



Crystal structure and Hirshfeld surface analysis of 2-amino-4-(4-methoxyphenyl)-6-oxo-1-phenyl-1,4,5,6-tetrahydropyridine-3-carbonitrile

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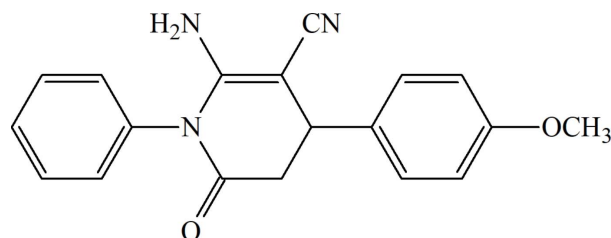
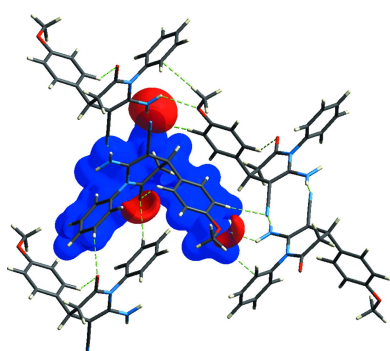
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The central tetrahydropyridine ring of the title compound, C₁₉H₁₇N₃O₂, adopts a screw-boat conformation. In the crystal, strong C—H···O and N—H···N hydrogen bonds form dimers with $R_2^2(14)$ and $R_2^2(12)$ ring motifs, respectively, between consecutive molecules along the *c*-axis direction. Intermolecular N—H···O and C—H···O hydrogen bonds connect these dimers, forming a three-dimensional network. C—H··· π interactions and π – π stacking interactions contribute to the stabilization of the molecular packing. A Hirshfeld surface analysis indicates that the contributions from the most prevalent interactions are H···H (47.1%), C···H/H···C (20.9%), O···H/H···O (15.3%) and N···H/H···N (11.4%).

1. Chemical context

Carbon–carbon and carbon–nitrogen bond-forming reactions represent an important synthetic class in organic chemistry (Yadigarov *et al.*, 2009; Abdelhamid *et al.*, 2011; Yin *et al.*, 2020; Khalilov *et al.*, 2021). Notably, pyridine derivatives are widely applied in the discovery of biologically active molecules and multifunctional materials (Magerramov *et al.*, 2018; Sherman & Murugan, 2015; Mamedov *et al.*, 2020). On the other hand, the tetrahydropyridine moiety is an essential part of diverse biologically active compounds, food additives and natural products (Mateeva *et al.*, 2005).



In the framework of ongoing structural studies (Safavora *et al.*, 2019; Naghiyev *et al.*, 2020; 2021a,b; Maharramov *et al.*, 2021), we report here the crystal structure and Hirshfeld surface analysis of the title compound, 2-amino-4-(4-methoxyphenyl)-6-oxo-1-phenyl-1,4,5,6-tetrahydropyridine-3-carbonitrile.



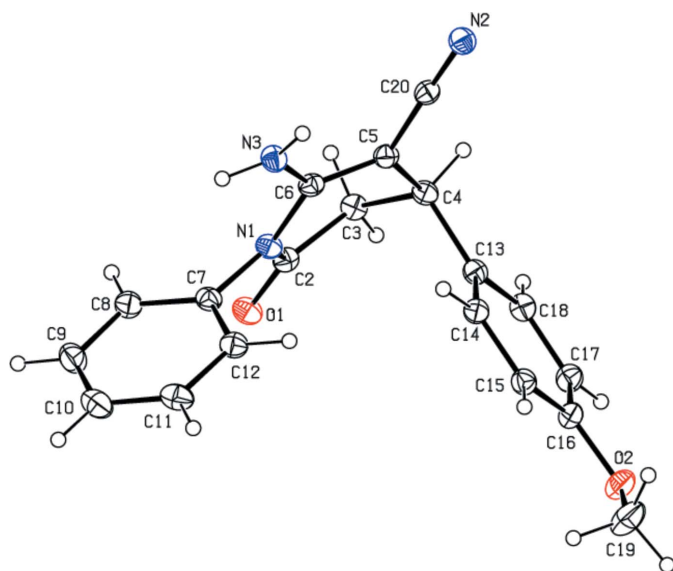


Figure 1
The molecular structure of the title compound with displacement ellipsoids drawn at the 30% probability level.

2. Structural commentary

The title compound (Fig. 1) crystallizes in the monoclinic space group $P2_1/n$ with $Z = 4$. The central N1/C2–C6 tetrahydropyridine ring of the molecule adopts a screw-boat conformation with puckering parameters (Cremer & Pople, 1975) $Q_T = 0.503$ (2) Å, $\theta = 66.1$ (2)°, $\varphi = 153.3$ (2)°. The C7–C12 phenyl ring, which is attached to N1, is in an equatorial position and makes a dihedral angle of 54.43 (9)° with the mean plane of the tetrahydropyridine ring. The C13–C18 methoxyphenyl ring, which is attached to C4, is in an axial position. The dihedral angle between the C7–C12 phenyl and C13–C18 methoxyphenyl rings is 68.61 (10)°.

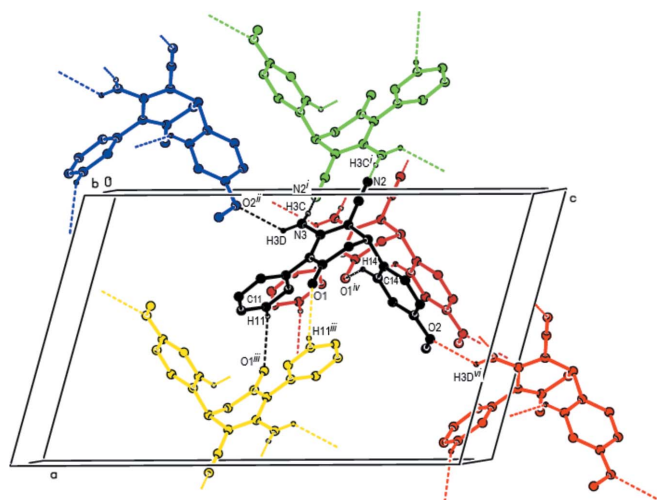


Figure 2
A general view of the N–H...N, N–H...O and C–H...O hydrogen bonds in the crystal packing of the title compound [symmetry codes: (i) $-x, -y, -z + 1$; (ii) $x - \frac{1}{2}, -y + \frac{1}{2}, z - \frac{1}{2}$; (iii) $-x + 1, -y + 1, -z + 1$; (iv) $x, y - 1, z$; (v) $\frac{1}{2} + x, \frac{1}{2} - y, \frac{1}{2} + z$].

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

Cg3 is the centroid of the C13–C18 benzene ring of the methoxyphenyl group.

$D-H \cdots A$	$D-H$	$H \cdots A$	$D \cdots A$	$D-H \cdots A$
N3–H3C...N2 ⁱ	0.91 (2)	2.10 (2)	2.996 (2)	166 (2)
N3–H3D...O2 ⁱⁱ	0.91 (2)	2.48 (2)	3.152 (2)	131.0 (19)
C11–H11...O1 ⁱⁱⁱ	0.95	2.55	3.210 (3)	127
C14–H14...O1 ^{iv}	0.95	2.48	3.199 (2)	133
C10–H10...Cg3 ⁱⁱⁱ	0.95	2.99	3.813 (3)	146
C18–H18...Cg3 ^v	0.95	2.87	3.716 (2)	150

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

3. Supramolecular features

As shown in Fig. 2, strong intermolecular C11–H11...O1 and N3–H3C...N2 hydrogen bonds (Table 1) form dimers with $R_2^2(14)$ and $R_2^2(12)$ ring motifs (Bernstein *et al.*, 1995), respectively, between adjacent molecules along the c -axis direction. These dimers are connected by N3–H3D...O2 and C14–H14...O1 hydrogen bonds, forming a three-dimensional network (Table 1; Fig. 3). Furthermore, C–H... π [C10–H10...Cg3ⁱⁱⁱ and C18–H18...Cg3^v; symmetry codes: (iii) $-x + 1, -y + 1, -z + 1$; (v) $-x + \frac{1}{2}, y + \frac{1}{2}, -z + \frac{3}{2}$; Cg3 is the centroid of the C13–C18 methoxyphenyl ring; Table 1] and π – π stacking interactions [$Cg2 \cdots Cg2^{iii} = 3.8918$ (15) Å and slippage = 1.551 Å; Cg2 is the centroid of the C7–C12 phenyl ring] contribute to the stabilization of the molecular packing (Figs. 4 and 5).

4. Hirshfeld surface analysis

The Hirshfeld surface analysis was performed and the associated two dimensional fingerprint plots generated using

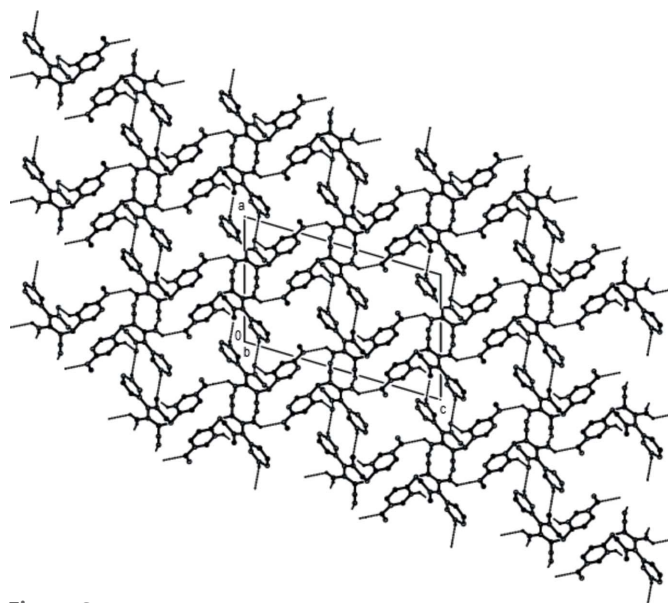


Figure 3
The crystal packing of the title compound, viewed along the b axis, showing the N–H...N, N–H...O and C–H...O hydrogen bonds as dashed lines.

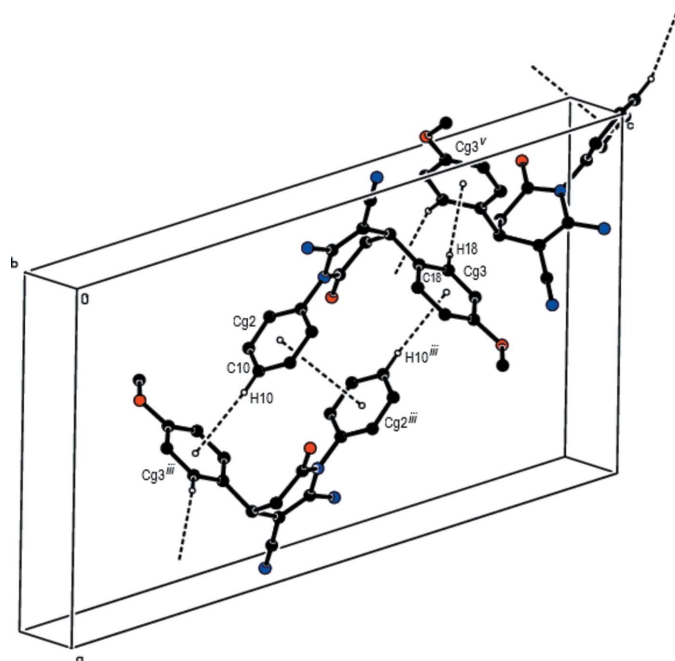


Figure 4
A general view of the C–H··· π interactions and π – π stacking interactions in the crystal packing of the title compound [symmetry codes: (iii) $-x + 1, -y + 1, -z + 1$; (v) $-x + \frac{1}{2}, y + \frac{1}{2}, -z + \frac{3}{2}$].

Crystal Explorer 17 (Turner *et al.*, 2017). The Hirshfeld surface was calculated using a standard (high) surface resolution with the three-dimensional d_{norm} surface plotted over a fixed colour scale mapped over the range -0.4835 (red) to 1.8469 (blue) a.u. The d_{norm} mapping indicates that strong hydrogen-bonding interactions, such as N–H···N, N–H···O and C–H···O hydrogen bonds (Tables 1 and 2), appear to be the

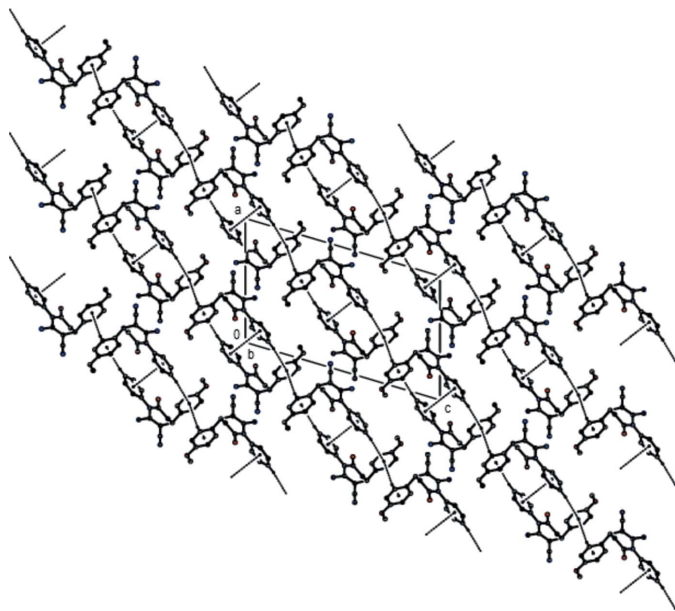


Figure 5
The crystal packing of the title compound, viewed along the b axis, showing the C–H··· π interactions and π – π stacking interactions as dashed lines.

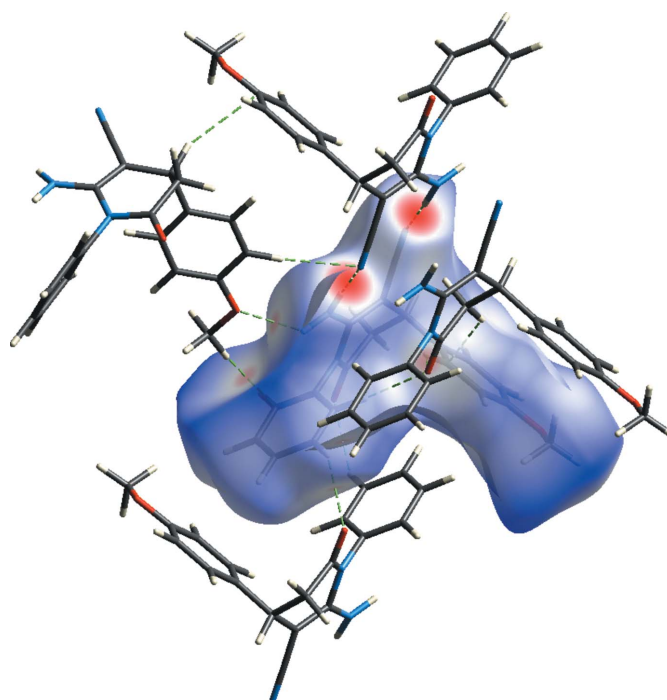


Figure 6
Hirshfeld surface mapped over d_{norm} showing the N–H···N, N–H···O and C–H···O intermolecular contacts.

primary interactions in the structure, seen as a bright-red area in the Hirshfeld surface (Fig. 6).

The Hirshfeld surface mapped over electrostatic potential (Spackman *et al.*, 2008) is shown in Fig. 7. The blue regions indicate positive electrostatic potential (hydrogen-bond

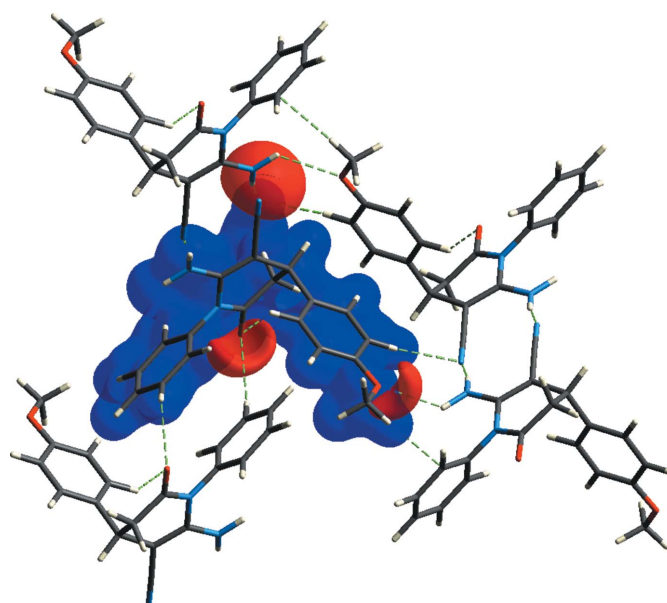


Figure 7
View of the three-dimensional Hirshfeld surface of the title compound, showing the hydrogen-bonding interactions, plotted over electrostatic potential energy in the range -0.0500 to 0.0500 a.u. using the STO-3 G basis set at the Hartree–Fock level of theory. Hydrogen-bond donors and acceptors are shown as blue and red regions, respectively, around the atoms, corresponding to positive and negative potentials.

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

Contact	Distance	Symmetry operation
O1...H14	2.48	$x, 1 + y, z$
H11...O1	2.55	$1 - x, 1 - y, 1 - z$
N2...H17	2.70	$\frac{1}{2} - x, -\frac{1}{2} + y, \frac{3}{2} - z$
O2...H3D	2.48 (2)	$\frac{1}{2} + x, \frac{1}{2} - y, \frac{1}{2} + z$
H3C...N2	2.10 (3)	$-x, -y, 1 - z$
C20...C6	3.318 (3)	$-x, 1 - y, 1 - z$

donors), while the red regions indicate negative electrostatic potential (hydrogen-bond acceptors).

The two-dimensional fingerprint plots are illustrated in Fig. 8. H...H contacts comprise 47.1% of the total interactions (Fig. 8*b*), followed by C...H/H...C (Fig. 8*c*; 20.9%), O...H/H...O (Fig. 8*d*; 15.3%) and N...H/H...N (Fig. 8*e*; 11.4%).

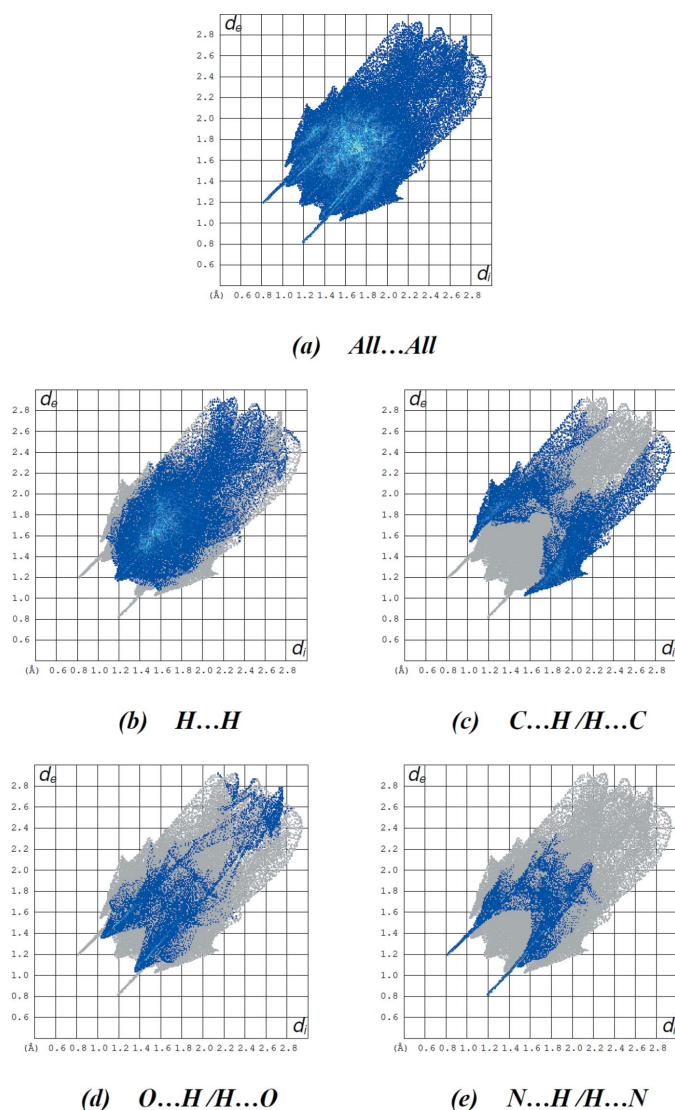


Figure 8
(a) The full two-dimensional fingerprint plot for the title compound and those delineated into (b) H...H (47.1%), (c) C...H/H...C (20.9%), (d) O...H/H...O (15.3%) and (e) N...H/H...N (11.4%) contacts.

The percentage contributions of the C...C, C...N/N...C and N...N contacts are negligible, at 3.1, 1.4 and 0.8%, respectively. The predominance of H...H, C...H/H...C, O...H/H...O and N...H/H...N contacts indicate that van der Waals interactions and hydrogen bonding play the major roles in the crystal packing (Hathwar *et al.*, 2015).

5. Database survey

A search of the Cambridge Structural Database (CSD, version 5.42, update of September 2021; Groom *et al.*, 2016) found four compounds with the 6-oxo-1-phenyl-1,4,5,6-tetrahydropyridine unit that are similar to the title compound, *viz.* 5-acetyl-2-amino-4-(4-bromophenyl)-6-oxo-1-phenyl-1,4,5,6-tetrahydropyridine-3-carbonitrile (**I**) (YAXQAT; Mamedov *et al.*, 2022), 2-amino-4-(2,6-dichlorophenyl)-5-(1-hydroxyethylidene)-6-oxo-1-phenyl-1,4,5,6-tetrahydropyridine-3-carbonitrile (**II**) (OZAKOS, Naghiyev *et al.*, 2021*c*), methyl 6-oxo-4-phenyl-2-[(*Z*)-2-(pyridin-2-yl)ethenyl]-1,4,5,6-tetrahydropyridine-3-carboxylate (**III**) (PEDFEL, Smits *et al.*, 2012) and ethyl 5-ethoxymethylene-2-methyl-6-oxo-4-phenyl-1,4,5,6-tetrahydropyridine-3-carboxylate (**IV**) (VAGXAD, Novoa de Armas *et al.*, 2003).

Compound (**I**) crystallizes in the monoclinic space group *Pc* with $Z = 4$, and with two molecules, *A* and *B*, in the asymmetric unit. These molecules are stereoisomers with an *R,R* absolute configuration at C3 and C4 in molecule *A*, whereas the corresponding atoms in *B*, C23 and C24, have an *S* configuration. In both molecules, the conformation of the central dihydropyridine ring is close to screw-boat. The molecular conformation is stabilized by N—H...O hydrogen bonds, forming a dimer with an $R_2^2(16)$ ring motif. Both molecules of the dimers are connected by intermolecular N—H...O and N—H...N hydrogen bonds with an $R_3^3(14)$ ring motif into chains along the *c*-axis direction. Furthermore C—Br... π and C=O... π stacking interactions between these ribbons contribute to the stabilization of the molecular packing.

Compound (**II**) crystallizes in the monoclinic space group $P2_1/c$ with $Z = 4$ and the asymmetric unit comprises one molecule. The central tetrahydropyridine ring is almost planar with a maximum deviation of 0.074 (3) Å for C4. The phenyl and dichlorophenyl rings are at an angle of 21.28 (15)°. They form dihedral angles of 86.10 (15) and 87.17 (14)°, respectively, with the central tetrahydropyridine ring. A strong intramolecular O2—H2...O1 hydrogen bond stabilizes the molecular conformation of the molecule, creating an $S(6)$ ring motif. In the crystal, molecules are linked by intermolecular N—H...N and C—H...N hydrogen bonds, and N—H... π and C—H... π interactions, forming a three-dimensional network.

In molecule (**III**) (monoclinic space group $P2_1/c$, $Z = 4$), the *cis* configuration of the pyridinyl-vinyl fragment is stabilized by a strong intramolecular N—H...N hydrogen bond. The phenyl and pyridine rings are inclined to one another by 77.3 (1)°. In the crystal, inversion dimers are present *via* pairs of C—H...O hydrogen bonds and are further linked by C—H...O hydrogen bonds and C—H... π interactions.

For compound (**IV**) (monoclinic space group $C2/c$, $Z = 8$), the molecules form dimers by means of a pair of $N-H \cdots O$ hydrogen bonds. The 2(*1H*)-pyridone ring displays a screw-boat conformation.

6. Synthesis and crystallization

To a solution of 2-(4-methoxybenzylidene)malononitrile (0.94 g; 5.1 mmol) and acetoacetanilide (0.92 g; 5.2 mmol) in methanol (25 mL), 3–4 drops of piperidine were added and the mixture was stirred at 328–333 K for 10 min and was kept at room temperature for 48 h. Then 15 mL of methanol were removed from the reaction mixture, which was left overnight. The precipitated crystals were separated by filtration and recrystallized from ethanol/water (1:1) solution (yield 61%; m.p. 471–472 K).

1H NMR (300 MHz, DMSO- d_6 , ppm.): 2.80 (*dd*–*dd*, 1H, CH₂); 3.19 (*dd*–*dd*, 1H, CH₂); 3.82 (*s*, 3H, OCH₃); 3.93 (*t*, 1H, CH); 5.85 (*s*, 2H, NH₂); 7.15–7.58 (*m*, 9H, 2Ar–H). ^{13}C NMR (75 MHz, DMSO- d_6 , ppm.): 36.06 (CH–Ar), 40.42 (CH₂), 53.78 (OCH₃), 59.05 (C_{quat.}), 112.89 (2CH_{ar.}), 121.21 (CN), 128.61 (CH_{ar.}), 128.88 (2CH_{ar.}), 130.44 (2CH_{ar.}), 130.51 (2CH_{ar.}), 136.06 (C_{ar. quat.}), 137.02 (C_{ar. quat.}), 154.59 (C_{ar. quat.}), 155.18 (C_{quat.}), 168.82 (N–C=O).

7. Refinement details

Crystal data, data collection and structure refinement details are summarized in Table 3. H atoms bonded to nitrogen were located in a difference-Fourier map, and only their positional parameters were refined [$N3-H3C = 0.91$ (2) and $N3-H3D = 0.91$ (2) Å with $U_{iso}(H) = 1.2U_{eq}(N)$]. C-bound H atoms were positioned geometrically, with $C-H = 0.95$ – 1.00 Å, and were refined with $U_{iso}(H) = 1.2U_{eq}(C)$ or $1.5U_{eq}(C\text{-methyl})$.

Acknowledgements

Authors' contributions are as follows. Conceptualization, KAA and EZH; methodology, EZH and KAA; investigation, KAA, MA and EVD; writing (original draft), MA and KAA; writing (review and editing of the manuscript), MA and EZH; visualization, MA, EZH and KAA; funding acquisition, VNK, ATH and AAA; resources, AAA, VNK and KAA; supervision, KAA and MA.

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Table 3
Experimental details.

Crystal data	
Chemical formula	C ₁₉ H ₁₇ N ₃ O ₂
M_r	319.36
Crystal system, space group	Monoclinic, $P2_1/n$
Temperature (K)	100
a, b, c (Å)	12.910 (3), 6.3200 (13), 21.170 (4)
β (°)	106.48 (3)
V (Å ³)	1656.3 (7)
Z	4
Radiation type	Synchrotron, $\lambda = 0.80246$ Å
μ (mm ⁻¹)	0.11
Crystal size (mm)	0.40 × 0.15 × 0.07
Data collection	
Diffractometer	Rayonix SX165 CCD
Absorption correction	Multi-scan (<i>SCALA</i> ; Evans, 2006)
T_{min}, T_{max}	0.950, 0.985
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	26143, 3603, 3125
R_{int}	0.049
$(\sin \theta/\lambda)_{max}$ (Å ⁻¹)	0.643
Refinement	
$R[F^2 > 2\sigma(F^2)], wR(F^2), S$	0.054, 0.143, 1.05
No. of reflections	3603
No. of parameters	225
H-atom treatment	H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}, \Delta\rho_{min}$ (e Å ⁻³)	0.29, -0.27

Computer programs: *Marccd* (Doyle, 2011), *iMosflm* (Battye et al., 2011), *SHELXT* (Sheldrick, 2015a), *SHELXL* (Sheldrick, 2015b), *ORTEP-3 for Windows* (Farrugia, 2012) and *PLATON* (Spek, 2020).

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supporting information

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Crystal structure and Hirshfeld surface analysis of 2-amino-4-(4-methoxyphenyl)-6-oxo-1-phenyl-1,4,5,6-tetrahydropyridine-3-carbonitrile

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Computing details

Data collection: *Marccd* (Doyle, 2011); cell refinement: *iMosflm* (Battye *et al.*, 2011); data reduction: *iMosflm* (Battye *et al.*, 2011); program(s) used to solve structure: SHELXT (Sheldrick, 2015a); program(s) used to refine structure: *SHELXL* (Sheldrick, 2015b); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 2012); software used to prepare material for publication: *PLATON* (Spek, 2020).

2-Amino-4-(4-methoxyphenyl)-6-oxo-1-phenyl-1,4,5,6-tetrahydropyridine-3-carbonitrile

Crystal data

$C_{19}H_{17}N_3O_2$

$M_r = 319.36$

Monoclinic, $P2_1/n$

$a = 12.910$ (3) Å

$b = 6.3200$ (13) Å

$c = 21.170$ (4) Å

$\beta = 106.48$ (3)°

$V = 1656.3$ (7) Å³

$Z = 4$

$F(000) = 672$

$D_x = 1.281$ Mg m⁻³

Synchrotron radiation, $\lambda = 0.80246$ Å

Cell parameters from 600 reflections

$\theta = 2.4$ – 30.0 °

$\mu = 0.11$ mm⁻¹

$T = 100$ K

Prism, colourless

$0.40 \times 0.15 \times 0.07$ mm

Data collection

Rayonix SX165 CCD
diffractometer

/f scan

Absorption correction: multi-scan
(Scala;Evans, 2006)

$T_{\min} = 0.950$, $T_{\max} = 0.985$

26143 measured reflections

3603 independent reflections

3125 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.049$

$\theta_{\max} = 31.1$ °, $\theta_{\min} = 2.3$ °

$h = -16 \rightarrow 16$

$k = -8 \rightarrow 7$

$l = -27 \rightarrow 27$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.054$

$wR(F^2) = 0.143$

$S = 1.05$

3603 reflections

225 parameters

0 restraints

Primary atom site location: difference Fourier
map

Secondary atom site location: difference Fourier
map

Hydrogen site location: mixed

H atoms treated by a mixture of independent
and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.059P)^2 + 1.3785P]$

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

$(\Delta/\sigma)_{\max} < 0.001$
 $\Delta\rho_{\max} = 0.29 \text{ e } \text{\AA}^{-3}$
 $\Delta\rho_{\min} = -0.27 \text{ e } \text{\AA}^{-3}$

Extinction correction: SHELXL,
 $F_c^* = kF_c [1 + 0.001 \times F_c^2 \lambda^3 / \sin(2\theta)]^{-1/4}$
 Extinction coefficient: 0.033 (3)

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 (\AA^2)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
O1	0.33220 (11)	0.8608 (2)	0.55256 (7)	0.0332 (3)
O2	0.55206 (12)	0.1472 (3)	0.79862 (7)	0.0413 (4)
N1	0.24126 (11)	0.5628 (2)	0.50951 (7)	0.0269 (3)
N2	-0.03015 (13)	0.1368 (3)	0.56257 (8)	0.0321 (4)
N3	0.12078 (12)	0.3027 (3)	0.45307 (8)	0.0300 (4)
H3C	0.0857 (18)	0.177 (4)	0.4520 (11)	0.036*
H3D	0.1458 (19)	0.336 (4)	0.4183 (12)	0.036*
C2	0.26361 (14)	0.7307 (3)	0.55371 (9)	0.0272 (4)
C3	0.19790 (14)	0.7407 (3)	0.60221 (9)	0.0287 (4)
H3A	0.1284	0.8122	0.5814	0.034*
H3B	0.2372	0.8254	0.6409	0.034*
C4	0.17589 (14)	0.5196 (3)	0.62495 (9)	0.0277 (4)
H4	0.1221	0.5339	0.6506	0.033*
C5	0.12391 (13)	0.3942 (3)	0.56355 (9)	0.0268 (4)
C6	0.15916 (13)	0.4149 (3)	0.50888 (9)	0.0262 (4)
C7	0.30690 (13)	0.5355 (3)	0.46514 (9)	0.0279 (4)
C8	0.31570 (15)	0.6969 (3)	0.42287 (9)	0.0338 (4)
H8	0.2765	0.8249	0.4215	0.041*
C9	0.38256 (17)	0.6690 (4)	0.38261 (11)	0.0408 (5)
H9	0.3906	0.7800	0.3541	0.049*
C10	0.43779 (17)	0.4806 (4)	0.38357 (11)	0.0421 (5)
H10	0.4827	0.4624	0.3554	0.051*
C11	0.42768 (16)	0.3194 (4)	0.42533 (10)	0.0379 (5)
H11	0.4651	0.1898	0.4256	0.045*
C12	0.36267 (15)	0.3466 (3)	0.46698 (9)	0.0314 (4)
H12	0.3564	0.2370	0.4964	0.038*
C13	0.27801 (14)	0.4188 (3)	0.66992 (9)	0.0266 (4)
C14	0.32417 (14)	0.2403 (3)	0.65102 (9)	0.0274 (4)
H14	0.2927	0.1822	0.6085	0.033*
C15	0.41514 (15)	0.1438 (3)	0.69241 (9)	0.0298 (4)
H15	0.4443	0.0197	0.6787	0.036*
C16	0.46259 (15)	0.2306 (3)	0.75379 (9)	0.0316 (4)
C17	0.41891 (15)	0.4125 (3)	0.77340 (9)	0.0333 (4)
H17	0.4520	0.4735	0.8153	0.040*
C18	0.32761 (15)	0.5043 (3)	0.73205 (9)	0.0301 (4)
H18	0.2980	0.6273	0.7460	0.036*

C19	0.5911 (2)	-0.0507 (4)	0.78134 (12)	0.0535 (6)
H19A	0.6153	-0.0321	0.7418	0.080*
H19B	0.5329	-0.1558	0.7726	0.080*
H19C	0.6517	-0.0995	0.8178	0.080*
C20	0.03871 (13)	0.2533 (3)	0.56225 (9)	0.0268 (4)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0328 (7)	0.0278 (7)	0.0408 (7)	-0.0063 (5)	0.0132 (6)	-0.0045 (6)
O2	0.0378 (7)	0.0485 (9)	0.0319 (7)	0.0068 (6)	0.0005 (6)	0.0031 (6)
N1	0.0262 (7)	0.0245 (7)	0.0304 (7)	-0.0025 (6)	0.0086 (6)	-0.0026 (6)
N2	0.0318 (8)	0.0294 (8)	0.0348 (8)	-0.0028 (6)	0.0093 (6)	0.0009 (7)
N3	0.0311 (8)	0.0297 (8)	0.0288 (8)	-0.0072 (6)	0.0079 (6)	-0.0025 (6)
C2	0.0272 (8)	0.0218 (8)	0.0310 (9)	0.0010 (6)	0.0058 (7)	0.0001 (7)
C3	0.0300 (8)	0.0248 (8)	0.0319 (9)	0.0000 (7)	0.0096 (7)	-0.0022 (7)
C4	0.0267 (8)	0.0268 (9)	0.0305 (9)	-0.0005 (7)	0.0098 (7)	-0.0023 (7)
C5	0.0240 (8)	0.0255 (8)	0.0300 (9)	-0.0003 (6)	0.0060 (6)	0.0015 (7)
C6	0.0233 (7)	0.0228 (8)	0.0303 (8)	-0.0006 (6)	0.0040 (6)	0.0003 (7)
C7	0.0243 (8)	0.0310 (9)	0.0278 (8)	-0.0053 (7)	0.0064 (6)	-0.0040 (7)
C8	0.0319 (9)	0.0351 (10)	0.0331 (9)	-0.0054 (8)	0.0072 (7)	0.0005 (8)
C9	0.0395 (10)	0.0479 (12)	0.0366 (10)	-0.0109 (9)	0.0135 (8)	0.0021 (9)
C10	0.0368 (10)	0.0531 (13)	0.0404 (11)	-0.0106 (9)	0.0173 (9)	-0.0088 (10)
C11	0.0304 (9)	0.0418 (11)	0.0426 (11)	-0.0044 (8)	0.0122 (8)	-0.0118 (9)
C12	0.0296 (9)	0.0313 (9)	0.0329 (9)	-0.0019 (7)	0.0083 (7)	-0.0033 (8)
C13	0.0279 (8)	0.0239 (8)	0.0286 (8)	-0.0032 (6)	0.0089 (7)	-0.0008 (7)
C14	0.0276 (8)	0.0259 (8)	0.0278 (8)	-0.0031 (7)	0.0067 (7)	-0.0011 (7)
C15	0.0308 (8)	0.0283 (9)	0.0312 (9)	0.0005 (7)	0.0105 (7)	0.0018 (7)
C16	0.0300 (9)	0.0356 (10)	0.0273 (9)	-0.0003 (7)	0.0047 (7)	0.0052 (7)
C17	0.0347 (9)	0.0368 (10)	0.0269 (9)	-0.0047 (8)	0.0063 (7)	-0.0033 (8)
C18	0.0342 (9)	0.0280 (9)	0.0290 (9)	-0.0020 (7)	0.0106 (7)	-0.0030 (7)
C19	0.0529 (13)	0.0586 (15)	0.0408 (12)	0.0229 (12)	0.0002 (10)	0.0033 (11)
C20	0.0264 (8)	0.0245 (8)	0.0280 (8)	0.0024 (7)	0.0054 (6)	0.0005 (7)

Geometric parameters (Å, °)

O1—C2	1.214 (2)	C8—H8	0.9500
O2—C16	1.375 (2)	C9—C10	1.385 (3)
O2—C19	1.434 (3)	C9—H9	0.9500
N1—C2	1.390 (2)	C10—C11	1.379 (3)
N1—C6	1.410 (2)	C10—H10	0.9500
N1—C7	1.443 (2)	C11—C12	1.390 (3)
N2—C20	1.156 (2)	C11—H11	0.9500
N3—C6	1.346 (2)	C12—H12	0.9500
N3—H3C	0.91 (2)	C13—C14	1.387 (2)
N3—H3D	0.91 (2)	C13—C18	1.398 (3)
C2—C3	1.507 (2)	C14—C15	1.391 (3)
C3—C4	1.530 (2)	C14—H14	0.9500

C3—H3A	0.9900	C15—C16	1.383 (3)
C3—H3B	0.9900	C15—H15	0.9500
C4—C5	1.508 (2)	C16—C17	1.395 (3)
C4—C13	1.529 (2)	C17—C18	1.381 (3)
C4—H4	1.0000	C17—H17	0.9500
C5—C6	1.365 (2)	C18—H18	0.9500
C5—C20	1.410 (2)	C19—H19A	0.9800
C7—C8	1.383 (3)	C19—H19B	0.9800
C7—C12	1.389 (3)	C19—H19C	0.9800
C8—C9	1.386 (3)		
C16—O2—C19	116.44 (16)	C8—C9—H9	119.7
C2—N1—C6	121.62 (15)	C11—C10—C9	120.19 (19)
C2—N1—C7	118.78 (14)	C11—C10—H10	119.9
C6—N1—C7	119.55 (14)	C9—C10—H10	119.9
C6—N3—H3C	122.5 (15)	C10—C11—C12	119.9 (2)
C6—N3—H3D	117.7 (15)	C10—C11—H11	120.0
H3C—N3—H3D	118 (2)	C12—C11—H11	120.0
O1—C2—N1	121.14 (16)	C7—C12—C11	119.35 (19)
O1—C2—C3	122.71 (16)	C7—C12—H12	120.3
N1—C2—C3	116.16 (15)	C11—C12—H12	120.3
C2—C3—C4	111.53 (14)	C14—C13—C18	117.78 (17)
C2—C3—H3A	109.3	C14—C13—C4	121.64 (16)
C4—C3—H3A	109.3	C18—C13—C4	120.58 (16)
C2—C3—H3B	109.3	C13—C14—C15	122.01 (17)
C4—C3—H3B	109.3	C13—C14—H14	119.0
H3A—C3—H3B	108.0	C15—C14—H14	119.0
C5—C4—C13	114.33 (15)	C16—C15—C14	119.18 (17)
C5—C4—C3	106.59 (15)	C16—C15—H15	120.4
C13—C4—C3	111.84 (14)	C14—C15—H15	120.4
C5—C4—H4	108.0	O2—C16—C15	123.92 (18)
C13—C4—H4	108.0	O2—C16—C17	116.22 (17)
C3—C4—H4	108.0	C15—C16—C17	119.86 (17)
C6—C5—C20	119.33 (16)	C18—C17—C16	120.13 (17)
C6—C5—C4	120.46 (15)	C18—C17—H17	119.9
C20—C5—C4	120.21 (16)	C16—C17—H17	119.9
N3—C6—C5	124.45 (16)	C17—C18—C13	121.01 (18)
N3—C6—N1	116.48 (16)	C17—C18—H18	119.5
C5—C6—N1	119.07 (16)	C13—C18—H18	119.5
C8—C7—C12	121.01 (17)	O2—C19—H19A	109.5
C8—C7—N1	120.29 (17)	O2—C19—H19B	109.5
C12—C7—N1	118.68 (16)	H19A—C19—H19B	109.5
C7—C8—C9	118.95 (19)	O2—C19—H19C	109.5
C7—C8—H8	120.5	H19A—C19—H19C	109.5
C9—C8—H8	120.5	H19B—C19—H19C	109.5
C10—C9—C8	120.5 (2)	N2—C20—C5	178.6 (2)
C10—C9—H9	119.7		

C6—N1—C2—O1	-177.75 (16)	C12—C7—C8—C9	-0.9 (3)
C7—N1—C2—O1	4.8 (3)	N1—C7—C8—C9	177.59 (17)
C6—N1—C2—C3	2.1 (2)	C7—C8—C9—C10	1.5 (3)
C7—N1—C2—C3	-175.40 (15)	C8—C9—C10—C11	-0.8 (3)
O1—C2—C3—C4	-143.36 (18)	C9—C10—C11—C12	-0.6 (3)
N1—C2—C3—C4	36.8 (2)	C8—C7—C12—C11	-0.4 (3)
C2—C3—C4—C5	-54.71 (18)	N1—C7—C12—C11	-178.91 (16)
C2—C3—C4—C13	70.92 (19)	C10—C11—C12—C7	1.1 (3)
C13—C4—C5—C6	-84.4 (2)	C5—C4—C13—C14	7.1 (2)
C3—C4—C5—C6	39.7 (2)	C3—C4—C13—C14	-114.15 (18)
C13—C4—C5—C20	95.23 (19)	C5—C4—C13—C18	-172.53 (16)
C3—C4—C5—C20	-140.67 (16)	C3—C4—C13—C18	66.2 (2)
C20—C5—C6—N3	-2.7 (3)	C18—C13—C14—C15	1.7 (3)
C4—C5—C6—N3	176.95 (17)	C4—C13—C14—C15	-177.99 (16)
C20—C5—C6—N1	177.30 (15)	C13—C14—C15—C16	-1.4 (3)
C4—C5—C6—N1	-3.1 (2)	C19—O2—C16—C15	-5.2 (3)
C2—N1—C6—N3	159.46 (16)	C19—O2—C16—C17	174.22 (19)
C7—N1—C6—N3	-23.1 (2)	C14—C15—C16—O2	179.53 (17)
C2—N1—C6—C5	-20.5 (2)	C14—C15—C16—C17	0.1 (3)
C7—N1—C6—C5	156.97 (16)	O2—C16—C17—C18	-178.57 (17)
C2—N1—C7—C8	-57.2 (2)	C15—C16—C17—C18	0.9 (3)
C6—N1—C7—C8	125.25 (18)	C16—C17—C18—C13	-0.6 (3)
C2—N1—C7—C12	121.29 (18)	C14—C13—C18—C17	-0.6 (3)
C6—N1—C7—C12	-56.3 (2)	C4—C13—C18—C17	179.03 (17)

Hydrogen-bond geometry (Å, °)

*Cg*3 is the centroid of the C13–C18 benzene ring of the methoxyphenyl group.

<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—H3C...N2 ⁱ	0.91 (2)	2.10 (2)	2.996 (2)	166 (2)
N3—H3D...O2 ⁱⁱ	0.91 (2)	2.48 (2)	3.152 (2)	131.0 (19)
C11—H11...O1 ⁱⁱⁱ	0.95	2.55	3.210 (3)	127
C14—H14...O1 ^{iv}	0.95	2.48	3.199 (2)	133
C10—H10... <i>Cg</i> 3 ⁱⁱⁱ	0.95	2.99	3.813 (3)	146
C18—H18... <i>Cg</i> 3 ^v	0.95	2.87	3.716 (2)	150

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