

Synthesis, crystal structure and Hirshfeld surface analysis of 2-{4-[(2-chlorophenyl)methyl]-3-methyl-6-oxopyridazin-1-yl}-*N*-phenylacetamide

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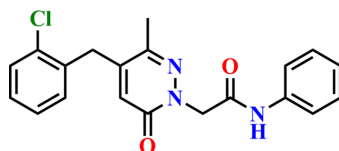
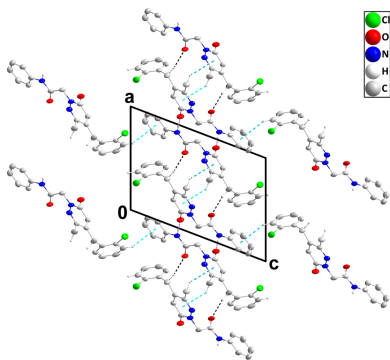
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In the title molecule, C₂₀H₁₈ClN₃O₂, the 2-chlorophenyl group is disordered to a small extent [occupancies 0.875 (2)/0.125 (2)]. The phenylacetamide moiety is nearly planar due to a weak, intramolecular C—H···O hydrogen bond. In the crystal, N—H···O hydrogen bonds and π -stacking interactions between pyridazine and phenyl rings form helical chains of molecules in the *b*-axis direction, which are linked by C—H···O hydrogen bonds and C—H··· π (ring) interactions. A Hirshfeld surface analysis was performed, which showed that H···H, C···H/H···C and O···H/H···O interactions to dominate the intermolecular contacts in the crystal.

1. Chemical context

Various classes of heterocyclic compounds have been widely proven to exhibit diverse biological activities (Ameziane El Hassani *et al.*, 2023; Missioui *et al.*, 2022a). Among them, pyridazin-3(2*H*)-one derivatives have emerged as one of the most studied scaffolds in recent decades (Akhtar *et al.*, 2016; Dubey & Bhosle, 2015). Known as a ‘wonder nucleus’, pyridazin-3(2*H*)-one has provided numerous derivatives with diverse pharmacological profiles. This heterocyclic compound has been shown to possess various biological activities, including anti-microbial (Özdemir *et al.*, 2019), anti-cancer (Bouchmaa *et al.*, 2019), butyrylcholinesterase inhibitors (Dundar *et al.*, 2019), anti-convulsant (Siddiqui *et al.*, 2020), anti-inflammatory (Boukharsa *et al.*, 2018; Zaoui *et al.*, 2021), anti-diabetic (Assila *et al.*, 2024; Boukharsa *et al.*, 2024). Acetamide derivatives, due to their wide range of activities (Missioui *et al.*, 2022a,b; Mortada *et al.*, 2023; Dahmani *et al.*, 2024), continue to hold significant importance as intermediates in organic chemistry. As a continuation of our work in synthesizing new *N*-arylacetamide derivatives (Guerrab *et al.*, 2021; Missioui *et al.*, 2020, 2021), and developing new pyridazine-3(2*H*)-one compounds (Zaoui *et al.*, 2022), the title compound, C₂₀H₁₈ClN₃O₂, was synthesized and its crystal structure is reported here. A Hirshfeld surface analysis was performed to analyze the intermolecular interactions.



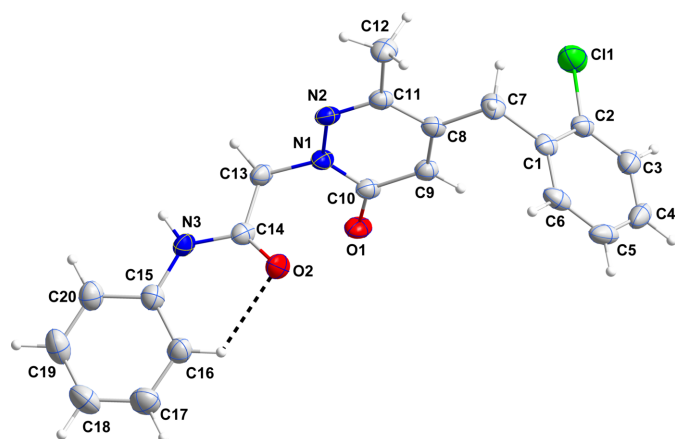


Figure 1
The title molecule with labeling scheme and 50% probability ellipsoids. The intramolecular C—H...O hydrogen bond is depicted by a dashed line. Only the major portion of the disordered 2-chlorophenyl group is shown.

2. Structural commentary

The dihedral angle between the mean planes of the C15–C20 and the pyridazine rings is 56.13 (13)° while that between the mean planes of the pyridazine and the major component of the disordered 2-chlorophenyl rings is 80.98 (11)°. The two components of the latter ring make a dihedral angle of 4.2 (12)°. The phenylacetamide moiety is nearly planar [largest deviation of an atom from the mean plane is 0.003 (3) Å] due to the weak, intramolecular C16—H16...O2 hydrogen bond (Table 1, Fig. 1) and the sum of the angles about N3 is 360° within experimental error. This suggests

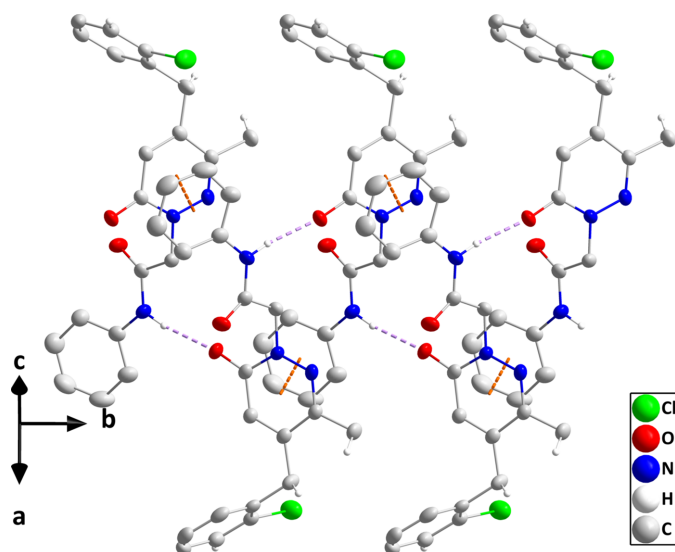


Figure 2
Perspective view of a portion of one chain of molecules. N—H...O hydrogen bonds and π -stacking interactions are depicted, respectively, by violet and orange dashed lines. Non-interacting hydrogen atoms and the minor portion of the disordered 2-chlorophenyl group are omitted for clarity.

Table 1
Hydrogen-bond geometry (Å, °).

Cg1 and Cg3 are the centroids of the C8/C9/C10/N1/N2/C11 and C15–C20 rings, respectively.

<i>D</i> —H... <i>A</i>	<i>D</i> —H	H... <i>A</i>	<i>D</i> ... <i>A</i>	<i>D</i> —H... <i>A</i>
N3—H3...O1 ⁱ	0.90 (1)	1.96 (2)	2.843 (4)	168 (4)
C3—H3A...Cg3 ⁱⁱ	0.95	2.94	3.791 (3)	149
C7—H7A...O2 ⁱⁱⁱ	0.99	2.33	3.303 (5)	167
C12—H12B...Cg1 ⁱⁱⁱ	0.98	2.99	3.670 (4)	128
C16—H16...O2	0.95	2.32	2.918 (4)	121

Symmetry codes: (i) $-x, y + \frac{1}{2}, -z + 1$; (ii) $x, y, z - 1$; (iii) $-x + 1, y + \frac{1}{2}, -z + 1$.

involvement of its lone pair in N→C π bonding in support of which, the N3—C14 and N3—C15 bond distances are, respectively, 1.363 (4) and 1.409 (5) Å.

3. Supramolecular features

In the crystal, N3—H3...O1 hydrogen bonds and π -stacking interactions between pyridazine and C15–C20 rings related by the symmetry operation $-x, y + \frac{1}{2}, -z + 1$ [centroid–centroid distance = 3.691 (2) Å, dihedral angle = 2.13 (18)°, slippage = 1.25 Å] form helical chains of molecules extending along the *b*-axis direction (Table 1 and Fig. 2). These are connected by C7—H7A...O2 hydrogen bonds and C3—H3A...Cg3 and C12—H12B...Cg1 interactions (Table 1; Cg1 and Cg3 are the centroids of the C8/C9/C10/N1/N2/C11 and C15–C20 rings, respectively), forming the full three-dimensional structure (Fig. 3).

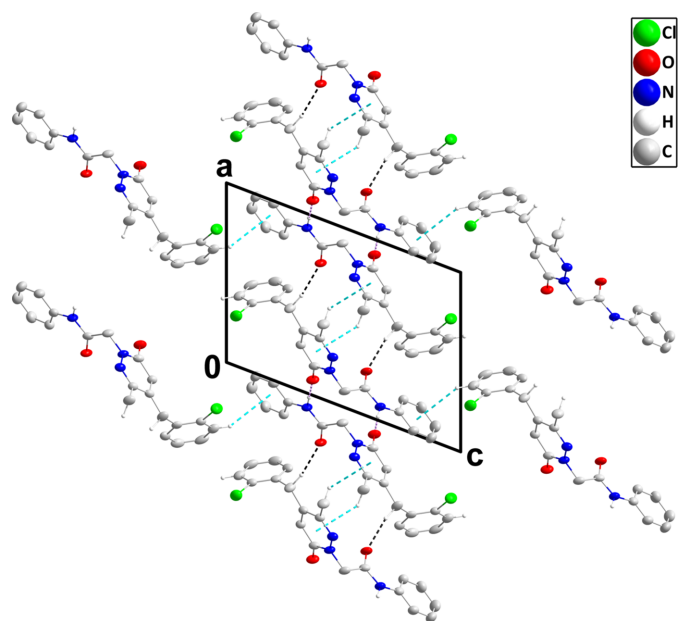


Figure 3
Packing viewed along the *b*-axis direction with N—H...O and C—H...O hydrogen bonds depicted, respectively, by violet and black dashed lines while C—H... π (ring) interactions are depicted by light-blue dashed lines. The π -stacking interactions, non-interacting hydrogen atoms and the minor portion of the disordered 2-chlorophenyl group are omitted for clarity.

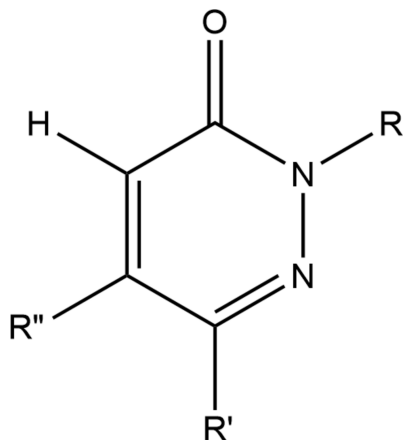


Figure 4
The search fragment used in the Cambridge Structural Database search.

4. Database survey

A search of the Cambridge Structural Database (CSD, updated to June 2024; Groom *et al.*, 2016) with the fragment shown in Fig. 4 ($R = R' = R'' = C$) gave 15 hits of which 12 were considered similar to the title molecule. The closest analog has $R = 4\text{-FC}_6\text{H}_4\text{NHC(=O)CH}_2$, $R' = \text{Me}$, $R'' = 2\text{-ClC}_6\text{H}_4$ (FITXUF; Assila *et al.*, 2023) and is largely the same in all respects, even down to the disorder in the 2-chlorophenyl group. The packing is somewhat different due to the presence of intermolecular $\text{C-H}\cdots\text{F}$ hydrogen bonds. Among the others is a group of structures having $R' = R'' = \text{Ph}$ and $R = \text{-CH}_2\text{COOH}$ (CIPTOL; Aydin *et al.*, 2007), (4-methyl)piperazin-1-yl- $\text{C(=O)CH}_2\text{CH}_2\text{-}$ (LOBTAY; Aydin *et al.*, 2008) and (4-chlorophenyl)piperazin-1-yl- $\text{C(=O)CH}_2\text{CH}_2\text{-}$ (QEDXXA; Aydin *et al.*, 2012). The remainder are those with $R = \text{-CH}_2\text{COOEt}$, $R' = \text{Me}$, $R'' = 4\text{-MeC}_6\text{H}_4\text{CH}_2\text{-}$ (EMOGUL; Zaoui *et al.*, 2021); $R = \text{-CH}_2\text{CH}_2\text{OH}$, $R' = \text{Me}$, $R'' = 2\text{-ClC}_6\text{H}_4\text{CH}_2\text{-}$ (IJEMOZ; Abourichaa *et al.*, 2003); $R = (5\text{-trifluoromethyl)benzo}[d]\text{thiazol-2-yl)CH}_2\text{-}$ (JOXVUN; Mylari *et al.*, 1992); $R = R'' = \text{Ph}$, $R' = 4\text{-ClC}_6\text{H}_4\text{-}$ (QOLLOU; Mantovani *et al.*, 2014); $R = \text{-CH}_2\text{COOEt}$, $R' = \text{Me}$, $R'' = 4\text{-ClC}_6\text{H}_4\text{CH}_2\text{-}$ (SIQXAV; Zaoui *et al.*, 2023); $R = \text{-CH}_2\text{COOEt}$, $R' = \text{Me}$, $R'' = \text{C}_6\text{H}_5\text{CH}_2\text{-}$ (WOCGON; Zaoui *et al.*, 2019); $R = \text{-CH}_2\text{COOEt}$, $R' = \text{Me}$, $R'' = 5\text{-chlorobenzofuran-2-yl-CH}_2\text{-}$ (XULSEE; Boukharsa *et al.*, 2015); $R = \text{-CH}_2\text{COOEt}$, $R' = \text{Me}$, $R'' = 4\text{-MeOC}_6\text{H}_4\text{CH}_2\text{-}$ (YAZLEU; Zaoui *et al.*, 2022). In EMOGUL, IJEMOZ and WOCGON, the pyridazine ring is planar with deviations from the mean plane by no more than 0.007 Å while in CIPTOL, XULSEE and YAZLEU the ring is more ‘ruffled’; with deviations ranging from 0.022 to 0.031 Å. The most non-planar pyridazine ring was found in QEDXAA where the largest deviation is 0.062 (2) Å. In those structures where a ring or ring system is attached to the pyridazine ring via a methylene group, that ring is nearly perpendicular to the mean plane of the pyridazine ring as is the 2-chlorophenyl group in the title molecule. Other flexible substituents are generally rotated well out of the mean plane of the pyridazine ring. In FITXUF and CIPTOL, the primary intermolecular interactions are classical hydrogen bonds ($\text{N-H}\cdots\text{O}$ and

$\text{O-H}\cdots\text{O}$, respectively), which generate chains of molecules as the basic building blocks of the 3-D structures. In the others, chains of molecules or chains of inversion dimers are formed in most cases by $\text{C-H}\cdots\text{O}$ hydrogen bonds with additional $\text{C-H}\cdots\text{O}$ and, in some instances, $\text{C-H}\cdots\text{N}$ hydrogen bonds serving to generate the complete 3-D structures.

5. Hirshfeld surface analysis

A Hirshfeld surface analysis of the intermolecular interactions in the crystal of the title molecule was performed with *CrystalExplorer* (Spackman *et al.*, 2021) with general details of the plots produced and their interpretation provided in a recent publication (Tan *et al.*, 2019). Fig. 5a shows the d_{norm} surface calculated over the range -0.5868 to 1.6936 in arbitrary units with neighboring molecules that are hydrogen bonded to it (green dashed lines). Fig. 5b shows the surface calculated over the shape function with one neighboring molecule showing the π -stacking interaction (red dashed lines). Fingerprint plots showing the major contributions to

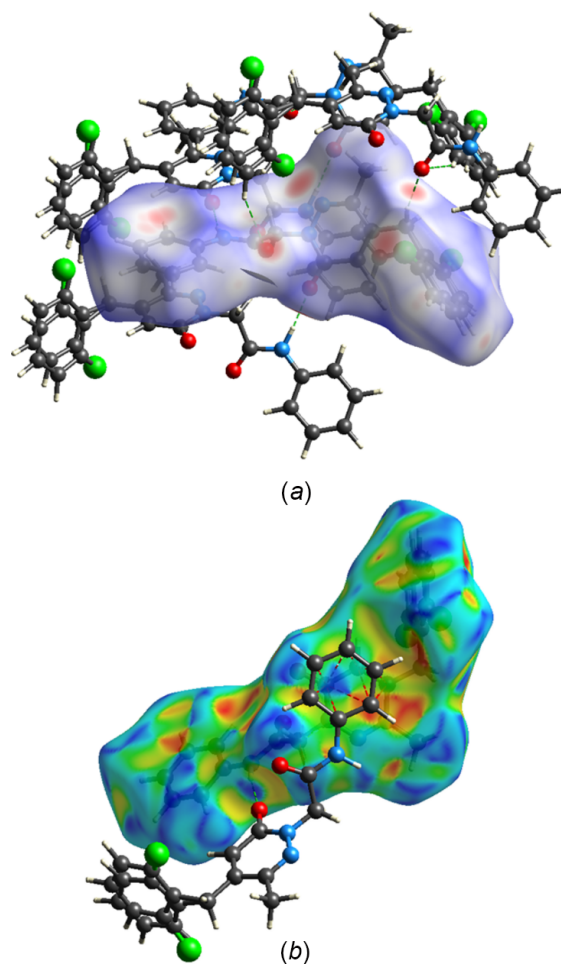


Figure 5
(a) The d_{norm} surface with neighboring molecules showing the hydrogen bonds as green dashed lines and (b) the surface calculated over the shape-index function with one neighboring molecule showing the π -stacking interactions as red dashed lines.

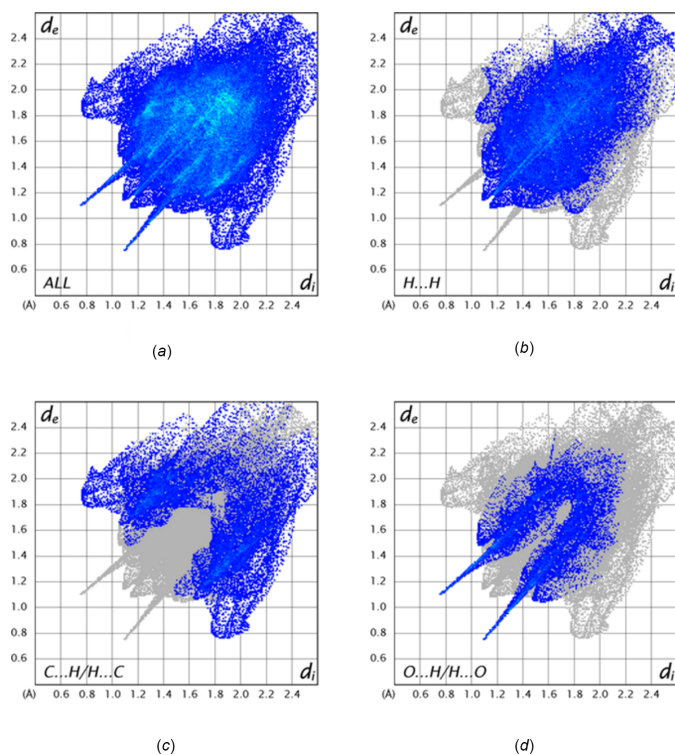


Figure 6 Fingerprint plots showing (a) all intermolecular interactions and those delineated into (b) H...H interactions, (c) C...H/H...C interactions and (d) O...H/H...O interactions.

the intermolecular interactions in the crystal are presented in Fig. 6. In Fig. 6a all these interactions are shown, while Fig. 6b–6d highlight the H...H, C...H/H...C and O...H/H...O interactions, respectively. The H...H contacts account for 43.8% of all intermolecular interactions, and result from the significant hydrogen content of the molecule and the fact that most of the hydrogen atoms comprise its periphery. The C...H/H...C contacts contribute 21.0% with those indicated by peaks having the highest density at $d_e + d_i = 3.3 \text{ \AA}$ coming primarily from the C—H... π (ring) interactions (Table 1). The O...H/H...O interactions contribute 13.7% and are represented by a pair of sharp spikes having $d_e + d_i = 2.2 \text{ \AA}$, which can be attributed to the N—H...O hydrogen bonds as well as a pair of rather broad peaks at longer distances. The latter likely represent the C—H...O hydrogen bonds, which have a wider distribution of H...O distances. Other atom–atom contacts each contribute less than 10% to the overall intermolecular interactions in the crystal.

6. Synthesis and crystallization

A mixture of the 3-benzylidene-4-oxopentanoic acid derivative (0.01 mol) and hydrazine monohydrate (0.02 mol) in 30 mL of ethanol was refluxed to produce the 5-(2-chlorobenzyl)-6-methylpyridazin-3(2H)-one precursor. To this pyridazin-3(2H)-one derivative (0.01 mol), 2-chloro-N-phenylacetamide (0.01 mol), potassium bicarbonate (0.02 mol), and a small amount of BTBA (benzyltributyl-

Table 2 Experimental details.

Crystal data	
Chemical formula	$C_{20}H_{18}ClN_3O_2$
M_r	367.82
Crystal system, space group	Monoclinic, $P2_1$
Temperature (K)	125
a, b, c (Å)	10.1898 (6), 6.7445 (4), 14.2538 (11)
β (°)	110.901 (2)
V (Å ³)	915.13 (10)
Z	2
Radiation type	Mo $K\alpha$
μ (mm ⁻¹)	0.23
Crystal size (mm)	0.35 × 0.32 × 0.05
Data collection	
Diffractometer	Bruker D8 QUEST PHOTON 3 diffractometer
Absorption correction	Numerical (SADABS; Krause <i>et al.</i> , 2015)
T_{\min}, T_{\max}	0.92, 0.99
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	21487, 3700, 3381
R_{int}	0.039
$(\sin \theta/\lambda)_{\text{max}}$ (Å ⁻¹)	0.626
Refinement	
$R[F^2 > 2\sigma(F^2)], wR(F^2), S$	0.049, 0.111, 1.09
No. of reflections	3700
No. of parameters	237
No. of restraints	4
H-atom treatment	H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{\text{max}}, \Delta\rho_{\text{min}}$ (e Å ⁻³)	0.49, -0.17
Absolute structure	Flack x determined using 1318 quotients $[(I^+) - (I^-)] / [(I^+) + (I^-)]$ (Parsons <i>et al.</i> , 2013)
Absolute structure parameter	0.06 (3)

Computer programs: APEX4 and SAINT (Bruker, 2021), SHELXT (Sheldrick, 2015a), SHELXL2019/1 (Sheldrick, 2015b), DIAMOND (Brandenburg & Putz, 2012) and SHELXTL (Sheldrick, 2008).

ammonium bromide) as a phase-transfer catalyst were added. The reaction mixture was stirred at room temperature for 24 h, and the reaction progress was monitored by TLC. Afterwards, 200 mL of distilled water were added, and the resulting precipitate was filtered, dried, and recrystallized from absolute acetone, yielding transparent crystals of the target compound.

Yield 90%; m.p: (461–463 K). IR (KBr, ν (cm⁻¹): 1597 (C=O pyridazinone), 1655 (C=O acetamide), 3279 (NH amide). ¹H NMR [500 MHz, DMSO- d_6 , δ (ppm)]: 2.25 (s, 3H, CH₃); 3.96 (s, 2H, phenyl-CH₂-pyridazinone); 4.80 (s, 2H, N-CH₂-CO); 6.07 (s, 1H, pyridazinone); 6.95–7.57 (m, 9H, two phenyl); 10.26 (s, 1H, NH). ¹³C NMR [126 MHz, DMSO- d_6 , δ (ppm)]: 19.07, 35.33, 54.69, 119.53, 123.94, 126.57, 128.31, 129.35, 129.72, 130.16, 132.08, 134.03, 134.87, 139.29, 144.85, 159.86, 165.74. MS (ESI⁺): $m/z = 368.11530 [M + H]^+$

7. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2. H atoms attached to carbon were placed in calculated positions and were included as riding

contributions with isotropic displacement parameters tied to those of the attached atoms. That attached to nitrogen was placed in a location derived from a difference map and refined with a DFIX 0.91 0.01 instruction. The 2-chlorophenyl ring is disordered over two sites by an approximate 180° rotation about the C1–C7 bond and a small translation in the plane of the ring. The two rings were refined as rigid hexagons and additional restraints were applied to render the geometries of the two components similar. The refined ratio for the disorder is 0.875 (2)/0.125 (2).

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supporting information

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Synthesis, crystal structure and Hirshfeld surface analysis of 2-{4-[(2-chlorophenyl)methyl]-3-methyl-6-oxopyridazin-1-yl}-*N*-phenylacetamide

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Computing details

2-{4-[(2-Chlorophenyl)methyl]-3-methyl-6-oxopyridazin-1-yl}-*N*-phenylacetamide

Crystal data

$C_{20}H_{18}ClN_3O_2$

$M_r = 367.82$

Monoclinic, $P2_1$

$a = 10.1898$ (6) Å

$b = 6.7445$ (4) Å

$c = 14.2538$ (11) Å

$\beta = 110.901$ (2)°

$V = 915.13$ (10) Å³

$Z = 2$

$F(000) = 384$

$D_x = 1.335$ Mg m⁻³

Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å

Cell parameters from 9927 reflections

$\theta = 3.0$ – 26.4 °

$\mu = 0.23$ mm⁻¹

$T = 125$ K

Plate, colourless

$0.35 \times 0.32 \times 0.05$ mm

Data collection

Bruker D8 QUEST PHOTON 3

diffractometer

Radiation source: fine-focus sealed tube

Graphite monochromator

Detector resolution: 7.3910 pixels mm⁻¹

φ and ω scans

Absorption correction: numerical
(*SADABS*; Krause *et al.*, 2015)

$T_{\min} = 0.92$, $T_{\max} = 0.99$

21487 measured reflections

3700 independent reflections

3381 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.039$

$\theta_{\max} = 26.4$ °, $\theta_{\min} = 2.1$ °

$h = -12 \rightarrow 12$

$k = -8 \rightarrow 8$

$l = -17 \rightarrow 17$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.049$

$wR(F^2) = 0.111$

$S = 1.09$

3700 reflections

237 parameters

4 restraints

Hydrogen site location: mixed

H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0454P)^2 + 0.4761P]$

where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} < 0.001$

$\Delta\rho_{\max} = 0.49$ e Å⁻³

$\Delta\rho_{\min} = -0.17$ e Å⁻³

Absolute structure: Flack x determined using

1318 quotients $[(F^+)-(F^-)]/[(F^+)+(F^-)]$ (Parsons *et al.*, 2013)

Absolute structure parameter: 0.06 (3)

Special details

Experimental. The diffraction data were obtained from 4 sets of frames, each of width 0.5° in ω or φ , collected with scan parameters determined by the "strategy" routine in *APEX4*. The scan time was 25 sec/frame.

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted R-factor wR and goodness of fit S are based on F^2 , conventional R-factors R are based on F, with F set to zero for negative F^2 . The threshold expression of $F^2 > 2\sigma(F^2)$ is used only for calculating R-factors(gt) etc. and is not relevant to the choice of reflections for refinement. R-factors based on F^2 are statistically about twice as large as those based on F, and R-factors based on ALL data will be even larger. H-atoms attached to carbon were placed in calculated positions (C—H = 0.95 - 0.99 Å) and were included as riding contributions with isotropic displacement parameters 1.2 - 1.5 times those of the attached atoms. That attached to nitrogen was placed in a location derived from a difference map and refined with a DFIX 0.91 0.01 instruction. The 2-chlorophenyl ring is disordered over two sites by an approximate 180° rotation about the C1—C7 bond and a small translation in the plane of the ring. The two rings were refined as rigid hexagons and additional restraints were applied to render the geometries of the two components similar. The refined ratio for the disorder is 0.875 (2)/0.125 (2).

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (\AA^2)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$	Occ. (<1)
C11	0.27934 (12)	0.6674 (2)	0.04167 (9)	0.0495 (3)	0.875 (2)
C11A	0.6147 (9)	0.3441 (14)	0.3447 (6)	0.0495 (3)	0.125 (2)
O1	0.0861 (3)	0.3089 (4)	0.3685 (2)	0.0370 (6)	
O2	0.2464 (2)	0.3392 (4)	0.5976 (2)	0.0374 (6)	
N1	0.1739 (3)	0.6043 (4)	0.4408 (2)	0.0291 (7)	
N2	0.2548 (3)	0.7696 (4)	0.4533 (2)	0.0298 (7)	
N3	0.0797 (3)	0.4603 (5)	0.6563 (2)	0.0307 (6)	
H3	0.016 (3)	0.559 (4)	0.646 (3)	0.037*	
C1	0.4556 (3)	0.4862 (3)	0.21149 (17)	0.0290 (9)	0.875 (2)
C2	0.3839 (3)	0.4761 (4)	0.10839 (17)	0.0290 (9)	0.875 (2)
C3	0.3975 (3)	0.3096 (4)	0.05509 (14)	0.0363 (10)	0.875 (2)
H3A	0.348463	0.302757	-0.015380	0.044*	0.875 (2)
C4	0.4828 (3)	0.1532 (4)	0.1049 (2)	0.0441 (12)	0.875 (2)
H4	0.492079	0.039454	0.068461	0.053*	0.875 (2)
C5	0.5545 (3)	0.1633 (3)	0.2080 (2)	0.0420 (10)	0.875 (2)
H5	0.612792	0.056389	0.242031	0.050*	0.875 (2)
C6	0.5409 (3)	0.3298 (4)	0.26129 (15)	0.0355 (10)	0.875 (2)
H6	0.589890	0.336628	0.331761	0.043*	0.875 (2)
C1A	0.418 (2)	0.519 (3)	0.1856 (11)	0.0290 (9)	0.125 (2)
C2A	0.506 (2)	0.356 (3)	0.2204 (7)	0.0290 (9)	0.125 (2)
C3A	0.508 (2)	0.205 (2)	0.1544 (14)	0.0363 (10)	0.125 (2)
H3B	0.567692	0.093525	0.178101	0.044*	0.125 (2)
C4A	0.423 (2)	0.218 (2)	0.0536 (12)	0.0441 (12)	0.125 (2)
H4A	0.424547	0.114648	0.008509	0.053*	0.125 (2)
C5A	0.336 (2)	0.381 (3)	0.0189 (6)	0.0420 (10)	0.125 (2)
H5A	0.277563	0.389180	-0.049963	0.050*	0.125 (2)
C6A	0.333 (2)	0.531 (2)	0.0849 (12)	0.0355 (10)	0.125 (2)
H6A	0.273723	0.642592	0.061157	0.043*	0.125 (2)

C7	0.4373 (4)	0.6682 (6)	0.2715 (3)	0.0377 (8)
H7A	0.530622	0.706460	0.320000	0.045*
H7B	0.401834	0.780294	0.224379	0.045*
C8	0.3387 (3)	0.6365 (5)	0.3282 (2)	0.0275 (7)
C9	0.2553 (3)	0.4749 (5)	0.3170 (3)	0.0282 (7)
H9	0.256025	0.375222	0.270067	0.034*
C10	0.1656 (3)	0.4513 (5)	0.3749 (3)	0.0293 (7)
C11	0.3333 (3)	0.7870 (5)	0.3987 (2)	0.0275 (7)
C12	0.4186 (4)	0.9741 (6)	0.4144 (3)	0.0377 (9)
H12A	0.394636	1.045273	0.350544	0.057*
H12B	0.518800	0.940812	0.439537	0.057*
H12C	0.397954	1.058368	0.463472	0.057*
C13	0.0906 (4)	0.5972 (5)	0.5049 (3)	0.0318 (8)
H13A	-0.006953	0.558849	0.464055	0.038*
H13B	0.087788	0.730915	0.532710	0.038*
C14	0.1493 (3)	0.4510 (5)	0.5906 (3)	0.0293 (7)
C15	0.0918 (3)	0.3325 (6)	0.7371 (3)	0.0308 (8)
C16	0.1674 (4)	0.1558 (6)	0.7552 (3)	0.0356 (8)
H16	0.221207	0.120335	0.715397	0.043*
C17	0.1635 (4)	0.0319 (7)	0.8320 (3)	0.0452 (10)
H17	0.214380	-0.089253	0.843960	0.054*
C18	0.0871 (5)	0.0822 (7)	0.8909 (3)	0.0505 (11)
H18	0.084005	-0.004487	0.942638	0.061*
C19	0.0149 (5)	0.2595 (8)	0.8743 (3)	0.0522 (12)
H19	-0.036243	0.295941	0.915817	0.063*
C20	0.0162 (4)	0.3842 (7)	0.7983 (3)	0.0400 (9)
H20	-0.034335	0.505612	0.787291	0.048*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.0450 (6)	0.0538 (7)	0.0516 (6)	0.0160 (6)	0.0197 (5)	0.0161 (6)
Cl1A	0.0450 (6)	0.0538 (7)	0.0516 (6)	0.0160 (6)	0.0197 (5)	0.0161 (6)
O1	0.0345 (13)	0.0240 (13)	0.0591 (16)	-0.0059 (11)	0.0247 (12)	0.0021 (12)
O2	0.0287 (13)	0.0395 (16)	0.0501 (15)	0.0115 (12)	0.0215 (11)	0.0096 (12)
N1	0.0264 (14)	0.0222 (15)	0.0441 (17)	-0.0008 (12)	0.0194 (13)	0.0030 (12)
N2	0.0280 (15)	0.0199 (14)	0.0423 (17)	0.0021 (12)	0.0137 (13)	0.0044 (13)
N3	0.0265 (14)	0.0277 (16)	0.0419 (16)	0.0062 (13)	0.0172 (13)	0.0015 (14)
C1	0.024 (2)	0.028 (2)	0.040 (2)	-0.0051 (18)	0.0176 (17)	0.0035 (19)
C2	0.0255 (19)	0.028 (2)	0.037 (2)	-0.0005 (16)	0.0149 (16)	0.0055 (19)
C3	0.039 (2)	0.032 (2)	0.046 (2)	-0.006 (2)	0.024 (2)	-0.003 (2)
C4	0.051 (3)	0.027 (2)	0.073 (3)	-0.003 (2)	0.045 (3)	-0.006 (2)
C5	0.037 (2)	0.030 (2)	0.067 (3)	0.005 (2)	0.028 (2)	0.018 (2)
C6	0.029 (2)	0.040 (3)	0.039 (2)	-0.0026 (19)	0.0138 (19)	0.015 (2)
C1A	0.024 (2)	0.028 (2)	0.040 (2)	-0.0051 (18)	0.0176 (17)	0.0035 (19)
C2A	0.0255 (19)	0.028 (2)	0.037 (2)	-0.0005 (16)	0.0149 (16)	0.0055 (19)
C3A	0.039 (2)	0.032 (2)	0.046 (2)	-0.006 (2)	0.024 (2)	-0.003 (2)
C4A	0.051 (3)	0.027 (2)	0.073 (3)	-0.003 (2)	0.045 (3)	-0.006 (2)

C5A	0.037 (2)	0.030 (2)	0.067 (3)	0.005 (2)	0.028 (2)	0.018 (2)
C6A	0.029 (2)	0.040 (3)	0.039 (2)	-0.0026 (19)	0.0138 (19)	0.015 (2)
C7	0.041 (2)	0.0331 (19)	0.046 (2)	-0.0146 (19)	0.0252 (17)	-0.0070 (19)
C8	0.0227 (15)	0.0263 (18)	0.0326 (17)	0.0000 (14)	0.0089 (13)	0.0054 (15)
C9	0.0264 (16)	0.0240 (18)	0.0350 (17)	-0.0009 (15)	0.0121 (13)	0.0015 (15)
C10	0.0233 (16)	0.0239 (17)	0.0426 (19)	0.0010 (15)	0.0139 (14)	0.0057 (16)
C11	0.0238 (16)	0.0235 (17)	0.0355 (19)	-0.0001 (14)	0.0109 (14)	0.0037 (15)
C12	0.0381 (19)	0.0272 (19)	0.053 (2)	-0.0102 (17)	0.0222 (17)	-0.0051 (18)
C13	0.0287 (17)	0.0249 (18)	0.049 (2)	0.0045 (14)	0.0221 (16)	0.0034 (15)
C14	0.0218 (15)	0.0254 (17)	0.0426 (19)	-0.0024 (15)	0.0140 (14)	0.0003 (16)
C15	0.0247 (16)	0.034 (2)	0.0339 (18)	-0.0034 (15)	0.0105 (15)	-0.0024 (15)
C16	0.0340 (18)	0.037 (2)	0.0361 (18)	0.0036 (18)	0.0130 (15)	0.0006 (18)
C17	0.045 (2)	0.046 (3)	0.040 (2)	0.0059 (19)	0.0106 (18)	0.0084 (18)
C18	0.052 (3)	0.057 (3)	0.043 (2)	-0.003 (2)	0.018 (2)	0.016 (2)
C19	0.047 (2)	0.075 (3)	0.042 (2)	0.002 (2)	0.025 (2)	0.003 (2)
C20	0.0321 (19)	0.047 (2)	0.044 (2)	0.0025 (18)	0.0175 (17)	-0.0005 (18)

Geometric parameters (Å, °)

C11—C2	1.726 (2)	C4A—H4A	0.9500
C11A—C2A	1.725 (3)	C5A—C6A	1.3900
O1—C10	1.238 (4)	C5A—H5A	0.9500
O2—C14	1.220 (4)	C6A—H6A	0.9500
N1—N2	1.360 (4)	C7—C8	1.512 (4)
N1—C10	1.377 (5)	C7—H7A	0.9900
N1—C13	1.454 (4)	C7—H7B	0.9900
N2—C11	1.305 (4)	C8—C9	1.356 (5)
N3—C14	1.363 (4)	C8—C11	1.444 (5)
N3—C15	1.409 (5)	C9—C10	1.443 (5)
N3—H3	0.902 (14)	C9—H9	0.9500
C1—C2	1.3900	C11—C12	1.503 (5)
C1—C6	1.3900	C12—H12A	0.9800
C1—C7	1.545 (4)	C12—H12B	0.9800
C2—C3	1.3900	C12—H12C	0.9800
C3—C4	1.3900	C13—C14	1.517 (5)
C3—H3A	0.9500	C13—H13A	0.9900
C4—C5	1.3900	C13—H13B	0.9900
C4—H4	0.9500	C15—C16	1.393 (5)
C5—C6	1.3900	C15—C20	1.397 (5)
C5—H5	0.9500	C16—C17	1.388 (6)
C6—H6	0.9500	C16—H16	0.9500
C1A—C2A	1.3900	C17—C18	1.377 (6)
C1A—C6A	1.3900	C17—H17	0.9500
C1A—C7	1.544 (5)	C18—C19	1.379 (7)
C2A—C3A	1.3900	C18—H18	0.9500
C3A—C4A	1.3900	C19—C20	1.376 (6)
C3A—H3B	0.9500	C19—H19	0.9500
C4A—C5A	1.3900	C20—H20	0.9500

N2—N1—C10	126.2 (3)	C1—C7—H7B	108.6
N2—N1—C13	114.0 (3)	H7A—C7—H7B	107.6
C10—N1—C13	119.7 (3)	C9—C8—C11	118.0 (3)
C11—N2—N1	117.8 (3)	C9—C8—C7	124.0 (3)
C14—N3—C15	128.2 (3)	C11—C8—C7	118.1 (3)
C14—N3—H3	116 (3)	C8—C9—C10	121.3 (3)
C15—N3—H3	115 (3)	C8—C9—H9	119.3
C2—C1—C6	120.0	C10—C9—H9	119.3
C2—C1—C7	120.2 (2)	O1—C10—N1	120.7 (3)
C6—C1—C7	119.8 (2)	O1—C10—C9	125.1 (3)
C1—C2—C3	120.0	N1—C10—C9	114.2 (3)
C1—C2—C11	122.29 (16)	N2—C11—C8	122.4 (3)
C3—C2—C11	117.70 (16)	N2—C11—C12	115.5 (3)
C4—C3—C2	120.0	C8—C11—C12	122.1 (3)
C4—C3—H3A	120.0	C11—C12—H12A	109.5
C2—C3—H3A	120.0	C11—C12—H12B	109.5
C3—C4—C5	120.0	H12A—C12—H12B	109.5
C3—C4—H4	120.0	C11—C12—H12C	109.5
C5—C4—H4	120.0	H12A—C12—H12C	109.5
C6—C5—C4	120.0	H12B—C12—H12C	109.5
C6—C5—H5	120.0	N1—C13—C14	112.1 (3)
C4—C5—H5	120.0	N1—C13—H13A	109.2
C5—C6—C1	120.0	C14—C13—H13A	109.2
C5—C6—H6	120.0	N1—C13—H13B	109.2
C1—C6—H6	120.0	C14—C13—H13B	109.2
C2A—C1A—C6A	120.0	H13A—C13—H13B	107.9
C2A—C1A—C7	110.6 (14)	O2—C14—N3	125.3 (3)
C6A—C1A—C7	129.4 (14)	O2—C14—C13	122.9 (3)
C1A—C2A—C3A	120.0	N3—C14—C13	111.8 (3)
C1A—C2A—C11A	119.8 (13)	C16—C15—C20	119.3 (3)
C3A—C2A—C11A	120.2 (13)	C16—C15—N3	123.8 (3)
C4A—C3A—C2A	120.0	C20—C15—N3	116.8 (3)
C4A—C3A—H3B	120.0	C17—C16—C15	119.5 (3)
C2A—C3A—H3B	120.0	C17—C16—H16	120.3
C3A—C4A—C5A	120.0	C15—C16—H16	120.3
C3A—C4A—H4A	120.0	C18—C17—C16	120.9 (4)
C5A—C4A—H4A	120.0	C18—C17—H17	119.5
C6A—C5A—C4A	120.0	C16—C17—H17	119.5
C6A—C5A—H5A	120.0	C17—C18—C19	119.4 (4)
C4A—C5A—H5A	120.0	C17—C18—H18	120.3
C5A—C6A—C1A	120.0	C19—C18—H18	120.3
C5A—C6A—H6A	120.0	C20—C19—C18	120.7 (4)
C1A—C6A—H6A	120.0	C20—C19—H19	119.6
C8—C7—C1A	114.4 (10)	C18—C19—H19	119.6
C8—C7—C1	114.7 (3)	C19—C20—C15	120.1 (4)
C8—C7—H7A	108.6	C19—C20—H20	120.0
C1—C7—H7A	108.6	C15—C20—H20	120.0

C8—C7—H7B	108.6		
C10—N1—N2—C11	-0.4 (5)	C1—C7—C8—C11	170.1 (3)
C13—N1—N2—C11	179.7 (3)	C11—C8—C9—C10	-1.1 (5)
C6—C1—C2—C3	0.0	C7—C8—C9—C10	178.7 (3)
C7—C1—C2—C3	-178.2 (3)	N2—N1—C10—O1	-178.7 (3)
C6—C1—C2—C11	-178.6 (2)	C13—N1—C10—O1	1.1 (5)
C7—C1—C2—C11	3.2 (3)	N2—N1—C10—C9	1.2 (5)
C1—C2—C3—C4	0.0	C13—N1—C10—C9	-179.0 (3)
C11—C2—C3—C4	178.6 (2)	C8—C9—C10—O1	179.6 (3)
C2—C3—C4—C5	0.0	C8—C9—C10—N1	-0.3 (5)
C3—C4—C5—C6	0.0	N1—N2—C11—C8	-1.3 (5)
C4—C5—C6—C1	0.0	N1—N2—C11—C12	179.0 (3)
C2—C1—C6—C5	0.0	C9—C8—C11—N2	2.0 (5)
C7—C1—C6—C5	178.2 (3)	C7—C8—C11—N2	-177.8 (3)
C6A—C1A—C2A—C3A	0.0	C9—C8—C11—C12	-178.3 (3)
C7—C1A—C2A—C3A	-178.3 (19)	C7—C8—C11—C12	1.9 (5)
C6A—C1A—C2A—C11A	178.2 (17)	N2—N1—C13—C14	-105.6 (3)
C7—C1A—C2A—C11A	-0.1 (16)	C10—N1—C13—C14	74.6 (4)
C1A—C2A—C3A—C4A	0.0	C15—N3—C14—O2	-8.2 (6)
C11A—C2A—C3A—C4A	-178.2 (17)	C15—N3—C14—C13	171.3 (3)
C2A—C3A—C4A—C5A	0.0	N1—C13—C14—O2	-7.1 (5)
C3A—C4A—C5A—C6A	0.0	N1—C13—C14—N3	173.5 (3)
C4A—C5A—C6A—C1A	0.0	C14—N3—C15—C16	-8.5 (6)
C2A—C1A—C6A—C5A	0.0	C14—N3—C15—C20	175.1 (3)
C7—C1A—C6A—C5A	178 (2)	C20—C15—C16—C17	1.6 (5)
C2A—C1A—C7—C8	-91.0 (12)	N3—C15—C16—C17	-174.7 (4)
C6A—C1A—C7—C8	90.9 (16)	C15—C16—C17—C18	-0.6 (6)
C2—C1—C7—C8	101.0 (3)	C16—C17—C18—C19	-0.9 (7)
C6—C1—C7—C8	-77.2 (3)	C17—C18—C19—C20	1.4 (7)
C1A—C7—C8—C9	9.9 (9)	C18—C19—C20—C15	-0.4 (6)
C1—C7—C8—C9	-9.7 (5)	C16—C15—C20—C19	-1.1 (6)
C1A—C7—C8—C11	-170.3 (8)	N3—C15—C20—C19	175.4 (4)

Hydrogen-bond geometry (\AA , $^\circ$)

Cg1 and Cg3 are the centroids of the C8/C9/C10/N1/N2/C11 and C15–C20 rings, respectively.

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
N3—H3 \cdots O1 ⁱ	0.90 (1)	1.96 (2)	2.843 (4)	168 (4)
C3—H3A \cdots Cg3 ⁱⁱ	0.95	2.94	3.791 (3)	149
C7—H7A \cdots O2 ⁱⁱⁱ	0.99	2.33	3.303 (5)	167
C12—H12B \cdots Cg1 ⁱⁱⁱ	0.98	2.99	3.670 (4)	128
C16—H16 \cdots O2	0.95	2.32	2.918 (4)	121

Symmetry codes: (i) $-x, y+1/2, -z+1$; (ii) $x, y, z-1$; (iii) $-x+1, y+1/2, -z+1$.