Acta Cryst E
CRYSTALLOGRAPHIC COMMUNICATIONS

ISSN 2056-9890

Crystal structure and Hirshfeld surface analysis of ethyl 2-amino-4-(4-chlorophenyl)-5,6,7,8,9,10-hexahydrocycloocta[*b*]pyridine-3-carboxylate

Srinivasan Pazhamalai,[a] Chandiran Jayakodi,[a] Velayutham Mahalakshmi,[a] Rajendran Arivu Selvan[a]* and Sivashanmugam Selvanayagam[b]‡

[a]Department of Chemistry, Annamalai University, Annamalainagar, Chidambaram 608 002, India, and [b]PG & Research Department of Physics, Government Arts College, Melur 625 106, India. *Correspondence e-mail: arivusamy007@gmail.com

Received 2 March 2026
Accepted 23 March 2026

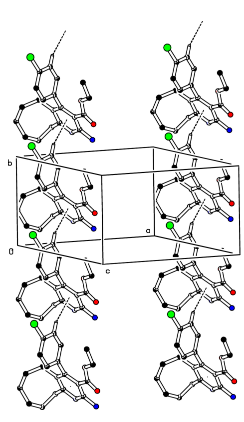
Edited by M. Weil, Vienna University of Technology, Austria

‡ Additional correspondence author, e-mail: sselvanayagam@gmail.com.

Keywords: pyridine derivative; intermolecular hydrogen bonds; Hirshfeld surface analysis; crystal structure..

CCDC reference: 2539747

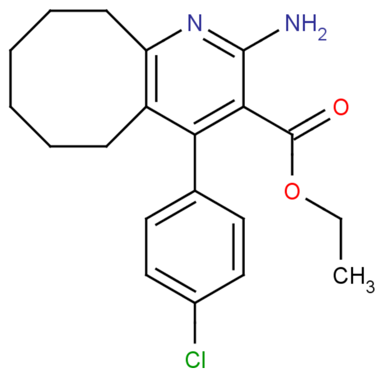
Supporting information: this article has supporting information at journals.iucr.org/e



In the title compound, $C_{20}H_{23}ClN_2O_2$, the cyclooctene ring has a boat–chair conformation. The ethyl formate moiety is equally disordered over two positions. The dihedral angle between the pyridine and chlorophenyl rings is 81.33 (13)°. An intramolecular N—H⋯O hydrogen bond helps to stabilize the molecular conformation. In the crystal, N—H⋯N hydrogen-bonding leads to the formation of dimers with an $R_2^2(8)$ graph-set motif. Additional consolidation of the packing in the crystal is achieved through C—H⋯π and π–π stacking interactions. The intermolecular interactions were quantified and analysed using Hirshfeld surface analysis, revealing that H⋯H interactions contribute by far the most to the crystal packing (63.6%).

1. Chemical context

The synthesis of functionalized N-heterocycles is an imprtant objective in organic and medicinal chemistry, as these moieties constitute the core of over 60% of US Food and Drug Administration (FDA) approved small-molecule drugs. Among these, the pyridine entity is arguably the most ubiquitous, appearing in essential vitamins such as nicotinic acid and various synthetic pharmaceuticals like Delafloxacin (Van Bambeke, 2015). Within this family, 2-aminonicotinate esters have garnered significant attention as privileged scaffolds. These multifunctional molecules possess a unique 1,2,3-arrangement of substituents – an amino group and a carboxylate ester – which provides a rich landscape for both molecular recognition and chemical transformation (Bagley *et al.*, 2015). The chemical appeal of 2-aminonicotinate esters lies in their synthetic versatility. They serve as precursors for the construction of fused heterocyclic systems, such as pyrido[2,3-*d*]pyrimidines and 1,8-naphthyridines, which are themselves pharmacologically active. Modern synthetic routes have shifted toward multicomponent reactions (MCRs), which allow for the one-pot assembly of these esters from readily available aldehydes, malononitriles, and alcohols. These methods are beneficial for their economy and compliance with green chemistry principles, often utilizing heterogeneous catalysts or aqueous media (Shaaban *et al.*, 2020). Biologically, these esters are highly multifunctional. The amino and ester groups provide critical hydrogen-bonding sites that facilitate high-affinity binding to various enzyme pockets. It has been demonstrated that derivatives of 2-aminonicotinates exhibit a broad spectrum of bio-activities, including anti-inflammatory and analgesic when acting as non-selective or COX-2 selective inhibitors (Bekhit *et al.*, 2017), and antimicrobial by demonstrating potency against Gram-positive and Gram-negative pathogens through disrupting metabolic pathways (El-Gazzar & Hafez, 2021). Given the rising challenge of drug resistance in both oncology and infectious diseases, the development of diverse 2-aminonicotinate derivatives offers a promising avenue for the discovery of next-generation therapeutic agents.



In this work, we describe the synthesis, structure and Hirshfeld surface analysis of the title compound, $C_{20}H_{23}ClN_2O_2$, (I).

2. Structural commentary

The molecular structure of (I) is displayed in Fig. 1. The pyridine ring (C1/C2/C9/N1/C10/C11) is essentially planar, with a maximum deviation of −0.016 (3) Å for atom C11, while its attached amino nitrogen atom N2 deviates by 0.005 (3) Å from this plane. The chlorophenyl ring is also almost planar, and its attached chlorine atom deviates by −0.031 (1) Å from this plane. The dihedral angle between the pyridine and chlorophenyl rings is 81.33 (13)°. The cyclooctene ring (C2–C9) has a boat–chair conformation based on a puckering analysis and endocyclic torsion angles (Evans & Boeyens, 1988). An intramolecular N—H⋯O hydrogen bond (Table 1) between atoms N2 and O1*A* contributes to the stability of the molecular conformation. This N2—H2*B*⋯O1*A* interaction generates an *S*(6) ring motif (Bernstein *et al.*, 1995), as shown in Fig. 1.

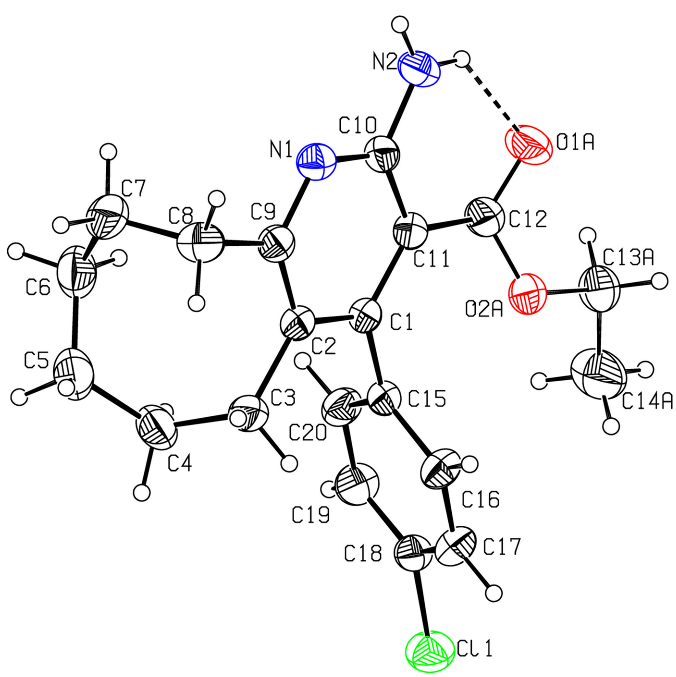


Figure 1
A view of the molecular structure of compound (I), showing the atom labelling. Displacement ellipsoids are drawn at the 30% probability level. Only one of the disordered parts of the ethyl formate chain is displayed for clarity; the intramolecular hydrogen bond is shown as a dashed line.

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

Cg is the centroid of pyridine ring (C1/C2/C9/N1/C10/C11).

D—H⋯*A*	*D*—H	H⋯*A*	*D*⋯*A*	*D*—H⋯*A*
N2—H2*B*⋯O1*A*	0.86	1.96	2.605 (10)	131
N2—H2*A*⋯N1^{i}	0.86	2.29	3.129 (3)	165
C17—H17⋯*Cg*^{ii}	0.93	2.84	3.657 (3)	147

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

3. Supramolecular features

In the crystal, molecules associate pairwise through N2—H2*A*⋯N1^{i} hydrogen bonds (Table 1) into inversion dimers with an $R_2^2(8)$ graph-set motif (Etter *et al.*, 1990; Bernstein *et al.*, 1995), as shown in Fig. 2. Molecules are linked into chains parallel to [010] by C—H⋯π interactions, C17—H17⋯*Cg*, where *Cg* is the centroid of the pyridine ring (Table 1, Fig. 3). Moreover, π–π interactions are observed between the centroids of inversion-related pyridine rings with a centroid-to-centroid distance of 3.764 (2) Å and a slippage of 0.711 Å.

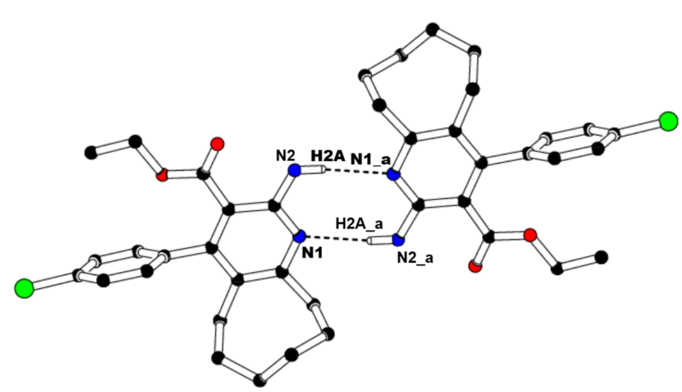


Figure 2
The formation of a centrosymmetric dimer in the crystal structure of (I) through N—H⋯N hydrogen bonds. For clarity, H atoms not involved in these interactions have been omitted, and only one of the disordered parts of the ethyl formate chain is displayed. [Symmetry code: (*a*) $-x, -y-1, -z+1$].

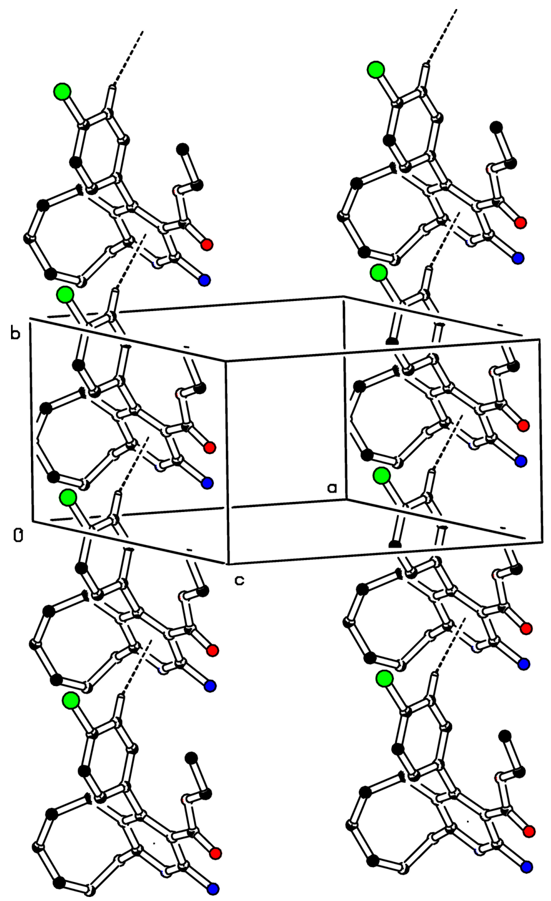


Figure 3
The crystal packing of (I). C—H$\cdots\pi$ interactions are shown as dashed lines. For clarity, H atoms not involved in these interactions have been omitted, and only one of the disordered parts of the ethyl formate chain is displayed.

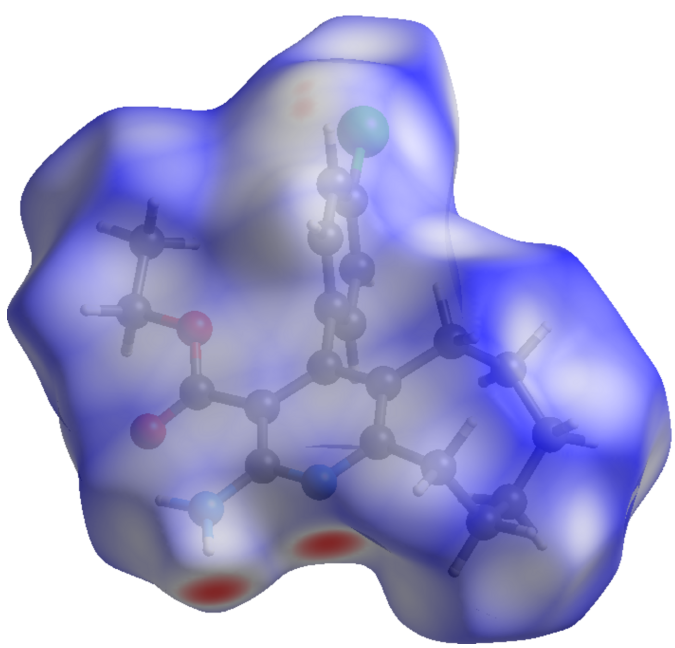


Figure 4
A view of the Hirshfeld surface mapped over $d_{\rm norm}$ for compound (I).

4. Hirshfeld surface analysis

A Hirshfeld surface (HS) analysis (Spackman & Jayatilaka, 2009) was carried out using *CrystalExplorer* (Spackman *et al.*, 2021) to characterize and quantify the intermolecular interactions in the title compound. For this purpose, a model without the disorder of the ethyl formate moiety was used (the ethyl formate moiety was set to full occupancy for the Hirshfeld surface analysis). The HS mapped over $d_{\rm norm}$ is illustrated in Fig. 4, where deep-red spots indicative of strong interactions occur at N1 and H2*A*, and these atoms are

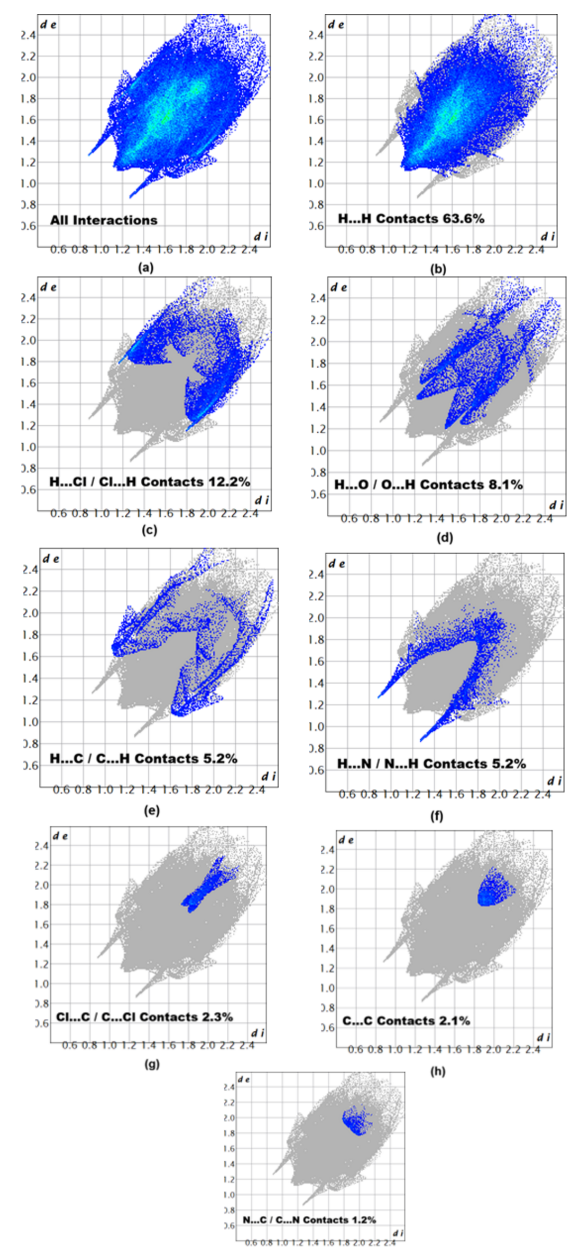


Figure 5
Two-dimensional fingerprint plots for the compound (I), showing (*a*) all interactions, and delineated into (*b*) H$\cdots$H, (*c*) H$\cdots$Cl/Cl$\cdots$H, (*d*) H$\cdots$O/O$\cdots$H, (*e*) H$\cdots$C/C$\cdots$H, (*f*) H$\cdots$N/N$\cdots$H, (*g*) Cl$\cdots$C/C$\cdots$Cl, (*h*) C$\cdots$C and (i) N$\cdots$C/C$\cdots$N interactions. The d_i and d_e values are the closest internal and external distances (in Å) from given points on the Hirshfeld surface.

responsible for the intermolecular hydrogen bonds discussed above. The associated two-dimensional fingerprint plots (McKinnon *et al.*, 2007) provide quantitative information about the non-covalent interactions in the crystal packing in terms of the percentage contribution of the interatomic contacts (Spackman & McKinnon, 2002). As shown in Fig. 5, the overall two-dimensional fingerprint plot for compound (I) is delineated into H···H, H···Cl/Cl···H, H···O/O···H, H···C/ C···H, H···N/N···H, Cl···C/C···Cl, C···C and N···C/C···N contacts, revealing that H···H interactions are by far the main contributor to the crystal packing.

5. Synthesis and crystallization

Compound (I) was prepared using a mixture of cyclooctanone (1.0 mmol, 0.126 g), 4-chlorobenzaldehyde (1.0 mmol, 0.140 g), ethyl cyanoacetate (1.0 mmol, 0.113 g) and ammonium acetate (1.5 mmol, 0.116 g) taken in a 100 ml round-bottom flask and dissolved using ethanol. The resulting solution was heated under reflux with stirring for 4–6 h. The progress of the reaction was periodically monitored by thin-layer chromatography using ethyl acetate:hexane (3:7) as the eluent. Upon completion of the reaction, the mixture was allowed to cool to room temperature, leading to the formation of a solid precipitate. The solid was collected by vacuum filtration and washed with cold ethanol to remove residual impurities. For final purification, the product was recrystallized from ethanol solution, yielding clean, well-formed crystals suitable for single crystal X-ray diffraction studies.

6. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2. Hydrogen atoms were placed in idealized positions and allowed to ride on their parent atoms, N—H = 0.86 Å and C—H = 0.93–0.97 Å, with $U_{iso}(H) = 1.5U_{eq}$ for methyl H atoms and $U_{iso}(H) = 1.2U_{eq}(C,N)$ for all other H atoms. The ethyl formate group (C12, O1, O2, C13, C14) is equally disordered over two sets of sites. For modelling of this disorder, pairs of C—O and C=O bond lengths were restrained to 1.31 (1) and 1.20 (1) Å, respectively, and the displacement parameters of all atoms involved were restrained to be within 0.01 Å of each other.

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{20}H_{23}ClN_2O_2$
M_r	358.85
Crystal system, space group	Monoclinic, $P2_1/n$
Temperature (K)	300
a, *b*, *c* (Å)	13.2386 (12), 7.2770 (7), 19.7223 (18)
β (°)	101.461 (3)
V (Å^3)	1862.1 (3)
Z	4
Radiation type	Mo *K*α
μ (mm^{-1})	0.22
Crystal size (mm)	0.26 × 0.12 × 0.08
Data collection	
Diffractometer	Bruker APEXII CCD
Absorption correction	Multi-scan (*SADABS*; Krause *et al.*, 2015)
T_{min}, T_{max}	0.945, 0.983
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	34945, 4649, 2682
R_{int}	0.050
$(\sin \theta/\lambda)_{max}$ (Å^{-1})	0.669
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.064, 0.207, 1.06
No. of reflections	4649
No. of parameters	264
No. of restraints	108
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.55, −0.27

Computer programs: *APEX3* and *SAINT* (Bruker, 2017), *SHELXT* (Sheldrick, 2015*a*), *SHELXL* (Sheldrick, 2015*b*), *ORTEP-3 for Windows* (Farrugia, 2012), *PLATON* (Spek, 2020) and *publCIF* (Westrip, 2010).

Acknowledgements

The authors thank the Single Crystal XRD Facility at VIT, Vellore, Tamil Nadu, India, for providing the instrumentation and support necessary for this study.

References

Bagley, M. C., Zhao, T. Z. & Smith, J. A. (2015). *J. Org. Chem.* **80**, 5800–5812.
Bekhit, A. A., Ashour, H. M. A., Guemei, A. A. & Bekhit, S. S. (2017). *Arch. Pharm.* **350**, e1600244.
Bernstein, J., Davis, R. E., Shimoni, L. & Chang, N.-L. (1995). *Angew. Chem. Int. Ed. Engl.* **34**, 1555–1573.
Bruker (2017). *APEX3* and *SAINT*. Bruker AXS Inc., Madison, Wisconsin, U. S. A.
El-Gazzar, A. B. & Hafez, H. N. (2021). *Chem. Pap.* **75**, 451–465.
Etter, M. C., MacDonald, J. C. & Bernstein, J. (1990). *Acta Cryst.* B**46**, 256–262.
Evans, D. G. & Boeyens, J. C. A. (1988). *Acta Cryst.* B**44**, 663–671.
Farrugia, L. J. (2012). *J. Appl. Cryst.* **45**, 849–854.
Krause, L., Herbst-Irmer, R., Sheldrick, G. M. & Stalke, D. (2015). *J. Appl. Cryst.* **48**, 3–10.
McKinnon, J. J., Jayatilaka, D. & Spackman, M. A. (2007). *Chem. Commun.* pp. 3814–3816.
Shaaban, M. R., Moustafa, A. H. & El-Hady, A. S. (2020). *Molecules* **25**, 3211.
Sheldrick, G. M. (2015*a*). *Acta Cryst.* A**71**, 3–8.
Sheldrick, G. M. (2015*b*). *Acta Cryst.* C**71**, 3–8.
Spackman, M. A. & Jayatilaka, D. (2009). *CrystEngComm* **11**, 19–32.
Spackman, M. A. & McKinnon, J. J. (2002). *CrystEngComm* **4**, 378–392.
Spackman, P. R., Turner, M. J., McKinnon, J. J., Wolff, S. K., Grimwood, D. J., Jayatilaka, D. & Spackman, M. A. (2021). *J. Appl. Cryst.* **54**, 1006–1011.
Spek, A. L. (2020). *Acta Cryst.* E**76**, 1–11.
Van Bambeke, F. (2015). *Fut. Microbiol.* **10**, 1111–1123.
Westrip, S. P. (2010). *J. Appl. Cryst.* **43**, 920–925.

supporting information

Acta Cryst. (2026). E**82**, 413-417 [https://doi.org/10.1107/S2056989026003087]

Crystal structure and Hirshfeld surface analysis of ethyl 2-amino-4-(4-chlorophenyl)-5,6,7,8,9,10-hexahydrocycloocta[*b*]pyridine-3-carboxylate

Srinivasan Pazhamalai, Chandiran Jayakodi, Velayutham Mahalakshmi, Rajendran Arivu Selvan and Sivashanmugam Selvanayagam

Computing details

Ethyl 2-amino-4-(4-chlorophenyl)-5,6,7,8,9,10-hexahydrocycloocta[*b*]pyridine-3-carboxylate

Crystal data

$C_{20}H_{23}ClN_2O_2$
M_r = 358.85
Monoclinic, $P2_1/n$
a = 13.2386 (12) Å
b = 7.2770 (7) Å
c = 19.7223 (18) Å
β = 101.461 (3)°
V = 1862.1 (3) Å^3
Z = 4
$F(000)$ = 760
D_x = 1.280 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 9916 reflections
θ = 3.0–26.3°
μ = 0.22 mm^{-1}
T = 300 K
Block, colourless
0.26 × 0.12 × 0.08 mm

Data collection

Bruker APEXII CCD diffractometer
Radiation source: i-mu-s microfocus source
φ and ω scans
Absorption correction: multi-scan (SADABS; Krause *et al.*, 2015)
T_{min} = 0.945, T_{max} = 0.983
34945 measured reflections
4649 independent reflections
2682 reflections with $I > 2\sigma(I)$
R_{int} = 0.050
θ_{max} = 28.4°, θ_{min} = 2.1°
h = −17→17
k = −9→9
l = −26→26

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.064
$wR(F^2)$ = 0.207
S = 1.06
4649 reflections
264 parameters
108 restraints
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0769P)^2 + 1.3578P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ < 0.001
$\Delta\rho_{max}$ = 0.55 e Å^{-3}
$\Delta\rho_{min}$ = −0.27 e Å^{-3}
Extinction correction: SHELXL (Sheldrick, 2015b), $Fc^* = kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.0053 (14)

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 (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}	Occ. (<1)
N1	0.07369 (17)	−0.2785 (3)	0.52707 (12)	0.0531 (6)	
N2	−0.00533 (19)	−0.3486 (3)	0.41712 (13)	0.0654 (7)	
H2A	−0.034366	−0.439014	0.433851	0.078*	
H2B	−0.017722	−0.329464	0.373218	0.078*	
Cl1	0.44196 (7)	0.62949 (12)	0.41161 (5)	0.0778 (3)	
C1	0.17789 (18)	0.0195 (3)	0.48544 (13)	0.0458 (6)	
C2	0.19016 (19)	−0.0236 (3)	0.55599 (13)	0.0485 (6)	
C3	0.2612 (2)	0.0864 (4)	0.61089 (15)	0.0604 (7)	
H3A	0.229762	0.094328	0.651235	0.072*	
H3B	0.265523	0.210315	0.593602	0.072*	
C4	0.3700 (2)	0.0133 (5)	0.63391 (17)	0.0750 (9)	
H4A	0.396761	−0.014633	0.592727	0.090*	
H4B	0.412006	0.111600	0.657960	0.090*	
C6	0.3400 (3)	−0.3296 (6)	0.6488 (2)	0.0872 (11)	
H6A	0.388293	−0.428382	0.664182	0.105*	
H6B	0.332028	−0.320339	0.598970	0.105*	
C8	0.1485 (3)	−0.2405 (4)	0.64719 (15)	0.0675 (8)	
H8A	0.084039	−0.293594	0.653997	0.081*	
H8B	0.163888	−0.136091	0.678107	0.081*	
C7	0.2338 (3)	−0.3826 (5)	0.66593 (18)	0.0817 (10)	
H7A	0.210848	−0.496052	0.641912	0.098*	
H7B	0.243729	−0.407132	0.715151	0.098*	
C5	0.3855 (3)	−0.1551 (5)	0.6802 (2)	0.0858 (11)	
H5A	0.458980	−0.173647	0.695896	0.103*	
H5B	0.356562	−0.129580	0.720655	0.103*	
C9	0.1357 (2)	−0.1747 (4)	0.57360 (14)	0.0499 (6)	
C10	0.05992 (19)	−0.2364 (4)	0.45992 (14)	0.0508 (6)	
C11	0.1101 (2)	−0.0837 (4)	0.43593 (13)	0.0501 (6)	
C15	0.24158 (19)	0.1722 (3)	0.46504 (13)	0.0471 (6)	
C16	0.2137 (2)	0.3543 (4)	0.46938 (16)	0.0581 (7)	
H16	0.152775	0.382814	0.483821	0.070*	
C17	0.2744 (2)	0.4943 (4)	0.45269 (15)	0.0605 (7)	
H17	0.254742	0.616217	0.455904	0.073*	
C18	0.3638 (2)	0.4525 (4)	0.43137 (14)	0.0548 (7)	
C19	0.3934 (2)	0.2739 (4)	0.42640 (17)	0.0650 (8)	
H19	0.454033	0.246819	0.411419	0.078*	
C20	0.3327 (2)	0.1336 (4)	0.44375 (16)	0.0594 (7)	
H20	0.353465	0.012167	0.441067	0.071*	
C12	0.0893 (2)	−0.0450 (4)	0.36086 (15)	0.0642 (8)	

O1A	0.0236 (10)	−0.1269 (16)	0.3190 (5)	0.085 (3)	0.51 (2)
O1B	0.0739 (13)	−0.1670 (13)	0.3166 (5)	0.088 (3)	0.49 (2)
O2A	0.1083 (12)	0.1269 (14)	0.3464 (11)	0.063 (2)	0.51 (2)
O2B	0.1305 (15)	0.1061 (19)	0.3421 (12)	0.081 (4)	0.49 (2)
C13A	0.0965 (17)	0.178 (3)	0.2723 (15)	0.075 (3)	0.51 (2)
H13A	0.104132	0.068990	0.245334	0.091*	0.51 (2)
H13B	0.028133	0.227566	0.255673	0.091*	0.51 (2)
C13B	0.1142 (19)	0.148 (3)	0.2708 (16)	0.080 (3)	0.49 (2)
H13C	0.044942	0.114708	0.247579	0.095*	0.49 (2)
H13D	0.163390	0.083931	0.248771	0.095*	0.49 (2)
C14A	0.1750 (15)	0.316 (3)	0.2633 (8)	0.112 (4)	0.51 (2)
H14A	0.166505	0.347352	0.215229	0.134*	0.51 (2)
H14B	0.166787	0.423928	0.289563	0.134*	0.51 (2)
H14C	0.242641	0.265654	0.279244	0.134*	0.51 (2)
C14B	0.1308 (13)	0.3638 (19)	0.2693 (8)	0.085 (3)	0.49 (2)
H14D	0.121116	0.404984	0.222240	0.103*	0.49 (2)
H14E	0.081895	0.423836	0.291869	0.103*	0.49 (2)
H14F	0.199453	0.393290	0.293052	0.103*	0.49 (2)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.0523 (12)	0.0505 (13)	0.0574 (14)	−0.0021 (10)	0.0134 (10)	−0.0020 (11)
N2	0.0636 (15)	0.0623 (15)	0.0640 (15)	−0.0174 (12)	−0.0026 (12)	−0.0024 (12)
Cl1	0.0826 (6)	0.0708 (5)	0.0807 (6)	−0.0202 (4)	0.0181 (4)	0.0063 (4)
C1	0.0449 (13)	0.0400 (13)	0.0505 (14)	0.0051 (10)	0.0044 (10)	−0.0004 (11)
C2	0.0524 (14)	0.0402 (13)	0.0507 (15)	0.0039 (11)	0.0053 (11)	−0.0012 (11)
C3	0.0747 (19)	0.0500 (16)	0.0521 (15)	−0.0059 (14)	0.0022 (14)	−0.0014 (12)
C4	0.0681 (19)	0.087 (2)	0.0620 (18)	−0.0129 (18)	−0.0056 (15)	0.0080 (17)
C6	0.083 (2)	0.088 (3)	0.082 (2)	0.026 (2)	−0.0030 (19)	0.001 (2)
C8	0.084 (2)	0.0660 (19)	0.0550 (17)	−0.0136 (16)	0.0210 (15)	−0.0035 (15)
C7	0.109 (3)	0.064 (2)	0.066 (2)	−0.0059 (19)	0.0022 (19)	0.0197 (16)
C5	0.072 (2)	0.097 (3)	0.081 (2)	0.006 (2)	−0.0015 (18)	0.016 (2)
C9	0.0498 (14)	0.0486 (14)	0.0520 (15)	0.0039 (11)	0.0115 (11)	−0.0022 (12)
C10	0.0449 (13)	0.0492 (15)	0.0558 (16)	0.0026 (11)	0.0038 (11)	−0.0024 (12)
C11	0.0517 (14)	0.0471 (14)	0.0491 (14)	0.0035 (12)	0.0044 (11)	0.0013 (12)
C15	0.0497 (14)	0.0438 (13)	0.0446 (13)	0.0015 (11)	0.0019 (10)	−0.0013 (11)
C16	0.0605 (16)	0.0469 (15)	0.0703 (19)	0.0066 (12)	0.0210 (14)	0.0046 (13)
C17	0.0732 (19)	0.0438 (14)	0.0666 (18)	0.0060 (13)	0.0186 (15)	0.0027 (13)
C18	0.0607 (16)	0.0505 (15)	0.0507 (15)	−0.0059 (13)	0.0050 (12)	0.0021 (12)
C19	0.0569 (16)	0.0631 (19)	0.078 (2)	0.0010 (14)	0.0210 (15)	−0.0009 (16)
C20	0.0596 (16)	0.0453 (15)	0.0739 (19)	0.0067 (12)	0.0145 (14)	−0.0015 (13)
C12	0.0742 (19)	0.0606 (18)	0.0526 (16)	−0.0057 (15)	0.0001 (14)	0.0008 (14)
O1A	0.105 (6)	0.081 (5)	0.058 (3)	−0.032 (4)	−0.015 (4)	0.001 (3)
O1B	0.127 (6)	0.074 (4)	0.058 (3)	−0.012 (4)	0.006 (4)	−0.013 (3)
O2A	0.077 (5)	0.059 (3)	0.048 (3)	−0.001 (3)	−0.001 (3)	0.005 (2)
O2B	0.098 (8)	0.091 (5)	0.048 (4)	−0.031 (5)	0.001 (5)	0.011 (5)
C13A	0.094 (6)	0.079 (6)	0.048 (3)	−0.003 (4)	0.000 (5)	0.009 (4)

C13B	0.098 (7)	0.085 (6)	0.049 (4)	−0.012 (5)	0.000 (5)	0.011 (5)
C14A	0.138 (10)	0.121 (9)	0.067 (5)	−0.043 (7)	0.002 (7)	0.016 (6)
C14B	0.104 (8)	0.086 (6)	0.063 (5)	−0.008 (5)	0.008 (5)	0.017 (4)

Geometric parameters (Å, º)

N1—C10	1.336 (3)	C10—C11	1.423 (4)
N1—C9	1.337 (3)	C11—C12	1.478 (4)
N2—C10	1.355 (3)	C15—C16	1.383 (4)
N2—H2A	0.8600	C15—C20	1.383 (4)
N2—H2B	0.8600	C16—C17	1.378 (4)
Cl1—C18	1.744 (3)	C16—H16	0.9300
C1—C2	1.404 (3)	C17—C18	1.367 (4)
C1—C11	1.405 (4)	C17—H17	0.9300
C1—C15	1.498 (4)	C18—C19	1.366 (4)
C2—C9	1.397 (4)	C19—C20	1.383 (4)
C2—C3	1.514 (4)	C19—H19	0.9300
C3—C4	1.518 (4)	C20—H20	0.9300
C3—H3A	0.9700	C12—O1A	1.228 (6)
C3—H3B	0.9700	C12—O2A	1.319 (9)
C4—C5	1.517 (5)	O2A—C13A	1.48 (3)
C4—H4A	0.9700	O2B—C13B	1.41 (3)
C4—H4B	0.9700	C13A—C14A	1.48 (3)
C6—C5	1.486 (5)	C13A—H13A	0.9700
C6—C7	1.558 (6)	C13A—H13B	0.9700
C6—H6A	0.9700	C13B—C14B	1.59 (3)
C6—H6B	0.9700	C13B—H13C	0.9700
C8—C9	1.505 (4)	C13B—H13D	0.9700
C8—C7	1.522 (5)	C14A—H14A	0.9600
C8—H8A	0.9700	C14A—H14B	0.9600
C8—H8B	0.9700	C14A—H14C	0.9600
C7—H7A	0.9700	C14B—H14D	0.9600
C7—H7B	0.9700	C14B—H14E	0.9600
C5—H5A	0.9700	C14B—H14F	0.9600
C5—H5B	0.9700		
C10—N1—C9	119.7 (2)	N2—C10—C11	123.0 (2)
C10—N2—H2A	120.0	C1—C11—C10	117.5 (2)
C10—N2—H2B	120.0	C1—C11—C12	124.0 (2)
H2A—N2—H2B	120.0	C10—C11—C12	118.4 (2)
C2—C1—C11	120.1 (2)	C16—C15—C20	118.3 (3)
C2—C1—C15	118.1 (2)	C16—C15—C1	121.5 (2)
C11—C1—C15	121.7 (2)	C20—C15—C1	120.2 (2)
C9—C2—C1	117.3 (2)	C17—C16—C15	121.2 (3)
C9—C2—C3	121.1 (2)	C17—C16—H16	119.4
C1—C2—C3	121.6 (2)	C15—C16—H16	119.4
C2—C3—C4	116.5 (3)	C18—C17—C16	119.4 (3)
C2—C3—H3A	108.2	C18—C17—H17	120.3

C4—C3—H3A	108.2	C16—C17—H17	120.3
C2—C3—H3B	108.2	C19—C18—C17	120.8 (3)
C4—C3—H3B	108.2	C19—C18—Cl1	119.7 (2)
H3A—C3—H3B	107.3	C17—C18—Cl1	119.5 (2)
C5—C4—C3	118.0 (3)	C18—C19—C20	119.7 (3)
C5—C4—H4A	107.8	C18—C19—H19	120.1
C3—C4—H4A	107.8	C20—C19—H19	120.1
C5—C4—H4B	107.8	C19—C20—C15	120.6 (3)
C3—C4—H4B	107.8	C19—C20—H20	119.7
H4A—C4—H4B	107.1	C15—C20—H20	119.7
C5—C6—C7	115.5 (3)	O1A—C12—O2A	117.1 (10)
C5—C6—H6A	108.4	O1A—C12—C11	123.1 (5)
C7—C6—H6A	108.4	O2A—C12—C11	113.1 (10)
C5—C6—H6B	108.4	C12—O2A—C13A	117.5 (15)
C7—C6—H6B	108.4	C14A—C13A—O2A	110.4 (17)
H6A—C6—H6B	107.5	C14A—C13A—H13A	109.6
C9—C8—C7	112.7 (3)	O2A—C13A—H13A	109.6
C9—C8—H8A	109.0	C14A—C13A—H13B	109.6
C7—C8—H8A	109.0	O2A—C13A—H13B	109.6
C9—C8—H8B	109.0	H13A—C13A—H13B	108.1
C7—C8—H8B	109.0	O2B—C13B—C14B	103.6 (17)
H8A—C8—H8B	107.8	O2B—C13B—H13C	111.0
C8—C7—C6	115.9 (3)	C14B—C13B—H13C	111.0
C8—C7—H7A	108.3	O2B—C13B—H13D	111.0
C6—C7—H7A	108.3	C14B—C13B—H13D	111.0
C8—C7—H7B	108.3	H13C—C13B—H13D	109.0
C6—C7—H7B	108.3	C13A—C14A—H14A	109.5
H7A—C7—H7B	107.4	C13A—C14A—H14B	109.5
C6—C5—C4	116.4 (3)	H14A—C14A—H14B	109.5
C6—C5—H5A	108.2	C13A—C14A—H14C	109.5
C4—C5—H5A	108.2	H14A—C14A—H14C	109.5
C6—C5—H5B	108.2	H14B—C14A—H14C	109.5
C4—C5—H5B	108.2	C13B—C14B—H14D	109.5
H5A—C5—H5B	107.3	C13B—C14B—H14E	109.5
N1—C9—C2	123.5 (2)	H14D—C14B—H14E	109.5
N1—C9—C8	114.7 (2)	C13B—C14B—H14F	109.5
C2—C9—C8	121.7 (2)	H14D—C14B—H14F	109.5
N1—C10—N2	115.1 (2)	H14E—C14B—H14F	109.5
N1—C10—C11	121.9 (2)		

C11—C1—C2—C9	2.2 (4)	N1—C10—C11—C1	2.0 (4)
C15—C1—C2—C9	−175.5 (2)	N2—C10—C11—C1	−178.5 (2)
C11—C1—C2—C3	−179.0 (2)	N1—C10—C11—C12	−179.0 (3)
C15—C1—C2—C3	3.2 (4)	N2—C10—C11—C12	0.5 (4)
C9—C2—C3—C4	84.5 (3)	C2—C1—C15—C16	−81.3 (3)
C1—C2—C3—C4	−94.3 (3)	C11—C1—C15—C16	101.0 (3)
C2—C3—C4—C5	−74.1 (4)	C2—C1—C15—C20	96.2 (3)
C9—C8—C7—C6	51.0 (4)	C11—C1—C15—C20	−81.5 (3)

C5—C6—C7—C8	56.5 (4)	C20—C15—C16—C17	0.3 (4)
C7—C6—C5—C4	−98.5 (4)	C1—C15—C16—C17	177.8 (3)
C3—C4—C5—C6	66.4 (5)	C15—C16—C17—C18	0.1 (5)
C10—N1—C9—C2	−1.5 (4)	C16—C17—C18—C19	0.1 (4)
C10—N1—C9—C8	−177.5 (2)	C16—C17—C18—Cl1	−178.9 (2)
C1—C2—C9—N1	0.2 (4)	C17—C18—C19—C20	−0.6 (5)
C3—C2—C9—N1	−178.6 (2)	Cl1—C18—C19—C20	178.4 (2)
C1—C2—C9—C8	175.9 (2)	C18—C19—C20—C15	1.0 (5)
C3—C2—C9—C8	−2.8 (4)	C16—C15—C20—C19	−0.9 (4)
C7—C8—C9—N1	86.3 (3)	C1—C15—C20—C19	−178.4 (3)
C7—C8—C9—C2	−89.8 (3)	C1—C11—C12—O1A	−173.8 (10)
C9—N1—C10—N2	−179.2 (2)	C10—C11—C12—O1A	7.3 (10)
C9—N1—C10—C11	0.4 (4)	C1—C11—C12—O2A	−23.5 (8)
C2—C1—C11—C10	−3.3 (4)	C10—C11—C12—O2A	157.6 (8)
C15—C1—C11—C10	174.4 (2)	O1A—C12—O2A—C13A	−31.6 (17)
C2—C1—C11—C12	177.8 (3)	C11—C12—O2A—C13A	176.1 (10)
C15—C1—C11—C12	−4.5 (4)	C12—O2A—C13A—C14A	−145 (2)

Hydrogen-bond geometry (Å, º)

Cg is the centroid of pyridine ring (C1/C2/C9/N1/C10/C11).

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H2*B*···O1*A*	0.86	1.96	2.605 (10)	131
N2—H2*A*···N1^{i}	0.86	2.29	3.129 (3)	165
C17—H17···*Cg*^{ii}	0.93	2.84	3.657 (3)	147

Symmetry codes: (i) −*x*, −*y*−1, −*z*+1; (ii) *x*, *y*+1, *z*.