



# Crystal structure and Hirshfeld surface analysis of (2Z)-3-oxo-N-phenyl-2-[(1H-pyrrol-2-yl)methylidene]butanamide monohydrate

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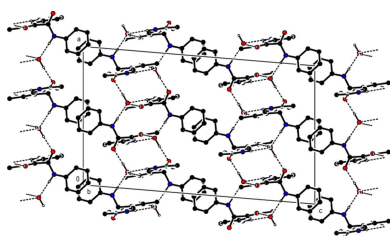
**Supporting information:** this article has supporting information at journals.iucr.org/e

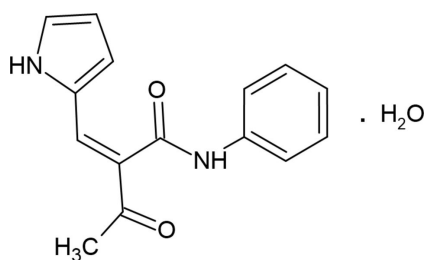
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In the title compound, C<sub>15</sub>H<sub>14</sub>N<sub>2</sub>O<sub>2</sub>·H<sub>2</sub>O, the 1H-pyrrole ring makes a dihedral angle of 59.95 (13)<sup>o</sup> with the phenyl ring. In the crystal, the molecules are connected by C—H···O hydrogen bonds into layers parallel to the (020) plane, while two molecules are connected to the water molecule by two N—H···O hydrogen bonds and one molecule by an O—H···O hydrogen bond. C—H···π and π–π interactions further link the molecules into chains extending in the [101] direction and stabilize the molecular packing. According to a Hirshfeld surface study, H···H (49.4%), C···H/H···C (23.2%) and O···H/H···O (20.0%) interactions are the most significant contributors to the crystal packing.

## 1. Chemical context

Heterocyclic and carbocyclic aromatic systems are the most important compounds in organic chemistry (Gurbanov *et al.*, 2017; Aliyeva *et al.*, 2023). Organic synthesis is developing enormously with newer aromatic compounds having been obtained for diverse medicinal and commercial purposes (Maharramov *et al.*, 2021; Poustforoosh *et al.*, 2022; Gurbanov *et al.*, 2022a,b). Nowadays, the application of five and six-membered heterocycles in particular has been expanded in different branches of chemistry, including coordination chemistry (Gurbanov *et al.*, 2021; Mahmoudi *et al.*, 2021), drug design and development (Çelik *et al.*, 2023) and material science (Velásquez *et al.*, 2019; Afkhami *et al.*, 2019). The pyrrole core is the most common five-membered heteroaromatic ring system in nitrogen heterocycles (Mahmoudi *et al.*, 2017). It is an essential structural motif present in many natural tetrapyrrole scaffolds of heme and related cofactors (chlorophyll *a*, heme *b*, vitamin B<sub>12</sub>, factor 430) and other bioactive molecules such as porphobilinogen, nargenicin and prodigiosin (Walsh *et al.*, 2006). The combination of different pharmacophores in a pyrrole ring system has led to the formation of more active compounds, such as elopiprazole, lorpiiprazole, isamoltane, obatoclax (Bhardwaj *et al.*, 2015). On the other hand, there have been a variety of significant examples of pyrrole derivatives used as target products as well as synthetic intermediates (Naghiyev *et al.*, 2020, 2021, 2022).

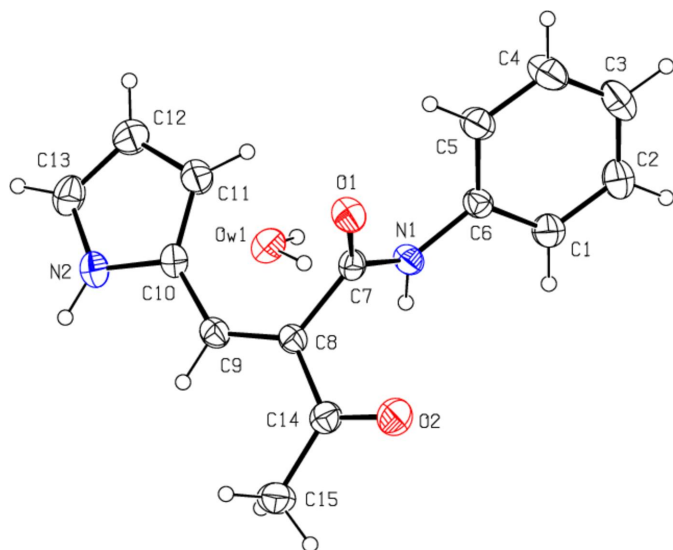




## 2. Structural commentary

The title compound crystallizes with one water molecule in the asymmetric unit (Fig. 1). The 1*H*-pyrrole ring (N2/C10–C13) makes a dihedral angle of 59.95 (13)° with the phenyl ring (C1–C6). The conformation is stabilized by an intramolecular C5–H5···O1 interaction (Table 1). In addition, an OW1–HW1···O1 hydrogen bond is observed between the main molecule and the water molecule in the asymmetric unit (Table 1). The 1*H*-pyrrole ring and *N*-phenylformamide substituents on the C8=C9 double bond are in a *cis* configuration [the C7–C8–C9–C10 torsion angle is 1.5 (3)°] and the 1*H*-pyrrole ring and the acetaldehyde substituents are in a *trans* configuration [the C14–C8–C9–C10 torsion angle is 179.17 (18)°].

The other torsion angles C5–C6–N1–C7, C6–N1–C7–O1, C6–N1–C7–C8, N1–C7–C8–C14, N1–C7–C8–C9, C7–C8–C14–C15 and C8–C9–C10–C11 are –30.7 (3), 6.7 (3), –172.19 (17), 85.2 (2), –97.0 (2), –176.03 (18) and –1.0 (4)°, respectively. The geometric parameters of the title compound are normal and comparable to that of related compound listed in the *Database survey* section.



**Figure 1**

The molecular structure of the title compound, showing the atom labeling and displacement ellipsoids drawn at the 30% probability level.

**Table 1**

Hydrogen-bond geometry (Å, °).

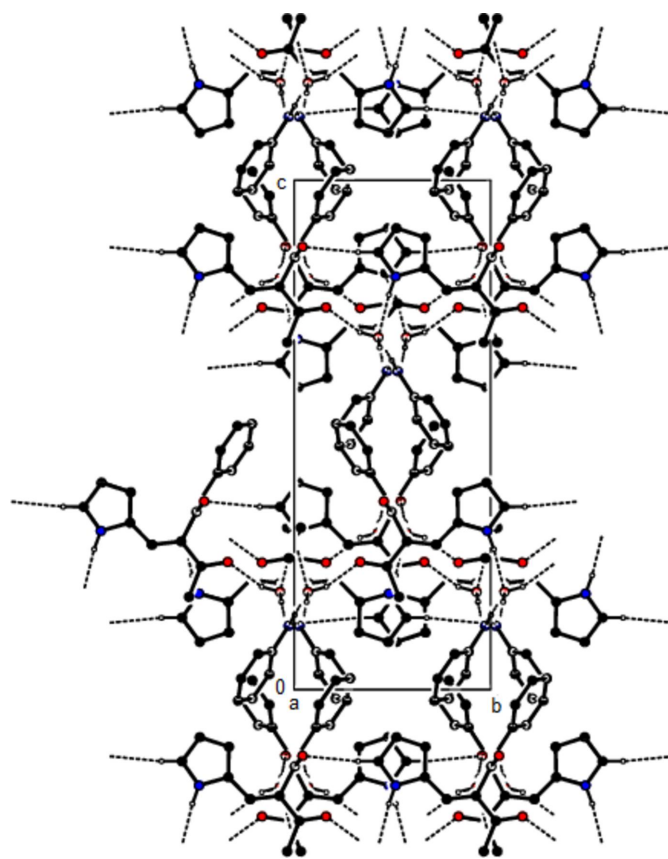
Cg1 is the centroid of the N2/C10–C13 pyrrole ring.

<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>
C5–H5···O1	0.93	2.41	2.906 (3)	113
C13–H13···O1 <sup>i</sup>	0.93	2.56	3.480 (3)	173
N1–HN1···OW1 <sup>ii</sup>	0.91 (2)	1.99 (2)	2.898 (2)	179 (2)
N2–HN2···OW1 <sup>iii</sup>	0.89 (2)	2.02 (2)	2.901 (2)	173 (2)
OW1–HW1···O1	0.92 (3)	1.80 (3)	2.718 (2)	177 (2)
OW1–HW2···O2 <sup>iv</sup>	0.87 (3)	1.92 (3)	2.750 (2)	160 (2)
C15–H15C···Cg1 <sup>iii</sup>	0.96	2.66	3.536 (3)	151

Symmetry codes: (i)  $x, y - 1, z$ ; (ii)  $x - \frac{1}{2}, -y + 1, z$ ; (iii)  $-x + \frac{3}{2}, -y + \frac{1}{2}, -z + \frac{1}{2}$ ; (iv)  $-x + \frac{3}{2}, -y + \frac{3}{2}, -z + \frac{1}{2}$ .

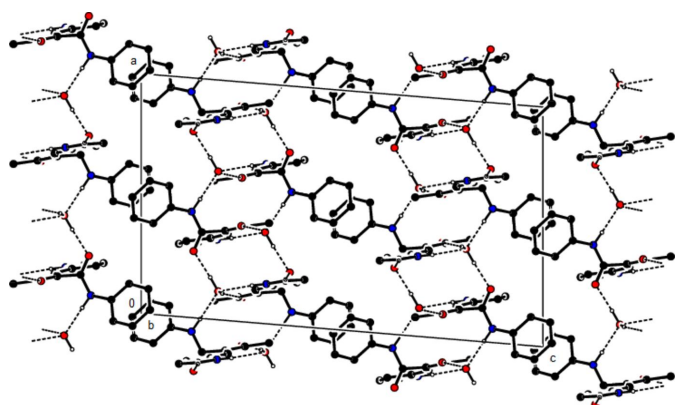
## 3. Supramolecular features and Hirshfeld surface analysis

In the crystal, the molecules are also connected by C–H···O hydrogen bonds in layers parallel to the (020) plane, while two molecules are connected to the water molecule by two N–H···O hydrogen bonds and one molecule by an O–H···O hydrogen bond (Table 1, Figs. 2 and 3). C–H··· $\pi$  and  $\pi$ – $\pi$  interactions [ $Cg2 \cdots Cg2(1 - x, 1 - y, 1 - z) = 3.8404(16)$  Å, slippage = 0.858 Å; Cg2 is the centroid of phenyl ring C1–C6] link the molecules into chains extending in



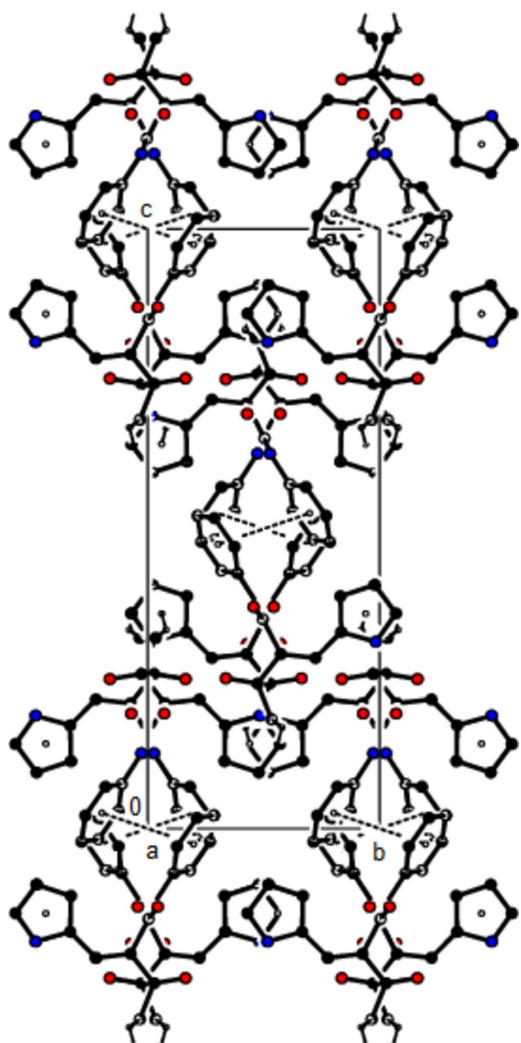
**Figure 2**

View of the crystal packing of the title compound along the *a*-axis showing N–H···O, C–H···O and O–H···O hydrogen bonds as dashed lines.



**Figure 3**  
View of the crystal packing of the title compound along the *b*-axis showing N–H···O, C–H···O and O–H···O hydrogen bonds as dashed lines.

the  $[\bar{1}01]$  direction and stabilize the molecular packing (Table 1, Figs. 4 and 5).

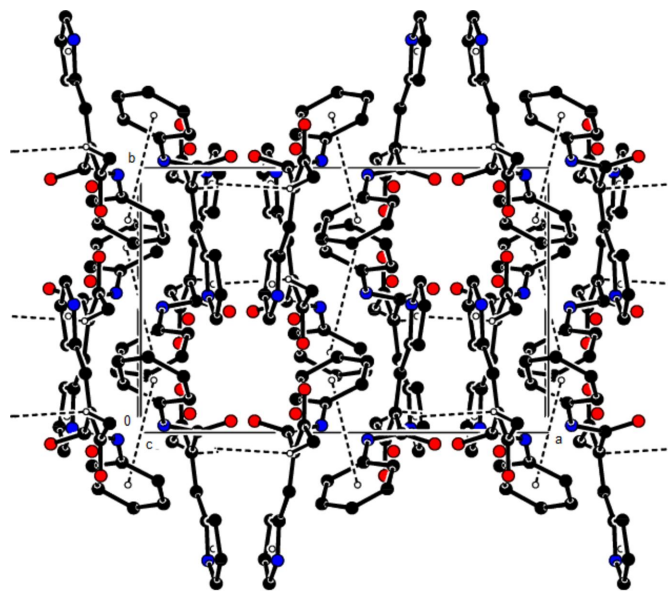


**Figure 4**  
View of the crystal packing of the title compound along the *a*-axis showing the C–H··· $\pi$  and  $\pi$ – $\pi$  interactions as dashed lines.

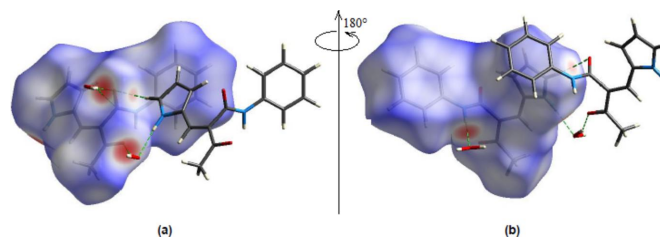
**Table 2**  
Summary of short interatomic contacts (Å) in the title compound.

Contact	Distance	Symmetry operation
O1···HW1	1.80	$x, y, z$
O1···H13	2.56	$x, 1 + y, z$
O2···HW2	1.92	$\frac{3}{2} - x, \frac{3}{2} - y, \frac{1}{2} - z$
HN1···OW1	1.99	$-\frac{1}{2} + x, 1 - y, z$
N2···H15B	2.92	$1 - x, -\frac{1}{2} + y, \frac{1}{2} - z$
HN2···OW1	2.02	$-x, \frac{1}{2} - y, \frac{1}{2} - z$
H15C···N2	2.76	$-x, \frac{1}{2} - y, \frac{1}{2} - z$
H5···H5	2.36	$-x, y, 1 - z$
H3···C11	3.06	$1 - x, 1 - y, 1 - z$
H3···H15A	2.59	$x, \frac{3}{2} - y, \frac{1}{2} + z$

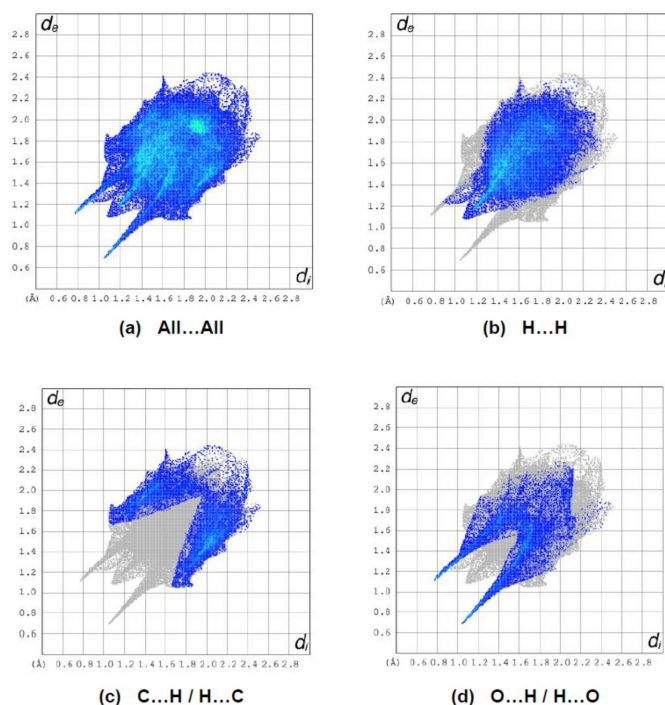
*Crystal Explorer 17.5* (Spackman *et al.*, 2021) was used to generate Hirshfeld surfaces and two-dimensional fingerprint plots in order to quantify the intermolecular interactions in the crystal. The Hirshfeld surfaces were mapped over  $d_{\text{norm}}$  in the range  $-0.6778$  (red) to  $+1.5015$  (blue) a.u. (Fig. 6). The interactions given in Table 2 play a key role in the molecular packing of the title compound. The most important interatomic contact is H···H as it makes the highest contribution to the crystal packing (49.4%, Fig. 7b). Other major contributors



**Figure 5**  
View of the crystal packing of the title compound along the *c*-axis showing the C–H··· $\pi$  and  $\pi$ – $\pi$  interactions as dashed lines.



**Figure 6**  
(*a*) Front and (*b*) back sides of the three-dimensional Hirshfeld surface of the title compound mapped over  $d_{\text{norm}}$ , with a fixed colour scale of  $-0.6778$  to  $+1.5015$  a.u.


**Figure 7**

The two-dimensional fingerprint plots of the title compound, showing (a) all interactions, and delineated into (b) H...H, (c) C...H/H...C and (d) O...H/H...O interactions. [ $d_e$  and  $d_i$  represent the distances from a point on the Hirshfeld surface to the nearest atoms outside (external) and inside (internal) the surface, respectively].

are C...H/H...C (23.2%, Fig. 7c) and O...H/H...O (20.0%, Fig. 7d) interactions. Other smaller contributions are made by C...C (3.4%), N...H/H...N (3.3%), C...N/N...C (0.4%) and C...O/O...C (0.3%) interactions.

#### 4. Database survey

A search of the Cambridge Structural Database (CSD, Version 5.43, last update November 2022; Groom *et al.*, 2016) for structures containing the fragment N—C—CH=C—C(=O)—NH, in which the N—C bond is part of a five-membered ring and the CH=C bond is acyclic, resulted in one hit, *N*-[(1,1-dimethylethoxy)carbonyl]-L-alanyl-[(2*Z*)-3-(pyrrolidin-2-yl)-2-methyl-2-propenoyl]-L-alanine methylamide dichloromethane solvate hydrate (CSD refcode SEFCUC; Grison *et al.*, 2005).

In the crystal of SEFCUC, molecules are connected by N—H...O and C—H...O hydrogen bonds, forming molecular layers parallel to the (001) plane. These layers are connected to each other by van der Waals forces. Torsion angles at the central C—C=C—C(=O)—NH unit in SEFCUC, *i.e.* the torsion angles C9—C13—C14—C16 and C13—C14—C16—N3 are  $-3.1$  (5) and  $-53.1$  (4)°, respectively. SEFCUC shows a folded conformation due to an intramolecular N—H...O hydrogen bond. The amide group is *trans*-planar, as in the title compound.

**Table 3**

Experimental details.

Crystal data	
Chemical formula	C <sub>15</sub> H <sub>14</sub> N <sub>2</sub> O <sub>2</sub> ·H <sub>2</sub> O
$M_r$	272.30
Crystal system, space group	Monoclinic, <i>I</i> 2/a
Temperature (K)	294
$a$ , $b$ , $c$ (Å)	13.7420 (13), 8.8912 (13), 23.114 (2)
$\beta$ (°)	94.742 (4)
$V$ (Å <sup>3</sup> )	2814.5 (6)
$Z$	8
Radiation type	Mo $K\alpha$
$\mu$ (mm <sup>-1</sup> )	0.09
Crystal size (mm)	0.29 × 0.24 × 0.21
Data collection	
Diffractometer	Bruker APEXII CCD
No. of measured, independent and observed [ $I > 2\sigma(I)$ ] reflections	98978, 2677, 1729
$R_{int}$	0.205
( $\sin \theta/\lambda$ ) <sub>max</sub> (Å <sup>-1</sup> )	0.611
Refinement	
$R[F^2 > 2\sigma(F^2)]$ , $wR(F^2)$ , $S$	0.041, 0.109, 1.05
No. of reflections	2677
No. of parameters	194
H-atom treatment	H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ , $\Delta\rho_{min}$ (e Å <sup>-3</sup> )	0.14, $-0.18$

Computer programs: APEX2 and SAINT (Bruker, 2018), SHELXT2014/5 (Sheldrick, 2015a), SHELXL2018/3 (Sheldrick, 2015b), ORTEP-3 for Windows (Farrugia, 2012) and PLATON (Spek, 2020).

#### 5. Synthesis and crystallization

To a solution of pyrrole-2-carboxaldehyde (1 g, 10 mmol) and acetoacetanilide (1.77 g, 10 mmol) in ethanol (80%, 20 mL), were added methylpiperazine (3–4 drops) and the mixture was stirred at room temperature for 2 h. The reaction mixture was then left overnight. The precipitated crystals were separated by filtration and recrystallized from an ethanol/water (1:1) solution (yield 69%; m.p. 513–514 K).

<sup>1</sup>H NMR (300 MHz, DMSO- $d_6$ ,  $\delta$ ): 2.34 (s, 3H, CH<sub>3</sub>), 6.21 (*d*, 1H, CH<sub>pyr.</sub>), 6.57 (1H, *d*, CH<sub>pyr.</sub>), 7.10 (*t*, 1H, CH<sub>pyr.</sub>), 7.14 (*t*, 1H, CH<sub>arom.</sub>), 7.35 (*m*, 2H; 2CH<sub>arom.</sub>), 7.57 (*s*, 1H, CH=), 7.70 (*d*, 2H, 2CH<sub>arom.</sub>), 10.41 (*s*, 1H, NH), 11.52 (*s*, 1H, NH). <sup>13</sup>C NMR (75 MHz, DMSO- $d_6$ ,  $\delta$ ): 26.45 (CH<sub>3</sub>), 112.12 (CH<sub>pyr.</sub>), 114.66 (CH<sub>pyr.</sub>), 119.74 (2CH<sub>arom.</sub>), 124.08 (CH<sub>pyr.</sub>), 126.70 (CH<sub>arom.</sub>), 129.37 (2CH<sub>arom.</sub>), 130.66 (C<sub>pyr.</sub>), 136.83 (CH=), 139.58 (C<sub>quat.</sub>), 139.70 (C<sub>quat.</sub>), 166.74 (C=O), 195.29 (C=O).

#### 6. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 3. The hydrogen atoms of the water molecule and the hydrogen atoms bound to nitrogen were located in difference-Fourier maps and refined with fixed positional thermal displacement parameters and with  $U_{iso}(H) = 1.2U_{eq}(N)$  or  $1.5U_{eq}(O)$ . All carbon-bound hydrogen atoms were positioned geometrically (C—H = 0.93–0.96 Å) and were included in the refinement in the riding-model approximation with  $U_{iso}(H) = 1.2$  or  $1.5U_{eq}(C)$ . One reflection (0 1 1),

affected by the beam stop, was omitted in the final cycles of refinement. Owing to poor agreement between observed and calculated intensities, fourteen outliers ( $\bar{1}5\ 1\ 10$ ,  $\bar{2}\ 3\ 15$ ,  $8\ 4\ 16$ ,  $16\ 0\ 0$ ,  $\bar{7}\ 3\ 18$ ,  $0\ 5\ 23$ ,  $2\ 3\ 7$ ,  $\bar{1}\ 2\ 21$ ,  $\bar{7}\ 7\ 18$ ,  $\bar{6}\ 6\ 8$ ,  $1\ 5\ 18$ ,  $\bar{1}\ 3\ 18$ ,  $0\ 2\ 14$ ,  $5\ 5\ 20$ ) were omitted during the final refinement cycle. The value of  $R(\text{int})$  should normally be considerably lower than 0.10. The value of  $R(\text{int})$  of 0.205 in this study may be high due to poor crystal quality.

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Authors' contributions are as follows. Conceptualization, ASS, ANK and FNN; methodology, ASS, ANK and MA; investigation, ASS and IB; writing (original draft), MA and AB; writing (review and editing of the manuscript), MA and ASS; visualization, MA and IB; funding acquisition, ASS, AB and IB; resources, AB, IB and MA; supervision, MA and IGM.

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## supporting information

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## Crystal structure and Hirshfeld surface analysis of (2Z)-3-oxo-N-phenyl-2-[(1H-pyrrol-2-yl)methylidene]butanamide monohydrate

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### Computing details

#### (2Z)-3-Oxo-N-phenyl-2-[(1H-pyrrol-2-yl)methylidene]butanamide monohydrate

##### Crystal data

$C_{15}H_{14}N_2O_2 \cdot H_2O$

$M_r = 272.30$

Monoclinic,  $I2/a$

$a = 13.7420$  (13) Å

$b = 8.8912$  (13) Å

$c = 23.114$  (2) Å

$\beta = 94.742$  (4)°

$V = 2814.5$  (6) Å<sup>3</sup>

$Z = 8$

$F(000) = 1152$

$D_x = 1.285$  Mg m<sup>-3</sup>

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

Cell parameters from 7717 reflections

$\theta = 3.2$ – $21.8$ °

$\mu = 0.09$  mm<sup>-1</sup>

$T = 294$  K

Prism, colourless

$0.29 \times 0.24 \times 0.21$  mm

##### Data collection

Bruker APEXII CCD

diffractometer

$\varphi$  and  $\omega$  scans

98978 measured reflections

2677 independent reflections

1729 reflections with  $I > 2\sigma(I)$

$R_{int} = 0.205$

$\theta_{max} = 25.7$ °,  $\theta_{min} = 2.7$ °

$h = -16$ → $16$

$k = -10$ → $10$

$l = -28$ → $28$

##### Refinement

Refinement on  $F^2$

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.041$

$wR(F^2) = 0.109$

$S = 1.05$

2677 reflections

194 parameters

0 restraints

Hydrogen site location: mixed

H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0357P)^2 + 2.2064P]$

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

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

$\Delta\rho_{max} = 0.14$  e Å<sup>-3</sup>

$\Delta\rho_{min} = -0.18$  e Å<sup>-3</sup>

##### Special details

**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.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters ( $\text{\AA}^2$ )

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
C1	0.45829 (16)	0.6822 (3)	0.43032 (9)	0.0540 (6)
H1	0.408073	0.670947	0.401110	0.065*
C2	0.4445 (2)	0.7687 (3)	0.47832 (10)	0.0710 (7)
H2	0.384992	0.816610	0.481260	0.085*
C3	0.5178 (2)	0.7850 (3)	0.52188 (11)	0.0699 (7)
H3	0.508515	0.845175	0.553880	0.084*
C4	0.60437 (19)	0.7124 (3)	0.51790 (10)	0.0669 (7)
H4	0.653634	0.721325	0.547795	0.080*
C5	0.61958 (16)	0.6259 (3)	0.47001 (9)	0.0532 (6)
H5	0.678910	0.576957	0.467656	0.064*
C6	0.54696 (14)	0.6122 (2)	0.42572 (8)	0.0388 (5)
C7	0.64024 (14)	0.5036 (2)	0.34901 (8)	0.0372 (4)
C8	0.62601 (13)	0.4259 (2)	0.29110 (8)	0.0361 (4)
C9	0.63778 (13)	0.2768 (2)	0.28452 (8)	0.0398 (5)
H9	0.628673	0.241390	0.246603	0.048*
C10	0.66233 (14)	0.1657 (2)	0.32780 (8)	0.0415 (5)
C11	0.68283 (17)	0.1689 (3)	0.38755 (9)	0.0547 (6)
H11	0.686480	0.254312	0.410856	0.066*
C12	0.69700 (18)	0.0209 (3)	0.40637 (10)	0.0614 (6)
H12	0.711383	-0.010172	0.444544	0.074*
C13	0.68602 (17)	-0.0701 (3)	0.35893 (10)	0.0571 (6)
H13	0.691324	-0.174348	0.359127	0.069*
C14	0.60180 (14)	0.5269 (2)	0.24196 (8)	0.0445 (5)
C15	0.57908 (17)	0.4655 (3)	0.18195 (9)	0.0588 (6)
H15A	0.556811	0.545433	0.156270	0.088*
H15B	0.529029	0.390312	0.182627	0.088*
H15C	0.636866	0.421282	0.168541	0.088*
N1	0.55800 (12)	0.52714 (18)	0.37468 (7)	0.0397 (4)
HN1	0.5032 (16)	0.497 (2)	0.3534 (9)	0.048*
N2	0.66617 (13)	0.01667 (19)	0.31163 (8)	0.0472 (4)
HN2	0.6547 (16)	-0.017 (2)	0.2755 (10)	0.057*
O1	0.72199 (10)	0.54315 (16)	0.36963 (6)	0.0486 (4)
O2	0.60026 (13)	0.66292 (18)	0.25062 (6)	0.0653 (5)
OW1	0.88137 (11)	0.57116 (18)	0.30826 (6)	0.0496 (4)
HW1	0.8260 (19)	0.564 (3)	0.3282 (10)	0.074*
HW2	0.8891 (18)	0.664 (3)	0.2981 (11)	0.074*

Atomic displacement parameters ( $\text{\AA}^2$ )

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
C1	0.0510 (13)	0.0643 (14)	0.0468 (12)	0.0125 (11)	0.0048 (10)	-0.0042 (11)
C2	0.0739 (17)	0.0797 (18)	0.0607 (16)	0.0279 (14)	0.0127 (13)	-0.0136 (14)
C3	0.0875 (19)	0.0703 (17)	0.0534 (14)	0.0020 (14)	0.0149 (13)	-0.0227 (13)
C4	0.0690 (16)	0.0836 (19)	0.0474 (13)	-0.0054 (14)	0.0004 (11)	-0.0176 (13)
C5	0.0487 (12)	0.0657 (15)	0.0445 (12)	0.0039 (11)	0.0004 (10)	-0.0096 (11)

C6	0.0448 (11)	0.0374 (11)	0.0347 (10)	-0.0020 (9)	0.0056 (8)	-0.0001 (8)
C7	0.0404 (11)	0.0299 (10)	0.0414 (11)	0.0011 (8)	0.0053 (8)	0.0011 (8)
C8	0.0360 (10)	0.0380 (11)	0.0348 (10)	-0.0003 (8)	0.0054 (8)	-0.0022 (8)
C9	0.0381 (10)	0.0442 (12)	0.0376 (11)	-0.0006 (9)	0.0058 (8)	-0.0054 (9)
C10	0.0440 (11)	0.0355 (11)	0.0460 (12)	0.0030 (9)	0.0086 (9)	-0.0039 (9)
C11	0.0725 (15)	0.0457 (13)	0.0462 (13)	0.0066 (11)	0.0074 (11)	-0.0015 (10)
C12	0.0794 (17)	0.0542 (15)	0.0508 (13)	0.0089 (12)	0.0066 (12)	0.0085 (12)
C13	0.0652 (15)	0.0375 (12)	0.0696 (16)	0.0041 (11)	0.0108 (12)	0.0073 (12)
C14	0.0416 (11)	0.0487 (14)	0.0439 (12)	0.0040 (9)	0.0071 (9)	0.0034 (10)
C15	0.0593 (14)	0.0750 (16)	0.0414 (12)	0.0077 (12)	0.0007 (10)	0.0015 (11)
N1	0.0361 (9)	0.0454 (10)	0.0376 (9)	-0.0012 (7)	0.0030 (7)	-0.0074 (8)
N2	0.0526 (10)	0.0371 (10)	0.0526 (11)	0.0007 (8)	0.0076 (8)	-0.0068 (9)
O1	0.0381 (8)	0.0556 (9)	0.0523 (8)	-0.0057 (7)	0.0049 (6)	-0.0094 (7)
O2	0.0966 (13)	0.0433 (10)	0.0563 (10)	0.0071 (8)	0.0070 (8)	0.0089 (8)
OW1	0.0450 (8)	0.0511 (9)	0.0533 (9)	0.0040 (7)	0.0078 (7)	0.0091 (7)

*Geometric parameters (Å, °)*

C1—C2	1.376 (3)	C9—H9	0.9300
C1—C6	1.380 (3)	C10—N2	1.379 (3)
C1—H1	0.9300	C10—C11	1.387 (3)
C2—C3	1.371 (4)	C11—C12	1.395 (3)
C2—H2	0.9300	C11—H11	0.9300
C3—C4	1.363 (4)	C12—C13	1.361 (3)
C3—H3	0.9300	C12—H12	0.9300
C4—C5	1.378 (3)	C13—N2	1.347 (3)
C4—H4	0.9300	C13—H13	0.9300
C5—C6	1.375 (3)	C14—O2	1.226 (2)
C5—H5	0.9300	C14—C15	1.500 (3)
C6—N1	1.420 (2)	C15—H15A	0.9600
C7—O1	1.235 (2)	C15—H15B	0.9600
C7—N1	1.336 (2)	C15—H15C	0.9600
C7—C8	1.505 (3)	N1—HN1	0.91 (2)
C8—C9	1.346 (3)	N2—HN2	0.89 (2)
C8—C14	1.465 (3)	OW1—HW1	0.92 (3)
C9—C10	1.427 (3)	OW1—HW2	0.87 (3)
C2—C1—C6	119.7 (2)	N2—C10—C9	119.18 (18)
C2—C1—H1	120.2	C11—C10—C9	134.50 (19)
C6—C1—H1	120.2	C10—C11—C12	107.6 (2)
C3—C2—C1	120.7 (2)	C10—C11—H11	126.2
C3—C2—H2	119.7	C12—C11—H11	126.2
C1—C2—H2	119.7	C13—C12—C11	107.8 (2)
C4—C3—C2	119.5 (2)	C13—C12—H12	126.1
C4—C3—H3	120.3	C11—C12—H12	126.1
C2—C3—H3	120.3	N2—C13—C12	108.4 (2)
C3—C4—C5	120.6 (2)	N2—C13—H13	125.8
C3—C4—H4	119.7	C12—C13—H13	125.8



C5—C4—H4	119.7	O2—C14—C8	118.96 (18)
C6—C5—C4	119.9 (2)	O2—C14—C15	120.36 (19)
C6—C5—H5	120.0	C8—C14—C15	120.68 (19)
C4—C5—H5	120.0	C14—C15—H15A	109.5
C5—C6—C1	119.57 (18)	C14—C15—H15B	109.5
C5—C6—N1	123.05 (18)	H15A—C15—H15B	109.5
C1—C6—N1	117.37 (17)	C14—C15—H15C	109.5
O1—C7—N1	124.06 (18)	H15A—C15—H15C	109.5
O1—C7—C8	121.43 (16)	H15B—C15—H15C	109.5
N1—C7—C8	114.50 (16)	C7—N1—C6	127.24 (17)
C9—C8—C14	122.57 (18)	C7—N1—HN1	114.1 (13)
C9—C8—C7	123.00 (17)	C6—N1—HN1	117.9 (13)
C14—C8—C7	114.39 (16)	C13—N2—C10	109.86 (18)
C8—C9—C10	128.81 (18)	C13—N2—HN2	125.1 (15)
C8—C9—H9	115.6	C10—N2—HN2	125.0 (15)
C10—C9—H9	115.6	HW1—OW1—HW2	109 (2)
N2—C10—C11	106.30 (18)		
C6—C1—C2—C3	-0.6 (4)	N2—C10—C11—C12	-1.0 (2)
C1—C2—C3—C4	-1.2 (4)	C9—C10—C11—C12	177.1 (2)
C2—C3—C4—C5	1.5 (4)	C10—C11—C12—C13	0.4 (3)
C3—C4—C5—C6	-0.1 (4)	C11—C12—C13—N2	0.3 (3)
C4—C5—C6—C1	-1.6 (3)	C9—C8—C14—O2	-173.92 (19)
C4—C5—C6—N1	178.9 (2)	C7—C8—C14—O2	3.9 (3)
C2—C1—C6—C5	2.0 (3)	C9—C8—C14—C15	6.2 (3)
C2—C1—C6—N1	-178.5 (2)	C7—C8—C14—C15	-176.03 (18)
O1—C7—C8—C9	84.1 (2)	O1—C7—N1—C6	6.7 (3)
N1—C7—C8—C9	-97.0 (2)	C8—C7—N1—C6	-172.19 (17)
O1—C7—C8—C14	-93.7 (2)	C5—C6—N1—C7	-30.7 (3)
N1—C7—C8—C14	85.2 (2)	C1—C6—N1—C7	149.9 (2)
C14—C8—C9—C10	179.17 (18)	C12—C13—N2—C10	-1.0 (3)
C7—C8—C9—C10	1.5 (3)	C11—C10—N2—C13	1.2 (2)
C8—C9—C10—N2	176.92 (19)	C9—C10—N2—C13	-177.18 (17)
C8—C9—C10—C11	-1.0 (4)		

### Hydrogen-bond geometry (Å, °)

Cg1 is the centroid of the N2/C10—C13 pyrrole ring.

<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>
C5—H5...O1	0.93	2.41	2.906 (3)	113
C13—H13...O1 <sup>i</sup>	0.93	2.56	3.480 (3)	173
N1—HN1...OW1 <sup>ii</sup>	0.91 (2)	1.99 (2)	2.898 (2)	179 (2)
N2—HN2...OW1 <sup>iii</sup>	0.89 (2)	2.02 (2)	2.901 (2)	173 (2)
OW1—HW1...O1	0.92 (3)	1.80 (3)	2.718 (2)	177 (2)
OW1—HW2...O2 <sup>iv</sup>	0.87 (3)	1.92 (3)	2.750 (2)	160 (2)
C15—H15C...Cg1 <sup>iii</sup>	0.96	2.66	3.536 (3)	151

Symmetry codes: (i)  $x, y-1, z$ ; (ii)  $x-1/2, -y+1, z$ ; (iii)  $-x+3/2, -y+1/2, -z+1/2$ ; (iv)  $-x+3/2, -y+3/2, -z+1/2$ .