength:	Mo $K\alpha$ radiation (0.71073 Å)
μ:	0.20 mm^{-1}
Diffractometer, scan mode:	SuperNova, ω
θ_{max}, completeness:	29.7°, >99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	17282, 7967, 0.039
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 5569
$N(param)_{refined}$:	533
Programs:	CrysAlis^{PRO} [1], SHELX [2, 3], WinGX/ORTEP [4]

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

Atom	x	y	z	U_{iso}*/U_{eq}
N1	0.81811(13)	0.74789(15)	0.8462(3)	0.0400(7)
N2	0.83674(13)	0.81117(15)	0.7963(3)	0.0398(7)
N3	0.06260(14)	0.75158(16)	0.8479(3)	0.0431(7)
N4	0.08813(14)	0.81248(15)	0.7989(3)	0.0428(7)
O1	1.04235(14)	0.63547(14)	0.3821(4)	0.0601(8)
O2	0.29649(15)	0.62024(15)	0.4201(3)	0.0622(8)
C1	0.99248(16)	0.73792(18)	0.4836(4)	0.0384(8)
C2	1.03887(17)	0.69742(19)	0.3894(4)	0.0427(8)
C3	1.07935(16)	0.74778(19)	0.3062(4)	0.0408(8)
C4	1.12964(19)	0.7355(2)	0.2039(4)	0.0485(10)
H4	1.143109	0.690972	0.181915	0.058*
C5	1.1592(2)	0.7909(2)	0.1356(4)	0.0533(11)
H5	1.192687	0.783807	0.065836	0.064*
C6	1.1393(2)	0.8566(2)	0.1704(5)	0.0521(10)
H6	1.159964	0.893400	0.123776	0.063*
C7	1.08948(19)	0.8691(2)	0.2729(4)	0.0494(9)
H7	1.076204	0.913712	0.294673	0.059*
C8	1.05960(17)	0.81350(19)	0.3428(4)	0.0411(8)
C9	1.00469(17)	0.81295(18)	0.4578(4)	0.0400(8)
H9A	1.020511	0.835516	0.544730	0.048*
H9B	0.963276	0.835431	0.424353	0.048*
C10	0.94833(17)	0.70713(19)	0.5740(4)	0.0409(8)
H10	0.948054	0.659466	0.571339	0.049*
C11	0.90116(17)	0.73834(19)	0.6748(4)	0.0375(8)
C12	0.85655(16)	0.70380(17)	0.7736(4)	0.0377(8)

Table 2 (continued)

Atom	x	y	z	U_{iso}*/U_{eq}
C13	0.88595(17)	0.80659(19)	0.6949(4)	0.0410(8)
H13	0.906156	0.843210	0.646871	0.049*
C14	0.84909(17)	0.63101(18)	0.8031(4)	0.0408(8)
C15	0.89497(19)	0.57704(18)	0.7839(4)	0.0459(9)
H15	0.938232	0.581796	0.742941	0.055*
C16	0.8680(2)	0.5143(2)	0.8339(5)	0.0583(11)
H16	0.892066	0.473195	0.830501	0.070*
C17	0.8038(2)	0.5199(2)	0.8871(5)	0.0603(11)
H17	0.778610	0.483258	0.923364	0.072*
S1	0.77468(5)	0.60175(6)	0.87961(14)	0.0585(3)
C18	0.80614(17)	0.87052(18)	0.8567(4)	0.0430(8)
C19	0.7736(2)	0.8670(2)	0.9871(5)	0.0535(10)
H19	0.770456	0.825233	1.034912	0.064*
C20	0.7456(2)	0.9247(3)	1.0477(5)	0.0655(12)
H20	0.723937	0.921903	1.136678	0.079*
C21	0.7493(2)	0.9862(2)	0.9781(6)	0.0689(13)
H21	0.730526	1.025292	1.019562	0.083*
C22	0.7808(3)	0.9899(2)	0.8474(7)	0.0816(16)
H22	0.783078	1.031773	0.799857	0.098*
C23	0.8095(2)	0.9327(2)	0.7842(6)	0.0660(13)
H23	0.830642	0.935670	0.694720	0.079*
C24	0.24134(17)	0.7250(2)	0.5006(4)	0.0431(9)
C25	0.28958(18)	0.6827(2)	0.4147(4)	0.0470(9)
C26	0.32794(17)	0.7300(2)	0.3212(4)	0.0431(9)
C27	0.3787(2)	0.7156(2)	0.2204(4)	0.0513(10)
H27	0.392392	0.670710	0.202823	0.062*
C28	0.4082(2)	0.7695(2)	0.1471(4)	0.0562(11)
H28	0.442575	0.761079	0.079939	0.067*
C29	0.3870(2)	0.8361(2)	0.1732(5)	0.0567(11)
H29	0.407064	0.871915	0.122212	0.068*
C30	0.33686(19)	0.8505(2)	0.2727(5)	0.0534(10)
H30	0.323206	0.895474	0.289477	0.064*
C31	0.30698(17)	0.79674(19)	0.3478(4)	0.0432(8)
C32	0.25205(17)	0.79913(19)	0.4622(4)	0.0434(9)
H32A	0.267213	0.825256	0.545135	0.052*
H32B	0.210198	0.819351	0.424984	0.052*
C33	0.19715(18)	0.6974(2)	0.5946(4)	0.0450(9)
H33	0.198058	0.649930	0.602792	0.054*
C34	0.14812(18)	0.7329(2)	0.6848(4)	0.0430(9)
C35	0.09869(17)	0.70368(18)	0.7799(4)	0.0416(8)
C36	0.13895(18)	0.8023(2)	0.7014(4)	0.0453(9)
H36	0.163394	0.836433	0.653963	0.054*
C37[a]	0.0847(7)	0.6312(2)	0.8080(9)	0.0446(12)
C38[a]	0.0840(6)	0.5769(5)	0.7127(12)	0.059(3)
H38[a]	0.093464	0.580654	0.614892	0.071*
C39[a]	0.0670(4)	0.5145(3)	0.7834(7)	0.0597(14)
H39[a]	0.067362	0.472051	0.737618	0.072*
C40[a]	0.0503(4)	0.5228(3)	0.9233(7)	0.0645(16)
H40[a]	0.036576	0.487340	0.983676	0.077*
S2[a]	0.05747(9)	0.60634(9)	0.97576(18)	0.0614(6)
C37A[b]	0.079(4)	0.6351(7)	0.827(5)	0.054(4)
C38A[b]	0.043(2)	0.6150(12)	0.949(3)	0.058(4)
H38A[b]	0.020552	0.644694	1.011504	0.069*
C39A[b]	0.0464(19)	0.5426(11)	0.967(2)	0.058(4)
H39A[b]	0.038416	0.520215	1.053783	0.070*
C40A[b]	0.062(2)	0.5100(9)	0.843(3)	0.055(4)

Table 2 (continued)

Atom	x	y	z	U_{iso}*/U_{eq}
H40A[b]	0.056163	0.463369	0.827059	0.066*
S2A[b]	0.0954(9)	0.5655(8)	0.7204(16)	0.061(3)
C41	0.06147(18)	0.8750(2)	0.8532(4)	0.0454(9)
C42	0.0016(2)	0.8749(2)	0.9313(5)	0.0541(10)
H42	−0.022648	0.834272	0.944071	0.065*
C43	−0.0224(2)	0.9354(2)	0.9911(6)	0.0654(12)
H43	−0.062948	0.935425	1.043924	0.079*
C44	0.0130(3)	0.9946(2)	0.9728(6)	0.0702(13)
H44	−0.002466	1.034658	1.016449	0.084*
C45	0.0718(2)	0.9960(2)	0.8899(6)	0.0714(14)
H45	0.094976	1.037023	0.875170	0.086*
C46	0.0960(2)	0.9361(2)	0.8289(5)	0.0581(11)
H46	0.135249	0.936729	0.771918	0.070*

[a]Occupancy: 0.831(5), [b]Occupancy: 0.169(5).

Source of material

The title compound was synthesized based on a literature procedure from reaction of equimolar amounts of 1-phenyl-3-(thiophen-2-yl)-1*H*-pyrazole-4-carbaldehyde and 2,3-dihydro-1*H*-inden-1-one in boiling ethanol containing few drops of piperidine as catalyst for 2 h [5]. Crystallization of the solid obtained using dimethylformamide gave colorless crystals of the title compound (87%).

Experimental details

All hydrogen atoms were placed in calculated positions and refined using a riding model. Aromatic C—H distances were set to 0.93 Å (AFIX 43 instruction in SHELXL [2, 3]) and their U_{iso} set to 1.2 times the U_{eq}(C). Methylene C—H distances were set to 0.97 Å and their U_{iso} to 1.2 times the U_{eq}(C) (AFIX 23). Crystal data, data collection and structure refinement details are summarized in Table 1.

Comment

Pyrazoles exhibit a wide range of biological activities and can be used as therapeutic agents such as celecoxib, rimonabant, difenamizole, betazole, and fezolamide [6–10]. Therefore, synthesis of heterocycles containing pyrazole ring systems is always important [11–13].

The molecule of the title compound comprises indanone (A), pyrazolyl (B), phenyl (C) and thiophenyl (D) ring systems. The asymmetric unit consists of two independent molecules: **1** (C1—C23) and **2** (C24—C46). The thiophenyl group is disordered in molecule **2** by a rotation of 180° about the $C_{thiophene}$–$C_{pyrazole}$ bond with component occupancies of 0.83(1) and 0.17(1). The twist angles between the planes through rings A–B, B–C, B–D are 2.3(1)°, 19.1(2)°, 23.0(2)° for molecule **1** and 5.4(2)°, 13.8(2)°, 34.4(3)° for molecule **2**. Bond lengths and angles are in the typical ranges [14].

In the structure, molecules **1** and **2** are arranged in pairs with indanone centroid-centroid separation of 4.87 Å. The pairs of molecules are stacked to form columns parallel to [100]. Neighbouring columns are related by translation along [001] and by 2_1 symmetry along [010]. C—H···O interactions form an extended network.

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References

1. Rigaku Oxford Diffraction: CrysAlis^{PRO}. Rigaku Oxford Diffraction, Yarnton, England (2015).
2. Sheldrick, G. M.: A short history of SHELX. Acta Crystallogr. **A64** (2008) 112–122.
3. Sheldrick, G. M.: Crystal structure refinement with SHELXL. Acta Crystallogr. **C71** (2015) 3–8.
4. Farrugia, L. J.: WinGX and ORTEP for Windows: an update. J. Appl. Crystallogr. **45** (2012) 849–854.
5. Huck, J.; Saladin, R.; Sierra, M.; Klotz, E.: Modulation of peroxisome proliferator activated receptors activity. PCT Int. Appl. (2004) WO 2004/037248 A2.
6. Karrouchi, K.; Radi, S.; Ramli, Y.; Taoufik, J.; Mabkhot, Y. N.; Al-Aizari, F. A.; Ansar, M.: Synthesis and pharmacological activities of pyrazole derivatives: a review. Molecules **23** (2018) 134.
7. Ganguly, S.; Jacob, S. K.: Therapeutic outlook of pyrazole analogs: a mini review. Mini-Rev. Med. Chem. **17** (2017) 959–983.
8. Faria, J. V.; Vegi, P. F.; Miguita, A. G. C.; Dos Santos, M. S.; Boechat, N.; Bernardino, A. M. R.: Recently reported biological activities of pyrazole compounds. Bioorg. Med. Chem. **25** (2017) 5891–5903.
9. Naim, M. J.; Alam, O.; Nawaz, F.; Alam, M. J.; Alam, P.: Current status of pyrazole and its biological activities. J. Pharm. Bioall. Sci. **8** (2016) 2–17.
10. Khan, M. F.; Alam, M. M.; Verma, G.; Akhtar, W.; Akhter, M.; Shaquiquzzaman, M.: The therapeutic voyage of pyrazole and its analogs: a review. Eur. J. Med. Chem. **120** (2016) 170–201.
11. Wen, J.; Fu, Y.; Hang, R.-Y.; Zhang, J.; Chen, S.-Y.; Yu, X.-Q.: A simple and efficient synthesis of pyrazoles in water. Tetrahedron **67** (2011) 9618–9621.
12. Sun, A.; Ye, J.-H.; Yu, H.; Zhang, W.; Wang, X.: A new efficient synthesis of pyrazoles from hydrazonoyl halides and β-oxophosphonates. Tetrahedron Lett. **55** (2014) 889–892.
13. Maddila, S.; Jonnalagadda, S. B.; Gangu, K. K.; Maddila, S. N.: Recent advances in the synthesis of pyrazole derivatives using multicomponent reactions. Curr. Org. Chem. **14** (2017) 634–653.
14. El-Hiti, G. A.; Abdel-wahab, B. F.; Hegazy, A. S.; Alamri, M.; Kariuki, B. M.: Crystal structure of 2-((3-(5-methyl-1-phenyl-1*H*-1,2,3-triazol-4-yl)-1-phenyl-1*H*-pyrazol-4-yl)methylene)-1*H*-indene-1,3(2*H*)-dione, $C_{28}H_{19}N_5O_2$. Z. Kristallogr. NCS **232** (2017) 19–20.