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

Ibrahim G. Mamedov,a Victor N. Khrustalev,b,c Mehmet Akkurt,d Anton P. Novikov,b Ayten R. Asgarova,a Khatira N. Aliyevaa and Anzurat A. Akobirshoeva*e

aDepartment of Chemistry, Baku State University, 23 Z. Khalilov str., Az, 1148 Baku, Azerbaijan, bPeoples’ Friendship University of Russia (RUDN University), 6 Miklukho-Maklay str., Moscow, 117198, Russian Federation, cN. D. Zelinsky Institute of Organic Chemistry RAS, Leninsky Prosp. 47, 119991 Moscow, Russian Federation, dDepartment of Physics, Faculty of Sciences, Erciyes University, 38039 Kayseri, Turkey, and eAcad. Sci. Republ. Tajikistan, Kh. Yu. Yusubbekov Pamir Biol. Inst., 1 Kholdorova str., 736002, Khorog, Gbao, Tajikistan. *Correspondence e-mail: anzurat2003@mail.ru

The crystal structure of the title compound, C20H16BrN3O2, was determined using an inversion twin. Its asymmetric unit comprises two crystallographically independent molecules (A and B) being the stereoisomers. Both molecules are linked by pairs of N—H···O hydrogen bonds, forming a dimer with an R22(16) ring motif. The dimers are connected by further N—H···O and N—H···N hydrogen bonds, forming chains along the c-axis direction C—Br···π interactions between these chains contribute to the stabilization of the molecular packing. Hirshfeld surface analysis showed that the most important contributions to the crystal packing are from H···H, C···H/H···C, O···H/H···O, Br···H/H···Br and N···H/H···N interactions.

1. Chemical context

Nitrogen-based heterocycles are an important class of organic molecules that are used extensively in different branches of chemistry (Yadigarov et al., 2009; Abdelhamid et al., 2011; Magerramov et al., 2018; Yin et al., 2020; Khalilov et al., 2021). In particular, the synthesis of heterocyclic systems comprising a bioactive pyridine core with a broad spectrum of biological activities is noteworthy (Mamedov et al., 2020; Wojcicka & Redzicka, 2021). On the other hand, the pyridine ring is an essential part of diverse natural products, such as nicotinic acid, nicotinamide, vitamin B3 and diverse alkaloids (Aida et al., 2009). In the framework of our ongoing structural studies (Safarova 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, 5-acetyl-2-amino-4-(4-bromophenyl)-6-oxo-1-phenyl-1,4,5,6-tetrahydropyridine-3-carbonitrile.
2. Structural commentary

The title compound crystallizes in the monoclinic space group \( \text{Pc} \) with \( Z = 4 \), and with two molecules, \( A \) and \( B \), in the asymmetric unit (Fig. 1). These molecules are stereoisomers with an \( R,R \) absolute configurations 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 puckering parameters (Cremer & Pople, 1975) are \( \phi = 63.9 (11) ^\circ \), \( \psi = 148.9 (12) ^\circ \) in \( A \) and \( \phi = 115.1 (11) ^\circ \), \( \psi = 339.4 (12) ^\circ \) in \( B \)]. In molecule \( A \), the phenyl (C7–C12) and bromophenyl (C14–C19) rings form dihedral angles of 64.0 (4) and 86.3 (4)\(^\circ \), respectively, with the mean plane of the central dihydropyridine ring. In molecule \( B \), the corresponding dihedral angles are 77.2 (4) and 83.9 (4)\(^\circ \). The acetyl groups in both molecules are almost planar [largest deviations of 0.005 (8) and 0.035 (8) \( \text{Å} \) for atoms C1 (\( A \)) and C23 (\( B \)), respectively] and they make the dihedral angles of 89.5 (5) and 87.7 (5)\(^\circ \) with the mean planes of the dihydropyridine rings in these molecules.

3. Supramolecular features

Strong N6–H6\( \cdot \cdot \cdot \)O2 hydrogen bonds (Fig. 1, Table 1) link molecules \( A \) and \( B \) into dimers with an \( R_{2}^{2}(16) \) ring motif (Bernstein et al., 1995). These dimers are additionally stabilized by C\( \cdots \)O interactions [O21\( \cdots \)Cg2 = 3.620 (8) \( \text{Å} \), C21\( \cdots \)O21\( \cdots \)Cg2 = 110.8 (6)\(^\circ \), O1\( \cdots \)Cg5 = 3.748 (8) \( \text{Å} \), Cl\( \cdots \)O1\( \cdots \)Cg5 = 125.1 (6)\(^\circ \), where Cg2 and Cg5 are the centroids of the C7–C12 phenyl ring in molecule \( A \) and the C27–C32 phenyl ring in molecule \( B \), respectively]. The dimers are connected by N–H\( \cdot \cdot \cdot \)O and N–H\( \cdot \cdot \cdot \)N hydrogen bonds with an \( R_{3}^{2}(14) \) ring motif into chains along the \( c \)-axis direction (Table 1; Figs. 2, 3, 4 and 5). C–Br\( \cdot \cdot \cdot \)\( \pi \) interactions

![Figure 1](image1.png)

Asymmetric unit of the title compounds showing two crystallographically independent molecules, \( A \) and \( B \). Displacement ellipsoids are drawn at the 30% probability level. The intermolecular N–H\( \cdot \cdot \cdot \)O hydrogen bonds are drawn with dashed lines.

![Figure 2](image2.png)

A general view of the N–H\( \cdot \cdot \cdot \)O and N–H\( \cdot \cdot \cdot \)N hydrogen bonds in the structure of the title compound.

![Figure 3](image3.png)

The crystal packing of the title compound viewed down the \( a \) axis, showing chains running along the \( c \)-axis direction formed through N–H\( \cdot \cdot \cdot \)O and N–H\( \cdot \cdot \cdot \)N hydrogen bonds.
are also observed [Br1⋅⋅⋅Cg6v = 3.407 (4) Å, C17—
Br1⋅⋅⋅Cg6v = 145.2 (3); symmetry code (v) −1 + x, 1 − y,
−1/2 + z; Cg6 is the centroid of the C34–C39 ring]. Together with
the other intermolecular contacts given in Table 2, these
interactions contribute to the stabilization of the molecular
packing, forming a three-dimensional network (Figs. 6 and 7).

4. Hirshfeld surface analysis
To visualize the intermolecular interactions for both inde-
pendent molecules A and B, CrystalExplorer17 (Turner et al.,
2017) was used to generate Hirshfeld surfaces and corre-
sponding two-dimensional fingerprint plots. The \( d_{\text{norm}} \)
mappings were performed in the range of −0.6596 to 1.4042
arbitrary units for molecule A and −0.5436 to 1.4926 arbitrary
units for molecule B. Bright red circles on the \( d_{\text{norm}} \) surfaces

---

**Table 2**

| Contact         | Distance | Symmetry operation |
|-----------------|----------|--------------------|
| O2···H30        | 2.63     | \( x - 1, -y + 1, z - \frac{1}{2} \) |
| O1···H26A       | 1.99     | \( x, y, z \) |
| H13C···H16      | 2.46     | \( x + 1, y, z \) |
| O2···H6A        | 1.87     | \( x, -y + 1, z - \frac{1}{2} \) |
| H18···N40       | 2.46     | \( x, y - 1, z \) |
| N20···H26B      | 2.43     | \( x, -y + 1, z + \frac{1}{2} \) |
| C9···Br2        | 3.377 (10) | \( x - 1, -y + 2, z - \frac{1}{2} \) |
| H13C···O22      | 2.79     | \( x, -y + 1, z - \frac{1}{2} \) |
| C16···H36       | 2.86     | \( x - 1, y - 1, z \) |
| H11···H26A      | 2.47     | \( x - 1, y, z \) |
| O21···H31       | 2.84     | \( x - 1, y, z \) |
| H23···N40       | 2.47     | \( x, -y + 2, z + \frac{1}{2} \) |
| H31···O21       | 2.84     | \( x + 1, y, z \) |

---

Figure 4
The crystal packing of the title compound viewed down the \( b \) axis, showing chains running along the \( c \) axis formed through N—H···O and N—H···N hydrogen bonds.

Figure 5
The crystal packing of the title compound viewed down the \( c \) axis, with intermolecular N—H···O, C—H···N and N—H···N hydrogen bonds.

Figure 6
The C—Br···π and C=O···π interactions in the structure of the title compound viewed down the \( a \) axis.
(Fig. 8a,b,c,d) indicate regions of N—H···O interactions. The N—H···N and C—H···N interactions (Tables 1 and 2) also cause red spots on the Hirshfeld surfaces.

The fingerprint plots (Fig. 9) reveal that while the H···H interactions make the greatest contributions (Table 3), as would be expected for a molecule with such a predominance

| Contact                  | Contribution for A | Contribution for B |
|--------------------------|--------------------|--------------------|
| H···H                    | 32.8               | 33.8               |
| C···H/H···C              | 19.6               | 18.9               |
| O···H/H···O              | 17.2               | 13.5               |
| Br···H/H···Br            | 10.6               | 11.3               |
| N···H/H···N              | 9.4                | 14.0               |
| Br···C/C···Br            | 4.8                | 4.6                |
| N···O/O···N              | 2.1                | –                  |
| C···O/O···C              | 1.4                | 1.3                |
| Br···O/O···Br            | 0.8                | 0.9                |
| C···C                    | 0.7                | 0.7                |
| N···N                    | 0.5                | 0.4                |
| Br···N/N···Br            | 0.1                | 0.6                |

The di and de values are the closest internal and external distances (in Å) from given points on the Hirshfeld surfaces.
### Table 4

| Crystal data | Chemical formula | $M_r$ | Monoclinic, $Pc$ |
|--------------|------------------|------|-----------------|
| Temperature (K) | 100 | $a$, $b$, $c$ (Å) | 9.5889 (7), 13.2144 (10), 14.4529 (10) |
| $\beta$ (°) | 103.9395 (18) | $V$ ($\text{Å}^3$) | 1777.4 (2) |
| Radiation type | Mo | $Z$ | 4 |
| $\mu$ (mm$^{-1}$) | 2.33 | Crystal size (mm) | 0.05 × 0.04 × 0.03 |
| Data collection | Bruker D8 QUEST PHOTON-III CCD | |
| Absorption correction | Multi-scan (SADABS; Krause et al., 2015) | |
| $T_{	ext{min}}$, $T_{	ext{max}}$ | 0.818, 0.926 | No. of measured, independent and observed [$F^2 > 2\sigma(F^2)$] reflections | 34410, 10756, 5403 |
| $R_{	ext{free}}$ | 0.099 | $\theta_{\text{max}}$ ($\text{Å}^{-1}$) | 0.714 |
| Refinement | |
| $R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, $S$ | 0.065, 0.132, 0.98 | No. of parameters | 471 |
| No. of restraints | 2 | H-atom treatment | H-atom parameters constrained |
| H-atom treatment | |
| Δρ$_{\text{max}}$, Δρ$_{\text{min}}$ (e Å$^{-3}$) | 0.50, -0.66 | Absolute structure | Refined as an inversion twin |
| Absolute structure parameter | 0.473 (14) |

Computer programs: CrysAlis PRO (Rigaku OD, 2021), SHELXT (Sheldrick, 2015a), SHELXL (Sheldrick, 2015b), ORTEP-3 for Windows (Farrugia, 2012) and PLATON (Spek, 2020).

of H atoms, C···H···C, O···H···O, Br···H···O and N···H···N contacts are also substantial. Table 3 gives the contributions of the other, less significant contacts. The fact that the same type of interactions provide different contributions to the Hirshfeld surface for molecules $A$ and $B$ can be attributed to the different environments of these molecules in the crystalline state.

### 5. Database survey

A search of the Cambridge Structural Database (CSD, Version 5.42, update of September 2021; Groom et al., 2016) for the tetrahydropyridine unit gave 1340 hits, and some of which, namely OZAKOS (Naghiyev et al., 2021c), JEBREQ (Mohana et al., 2017), JEBRAM (Mohana et al., 2017), SETWUK (Suresh et al., 2007) and SETWOE (Suresh et al., 2007) closely resemble the title compound.

In OZAKOS (space group: $Pc$), the molecular conformation of the title compound is stabilized by an intramolecular O···H···O hydrogen bond, forming 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 both the related salts, JEBREQ (space group: $P\overline{1}$) and JEBRAM (space group: $P\overline{1}$), the N atom in the 1-position of the pyrimidine ring is protonated. In the hydrated salt JEBREQ, the presence of the water molecule prevents the formation of the familiar $R_2^2(8)$ ring motif. Instead, an expanded ring [i.e. $R_2^2(8)$] is formed involving the sulfonate group, the pyrimidinium cation and the water molecule. Both salts form a supramolecular homosynthon [$R_2^2(8)$ ring motif] through N···H···N hydrogen bonds. The molecular structures are further stabilized by π···π stacking, and C=O···π, C—H···Cl interactions. It appears that the protonation state of the pyrimidine ring influences the intermolecular interactions within the crystal lattice to a substantial extent. In JEBRAM, the protonated N atom and the amino group of the pyrimidinium cation interact with the carboxylate group of the anion through N···H···O hydrogen bonds, forming a heterosynthon with an $R_2^2(8)$ ring motif.

The polysubstituted pyridines, SETWUK (space group: $P_2_1/n$) and SETWOE (space group: $P_2_1/c$), adopt nearly planar structures. The crystal structure of SETWUK is stabilized by intermolecular C···H···F and C···H···π interactions. The C···H···F bond generates a linear chain with a C(14) motif. The crystal structure of SETWOE is stabilized by intermolecular C···H···O and C···H···π interactions. The C···H···O hydrogen bonds generate rings with $R_2^2(14)$ and $R_2^2(20)$ motifs. In addition, in SETWOE and SETWUK, intramolecular O···H···O interactions are found, which generate an S(6) graph-set motif. No significant aryl···aryl or π···π interactions exist in these structures. All this bears some resemblance to the title compound.

### 6. Synthesis and crystallization

To a solution of 2-(4-bromobenzylidene)malononitrile (1.19 g, 5.1 mmol) and acetoacetanilide (0.92 g, 5.2 mmol) in methanol (25 mL), piperidine (2–3 drops) was added and the mixture was stirred at room temperature for 48 h. Then 15 mL of methanol were removed by rotary evaporation from the reaction mixture, which was left overnight. The precipitated crystals were separated by filtration and recrystallized from ethanol/water (1:1) solution (yield 66%; m.p. 536–537 K).

$^1$H NMR (300 MHz, DMSO-d$_6$, m.h.): 2.29 (s, 3H, CH$_3$–C(O)); 4.15 (d, 1H, CH–Ar); 4.34 (d, 1H, CH–C(O)); 5.98 (s, 2H, NH$_2$); 7.12–7.35 (m, 5H, 5CH$_{ar}$); 7.40 (d, 2H, 2CH$_{ar}$); 7.61 (d, 2H, 2CH$_{ar}$).

$^{13}$C NMR (75 MHz, DMSO-d$_6$, m.h.): 27.86 (CH$_3$–C(O)); 37.94 (CH–Ar); 57.24 (CH–C(O)); 117.21 (CN); 121.25 (Br–Car); 127.67 (CH$_{ar}$); 128.19 (2CH$_{ar}$); 129.58 (2CH$_{ar}$); 130.15 (2CH$_{ar}$); 136.98 (C$_{ar}$); 140.37 (C$_{ar}$); 154.14 (C$_{quat}$); 166.20 (N–C(O)); 202.55 (C(O)).

### 7. Refinement details

Crystal data, data collection and structure refinement details are summarized in Table 4. All H atoms were positioned geometrically (N–H = 0.90 Å, C–H = 0.95–1.00 Å) and refined as riding with $U_{iso}$(H) = 1.2$U_{eq}$(C, N) or 1.5$U_{eq}$(C-methyl).
Acknowledgements

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

Funding information

This work was supported by the Baku State University, and RUDN University Strategic Academic Leadership Program.

References

Abdelhamid, A. A., Mohamed, S. K., Khalilov, A. N., Gurbanov, A. V. & Ng, S. W. (2011). Acta Cryst. E67, o744.
Aida, W., Ohtsuki, T., Li, X. & Ishibashi, M. (2009). Tetrahedron, 65, 369–373.
Bernstein, J., Davis, R. E., Shimoni, L. & Chang, N.-L. (1995). Angew. Chem. Int. Ed. Engl. 34, 1555–1573.
Cremer, D. & Pople, J. A. (1975). J. Am. Chem. Soc. 97, 1354–1358.
Farrugia, L. J. (2012). J. Appl. Cryst. 45, 849–854.
Groom, C. R., Bruno, I. J., Lightfoot, M. P. & Ward, S. C. (2016). Acta Cryst. B72, 171–179.
Khalilov, A. N., Tüzün, B., Taslimi, P., Tas, A., Tunbilek, Z. & Cakmak, N. K. (2021). J. Mol. Liq. 344, 117761.
Krause, L., Herbst-Irmer, R., Sheldrick, G. M. & Stalke, D. (2015). J. Appl. Cryst. 48, 3–10.
Magerramov, A. M., Naghiyev, F. N., Mamedova, G. Z., Asadov, Kh. A. & Mamedov, I. G. (2018). Russ. J. Org. Chem. 54, 1731–1734.
Maharramov, A. M., Shikhaliyev, N. G., Zeynalli, N. R., Niyazova, A. A., Garazade, Kh. A. & Shikhaliyeva, I. M. (2021). UNEC J. Engineer. Appl. Sci. 1, 5-11.
Mamedov, I., Naghiyev, F., Maharramov, A., Uwangue, O., Farewell, A., Sunnerhagen, P. & Erdelyi, M. (2020). Mendeleev Commun. 30, 498–499.
Mohana, M., Thomas Muthiah, P. & Butcher, R. J. (2017). Acta Cryst. C73, 536–540.
Naghiyev, F. N., Cisterna, J., Khalilov, A. N., Maharramov, A. M., Askerov, R. K., Asadov, K. A., Mamedov, I. G., Salmanli, K. S., Cárdenas, A. & Brito, I. (2020). Molecules, 25, 2235–2248.
Naghiyev, F. N., Grishina, M. M., Khrustalev, V. N., Khalilov, A. N., Akkurt, M., Akobirshoeva, A. A. & Mamedov, I. G. (2021a). Acta Cryst. E77, 195–199.
Naghiyev, F. N., Pavlova, A. V., Khrustalev, V. N., Akkurt, M., Khalilov, A. N., Akobirshoeva, A. A. & Mamedov, I. G. (2021c). Acta Cryst. E77, 930–934.
Naghiyev, F. N., Tereshina, T. A., Khrustalev, V. N., Akkurt, M., Khalilov, A. N., Akobirshoeva, A. A. & Mamedov, I. G. (2021b). Acta Cryst. E77, 512–515.
Rigaku OD (2021). CrysAlis PRO. Rigaku Oxford Diffraction, Yarnton, England.
Safavora, A. S., Brito, I., Cisterna, J., Cárdenas, A., Huseynov, E. Z., Khalilov, A. N., Naghiyev, F. N., Askerov, R. K. & Maharramov, A. M. Z. (2019). Z. Kristallogr. New Cryst. Struct. 234, 1183–1185.
Sheldrick, G. M. (2015a). Acta Cryst. A71, 3–8.
Sheldrick, G. M. (2015b). Acta Cryst. C71, 3–8.
Spek, A. L. (2020). Acta Cryst. E76, 1–11.
Suresh, J., Suresh Kumar, R., Perumal, S., Mostad, A. & Natarajan, S. (2007). Acta Cryst. C63, o141–o144.
Turner, M. J., McKinnon, J. J., Wolff, S. K., Grimwood, D. J., Spackman, P. R., Jayatilaka, D. & Spackman, M. A. (2017). CrystalExplorer17. The University of Western Australia.
Wojcicka, A. & Redzicka, A. (2021). Pharmaceuticals, 14, 354.
Yadigarov, R. R., Khalilov, A. N., Mamedov, I. G., Nagiev, F. N., Magerramov, A. M. & Allakhverdiev, M. A. (2009). Russ. J. Org. Chem. 45, 1856–1858.
Yin, J., Khalilov, A. N., Muthupandi, P., Ladd, R. & Birman, V. B. (2020). J. Am. Chem. Soc. 142, 60–63.
Crystal structure and Hirshfeld surface analysis of 5-acetyl-2-amino-4-(4-bromophenyl)-6-oxo-1-phenyl-1,4,5,6-tetrahydropyridine-3-carbonitrile

Ibrahim G. Mamedov, Victor N. Khrustalev, Mehmet Akkurt, Anton P. Novikov, Ayten R. Asgarova, Khatira N. Aliyeva and Anzurat A. Akobirshoeva

Computing details

Data collection: CrysAlis PRO (Rigaku OD, 2021); cell refinement: CrysAlis PRO (Rigaku OD, 2021); data reduction: CrysAlis PRO (Rigaku OD, 2021); 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).

5-Acetyl-2-amino-4-(4-bromophenyl)-6-oxo-1-phenyl-1,4,5,6-tetrahydropyridine-3-carbonitrile

Crystal data

C_{20}H_{16}BrN_{3}O_{2}

Mr = 410.26

Monoclinic, P2₁

a = 9.5889 (7) Å
b = 13.2144 (10) Å
c = 14.4529 (10) Å
β = 103.9395 (18)°
V = 1777.4 (2) Å³
Z = 4

F(000) = 832
Dc = 1.533 Mg m⁻³
Mo Kα radiation, λ = 0.71073 Å
Cell parameters from 3126 reflections
θ = 2.7–24.0°
µ = 2.33 mm⁻¹
T = 100 K
Prism, colourless
0.05 × 0.04 × 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)
Tmin = 0.818, Tmax = 0.926
34410 measured reflections
10756 independent reflections
5403 reflections with I > 2σ(I)
Rint = 0.099
θmax = 30.5°, θmin = 2.1°
h = −13→13
k = −18→18
l = −20→20

Refinement

Refinement on F²
Least-squares matrix: full
R[F² > 2σ(F²)] = 0.065
wR(F²) = 0.132
S = 0.98
10756 reflections
471 parameters
2 restraints

Primary atom site location: difference Fourier map
Secondary atom site location: difference Fourier map
Hydrogen site location: mixed
H-atom parameters constrained
w = 1/[σ²(Fo²) + (0.0401P)^2]
where P = (Fo² + 2Fc²)/3
(Δ/σ)max < 0.001
\[ \Delta \rho_{\text{max}} = 0.50 \text{ e Å}^{-3} \]
\[ \Delta \rho_{\text{min}} = -0.66 \text{ e Å}^{-3} \]
Extinction correction: SHELXL,
\[ F_c^* = kF_c[1 + 0.001xF_c^2/\sin(2\theta)]^{1/4} \]
Extinction coefficient: 0.0039 (3)
Absolute structure: Refined as an inversion twin
Absolute structure parameter: 0.473 (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.

**Refinement.** Refined as a two-component inversion twin.

**Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)**

| Br1  | x       | y       | z       | Ueq   |
|------|---------|---------|---------|-------|
| Br1  | -0.05184 (9) | 0.02892 (6) | 0.27044 (7) | 0.0328 (2) |
| N1   | 0.3304 (7) | 0.5094 (5) | 0.4197 (5) | 0.0194 (15) |
| C1   | 0.6385 (9) | 0.4260 (6) | 0.3992 (6) | 0.0216 (19) |
| O1   | 0.6581 (7) | 0.5145 (5) | 0.4155 (5) | 0.0456 (19) |
| C2   | 0.3768 (9) | 0.4655 (6) | 0.3455 (6) | 0.0219 (18) |
| O2   | 0.3284 (7) | 0.4954 (4) | 0.2642 (4) | 0.0273 (14) |
| C3   | 0.4864 (9) | 0.3816 (6) | 0.3708 (6) | 0.0211 (18) |
| H3   | 0.4792 | 0.3389 | 0.3127 | 0.025* |
| C4   | 0.4547 (9) | 0.3140 (6) | 0.4498 (6) | 0.0221 (19) |
| H4   | 0.5405 | 0.2699 | 0.4736 | 0.026* |
| C5   | 0.4402 (9) | 0.3820 (6) | 0.5310 (6) | 0.0229 (19) |
| C6   | 0.3781 (9) | 0.4741 (6) | 0.5141 (6) | 0.0213 (18) |
| N6   | 0.3596 (7) | 0.5398 (5) | 0.5816 (5) | 0.0219 (15) |
| H6A  | 0.3529 | 0.5257 | 0.6413 | 0.026* |
| H6B  | 0.3534 | 0.6055 | 0.5646 | 0.026* |
| C7   | 0.2213 (9) | 0.5898 (6) | 0.3983 (6) | 0.0212 (18) |
| C8   | 0.2595 (10) | 0.6844 (7) | 0.3737 (7) | 0.031 (2) |
| H8   | 0.3538 | 0.6970 | 0.3663 | 0.037* |
| C9   | 0.1581 (10) | 0.7610 (7) | 0.3600 (7) | 0.036 (2) |
| H9   | 0.1814 | 0.8267 | 0.3416 | 0.044* |
| C10  | 0.0223 (11) | 0.7409 (8) | 0.3732 (7) | 0.036 (2) |
| H10  | -0.0463 | 0.7940 | 0.3661 | 0.044* |
| C11  | -0.0144 (11) | 0.6464 (7) | 0.3963 (6) | 0.035 (2) |
| H11  | -0.1089 | 0.6334 | 0.4031 | 0.042* |
| C12  | 0.0861 (10) | 0.5691 (7) | 0.4099 (6) | 0.030 (2) |
| H12  | 0.0620 | 0.5030 | 0.4269 | 0.036* |
| C13  | 0.7611 (10) | 0.3551 (6) | 0.4072 (7) | 0.034 (2) |
| H13A | 0.7314 | 0.2868 | 0.4207 | 0.050* |
| H13B | 0.8413 | 0.3770 | 0.4591 | 0.050* |
| H13C | 0.7915 | 0.3546 | 0.3472 | 0.050* |
| C14  | 0.3267 (9) | 0.2446 (6) | 0.4069 (6) | 0.0213 (17) |
| C15  | 0.1891 (10) | 0.2670 (6) | 0.4115 (7) | 0.029 (2) |
| H15  | 0.1720 | 0.3262 | 0.4445 | 0.034* |
| C16  | 0.0740 (10) | 0.2057 (6) | 0.3695 (6) | 0.029 (2) |
H16  -0.0213  0.2229  0.3717  0.034*
C17  0.1025 (10)  0.1185 (6)  0.3240 (6)  0.026 (2)
C18  0.2390 (10)  0.0940 (6)  0.3159 (7)  0.029 (2)
H18  0.2561  0.0350  0.2828  0.035*
C19  0.3494 (10)  0.1581 (6)  0.3576 (6)  0.026 (2)
H19  0.4441  0.1428  0.3525  0.031*
C20  0.4925 (10)  0.3499 (6)  0.6274 (7)  0.024 (2)
N20  0.5390 (9)  0.3255 (6)  0.7052 (5)  0.0335 (19)
Br2  1.05193 (11)  1.25495 (8)  0.62011 (8)  0.0409 (3)
N2  0.6616 (7)  0.7734 (5)  0.5811 (5)  0.0220 (16)
C21  0.3521 (10)  0.8636 (7)  0.5959 (6)  0.028 (2)
O21  0.3329 (8)  0.7728 (5)  0.6051 (5)  0.0403 (18)
C22  0.6085 (10)  0.8245 (6)  0.6505 (6)  0.027 (2)
O22  0.6450 (7)  0.8015 (4)  0.7343 (4)  0.0305 (15)
C23  0.5025 (9)  0.9077 (6)  0.6148 (6)  0.0238 (19)
H23  0.5119  0.9588  0.6670  0.029*
C24  0.5288 (9)  0.9623 (6)  0.5266 (6)  0.0226 (19)
H24  0.4424  1.0048  0.4993  0.027*
C25  0.5389 (9)  0.8820 (6)  0.4542 (6)  0.0230 (19)
C26  0.6117 (9)  0.7941 (6)  0.4838 (6)  0.0210 (18)
N26  0.6426 (8)  0.7245 (5)  0.4234 (5)  0.0279 (17)
H26A  0.6602  0.6614  0.4466  0.034*
H26B  0.6454  0.7409  0.3634  0.034*
C27  0.7687 (10)  0.6958 (6)  0.6136 (6)  0.0234 (19)
C28  0.7276 (10)  0.6009 (6)  0.6383 (7)  0.030 (2)
H28  0.6293  0.5858  0.6336  0.036*
C29  0.8324 (11)  0.5287 (7)  0.6698 (7)  0.040 (3)
H29  0.8056  0.4632  0.6865  0.048*
C30  0.9761 (11)  0.5507 (6)  0.6776 (7)  0.033 (2)
H30  1.0476  0.5007  0.6998  0.040*
C31  1.0139 (11)  0.6444 (7)  0.6530 (7)  0.035 (2)
H31  1.1123  0.6590  0.6575  0.042*
C32  0.9120 (10)  0.7187 (6)  0.6216 (6)  0.028 (2)
H32  0.9398  0.7842  0.6059  0.034*
C33  0.2270 (11)  0.9361 (7)  0.5651 (8)  0.041 (3)
H33A  0.2541  1.0027  0.5936  0.061*
H33B  0.1440  0.9103  0.5862  0.061*
H33C  0.2021  0.9422  0.4955  0.061*
C34  0.6606 (8)  1.0327 (6)  0.5511 (6)  0.0188 (17)
C35  0.7743 (10)  1.0222 (7)  0.5076 (6)  0.028 (2)
H35  0.7720  0.9690  0.4630  0.034*
C36  0.8913 (10)  1.0875 (7)  0.5276 (7)  0.029 (2)
H36  0.9687  1.0787  0.4980  0.035*
C37  0.8931 (10)  1.1659 (6)  0.5917 (6)  0.026 (2)
C38  0.7802 (10)  1.1788 (6)  0.6350 (6)  0.027 (2)
H38  0.7820  1.2325  0.6789  0.033*
C39  0.6654 (10)  1.1133 (6)  0.6139 (6)  0.025 (2)
H39  0.5874  1.1232  0.6429  0.030*
C40  |  0.4888 (10) |  0.8997 (6) |  0.3553 (7) |  0.025 (2)  
N40  |  0.4482 (10) |  0.9142 (5) |  0.2749 (6) |  0.0307 (16)

Atomic displacement parameters (Å²)

|    | U₁₁ | U₂₂ | U₃₃ | U₁₂ | U₁₃ | U₂₃ |
|----|-----|-----|-----|-----|-----|-----|
| Br1| 0.0341 (5) | 0.0228 (4) | 0.0388 (5) | -0.0033 (5) | 0.0036 (4) | -0.0037 (5) |
| N1 | 0.027 (4) | 0.020 (3) | 0.012 (3) | 0.005 (3) | 0.007 (3) | 0.002 (3) |
| C1 | 0.027 (5) | 0.016 (4) | 0.025 (5) | 0.002 (4) | 0.013 (4) | -0.003 (3) |
| O1 | 0.030 (4) | 0.026 (4) | 0.081 (5) | -0.002 (3) | 0.013 (4) | -0.012 (4) |
| C2 | 0.025 (5) | 0.017 (4) | 0.026 (5) | -0.001 (4) | 0.013 (4) | -0.005 (4) |
| O2 | 0.035 (4) | 0.025 (3) | 0.021 (3) | -0.001 (3) | 0.006 (3) | 0.004 (3) |
| C3 | 0.028 (5) | 0.018 (4) | 0.018 (4) | 0.001 (4) | 0.006 (4) | -0.005 (3) |
| C4 | 0.031 (5) | 0.019 (4) | 0.015 (4) | -0.002 (4) | 0.004 (4) | 0.001 (3) |
| C5 | 0.031 (5) | 0.021 (4) | 0.018 (4) | -0.001 (4) | 0.008 (4) | 0.001 (3) |
| C6 | 0.026 (5) | 0.020 (4) | 0.020 (4) | -0.004 (4) | 0.010 (4) | -0.006 (4) |
| N6 | 0.031 (4) | 0.021 (4) | 0.015 (4) | -0.003 (3) | 0.008 (3) | -0.001 (3) |
| C7 | 0.024 (5) | 0.020 (4) | 0.019 (4) | 0.004 (3) | 0.003 (4) | 0.000 (3) |
| C8 | 0.027 (5) | 0.023 (4) | 0.043 (6) | -0.001 (4) | 0.010 (5) | 0.002 (4) |
| C9 | 0.039 (6) | 0.031 (5) | 0.035 (6) | 0.012 (5) | 0.000 (5) | 0.003 (4) |
| C10| 0.036 (6) | 0.042 (6) | 0.030 (6) | 0.014 (5) | 0.006 (5) | -0.005 (5) |
| C11| 0.039 (6) | 0.033 (5) | 0.037 (6) | 0.010 (5) | 0.017 (5) | 0.000 (4) |
| C12| 0.026 (5) | 0.030 (5) | 0.033 (5) | 0.002 (4) | 0.008 (4) | 0.005 (4) |
| C13| 0.029 (5) | 0.028 (5) | 0.044 (6) | -0.001 (4) | 0.010 (5) | -0.002 (4) |
| C14| 0.024 (5) | 0.019 (4) | 0.021 (4) | 0.000 (4) | 0.006 (4) | 0.002 (4) |
| C15| 0.041 (6) | 0.015 (4) | 0.032 (5) | -0.002 (4) | 0.012 (5) | 0.000 (4) |
| C16| 0.031 (5) | 0.018 (4) | 0.040 (6) | -0.003 (4) | 0.014 (5) | -0.006 (4) |
| C17| 0.027 (5) | 0.025 (5) | 0.023 (5) | -0.008 (4) | 0.001 (4) | 0.002 (4) |
| C18| 0.031 (5) | 0.020 (4) | 0.037 (6) | 0.002 (4) | 0.008 (5) | -0.003 (4) |
| C19| 0.022 (5) | 0.030 (5) | 0.029 (5) | -0.001 (4) | 0.010 (4) | -0.007 (4) |
| C20| 0.032 (5) | 0.015 (4) | 0.024 (5) | 0.002 (4) | 0.006 (4) | -0.001 (4) |
| N20| 0.045 (5) | 0.031 (4) | 0.025 (5) | 0.011 (4) | 0.009 (4) | 0.001 (3) |
| Br2| 0.0417 (6) | 0.0460 (6) | 0.0350 (5) | -0.0143 (5) | 0.0092 (4) | -0.0033 (5) |
| N2 | 0.020 (4) | 0.017 (4) | 0.026 (4) | 0.006 (3) | 0.000 (3) | 0.002 (3) |
| C21| 0.035 (6) | 0.027 (5) | 0.023 (5) | 0.004 (4) | 0.008 (4) | 0.000 (4) |
| O21| 0.051 (5) | 0.026 (4) | 0.048 (5) | -0.004 (3) | 0.021 (4) | 0.000 (3) |
| C22| 0.038 (6) | 0.016 (4) | 0.025 (5) | -0.003 (4) | 0.005 (4) | 0.001 (4) |
| O22| 0.044 (4) | 0.025 (3) | 0.023 (3) | 0.003 (3) | 0.008 (3) | 0.003 (3) |
| C23| 0.027 (5) | 0.018 (4) | 0.029 (5) | 0.002 (4) | 0.012 (4) | -0.003 (4) |
| C24| 0.029 (5) | 0.013 (4) | 0.025 (5) | 0.003 (4) | 0.007 (4) | -0.001 (3) |
| C25| 0.031 (5) | 0.021 (4) | 0.019 (4) | 0.004 (4) | 0.010 (4) | 0.001 (4) |
| C26| 0.028 (5) | 0.013 (4) | 0.023 (5) | 0.001 (3) | 0.007 (4) | -0.004 (3) |
| N26| 0.034 (4) | 0.022 (4) | 0.030 (4) | 0.006 (3) | 0.011 (4) | 0.000 (3) |
| C27| 0.031 (5) | 0.018 (4) | 0.022 (5) | 0.008 (4) | 0.007 (4) | -0.001 (3) |
| C28| 0.026 (5) | 0.019 (4) | 0.040 (6) | -0.002 (4) | 0.003 (4) | 0.006 (4) |
| C29| 0.041 (6) | 0.023 (5) | 0.050 (6) | 0.004 (5) | -0.001 (5) | 0.012 (5) |
| C30| 0.039 (6) | 0.023 (5) | 0.038 (6) | 0.011 (4) | 0.009 (5) | -0.001 (4) |
| C31| 0.033 (6) | 0.038 (6) | 0.034 (6) | 0.005 (4) | 0.005 (5) | -0.003 (4) |
C32  0.035 (5)  0.022 (4)  0.028 (5)  0.006 (4)  0.009 (4)  0.002 (4)
C33  0.039 (6)  0.035 (5)  0.050 (7)  0.006 (5)  0.015 (5)  0.004 (5)
C34  0.017 (4)  0.018 (4)  0.020 (4)  0.003 (3)  0.003 (3)  0.005 (3)
C35  0.037 (6)  0.022 (5)  0.025 (5)  0.003 (4)  0.009 (4)  0.001 (4)
C36  0.028 (5)  0.031 (5)  0.030 (5)  0.003 (4)  0.010 (4)  0.001 (4)
C37  0.031 (5)  0.020 (4)  0.026 (5)  −0.004 (4)  0.003 (4)  0.003 (4)
C38  0.039 (6)  0.024 (4)  0.018 (5)  0.000 (4)  0.005 (4)  0.000 (4)
C39  0.031 (5)  0.017 (4)  0.025 (5)  0.004 (4)  0.004 (4)  0.002 (4)
C40  0.036 (5)  0.013 (4)  0.028 (5)  0.008 (4)  0.012 (4)  0.000 (4)
N40  0.039 (4)  0.027 (4)  0.030 (4)  0.010 (4)  0.014 (3)  0.008 (4)

| Geometric parameters (Å, °)       |
|-----------------------------------|
| Br1—C17: 1.908 (9)                |
| N1—C2: 1.383 (10)                 |
| N1—C6: 1.410 (10)                 |
| N1—C7: 1.470 (10)                 |
| C1—C13: 1.486 (12)                |
| C1—C3: 1.533 (12)                 |
| C2—C3: 1.511 (11)                 |
| C3—C4: 1.537 (11)                 |
| C3—H3: 1.0000                     |
| C4—C5: 1.510 (11)                 |
| C4—C14: 1.538 (11)                |
| C4—H4: 1.0000                     |
| C5—C6: 1.351 (11)                 |
| C5—C20: 1.428 (12)                |
| C6—N6: 1.349 (10)                 |
| N6—H6A: 0.8999                    |
| N6—H6B: 0.9000                    |
| C7—C8: 1.374 (11)                 |
| C7—C12: 1.374 (12)                |
| C8—C9: 1.385 (12)                 |
| C8—H8: 0.9500                     |
| C9—C10: 1.387 (13)                |
| C9—H9: 0.9500                     |
| C10—C11: 1.361 (13)               |
| C10—H10: 0.9500                   |
| C11—C12: 1.386 (12)               |
| C11—H11: 0.9500                   |
| C12—H12: 0.9500                   |
| C13—H13A: 0.9800                  |
| C13—H13B: 0.9800                  |
| C13—H13C: 0.9800                  |
| C14—C15: 1.370 (12)               |
| C14—C19: 1.391 (11)               |

Acta Cryst. (2022). E78, 291-296
| Bond          | Distance (Å) | Bond          | Distance (Å) | Bond          | Distance (Å) |
|---------------|--------------|---------------|--------------|---------------|--------------|
| C15—C16       | 1.386 (12)   | C35—C36       | 1.389 (13)   | C15—H15      | 0.9500       |
| C15—H15      | 0.9500       | C36—H35       | 0.9500       | C16—C17      | 1.386 (12)   |
| C16—C17      | 1.386 (12)   | C36—C37       | 1.386 (12)   | C16—H16      | 0.9500       |
| C16—H16      | 0.9500       | C37—C38       | 1.385 (12)   | C17—C18      | 1.380 (12)   |
| C17—C18      | 1.380 (12)   | C38—C39       | 1.375 (12)   | C18—C19      | 1.376 (12)   |
| C18—C19      | 1.376 (12)   | C38—H38       | 0.9500       | C18—H18      | 0.9500       |
| C19—C19      | 0.9500       | C39—C39       | 0.9500       | C20—N20      | 1.151 (11)   |
| C20—N20      | 1.151 (11)   | C40—C40       | 1.149 (11)   |               |              |
| C2—N1—C6     | 121.4 (7)    | C26—N2—C22    | 121.9 (7)    | C2—N1—C7     | 119.0 (7)    |
| C2—N1—C7     | 119.0 (7)    | C26—N2—C27    | 120.6 (7)    | C6—N1—C7     | 119.4 (7)    |
| O1—C1—C13    | 121.1 (8)    | O21—C21—C33   | 121.4 (9)    | O1—C1—C3     | 121.3 (8)    |
| O1—C1—C3     | 121.3 (8)    | O21—C21—C23   | 121.1 (8)    | C13—C1—C3    | 117.6 (7)    |
| O2—C2—N1     | 119.5 (7)    | O22—C22—N2    | 121.6 (8)    | O2—C2—C3     | 123.4 (7)    |
| O2—C2—C3     | 123.4 (7)    | O22—C22—C23   | 122.3 (8)    | N1—C2—C3     | 117.1 (7)    |
| C2—C3—C1     | 110.2 (6)    | C22—C23—C21   | 108.4 (7)    | C2—C3—C4     | 110.8 (7)    |
| C2—C3—C4     | 110.8 (7)    | C22—C23—C24   | 113.3 (7)    | C1—C3—C4     | 111.6 (7)    |
| C1—C3—H3     | 108.0        | C21—C23—H23   | 107.8        | C2—C3—H3     | 108.0        |
| C1—C3—H3     | 108.0        | C21—C23—H23   | 107.8        | C4—C3—H3     | 108.0        |
| C5—C4—C3     | 107.7 (6)    | C25—C24—C23   | 107.2 (6)    | C5—C4—C3     | 107.5        |
| C5—C4—C14    | 117.0 (7)    | C25—C24—C34   | 113.3 (7)    | C3—C4—C14    | 109.2 (7)    |
| C3—C4—C14    | 109.2 (7)    | C23—C24—C34   | 112.5 (7)    | C5—C4—H4     | 107.5        |
| C3—C4—H4     | 107.5        | C23—C24—H24   | 107.8        | C4—C3—H3     | 108.0        |
| C14—C4—H4    | 107.5        | C34—C24—H24   | 107.8        | C5—C4—H4     | 107.5        |
| C6—C5—C20    | 118.7 (8)    | C26—C25—C40   | 118.6 (8)    | C6—C5—C4     | 120.9 (8)    |
| C6—C5—C4     | 120.9 (8)    | C26—C25—C24   | 119.5 (7)    | C20—C5—C4    | 120.4 (7)    |
| N6—C6—C5     | 125.3 (8)    | N26—C26—C25   | 123.4 (8)    | N6—C6—N1     | 114.7 (7)    |
| N6—C6—N1     | 114.7 (7)    | N26—C26—N2    | 116.2 (7)    | C5—C6—N1     | 119.9 (7)    |
| C6—N6—H6A    | 127.7        | C26—N26—H26A  | 116.2        | C6—N6—H6B    | 115.8        |
| H6A—N6—H6B   | 116.5        | H26A—N26—H26B | 122.3        | C8—C7—C12    | 122.1 (8)    |
| C8—C7—C12    | 122.1 (8)    | C32—C27—C28   | 120.9 (8)    | C8—C7—N1     | 119.4 (8)    |
| C12—C7—N1    | 118.4 (7)    | C28—C27—N2    | 120.0 (8)    | C12—C7—N1    | 118.4 (7)    |
| H7—C8—H8     | 118.7 (9)    | C29—C28—C27   | 118.7 (9)    | C7—C8—H8     | 120.6        |
| C9—C8—H8     | 120.6        | C27—C28—H28   | 120.6        | C9—C8—H8     | 120.6        |
| C8—C9—C10    | 119.4 (9)    | C28—C29—C30   | 120.8 (9)    | C8—C9—H9     | 120.3        |
| C8—C9—H9     | 120.3        | C28—C29—H29   | 119.6        |               |              |
C10—C9—H9 120.3  C30—C29—H29 119.6
C11—C10—C9 121.1 (9)  C31—C30—C29 119.4 (9)
C11—C10—H10 119.5  C31—C30—H30 120.3
C9—C10—H10 119.5  C29—C30—H30 120.3
C10—C11—C12 120.0 (10)  C30—C31—C32 121.3 (9)
C10—C11—H11 120.0  C30—C31—H31 119.3
C12—C11—H11 120.0  C32—C31—H31 119.3
C7—C12—C11 118.7 (9)  C31—C32—C27 118.8 (8)
C7—C12—H12 120.7  C31—C32—H32 120.6
C11—C12—H12 120.7  C27—C32—H32 120.6
C1—C13—H13A 109.5  C21—C33—H33A 109.5
C1—C13—H13B 109.5  C21—C33—H33B 109.5
H13A—C13—H13B 109.5  H33A—C33—H33B 109.5
C1—C13—H13C 109.5  C21—C33—H33C 109.5
H13A—C13—H13C 109.5  H33A—C33—H33C 109.5
C15—C14—C19 118.3 (8)  C35—C34—C39 117.7 (8)
C15—C14—C4 122.4 (7)  C35—C34—C24 121.7 (7)
C19—C14—C4 119.3 (7)  C39—C34—C24 120.5 (7)
C14—C15—C16 121.8 (8)  C36—C35—C34 121.7 (8)
C14—C15—H15 119.1  C36—C35—H35 119.2
C16—C15—H15 119.1  C34—C35—H35 119.2
C15—C16—C17 117.8 (9)  C37—C36—C35 118.8 (9)
C15—C16—H16 121.1  C37—C36—H36 120.6
C17—C16—H16 121.1  C35—C36—H36 120.6
C18—C17—C16 122.3 (8)  C38—C37—C36 120.6 (9)
C18—C17—Br1 118.6 (7)  C38—C37—Br2 120.1 (7)
C16—C17—Br1 119.1 (7)  C36—C37—Br2 119.3 (7)
C19—C18—C17 117.6 (8)  C39—C38—C37 119.5 (8)
C19—C18—H18 121.2  C39—C38—H38 120.2
C17—C18—H18 121.2  C37—C38—H38 120.2
C18—C19—C14 122.1 (8)  C38—C39—C34 121.7 (8)
C18—C19—H19 119.0  C38—C39—H39 119.2
C14—C19—H19 119.0  C34—C39—H39 119.2
N20—C20—C5 177.7 (10)  N40—C40—C25 180.0 (14)
N6—N1—C2—O2 176.9 (8)  C26—N2—C22—O2 175.0 (8)
N7—N1—C2—O2 13.1 (11)  C27—N2—C22—C23 −3.7 (12)
C6—N1—C2—C3 −3.0 (11)  C26—N2—C22—C23 −4.0 (11)
C7—N1—C2—C3 −178.6 (7)  C27—N2—C22—C23 177.3 (7)
O2—C2—C3—C1 94.4 (10)  O22—C22—C23—C21 −86.3 (10)
N1—C2—C3—C1 −85.7 (9)  N2—C22—C23—C21 92.7 (9)
O2—C2—C3—C4 −141.5 (8)  O22—C22—C23—C24 149.1 (8)
N1—C2—C3—C4 38.4 (10)  N2—C22—C23—C24 −3.19 (10)
O1—C1—C3—C2 13.1 (12)  O21—C21—C23—C22 −3.8 (12)
C13—C1—C3—C2 −167.8 (7)  C33—C21—C23—C22 176.2 (8)
O1—C1—C3—C4 −110.6 (10)  O21—C21—C23—C24 121.7 (9)
C13—C1—C3—C4 68.5 (10)  C33—C21—C23—C24 −58.3 (10)
C2—C3—C4—C5  −52.2 (9)  C22—C23—C24—C25  51.6 (9)
C1—C3—C4—C5  71.1 (9)   C21—C23—C24—C25 −71.1 (8)
C2—C3—C4—C14  75.8 (8)  C22—C23—C24—C34 −73.7 (9)
C1—C3—C4—C14 −160.9 (7) C21—C23—C24—C34 163.5 (7)
C3—C4—C5—C6  36.6 (11) C23—C24—C25—C26 83.9 (9)
C14—C4—C5—C6 −86.8 (10) C23—C24—C25—C26 −40.8 (10)
C3—C4—C5—C20 −143.3 (8) C23—C24—C25—C40 146.4 (8)
C14—C4—C5—C20 93.4 (10) C23—C24—C25—C40 −88.9 (10)
C20—C5—C6—N6 −0.5 (14) C40—C25—C26—N26 2.2 (13)
C4—C5—C6—N6 179.6 (8) C24—C25—C26—N26 −170.8 (8)
C20—C5—C6—N1 177.6 (8) C40—C25—C26—N2 −179.3 (8)
C4—C5—C6—N1 −2.3 (13) C24—C25—C26—N2 7.7 (12)
C2—N1—C6—N6 161.5 (7) C22—N2—C26—N26 −163.9 (7)
C7—N1—C6—N6 −22.9 (10) C27—N2—C26—N26 14.7 (11)
C2—N1—C6—C5 −16.8 (12) C22—N2—C26—C25 17.5 (12)
C7—N1—C6—C5 158.8 (8) C27—N2—C26—C25 −163.9 (8)
C2—N1—C7—C8 −73.8 (10) C22—N2—C26—C34 81.3 (10)
C6—N1—C7—C8 110.5 (9) C22—N2—C26—C34 −100.0 (9)
C2—N1—C7—C12 110.5 (9) C26—N2—C27—C28 −100.2 (10)
C6—N1—C7—C12 −65.2 (10) C22—N2—C27—C28 78.4 (10)
C12—C7—C8—C9 −0.2 (14) C32—C27—C28—C29 −1.0 (14)
N1—C7—C8—C9 −175.7 (8) N2—C27—C28—C29 −179.4 (8)
C7—C8—C9—C10 1.3 (14) C27—C28—C29—C30 0.6 (15)
C8—C7—C12—C11 2.0 (15) C28—C29—C30—C31 0.8 (15)
C8—C7—C12—C11 −65.2 (10) C29—C30—C31—C32 −1.2 (14)
C10—C11—C12—C7 −0.9 (14) C29—C30—C31—C32 1.3 (13)
C9—C10—C11—C12 −97.2 (9) C30—C31—C32—C33 1.9 (13)
C5—C4—C14—C15 25.4 (12) C32—C33—C34—C35 1.3 (11)
C3—C4—C14—C15 −97.2 (9) C32—C33—C34—C35 123.2 (8)
C5—C4—C14—C19 −157.7 (8) C32—C33—C34—C39 177.4 (7)
C3—C4—C14—C19 79.7 (9) C32—C33—C34—C39 −60.7 (10)
C19—C14—C15—C16 0.6 (13) C32—C33—C34—C39 1.9 (13)
C4—C14—C15—C16 177.5 (8) C32—C33—C34—C39 178.1 (8)
C14—C15—C16—C17 1.6 (13) C34—C35—C36—C37 0.9 (14)
C15—C16—C17—Br1 −2.9 (14) C34—C35—C36—C37 −0.9 (14)
C15—C16—C17—Br1 177.1 (7) C35—C36—C37—Br2 179.7 (7)
C16—C17—C18—C19 1.9 (14) C36—C37—C38—C39 −0.1 (13)
Br1—C17—C18—C19 −178.0 (7) Br2—C37—C38—C39 −179.8 (6)
C17—C18—C19—C14 0.4 (14) C37—C38—C39—C40 1.2 (13)
C15—C14—C19—C18 −1.6 (13) C35—C34—C39—C38 −2.0 (12)
C4—C14—C19—C18 −178.7 (8) C24—C34—C39—C38 −178.3 (8)

**Hydrogen-bond geometry (Å, °)**

| D—H···A       | D—H  | H···A  | D···A    | D—H···A |
|---------------|------|-------|---------|---------|
| N6—H6A···O2i  | 0.90 | 1.87  | 2.766 (9) | 175     |
| N6—H6B···O21  | 0.90 | 2.31  | 3.115 (9) | 149     |

*Acta Cryst. (2022). E78, 291-296* sup-8
|                  | d     | r     | D     | θ     |
|------------------|-------|-------|-------|-------|
| C18—H18···N40<sup>ii</sup> | 0.95  | 2.46  | 3.256 (12) | 141   |
| C23—H23···N40<sup>iii</sup> | 1.00  | 2.47  | 3.426 (11) | 161   |
| N26—H26<sup>a</sup>···O1    | 0.90  | 1.99  | 2.784 (9)  | 146   |
| N26—H26<sup>b</sup>···N20<sup>iv</sup> | 0.90  | 2.43  | 3.139 (10) | 136   |

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