



Crystal structure and Hirshfeld surface analysis of 6-amino-8-(2,6-dichlorophenyl)-1,3,4,8-tetrahydro-2H-pyrido[1,2-a]pyrimidine-7,9-dicarbonitrile

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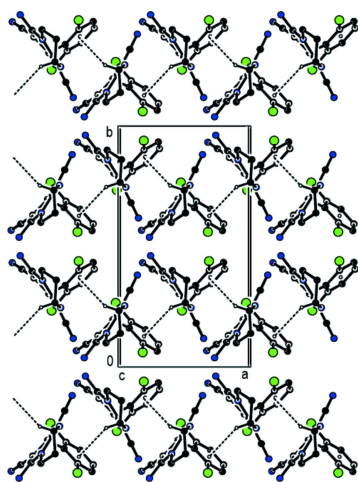
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In the molecular structure of the title compound, C₁₆H₁₃Cl₂N₅, the 1,4-dihydropyridine ring of the 1,3,4,8-tetrahydro-2H-pyrido[1,2-a]pyrimidine ring system adopts a screw-boat conformation, while the 1,3-diazinane ring is puckered. In the crystal, intermolecular N—H···N and C—H···N hydrogen bonds form molecular sheets parallel to the (110) and ($\bar{1}10$) planes, crossing each other. Adjacent molecules are further linked by C—H··· π interactions, which form zigzag chains propagating parallel to [100]. A Hirshfeld surface analysis indicates that the most significant contributions to the crystal packing are from N···H/H···N (28.4%), H···H (24.5%), C···H/H···C (21.4%) and Cl···H/H···Cl (16.1%) contacts.

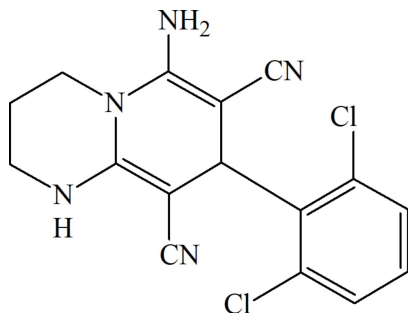
1. Chemical context

Chemical transformations comprising carbon–carbon and carbon–heteroatom bond-formation reactions are fundamental tools in modern synthetic organic chemistry (Yadigarov *et al.*, 2009; Abdelhamid *et al.*, 2011; Khalilov *et al.*, 2011; Yin *et al.*, 2020). They are also used for the synthesis of valuable building blocks in medicinal chemistry, coordination chemistry and material science (Mahmoudi *et al.*, 2017, 2019; Viswanathan *et al.*, 2019).

Pyrido[1,2-a]pyrimidines constitute a valuable class of heterocycles because many of them possess broad biological activities, such as monoamine oxidase inhibition, anti-hypertensive, insecticide, serotonergic antagonist, analgesic, anti-inflammatory, cytoprotective, bronchodilatory, phosphodiesterase-inhibitory, antithrombotic, antiallergic, anti-atherosclerotic and hypoglycaemic activities, as well as antitumor effects (Hermez & Mészáros, 1988; Ukrainets *et al.*, 2018). The pyrido[1,2-a]pyrimidine motif is incorporated into the structure of some marketed drugs, including the anti-asthmatic agent pemirolast, the tranquilizer pirenperone, the antiallergic agent ramastine, and the psychotropic agents risperidone and paliperidone (Awouters *et al.*, 1986; Blaton *et al.*, 1995; Yahata *et al.*, 2006; Riva *et al.*, 2011). Over recent decades, a number of synthetic protocols for the synthesis of pyrido[1,2-a]pyrimidines have been reported, and these



approaches have focused on two-component reactions (Wu *et al.*, 2003; Pryadaina *et al.*, 2005). Multi-component reactions have developed as powerful tools for the design of complex molecules, natural products and drug-like molecules in a minimum number of synthetic steps (Abdelhamid *et al.*, 2014; McLaughlin *et al.*, 2014; Janssen *et al.*, 2018).



As part of our studies on the chemistry of bridgehead nitrogen heterocycles, as well as taking into account our ongoing structural studies (Mamedov *et al.*, 2013; Naghiyev *et al.*, 2020*a,b,c*; Naghiyev *et al.*, 2021), we report here the crystal structure and Hirshfeld surface analysis of the title compound, $C_{16}H_{13}Cl_2N_5$, obtained by an efficient three-component synthetic protocol.

2. Structural commentary

In the molecular structure of the title compound, (Fig. 1), the 1,4-dihydropyridine ring (N5/C6–C9/C9A) of the 1,3,4,8-tetrahydro-2*H*-pyrido[1,2-*a*]pyrimidine ring system (N1/C2–C4/N5/C6–C9/C9A) adopts a screw-boat conformation with puckering parameters (Cremer & Pople, 1975) $Q_T = 0.520$ (3) Å, $\theta = 120.8$ (3)° and $\varphi = 270.4$ (3)°, while the 1,3-diazinane ring (N1/C2–C4/N5/C9A) is puckered [$Q_T = 0.160$ (3) Å, $\theta = 75.2$ (11)° and $\varphi = 169.4$ (10)°]. The dichlorophenyl ring (C11–C16) makes a dihedral angle of

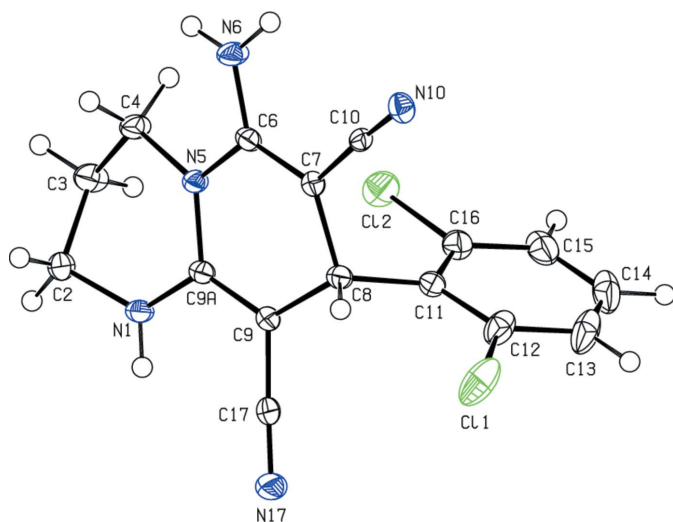


Figure 1
The molecular structure of the title compound. Displacement ellipsoids are drawn at the 50% probability level.

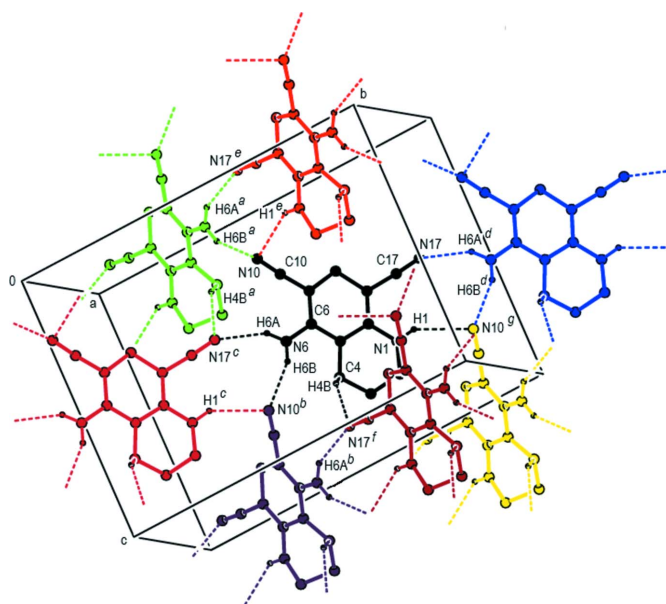


Figure 2
A view showing details of the intermolecular N–H...N and C–H...N hydrogen bonds in the unit cell of the title compound. The dichlorophenyl group and H atoms not involved in hydrogen bonding have been omitted for clarity. [Symmetry codes: (a) $x, 1 - y, -\frac{1}{2} + z$; (b) $x, 1 - y, \frac{1}{2} + z$; (c) $-\frac{1}{2} + x, -\frac{1}{2} + y, z$; (d) $\frac{1}{2} + x, \frac{1}{2} + y, z$; (e) $-\frac{1}{2} + x, \frac{3}{2} - y, -\frac{1}{2} + z$; (f) $-\frac{1}{2} + x, \frac{3}{2} - y, \frac{1}{2} + z$; (g) $\frac{1}{2} + x, \frac{3}{2} - y, \frac{1}{2} + z$].

80.82 (12)° with the mean plane of the 1,3,4,8-tetrahydro-2*H*-pyrido[1,2-*a*]pyrimidine ring system.

3. Supramolecular features

In the crystal, intermolecular N–H...N hydrogen bonds between the amine functions as donor groups and the nitrile N atoms as acceptor groups and intermolecular C–H...N hydrogen bonds lead to the formation of sheets extending parallel to (110) and ($\bar{1}10$) (Table 1; Figs. 2, 3 and 4). These hydrogen-bonded sheets cross each other (Fig. 5). C–H... π

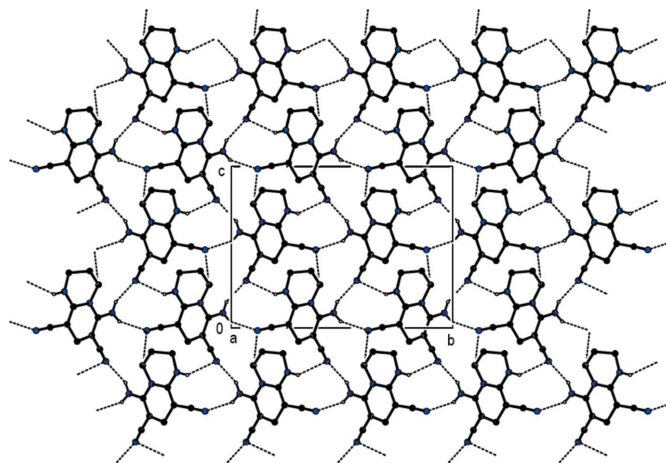


Figure 3
A view along [100] showing the intermolecular N–H...N and C–H...N hydrogen bonds of the title compound. The dichlorophenyl group and H atoms not involved in hydrogen bonding have been omitted for clarity.

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

Cg3 is the centroid of the C11–C16 dichlorophenyl ring.

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
N1–H1 \cdots N10 ⁱ	0.85 (3)	2.43 (3)	3.152 (3)	143 (3)
N6–H6A \cdots N17 ⁱⁱ	0.85 (4)	2.17 (3)	2.927 (3)	149 (3)
N6–H6B \cdots N10 ⁱⁱⁱ	0.85 (4)	2.16 (4)	2.953 (3)	156 (3)
C4–H4B \cdots N17 ^{iv}	0.99	2.59	3.440 (4)	144
C2–H2A \cdots Cg3 ^{iv}	0.99	2.87	3.653 (3)	136

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

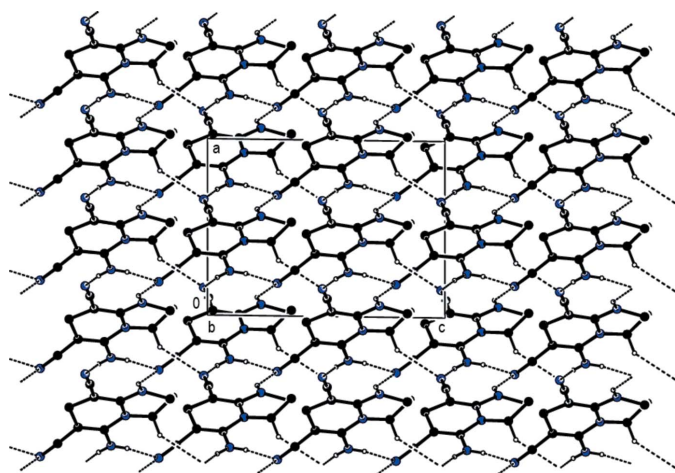


Figure 4
A view along [010] showing the intermolecular N–H \cdots N and C–H \cdots N hydrogen bonds of the title compound. The dichlorophenyl group and H atoms not involved in hydrogen bonding have been omitted for clarity.

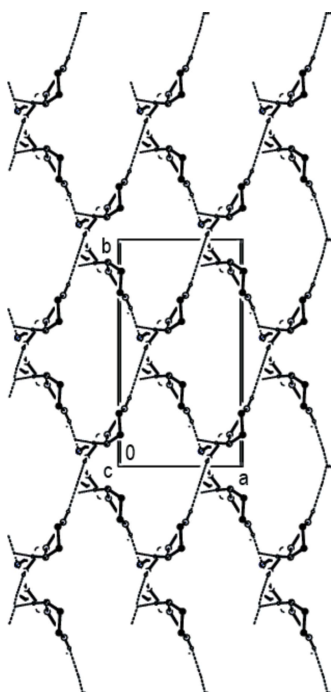


Figure 5
A view along [001] showing the intermolecular N–H \cdots N and C–H \cdots N hydrogen bonds of the title compound. The dichlorophenyl group and H atoms not involved in hydrogen bonding have been omitted for clarity.

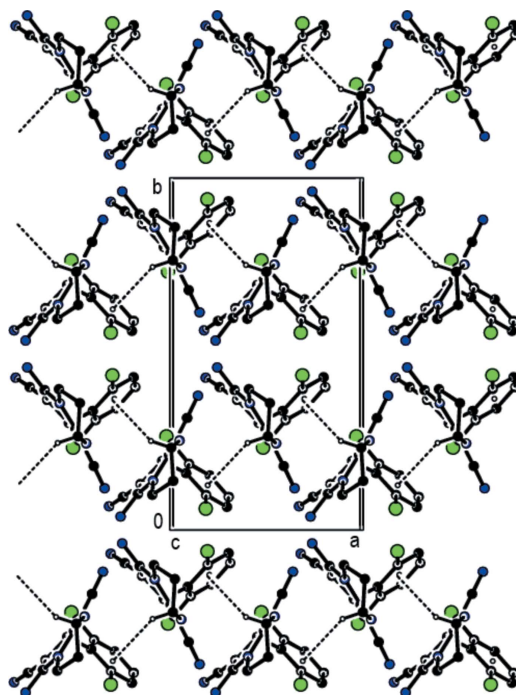


Figure 6
A view along [010] showing the C–H \cdots π interactions in the title compound.

interactions (Table 1), which form zigzag chains propagating parallel to [100] (Fig. 6), are also involved in the packing.

4. Hirshfeld surface analysis

In order to visualize the intermolecular interactions in the crystal of the title compound, a Hirshfeld surface analysis (Spackman & Jayatilaka, 2009) was performed with *Crystal-Explorer17.5* (Turner *et al.*, 2017). Fig. 7(a) and Fig. 7(b) show the front and back sides of the three-dimensional Hirshfeld surface of the title molecule plotted over d_{norm} in the range -0.4776 to $+1.4517$ a.u., using a ‘high standard’ surface resolution colour-mapped over the normalized contact distance. The red, white and blue regions visible on the d_{norm} surfaces indicate contacts with distances shorter, longer and equal to the van der Waals separations. The red spots highlight the

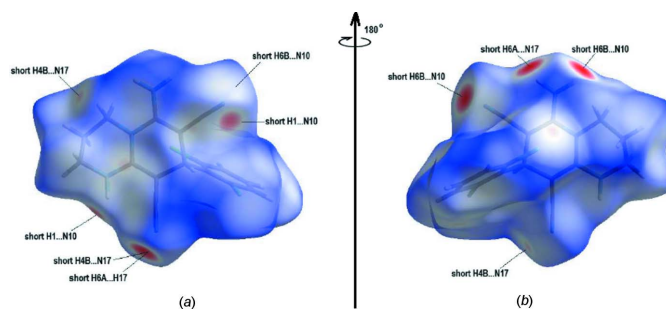


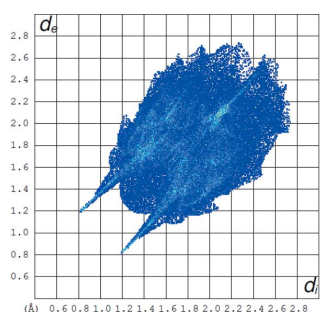
Figure 7
(a) Front and (b) back sides of the three-dimensional Hirshfeld surface of the title compound plotted over d_{norm} in the range -0.4776 to $+1.4517$ a.u.

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

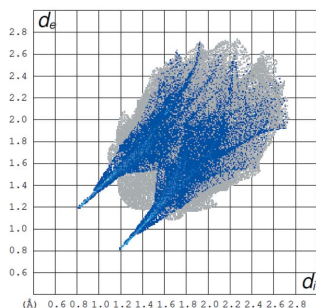
Contact	Distance	Symmetry operation
H6B···N10	2.16	$x, 1 - y, \frac{1}{2} + z$
H1···N10	2.43	$\frac{1}{2} + x, \frac{3}{2} - y, \frac{1}{2} + z$
H4B···N17	2.59	$-\frac{1}{2} + x, \frac{3}{2} - y, \frac{1}{2} + z$
H6A···N17	2.16	$-\frac{1}{2} + x, -\frac{1}{2} + y, z$
N10···H15	2.81	$-1 + x, y, z$
H3B···H13	2.57	$x, y, 1 + z$

interatomic contacts, including the N—H···N and C—H···N hydrogen bonds.

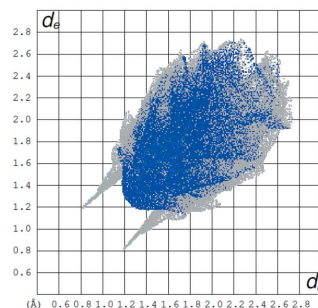
The overall two-dimensional fingerprint plot for the title compound and those delineated into N···H/H···N, H···H, C···H/H···C and Cl···H/H···Cl contacts are illustrated in



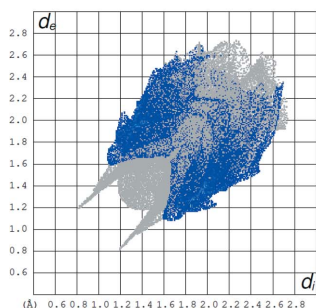
(a) All...All



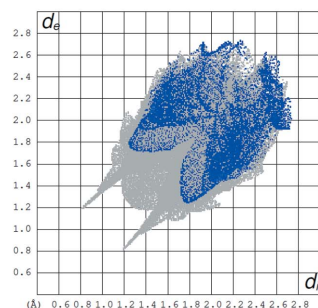
(b) N···H/H···N



(c) H···H



(d) C···H/H···C



(e) Cl···H/H···Cl

Figure 8
The two-dimensional fingerprint plots of the title compound, showing (a) all interactions, and delineated into (b) N···H/H···N, (c) H···H, (d) C···H/H···C and (e) Cl···H/H···Cl 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].

Table 3
Percentage contributions of interatomic contacts to the Hirshfeld surface for the title compound.

Contact	Percentage contribution
N···H/H···N	28.4
H···H	24.5
C···H/H···C	21.4
Cl···H/H···Cl	16.1
Cl···C/C···Cl	3.3
Cl···Cl	2.5
Cl···N/N···Cl	2.3
C···N/N···C	0.8
C···C	0.6
N···N	0.2

Fig. 8. Numerical details of the various contacts are given in Table 2 and their percentage contributions to the Hirshfeld surfaces are collated in Table 3. N···H/H···N (28.4%), H···H (24.5%), C···H/H···C (21.4%) and Cl···H/H···Cl (16.1%) contribute significantly to the packing while Cl···C/C···Cl, Cl···Cl, Cl···N/N···Cl, C···N/N···C, C···C and N···N contacts have a negligible directional impact.

The large number of N···H/H···N, H···H, C···H/H···C and Cl···H/H···Cl interactions suggest that van der Waals interactions and hydrogen bonding play the major roles in the crystal packing (Hathwar *et al.*, 2015).

5. Database survey

Four related compounds, which have the 1,3,4,8-tetrahydro-2H-pyrido[1,2-*a*]pyrimidine ring system of the title compound, were found in a search of the Cambridge Structural Database (CSD version 5.42, update of November 2020; Groom *et al.*, 2016): 9-(4-nitrobenzylidene)-8,9-dihydropyrido[2,3-*d*]pyrrolo[1,2-*a*]pyrimidin-5(7*H*)-one (refcode VAMBET; Khodjaniazov & Ashurov, 2016), 11-(aminomethylidene)-8,9,10,11-tetrahydropyrido[2',3':4,5]pyrimido[1,2-*a*]azepin-5(7*H*)-one (HECLUZ; Khodjaniazov *et al.*, 2017), 7'-amino-1'*H*-spiro[cycloheptane-1,2'-pyrimido[4,5-*d*]pyrimidin]-4'(3'*H*)-one (LEGLIU; Chen *et al.*, 2012) and 11-(2-oxopyrrolidin-1-ylmethyl)-1,2,3,4,5,6,11,11a-octahydropyrido[2,1-*b*]quinazolin-6-one dihydrate (KUTPEV; Samarov *et al.*, 2010).

In the crystal of VAMBET, molecules are linked *via* C—H···O and C—H···N hydrogen bonds, forming layers parallel to (101). In the molecule of HECLUZ, the seven-membered pentamethylene ring adopts a twist-boat conformation. In the crystal, hydrogen bonds with 16-membered ring and three chain motifs are generated by N—H···N and N—H···O contacts. The amino group is located close to the nitrogen atoms, forming hydrogen bonds with $R_2^1(4)$ and $R_2^2(12)$ graph-set motifs. This amino group also forms a hydrogen bond with the C=O oxygen atom of a molecule translated parallel to [100], which links the molecules into $R_4^4(16)$ rings. Hydrogen-bonded chains are formed along [100] by alternating $R_2^2(12)$ and $R_4^4(16)$ rings. These chains are stabilized by intermolecular π - π stacking interactions observed between the pyridine and pyrimidine rings. In LEGLIU, the molecular structure is built up from two fused six-membered rings and one seven-

Table 4
Experimental details.

Crystal data	
Chemical formula	C ₁₆ H ₁₃ Cl ₂ N ₅
<i>M_r</i>	346.21
Crystal system, space group	Monoclinic, <i>Cc</i>
Temperature (K)	100
<i>a</i> , <i>b</i> , <i>c</i> (Å)	8.6598 (2), 16.0275 (5), 11.6590 (3)
β (°)	90.7364 (9)
<i>V</i> (Å ³)	1618.08 (8)
<i>Z</i>	4
Radiation type	Mo <i>K</i> α
μ (mm ⁻¹)	0.41
Crystal size (mm)	0.30 × 0.03 × 0.03
Data collection	
Diffractometer	Bruker D8 QUEST PHOTON-III CCD
Absorption correction	Multi-scan (<i>SADABS</i> ; Krause <i>et al.</i> , 2015)
<i>T_{min}</i> , <i>T_{max}</i>	0.880, 0.980
No. of measured, independent and observed [<i>I</i> > 2 σ (<i>I</i>)] reflections	21346, 5861, 4528
<i>R_{int}</i>	0.064
($\sin \theta/\lambda$) _{max} (Å ⁻¹)	0.758
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, <i>S</i>	0.044, 0.090, 1.03
No. of reflections	5861
No. of parameters	217
No. of restraints	2
H-atom treatment	H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{\max}$, $\Delta\rho_{\min}$ (e Å ⁻³)	0.25, -0.32
Absolute structure	Flack <i>x</i> determined using 1774 quotients [(<i>I</i> ⁺) - (<i>I</i> ⁻)] / [(<i>I</i> ⁺) + (<i>I</i> ⁻)] (Parsons <i>et al.</i> , 2013).
Absolute structure parameter	0.27 (3)

Computer programs: *APEX3* (Bruker, 2018), *SAINT* (Bruker, 2013), *SHELXT2014/5* (Sheldrick, 2015a), *SHELXL2018/3* (Sheldrick, 2015b), *ORTEP-3 for Windows* (Farrugia, 2012) and *PLATON* (Spek, 2020).

membered ring linked through a spiro C atom. The crystal packing is stabilized by intermolecular N—H...O hydrogen bonds between the two N—H groups and the ketone O atoms of the neighbouring molecules. In KUTPEV, water molecules are mutually O—H...O hydrogen bonded and form infinite chains propagating parallel to [010]. Neighbouring chains are linked by the quinazoline molecules by means of O—H...O=C hydrogen bonds, forming a two-dimensional network.

6. Synthesis and crystallization

To a dissolved mixture of 2-(2,6-dichlorobenzylidene)malononitrile (1.14 g; 5.1 mmol) and malononitrile (0.34 g; 5.2 mmol) in methanol (40 mL), 1,3-diaminopropane (0.38 g; 5.2 mmol) was added and was stirred at room temperature for 10 min. Then 25 mL of methanol were removed from the reaction mixture that was left overnight. The precipitated crystals were separated by filtration and recrystallized from ethanol (yield 78%; m.p. 541–542 K).

¹H NMR (300 MHz, DMSO-*d*₆): 1.89 (*m*, 2H, CH₂); 3.13 (*m*, 2H, CH₂); 3.67 (*m*, 2H, CH₂); 5.31 (*s*, 1H, CH-Ar); 6.14 (*s*, 2H,

NH₂); 6.78 (*s*, 1H, NH); 7.25 (*t*, 1H, Ar-H, ³*J*_{H-H} = 7.9); 7.42 (*d*, 2H, 2Ar-H, ³*J*_{H-H} = 7.8). ¹³C NMR (75 MHz, DMSO-*d*₆): 22.30 (CH₂), 36.32 (Ar-CH), 38.62 (CH₂), 42.92 (CH₂), 51.70 (=C_{quar}), 55.06 (=C_{quar}), 121.61 (CN), 122.04 (CN), 129.56 (3CH_{arom}), 138.25 (3C_{ar}), 152.11 (=C_{quar}), 154.17 (=C_{quar}).

7. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 4. The C-bound H atoms were placed in calculated positions (C—H = 0.95–1.00 Å) and refined as riding with *U*_{iso}(H) = 1.2*U*_{eq}(C). All N-bound H atoms were located in a difference map [N1—H1 = 0.85 (3) Å, N6—H6A = 0.85 (4) Å and N6—H6B = 0.85 (4) Å] and they were refined with the constraint *U*_{iso}(H) = 1.2*U*_{eq}(N).

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Authors contributions are as follows. Conceptualization, FNN and IGM; methodology, FNN and IGM; investigation, VNK, FNN, TAT and AAA; writing (original draft), MA and IGM; writing (review and editing of the manuscript), MA and IGM; visualization, MA, FNN and IGM; funding acquisition, VNK and FNN; resources, RMR, AAA and FNN; supervision, IGM and MA.

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supporting information

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Crystal structure and Hirshfeld surface analysis of 6-amino-8-(2,6-dichlorophenyl)-1,3,4,8-tetrahydro-2H-pyrido[1,2-a]pyrimidine-7,9-dicarbonitrile

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Computing details

Data collection: *APEX3* (Bruker, 2018); cell refinement: *SAINTE* (Bruker, 2013); data reduction: *SAINTE* (Bruker, 2013); program(s) used to solve structure: *SHELXT2014/5* (Sheldrick, 2015a); program(s) used to refine structure: *SHELXL2018/3* (Sheldrick, 2015b); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 2012); software used to prepare material for publication: *PLATON* (Spek, 2020).

6-Amino-8-(2,6-dichlorophenyl)-1,3,4,8-tetrahydro-2H-pyrido[1,2-a]pyrimidine-7,9-dicarbonitrile

Crystal data

$C_{16}H_{13}Cl_2N_5$

$M_r = 346.21$

Monoclinic, *Cc*

$a = 8.6598$ (2) Å

$b = 16.0275$ (5) Å

$c = 11.6590$ (3) Å

$\beta = 90.7364$ (9)°

$V = 1618.08$ (8) Å³

$Z = 4$

$F(000) = 712$

$D_x = 1.421$ Mg m⁻³

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

Cell parameters from 4611 reflections

$\theta = 2.5$ – 32.2 °

$\mu = 0.41$ mm⁻¹

$T = 100$ K

Needle, colourless

$0.30 \times 0.03 \times 0.03$ mm

Data collection

Bruker D8 QUEST PHOTON-III CCD diffractometer

φ and ω scans

Absorption correction: multi-scan (SADABS; Krause *et al.*, 2015)

$T_{\min} = 0.880$, $T_{\max} = 0.980$

21346 measured reflections

5861 independent reflections

4528 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.064$

$\theta_{\max} = 32.6$ °, $\theta_{\min} = 2.5$ °

$h = -13 \rightarrow 13$

$k = -24 \rightarrow 24$

$l = -17 \rightarrow 17$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.044$

$wR(F^2) = 0.090$

$S = 1.02$

5861 reflections

217 parameters

2 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

$w = 1/[\sigma^2(F_o^2) + (0.0315P)^2 + 0.2854P]$

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

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

$$\Delta\rho_{\max} = 0.25 \text{ e } \text{\AA}^{-3}$$

$$\Delta\rho_{\min} = -0.32 \text{ e } \text{\AA}^{-3}$$

Absolute structure: Flack x determined using
1774 quotients $[(F^+)-(F^-)]/[(F^+)+(F^-)]$ (Parsons et
al., 2013).

Absolute structure parameter: 0.27 (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	$U_{\text{iso}}^*/U_{\text{eq}}$
C11	0.48473 (9)	0.76604 (6)	0.16871 (7)	0.0429 (2)
C12	0.69787 (9)	0.56058 (4)	0.51124 (7)	0.03080 (17)
N1	0.5635 (3)	0.75406 (14)	0.7250 (2)	0.0199 (5)
H1	0.604 (4)	0.801 (2)	0.709 (3)	0.024*
C2	0.5218 (3)	0.73373 (16)	0.8418 (2)	0.0207 (5)
H2A	0.4154	0.7528	0.8576	0.025*
H2B	0.5934	0.7611	0.8969	0.025*
C3	0.5326 (4)	0.63931 (17)	0.8531 (2)	0.0248 (6)
H3A	0.6392	0.6206	0.8373	0.030*
H3B	0.5064	0.6222	0.9321	0.030*
C4	0.4209 (3)	0.59988 (16)	0.7681 (2)	0.0218 (5)
H4A	0.4471	0.5401	0.7595	0.026*
H4B	0.3151	0.6033	0.7990	0.026*
N5	0.4232 (3)	0.64027 (13)	0.65394 (19)	0.0167 (4)
C6	0.3418 (3)	0.60234 (14)	0.5649 (2)	0.0163 (5)
N6	0.2511 (3)	0.53821 (15)	0.5930 (2)	0.0236 (5)
H6A	0.214 (4)	0.506 (2)	0.541 (3)	0.028*
H6B	0.238 (4)	0.522 (2)	0.661 (3)	0.028*
C7	0.3535 (3)	0.63028 (15)	0.4540 (2)	0.0155 (5)
C8	0.4667 (3)	0.69614 (15)	0.4164 (2)	0.0167 (5)
H8	0.4066	0.7379	0.3704	0.020*
C9	0.5266 (3)	0.74104 (16)	0.5222 (2)	0.0179 (5)
C9A	0.5062 (3)	0.71289 (15)	0.6322 (2)	0.0161 (5)
C10	0.2600 (3)	0.59393 (15)	0.3682 (2)	0.0147 (5)
N10	0.1851 (3)	0.56730 (13)	0.2941 (2)	0.0194 (5)
C11	0.5914 (3)	0.66149 (18)	0.3380 (3)	0.0200 (5)
C12	0.6051 (3)	0.6880 (2)	0.2246 (3)	0.0283 (6)
C13	0.7126 (4)	0.6549 (3)	0.1492 (3)	0.0375 (8)
H13	0.7167	0.6742	0.0723	0.045*
C14	0.8127 (4)	0.5940 (2)	0.1873 (3)	0.0383 (8)
H14	0.8869	0.5715	0.1367	0.046*
C15	0.8058 (3)	0.56565 (19)	0.2989 (3)	0.0314 (7)
H15	0.8746	0.5235	0.3254	0.038*
C16	0.6971 (3)	0.59938 (18)	0.3718 (3)	0.0240 (6)
C17	0.5995 (3)	0.81835 (17)	0.5040 (2)	0.0228 (6)

N17 0.6562 (4) 0.88151 (16) 0.4841 (2) 0.0362 (7)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C11	0.0391 (4)	0.0681 (6)	0.0215 (4)	-0.0012 (4)	0.0009 (3)	0.0190 (4)
C12	0.0305 (4)	0.0267 (3)	0.0353 (4)	0.0081 (3)	0.0050 (3)	0.0077 (3)
N1	0.0294 (12)	0.0147 (10)	0.0156 (11)	-0.0072 (9)	0.0006 (9)	0.0002 (8)
C2	0.0278 (14)	0.0215 (13)	0.0127 (12)	-0.0055 (10)	-0.0012 (10)	-0.0013 (10)
C3	0.0353 (16)	0.0207 (13)	0.0180 (14)	-0.0050 (11)	-0.0071 (12)	0.0033 (10)
C4	0.0328 (14)	0.0189 (12)	0.0136 (13)	-0.0088 (11)	-0.0029 (11)	0.0027 (9)
N5	0.0237 (11)	0.0145 (10)	0.0120 (10)	-0.0050 (8)	0.0000 (8)	0.0003 (7)
C6	0.0200 (12)	0.0127 (10)	0.0161 (12)	-0.0016 (9)	-0.0006 (9)	-0.0018 (9)
N6	0.0354 (13)	0.0211 (11)	0.0142 (11)	-0.0141 (10)	-0.0024 (10)	0.0009 (9)
C7	0.0179 (11)	0.0150 (11)	0.0137 (12)	-0.0006 (9)	0.0007 (9)	-0.0013 (9)
C8	0.0216 (13)	0.0135 (10)	0.0151 (12)	-0.0022 (9)	0.0001 (10)	0.0002 (9)
C9	0.0242 (13)	0.0149 (11)	0.0148 (13)	-0.0040 (9)	0.0029 (10)	-0.0025 (9)
C9A	0.0189 (12)	0.0131 (10)	0.0163 (12)	-0.0025 (9)	0.0001 (9)	-0.0007 (9)
C10	0.0179 (11)	0.0123 (10)	0.0140 (12)	0.0010 (9)	0.0034 (9)	0.0018 (8)
N10	0.0226 (11)	0.0193 (11)	0.0163 (12)	0.0030 (9)	-0.0013 (9)	-0.0016 (8)
C11	0.0220 (12)	0.0199 (11)	0.0181 (12)	-0.0082 (9)	0.0022 (10)	-0.0051 (9)
C12	0.0257 (15)	0.0417 (17)	0.0175 (15)	-0.0130 (13)	0.0008 (11)	-0.0018 (12)
C13	0.0281 (16)	0.067 (2)	0.0179 (15)	-0.0197 (16)	0.0048 (12)	-0.0117 (15)
C14	0.0248 (15)	0.053 (2)	0.038 (2)	-0.0131 (15)	0.0123 (13)	-0.0255 (16)
C15	0.0214 (14)	0.0300 (15)	0.043 (2)	-0.0064 (12)	0.0079 (13)	-0.0150 (14)
C16	0.0228 (14)	0.0229 (13)	0.0264 (15)	-0.0039 (10)	0.0037 (11)	-0.0039 (11)
C17	0.0329 (15)	0.0213 (12)	0.0144 (13)	-0.0073 (11)	0.0054 (11)	-0.0053 (10)
N17	0.064 (2)	0.0259 (12)	0.0189 (13)	-0.0208 (13)	0.0093 (12)	-0.0046 (10)

Geometric parameters (Å, °)

C11—C12	1.749 (4)	N6—H6B	0.85 (4)
C12—C16	1.741 (3)	C7—C10	1.406 (4)
N1—C9A	1.355 (3)	C7—C8	1.509 (3)
N1—C2	1.451 (3)	C8—C9	1.514 (4)
N1—H1	0.85 (3)	C8—C11	1.529 (4)
C2—C3	1.522 (4)	C8—H8	1.0000
C2—H2A	0.9900	C9—C9A	1.373 (4)
C2—H2B	0.9900	C9—C17	1.408 (4)
C3—C4	1.514 (4)	C10—N10	1.155 (3)
C3—H3A	0.9900	C11—C12	1.395 (4)
C3—H3B	0.9900	C11—C16	1.405 (4)
C4—N5	1.481 (3)	C12—C13	1.392 (4)
C4—H4A	0.9900	C13—C14	1.375 (5)
C4—H4B	0.9900	C13—H13	0.9500
N5—C6	1.387 (3)	C14—C15	1.380 (5)
N5—C9A	1.393 (3)	C14—H14	0.9500
C6—N6	1.337 (3)	C15—C16	1.385 (4)

C6—C7	1.373 (4)	C15—H15	0.9500
N6—H6A	0.85 (4)	C17—N17	1.150 (3)
C9A—N1—C2	123.1 (2)	C7—C8—C9	108.2 (2)
C9A—N1—H1	114 (2)	C7—C8—C11	112.7 (2)
C2—N1—H1	121 (2)	C9—C8—C11	115.0 (2)
N1—C2—C3	106.8 (2)	C7—C8—H8	106.8
N1—C2—H2A	110.4	C9—C8—H8	106.8
C3—C2—H2A	110.4	C11—C8—H8	106.8
N1—C2—H2B	110.4	C9A—C9—C17	119.5 (2)
C3—C2—H2B	110.4	C9A—C9—C8	123.9 (2)
H2A—C2—H2B	108.6	C17—C9—C8	116.4 (2)
C4—C3—C2	108.7 (2)	N1—C9A—C9	122.4 (2)
C4—C3—H3A	110.0	N1—C9A—N5	116.5 (2)
C2—C3—H3A	110.0	C9—C9A—N5	121.1 (2)
C4—C3—H3B	110.0	N10—C10—C7	176.6 (3)
C2—C3—H3B	110.0	C12—C11—C16	114.7 (3)
H3A—C3—H3B	108.3	C12—C11—C8	121.7 (3)
N5—C4—C3	113.0 (2)	C16—C11—C8	123.5 (3)
N5—C4—H4A	109.0	C13—C12—C11	123.2 (3)
C3—C4—H4A	109.0	C13—C12—C11	115.9 (3)
N5—C4—H4B	109.0	C11—C12—C11	120.8 (2)
C3—C4—H4B	109.0	C14—C13—C12	119.3 (3)
H4A—C4—H4B	107.8	C14—C13—H13	120.3
C6—N5—C9A	119.3 (2)	C12—C13—H13	120.3
C6—N5—C4	117.9 (2)	C13—C14—C15	120.2 (3)
C9A—N5—C4	122.8 (2)	C13—C14—H14	119.9
N6—C6—C7	122.1 (2)	C15—C14—H14	119.9
N6—C6—N5	116.6 (2)	C14—C15—C16	119.2 (3)
C7—C6—N5	121.2 (2)	C14—C15—H15	120.4
C6—N6—H6A	120 (2)	C16—C15—H15	120.4
C6—N6—H6B	124 (2)	C15—C16—C11	123.3 (3)
H6A—N6—H6B	115 (3)	C15—C16—C12	116.0 (3)
C6—C7—C10	119.1 (2)	C11—C16—C12	120.6 (2)
C6—C7—C8	123.9 (2)	N17—C17—C9	176.9 (3)
C10—C7—C8	117.0 (2)		
C9A—N1—C2—C3	46.5 (3)	C17—C9—C9A—N5	174.5 (3)
N1—C2—C3—C4	-60.3 (3)	C8—C9—C9A—N5	-1.8 (4)
C2—C3—C4—N5	43.2 (3)	C6—N5—C9A—N1	170.2 (2)
C3—C4—N5—C6	171.2 (2)	C4—N5—C9A—N1	-11.5 (4)
C3—C4—N5—C9A	-7.1 (4)	C6—N5—C9A—C9	-9.1 (4)
C9A—N5—C6—N6	-173.0 (2)	C4—N5—C9A—C9	169.2 (2)
C4—N5—C6—N6	8.6 (4)	C7—C8—C11—C12	116.4 (3)
C9A—N5—C6—C7	6.4 (4)	C9—C8—C11—C12	-118.9 (3)
C4—N5—C6—C7	-172.0 (2)	C7—C8—C11—C16	-61.5 (3)
N6—C6—C7—C10	4.1 (4)	C9—C8—C11—C16	63.2 (3)
N5—C6—C7—C10	-175.2 (2)	C16—C11—C12—C13	1.1 (4)

N6—C6—C7—C8	-173.2 (2)	C8—C11—C12—C13	-176.9 (3)
N5—C6—C7—C8	7.4 (4)	C16—C11—C12—C11	-179.0 (2)
C6—C7—C8—C9	-16.0 (3)	C8—C11—C12—C11	3.0 (4)
C10—C7—C8—C9	166.6 (2)	C11—C12—C13—C14	-1.0 (5)
C6—C7—C8—C11	112.3 (3)	C11—C12—C13—C14	179.1 (2)
C10—C7—C8—C11	-65.1 (3)	C12—C13—C14—C15	0.5 (5)
C7—C8—C9—C9A	13.2 (3)	C13—C14—C15—C16	-0.2 (5)
C11—C8—C9—C9A	-113.8 (3)	C14—C15—C16—C11	0.4 (4)
C7—C8—C9—C17	-163.2 (2)	C14—C15—C16—C12	-179.4 (2)
C11—C8—C9—C17	69.8 (3)	C12—C11—C16—C15	-0.8 (4)
C2—N1—C9A—C9	169.2 (3)	C8—C11—C16—C15	177.2 (3)
C2—N1—C9A—N5	-10.1 (4)	C12—C11—C16—C12	179.0 (2)
C17—C9—C9A—N1	-4.7 (4)	C8—C11—C16—C12	-3.1 (4)
C8—C9—C9A—N1	179.0 (3)		

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

Cg3 is the centroid of the C11–C16 dichlorophenyl ring.

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
N1—H1 \cdots N10 ⁱ	0.85 (3)	2.43 (3)	3.152 (3)	143 (3)
N6—H6A \cdots N17 ⁱⁱ	0.85 (4)	2.17 (3)	2.927 (3)	149 (3)
N6—H6B \cdots N10 ⁱⁱⁱ	0.85 (4)	2.16 (4)	2.953 (3)	156 (3)
C4—H4B \cdots N17 ^{iv}	0.99	2.59	3.440 (4)	144
C2—H2A \cdots Cg3 ^{iv}	0.99	2.87	3.653 (3)	136

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