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Synthesis and structure of 6-bromo-2-(diethoxymethyl)-2-hydroxy-3-phenyl-2,3-dihydro-1*H*-imidazo[1,2-*a*]pyridin-4-ium chloride acetonitrile monosolvate

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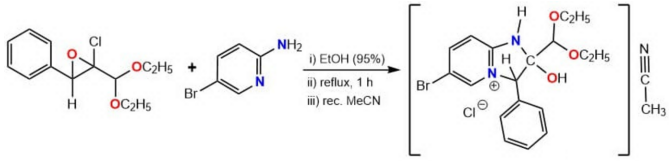
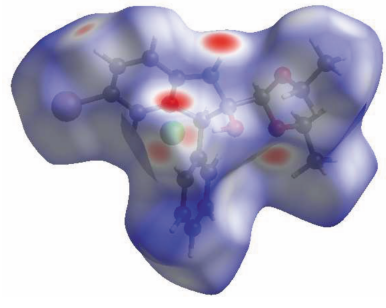
In the title solvated molecular salt, $C_{18}H_{22}BrN_2O_3^+\cdot Cl^-\cdot CH_3CN$, the imidazole ring is in envelope conformation and the pyridine and phenyl rings are oriented at a dihedral angle of 72.52 (5)°. In the crystal, O—H···Cl and N—H···Cl hydrogen bonds link the cations and anions into centrosymmetric tetramers enclosing $R_4^2(12)$ loops. Short Br···Cl [3.2313 (4) Å] and O···Cl [3.0490 (10) Å] contacts are observed. A Hirshfeld surface analysis of the structure indicates that the most important contributions for the crystal packing are from H···H (52.4%), H···C/C···H (12.1%), H···Br/Br···H (11.0%) and H···Cl/Cl···H (10.2%) interactions.

1. Chemical context

Nitrogen-containing heterocycles, cyclic molecules with one or more nitrogen atoms in the cyclic scaffold, are ubiquitous in pharmaceutical drugs, agrochemicals, metalloenzymes and biologically active natural products (Li *et al.*, 2023). The synthetic chemistry of N-heterocycles is not limited to organic chemistry (Guseinov *et al.*, 2017, 2020, 2024), they have been well explored in the spectrophotometric determination of metal ions (Alieva *et al.*, 2008), synthesis of cyclic carbonates from cycloaddition of CO_2 with epoxides (Aliyeva *et al.*, 2024), crystal engineering (Naghiyev *et al.*, 2023) and catalysis (Kerimli *et al.*, 2021). N-heterocycles have many advantages, such as easy modification and functionalization (Khalilov *et al.*, 2021), immobilization on solid materials through supramolecular interactions (Mammadov *et al.*, 2023) and crystal growth and design (Hajiyeva *et al.*, 2024). As part of our work in this area, we now describe the synthesis and structure of the title solvated molecular salt, $C_{18}H_{22}BrN_2O_3^+\cdot Cl^-\cdot C_2H_3N$ (**1**).





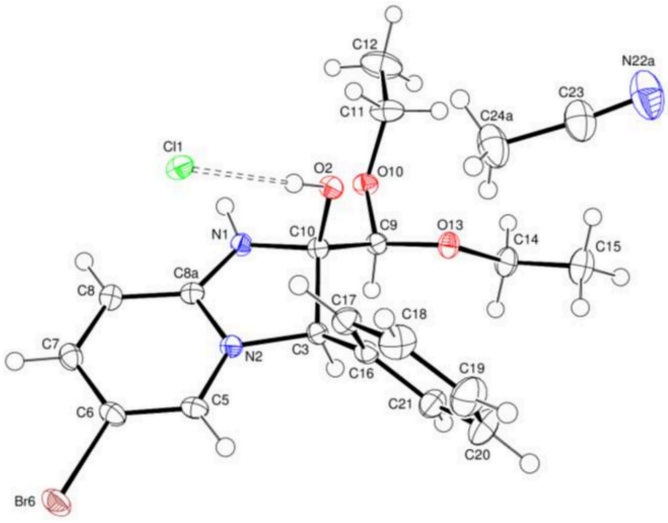


Figure 1
The asymmetric unit of the title compound with 50% probability ellipsoids. Only one part of the disordered atoms is shown for clarity. The O—H···Cl hydrogen bond is shown as a dashed line.

2. Structural commentary

The molecular structure of (**1**) is illustrated in Fig. 1. In the cation, the five-membered imidazole N1/N2/C3/C8*A*/C10 ring is non-planar due to the substituents bonded to atoms C3 and C10. It adopts an envelope conformation with puckering parameter φ = 137.38 (5)° where atom C10 is at the flap position and is displaced by −0.4138 (13) Å from the best least-squares plane of the other four atoms. The pyridine N2/C5–C8/C8a and phenyl C16–C21 rings are oriented at a dihedral angle of 72.52 (5)°. Atom Br6 is displaced by −0.0523 (1) Å from the best least-squares plane of the pyridine ring. The pendant C12 ethoxymethyl group has a *gauche*–*anti* conformation as indicated by the following torsion angles: C10—C9—O10—C11 = 75.76 (15)°; C9—O10—C11—C12 = 170.67 (13)°. Conversely, the C15 chain is *anti*–*anti*: C10—C9—O13—C14 = 163.79 (11)°; C9—O13—C14—C15 = −176.75 (12)°. Atom N1 of the imidazole ring is *anti* to O13 [N1—C10—C9—O13 = 178.28 (10)°] and *gauche* to O10 [N1—C10—C9—O10 = 51.69 (14)°]. Atoms C3 and C10 of the cation are stereogenic centres: in the arbitrarily chosen asymmetric unit, they both have *R* configurations, but crystal symmetry generates a racemic mixture.

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O2—H2···Cl1	0.80 (2)	2.25 (2)	3.0489 (10)	177 (2)
N1—H1···Cl1^{i}	0.86 (2)	2.37 (2)	3.1829 (12)	157.0 (17)
C7—H7···N22*A*^{ii}	0.95	2.51	3.410 (15)	157
C24*A*—H24*F*···O13	0.98	2.34	3.260 (12)	156
C3—H3···Cl1^{iii}	1.00	2.62	3.6125 (13)	171
C9—H9···Cl1^{iii}	1.00	2.72	3.6643 (14)	157
C17—H17···Cl1	0.95	2.70	3.6259 (15)	165

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

3. Supramolecular features

In the crystal, the cation and the anion are linked by a strong O—H···Cl hydrogen bond (Table 1). The chloride ion also accepts an N—H···Cl hydrogen bond from another cation, which generates centrosymmetric tetramers (two cations, two anions) enclosing $R_4^2(12)$ loops (Fig. 2). Various weak C—H···N, C—H···O and C—H···Cl hydrogen bonds are also observed (Table 1). Short Br6···Cl1 [3.2313 (4) Å, compared to a van der Waals separation of about 3.60 Å] and O2···Cl1 [3.0490 (10), 3.27 Å] contacts occur.

4. Hirshfeld surface analysis

To visualize the intermolecular interactions in the crystal a Hirshfeld surface (HS) analysis was carried out using *Crystal Explorer 17.5* (Spackman *et al.*, 2021) following the protocol of

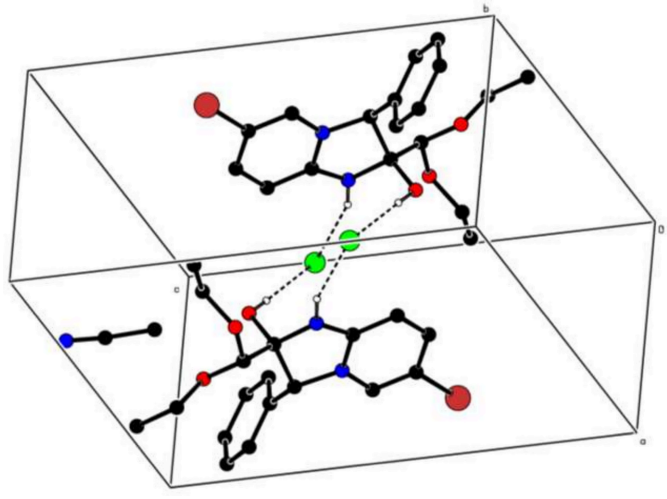


Figure 2
A partial packing diagram with O—H···Cl and N—H···Cl hydrogen bonds shown as dashed lines. The other hydrogen atoms have been omitted for clarity.

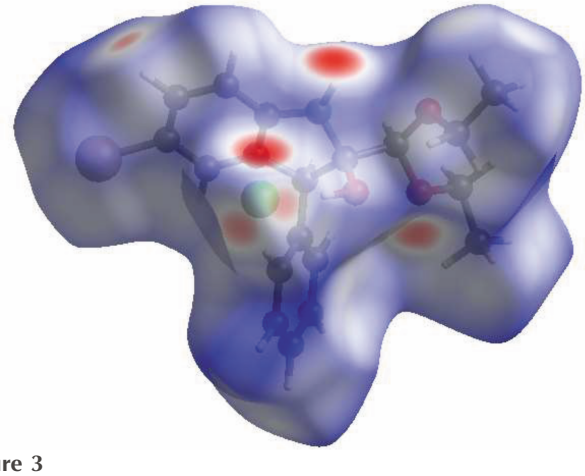


Figure 3
View of the three-dimensional Hirshfeld surface of the title compound plotted over d_{norm}.

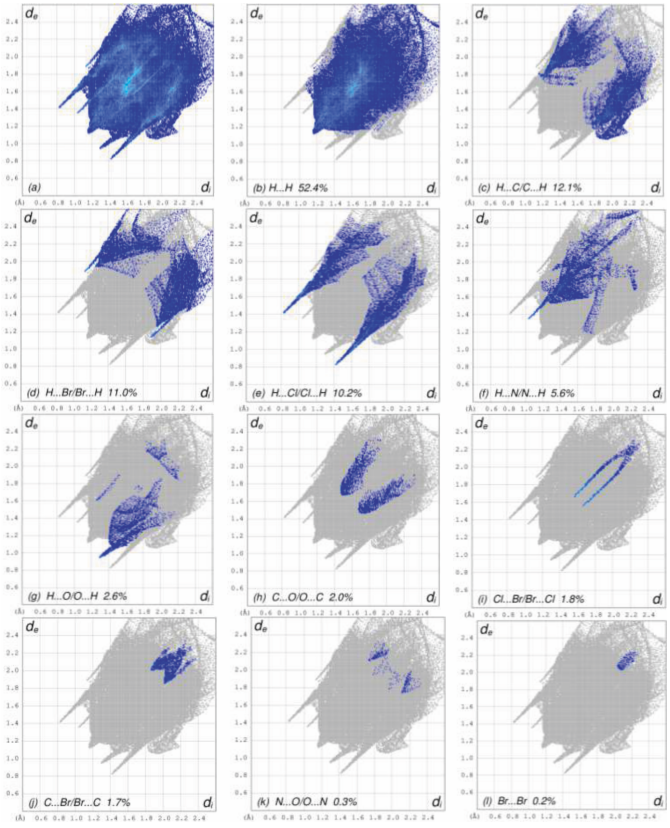


Figure 4
The two-dimensional fingerprint plots for the title compound, showing (*a*) all interactions and (*b*)–(*n*) different contact types. The d_i and d_e values are the closest internal and external distances (in Å) from given points on the Hirshfeld surface. Both disordered components of the acetonitrile molecule were omitted.

Tan *et al.* (2019) after removal of the disordered acetonitrile solvent molecule. In the surface plotted over d_{norm} (Fig. 3), the contact distances equal, shorter and longer with respect to the sum of van der Waals radii are shown by the white, red and blue colours, respectively, where the bright-red spots correspond to the respective donors and/or acceptors. The overall two-dimensional fingerprint plot, Fig. 4*a*, and those delineated into the different contact types are illustrated in Fig. 4*b*–*n*, together with their relative contributions to the HS.The most important contributors to the surface are H···H (52.4%) H···C/C···H (12.1%), H···Br/Br···H (11.0%) and H···Cl/Cl···H (10.2%) contacts. The remaining contacts have a very low density of points.

5. Synthesis and crystallization

A solution of 2-chloro-2-(diethoxymethyl)-3-phenyloxirane (128 mg, 0.5 mmol) and 5-bromopyridin-2-amine (82 mg, 0.5 mmol) in 20 ml of ethanol (95%) was boiled for 1 h. The solvent was distilled off *in vacuo* and the remaining powder was recrystallized from acetonitrile solution. Yield: 166 mg (79%), m.p. 485–487 K. Analysis calculated (%) for $C_{20}H_{25}BrClN_3O_3$: C 51.02, H 5.35, N 8.93; found C 51.00, H 5.32, N 8.91. ^{1}H NMR (300 MHz, DMSO-d_6): 1.22 (6H), 2.07 (3H), 3.63–3.94 (4H), 4.77 (1H), 6.43 (1H), 6.89 (OH), 7.43–7.93 (8H), 8.25 (NH), ^{13}C NMR (200 MHz, DMSO-d^6): 1.03, 15.55, 63.56, 76.28, 101.78, 104.25, 111.25, 117.90, 125.58, 126.77, 128.88, 129.25, 143.89, 147.25, 151.26, 160.99.

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{18}H_{22}BrN_2O_3^+\cdot Cl^-\cdot C_2H_3N$
M_r	470.79
Crystal system, space group	Triclinic, $P\overline{1}$
Temperature (K)	100
a, b, c (Å)	7.59344 (7), 11.07365 (13), 13.73164 (16)
α, β, γ (°)	75.4564 (10), 85.3656 (8), 88.4658 (8)
V (Å^3)	1113.98 (2)
Z	2
Radiation type	Cu $K\alpha$
μ (mm^{-1})	3.82
Crystal size (mm)	0.29 × 0.22 × 0.18
Data collection	
Diffractometer	XtaLAB Synergy, Dualflex, HyPix
Absorption correction	Gaussian (*CrysAlis PRO*; Rigaku OD, 2023)
T_{min}, T_{max}	0.345, 1.000
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	28449, 4661, 4621
R_{int}	0.025
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.632
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.023, 0.058, 1.07
No. of reflections	4661
No. of parameters	273
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})	0.34, −0.36

Computer programs: *CrysAlis PRO* (Rigaku OD, 2023), *SHELXT2014/5* (Sheldrick, 2015*a*), *SHELXL2018/3* (Sheldrick, 2015*b*) and *OLEX2* (Dolomanov *et al.*, 2009).

6. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2. The OH and NH hydrogen atoms were located in a difference-Fourier map and refined isotropically. The C-bound hydrogen-atom positions were calculated geometrically at distances of 0.95–1.00 Å depending on hybridization and refined using a riding model by applying the constraints of U_{iso} = 1.2U_{eq}(C) or 1.5U_{eq}(methyl C). Atoms N22 and C24 and its attached H atoms of the acetonitrile solvent molecule are disordered over two adjacent orientations, and they were refined with an occupancy ratio of 0.443 (19):0.557 (19).

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sity (Azerbaijan). TH is also grateful to Hacettepe University Scientific Research Project Unit (grant No. 013 D04 602 004). The authors contributions are as follows. Conceptualization, FIG, TH and ANB; synthesis, FIG and SZH; X-ray analysis, AIS; Hirshfeld surface analysis, TH; writing (review and editing of the manuscript), JL, TH and KIH; funding acquisition, KIH and TAJ; supervision, FIG, TH and ANB.

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supporting information

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Synthesis and structure of 6-bromo-2-(diethoxymethyl)-2-hydroxy-3-phenyl-2,3-dihydro-1*H*-imidazo[1,2-*a*]pyridin-4-ium chloride acetonitrile monosolvate

Firudin I. Guseinov, Aida I. Samigullina, Tuncer Hökelek, Sahil Z. Hamidov, Jamal Lasri, Khudayar I. Hasanov, Tahir A. Javadzade and Alebel N. Belay

Computing details

6-Bromo-2-(diethoxymethyl)-2-hydroxy-3-phenyl-2,3-dihydro-1*H*-imidazo[1,2-*a*]pyridin-4-ium chloride acetonitrile monosolvate

Crystal data

$C_{18}H_{22}BrN_2O_3^+\cdot Cl^-\cdot C_2H_3N$
M_r = 470.79
Triclinic, $P\overline{1}$
a = 7.59344 (7) Å
b = 11.07365 (13) Å
c = 13.73164 (16) Å
α = 75.4564 (10)°
β = 85.3656 (8)°
γ = 88.4658 (8)°
V = 1113.98 (2) Å^3
Z = 2
$F(000)$ = 484
D_x = 1.404 Mg m^{-3}
Cu $K\alpha$ radiation, λ = 1.54184 Å
Cell parameters from 22359 reflections
θ = 3.3–76.8°
μ = 3.82 mm^{-1}
T = 100 K
Prism, colorless
0.29 × 0.22 × 0.18 mm

Data collection

XtaLAB Synergy, Dualflex, HyPix diffractometer
Radiation source: micro-focus sealed X-ray tube, PhotonJet (Cu) X-ray Source
Mirror monochromator
Detector resolution: 10.0000 pixels mm^{-1}
ω scans
Absorption correction: gaussian (CrysAlisPro; Rigaku OD, 2023)
T_{min} = 0.345, T_{max} = 1.000
28449 measured reflections
4661 independent reflections
4621 reflections with $I > 2\sigma(I)$
R_{int} = 0.025
θ_{max} = 77.0°, θ_{min} = 3.3°
h = −9→8
k = −13→13
l = −17→17

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.023
$wR(F^2)$ = 0.058
S = 1.07
4661 reflections
273 parameters
2 restraints
Primary atom site location: dual
Secondary atom site location: difference Fourier map
Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0263P)^2 + 0.6353P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.002
$\Delta\rho_{max}$ = 0.34 e Å^{-3}
$\Delta\rho_{min}$ = −0.36 e Å^{-3}

Extinction correction: *SHELXL2018/3* (Sheldrick, 2015b), $Fc^{*}=kFc[1+0.001xFc^{2}\lambda^{3}/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.00204 (13)

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 (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}	Occ. (<1)
Br6	0.71051 (2)	−0.02116 (2)	0.40214 (2)	0.02342 (6)	
O2	0.63388 (13)	0.39419 (9)	0.70984 (7)	0.01627 (19)	
H2	0.555 (3)	0.369 (2)	0.6849 (16)	0.034 (5)*	
O10	0.90770 (13)	0.59295 (9)	0.65996 (7)	0.01821 (19)	
O13	0.94075 (12)	0.41038 (9)	0.78971 (7)	0.01768 (19)	
N1	0.77063 (15)	0.44100 (11)	0.54370 (8)	0.0156 (2)	
H1	0.735 (2)	0.5163 (19)	0.5181 (14)	0.023 (4)*	
N2	0.79268 (14)	0.23811 (10)	0.55778 (8)	0.0148 (2)	
C3	0.86257 (17)	0.25545 (12)	0.65138 (10)	0.0149 (2)	
H3	0.994348	0.259107	0.640632	0.018*	
C5	0.78508 (17)	0.12883 (13)	0.53030 (10)	0.0174 (3)	
H5	0.818893	0.052418	0.574201	0.021*	
C6	0.72782 (18)	0.13148 (13)	0.43832 (11)	0.0184 (3)	
C7	0.67733 (18)	0.24541 (14)	0.37389 (10)	0.0191 (3)	
H7	0.637428	0.246615	0.309840	0.023*	
C8	0.68567 (17)	0.35439 (13)	0.40341 (10)	0.0177 (3)	
H8	0.650962	0.431486	0.360816	0.021*	
C8A	0.74689 (17)	0.34921 (12)	0.49836 (10)	0.0150 (2)	
C9	0.93386 (17)	0.46309 (12)	0.68605 (10)	0.0153 (2)	
H9	1.049840	0.445590	0.652129	0.018*	
C10	0.79212 (17)	0.39016 (12)	0.65150 (10)	0.0143 (2)	
C11	0.7717 (2)	0.64392 (14)	0.71812 (13)	0.0272 (3)	
H11A	0.785968	0.611957	0.791164	0.033*	
H11B	0.653457	0.619906	0.704179	0.033*	
C12	0.7906 (3)	0.78235 (16)	0.68768 (17)	0.0405 (4)	
H12A	0.781124	0.812412	0.614806	0.061*	
H12B	0.906170	0.805053	0.704541	0.061*	
H12C	0.697028	0.820516	0.723683	0.061*	
C14	1.09708 (19)	0.44236 (15)	0.83005 (11)	0.0238 (3)	
H14A	1.100746	0.533414	0.823262	0.029*	
H14B	1.204216	0.417686	0.793453	0.029*	
C15	1.0887 (2)	0.37254 (18)	0.94002 (13)	0.0331 (4)	
H15A	1.085856	0.282624	0.945606	0.050*	
H15B	0.981776	0.397466	0.975210	0.050*	
H15C	1.193030	0.392261	0.970583	0.050*	

C16	0.81484 (18)	0.15009 (12)	0.74240 (10)	0.0170 (3)	
C17	0.64230 (19)	0.10643 (13)	0.76702 (11)	0.0204 (3)	
H17	0.550314	0.143950	0.726404	0.024*	
C18	0.6051 (2)	0.00806 (15)	0.85096 (12)	0.0287 (3)	
H18	0.487323	−0.021077	0.868097	0.034*	
C19	0.7394 (3)	−0.04782 (16)	0.90987 (13)	0.0360 (4)	
H19	0.713307	−0.114718	0.967518	0.043*	
C20	0.9118 (2)	−0.00599 (16)	0.88460 (13)	0.0338 (4)	
H20	1.003978	−0.045215	0.924294	0.041*	
C21	0.9496 (2)	0.09330 (14)	0.80120 (11)	0.0239 (3)	
H21	1.067421	0.122388	0.784344	0.029*	
Cl1	0.33366 (4)	0.30609 (3)	0.61057 (2)	0.01733 (8)	
N22	0.602 (7)	0.357 (3)	1.1158 (12)	0.046 (3)	0.443 (19)
N22A	0.594 (5)	0.331 (2)	1.1255 (9)	0.046 (3)	0.557 (19)
C24	0.611 (2)	0.2884 (16)	0.9482 (9)	0.0410 (19)	0.443 (19)
H24A	0.686904	0.347333	0.898044	0.061*	0.443 (19)
H24B	0.493588	0.288120	0.923808	0.061*	0.443 (19)
H24C	0.663150	0.204517	0.959064	0.061*	0.443 (19)
C24A	0.5848 (18)	0.3253 (13)	0.9381 (7)	0.0410 (19)	0.557 (19)
H24D	0.517870	0.398668	0.904347	0.061*	0.557 (19)
H24E	0.524540	0.249411	0.934754	0.061*	0.557 (19)
H24F	0.703877	0.327211	0.904437	0.061*	0.557 (19)
C23	0.5968 (2)	0.32604 (18)	1.04328 (13)	0.0337 (4)	

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Br6	0.02516 (9)	0.02146 (9)	0.02878 (9)	−0.00157 (6)	−0.00279 (6)	−0.01548 (6)
O2	0.0129 (4)	0.0182 (5)	0.0193 (5)	−0.0019 (3)	−0.0004 (4)	−0.0077 (4)
O10	0.0188 (5)	0.0135 (4)	0.0231 (5)	−0.0011 (3)	−0.0015 (4)	−0.0059 (4)
O13	0.0174 (5)	0.0188 (5)	0.0175 (5)	−0.0031 (4)	−0.0041 (4)	−0.0047 (4)
N1	0.0177 (5)	0.0134 (5)	0.0162 (5)	−0.0005 (4)	−0.0033 (4)	−0.0038 (4)
N2	0.0138 (5)	0.0148 (5)	0.0163 (5)	−0.0006 (4)	−0.0013 (4)	−0.0045 (4)
C3	0.0150 (6)	0.0143 (6)	0.0164 (6)	−0.0010 (5)	−0.0025 (5)	−0.0054 (5)
C5	0.0167 (6)	0.0145 (6)	0.0220 (7)	−0.0004 (5)	0.0007 (5)	−0.0072 (5)
C6	0.0169 (6)	0.0184 (6)	0.0224 (7)	−0.0025 (5)	0.0009 (5)	−0.0106 (5)
C7	0.0169 (6)	0.0241 (7)	0.0175 (6)	−0.0030 (5)	−0.0001 (5)	−0.0075 (5)
C8	0.0168 (6)	0.0192 (6)	0.0164 (6)	−0.0018 (5)	−0.0013 (5)	−0.0030 (5)
C8A	0.0119 (6)	0.0148 (6)	0.0178 (6)	−0.0015 (4)	0.0008 (5)	−0.0038 (5)
C9	0.0152 (6)	0.0142 (6)	0.0174 (6)	−0.0012 (5)	−0.0022 (5)	−0.0050 (5)
C10	0.0138 (6)	0.0139 (6)	0.0154 (6)	−0.0010 (4)	−0.0010 (5)	−0.0038 (5)
C11	0.0204 (7)	0.0205 (7)	0.0425 (9)	0.0018 (5)	0.0010 (6)	−0.0125 (6)
C12	0.0440 (10)	0.0218 (8)	0.0572 (12)	−0.0017 (7)	0.0097 (9)	−0.0166 (8)
C14	0.0205 (7)	0.0283 (7)	0.0244 (7)	−0.0030 (6)	−0.0086 (6)	−0.0075 (6)
C15	0.0329 (9)	0.0399 (9)	0.0268 (8)	−0.0021 (7)	−0.0124 (7)	−0.0056 (7)
C16	0.0214 (7)	0.0136 (6)	0.0168 (6)	0.0009 (5)	−0.0020 (5)	−0.0049 (5)
C17	0.0218 (7)	0.0159 (6)	0.0228 (7)	−0.0004 (5)	−0.0025 (5)	−0.0034 (5)
C18	0.0308 (8)	0.0215 (7)	0.0293 (8)	−0.0035 (6)	0.0028 (6)	0.0005 (6)

C19	0.0466 (10)	0.0263 (8)	0.0271 (8)	0.0012 (7)	−0.0019 (7)	0.0076 (6)
C20	0.0390 (9)	0.0311 (9)	0.0271 (8)	0.0068 (7)	−0.0113 (7)	0.0027 (7)
C21	0.0241 (7)	0.0234 (7)	0.0241 (7)	0.0030 (6)	−0.0062 (6)	−0.0046 (6)
Cl1	0.01411 (14)	0.01354 (14)	0.02489 (16)	−0.00020 (10)	−0.00402 (11)	−0.00502 (11)
N22	0.046 (3)	0.065 (9)	0.026 (2)	−0.015 (7)	0.003 (3)	−0.010 (4)
N22A	0.046 (3)	0.065 (9)	0.026 (2)	−0.015 (7)	0.003 (3)	−0.010 (4)
C24	0.028 (4)	0.071 (7)	0.0266 (18)	−0.010 (3)	0.0026 (16)	−0.018 (3)
C24A	0.028 (4)	0.071 (7)	0.0266 (18)	−0.010 (3)	0.0026 (16)	−0.018 (3)
C23	0.0292 (8)	0.0458 (10)	0.0243 (8)	−0.0118 (7)	0.0026 (6)	−0.0056 (7)

Geometric parameters (Å, º)

Br6—C6	1.8882 (13)	C12—H12B	0.9800
O2—H2	0.80 (2)	C12—H12C	0.9800
O2—C10	1.3954 (16)	C14—H14A	0.9900
O10—C9	1.4047 (16)	C14—H14B	0.9900
O10—C11	1.4438 (18)	C14—C15	1.511 (2)
O13—C9	1.4010 (16)	C15—H15A	0.9800
O13—C14	1.4383 (16)	C15—H15B	0.9800
N1—H1	0.86 (2)	C15—H15C	0.9800
N1—C8A	1.3413 (18)	C16—C17	1.395 (2)
N1—C10	1.4664 (16)	C16—C21	1.392 (2)
N2—C3	1.4867 (16)	C17—H17	0.9500
N2—C5	1.3590 (17)	C17—C18	1.389 (2)
N2—C8A	1.3487 (17)	C18—H18	0.9500
C3—H3	1.0000	C18—C19	1.387 (2)
C3—C10	1.5715 (18)	C19—H19	0.9500
C3—C16	1.5072 (18)	C19—C20	1.388 (3)
C5—H5	0.9500	C20—H20	0.9500
C5—C6	1.362 (2)	C20—C21	1.391 (2)
C6—C7	1.411 (2)	C21—H21	0.9500
C7—H7	0.9500	N22—C23	1.132 (12)
C7—C8	1.371 (2)	N22A—C23	1.141 (9)
C8—H8	0.9500	C24—H24A	0.9800
C8—C8A	1.4065 (19)	C24—H24B	0.9800
C9—H9	1.0000	C24—H24C	0.9800
C9—C10	1.5352 (17)	C24—C23	1.462 (11)
C11—H11A	0.9900	C24A—H24D	0.9800
C11—H11B	0.9900	C24A—H24E	0.9800
C11—C12	1.492 (2)	C24A—H24F	0.9800
C12—H12A	0.9800	C24A—C23	1.457 (8)
C10—O2—H2	109.4 (15)	C11—C12—H12B	109.5
C9—O10—C11	117.69 (11)	C11—C12—H12C	109.5
C9—O13—C14	113.71 (10)	H12A—C12—H12B	109.5
C8A—N1—H1	121.0 (12)	H12A—C12—H12C	109.5
C8A—N1—C10	110.85 (11)	H12B—C12—H12C	109.5
C10—N1—H1	123.7 (12)	O13—C14—H14A	110.3

C5—N2—C3	126.31 (11)	O13—C14—H14B	110.3
C8A—N2—C3	110.27 (11)	O13—C14—C15	106.88 (12)
C8A—N2—C5	123.28 (12)	H14A—C14—H14B	108.6
N2—C3—H3	108.0	C15—C14—H14A	110.3
N2—C3—C10	101.08 (10)	C15—C14—H14B	110.3
N2—C3—C16	112.88 (10)	C14—C15—H15A	109.5
C10—C3—H3	108.0	C14—C15—H15B	109.5
C16—C3—H3	108.0	C14—C15—H15C	109.5
C16—C3—C10	118.46 (11)	H15A—C15—H15B	109.5
N2—C5—H5	120.8	H15A—C15—H15C	109.5
N2—C5—C6	118.41 (13)	H15B—C15—H15C	109.5
C6—C5—H5	120.8	C17—C16—C3	122.00 (12)
C5—C6—Br6	118.27 (11)	C21—C16—C3	118.21 (13)
C5—C6—C7	120.32 (13)	C21—C16—C17	119.76 (13)
C7—C6—Br6	121.38 (10)	C16—C17—H17	120.0
C6—C7—H7	119.9	C18—C17—C16	119.93 (14)
C8—C7—C6	120.20 (13)	C18—C17—H17	120.0
C8—C7—H7	119.9	C17—C18—H18	119.9
C7—C8—H8	120.8	C19—C18—C17	120.21 (15)
C7—C8—C8A	118.33 (13)	C19—C18—H18	119.9
C8A—C8—H8	120.8	C18—C19—H19	120.0
N1—C8A—N2	110.41 (12)	C18—C19—C20	120.01 (15)
N1—C8A—C8	130.13 (13)	C20—C19—H19	120.0
N2—C8A—C8	119.46 (12)	C19—C20—H20	120.0
O10—C9—H9	107.4	C19—C20—C21	120.04 (15)
O10—C9—C10	113.93 (11)	C21—C20—H20	120.0
O13—C9—O10	114.38 (11)	C16—C21—H21	120.0
O13—C9—H9	107.4	C20—C21—C16	120.04 (15)
O13—C9—C10	105.91 (10)	C20—C21—H21	120.0
C10—C9—H9	107.4	H24A—C24—H24B	109.5
O2—C10—N1	111.60 (10)	H24A—C24—H24C	109.5
O2—C10—C3	115.02 (10)	H24B—C24—H24C	109.5
O2—C10—C9	109.37 (10)	C23—C24—H24A	109.5
N1—C10—C3	100.50 (10)	C23—C24—H24B	109.5
N1—C10—C9	110.23 (10)	C23—C24—H24C	109.5
C9—C10—C3	109.84 (10)	H24D—C24A—H24E	109.5
O10—C11—H11A	110.3	H24D—C24A—H24F	109.5
O10—C11—H11B	110.3	H24E—C24A—H24F	109.5
O10—C11—C12	107.15 (13)	C23—C24A—H24D	109.5
H11A—C11—H11B	108.5	C23—C24A—H24E	109.5
C12—C11—H11A	110.3	C23—C24A—H24F	109.5
C12—C11—H11B	110.3	N22—C23—C24	174 (3)
C11—C12—H12A	109.5	N22A—C23—C24A	175 (2)

Br6—C6—C7—C8	177.82 (10)	C7—C8—C8A—N2	−1.00 (19)
O10—C9—C10—O2	−71.36 (13)	C8A—N1—C10—O2	−97.51 (13)
O10—C9—C10—N1	51.69 (14)	C8A—N1—C10—C3	24.90 (13)
O10—C9—C10—C3	161.53 (10)	C8A—N1—C10—C9	140.75 (11)

O13—C9—C10—O2	55.23 (13)	C8A—N2—C3—C10	17.62 (13)
O13—C9—C10—N1	178.28 (10)	C8A—N2—C3—C16	145.19 (11)
O13—C9—C10—C3	−71.88 (13)	C8A—N2—C5—C6	−0.23 (19)
N2—C3—C10—O2	95.88 (12)	C9—O10—C11—C12	170.67 (13)
N2—C3—C10—N1	−24.09 (11)	C9—O13—C14—C15	−176.75 (12)
N2—C3—C10—C9	−140.24 (10)	C10—N1—C8A—N2	−15.17 (15)
N2—C3—C16—C17	−49.31 (17)	C10—N1—C8A—C8	165.20 (13)
N2—C3—C16—C21	128.79 (13)	C10—C3—C16—C17	68.46 (17)
N2—C5—C6—Br6	−178.08 (9)	C10—C3—C16—C21	−113.43 (14)
N2—C5—C6—C7	−0.2 (2)	C11—O10—C9—O13	−46.27 (16)
C3—N2—C5—C6	−175.57 (12)	C11—O10—C9—C10	75.76 (15)
C3—N2—C8A—N1	−2.82 (15)	C14—O13—C9—O10	−69.90 (14)
C3—N2—C8A—C8	176.85 (11)	C14—O13—C9—C10	163.79 (11)
C3—C16—C17—C18	179.20 (13)	C16—C3—C10—O2	−27.96 (16)
C3—C16—C21—C20	−178.63 (14)	C16—C3—C10—N1	−147.93 (11)
C5—N2—C3—C10	−166.53 (12)	C16—C3—C10—C9	95.92 (13)
C5—N2—C3—C16	−38.96 (17)	C16—C17—C18—C19	−0.7 (2)
C5—N2—C8A—N1	−178.82 (11)	C17—C16—C21—C20	−0.5 (2)
C5—N2—C8A—C8	0.85 (19)	C17—C18—C19—C20	−0.4 (3)
C5—C6—C7—C8	0.0 (2)	C18—C19—C20—C21	1.1 (3)
C6—C7—C8—C8A	0.6 (2)	C19—C20—C21—C16	−0.6 (3)
C7—C8—C8A—N1	178.60 (13)	C21—C16—C17—C18	1.1 (2)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O2—H2···Cl1	0.80 (2)	2.25 (2)	3.0489 (10)	177 (2)
N1—H1···Cl1^{i}	0.86 (2)	2.37 (2)	3.1829 (12)	157.0 (17)
C7—H7···N22*A*^{ii}	0.95	2.51	3.410 (15)	157
C24*A*—H24*F*···O13	0.98	2.34	3.260 (12)	156
C3—H3···Cl1^{iii}	1.00	2.62	3.6125 (13)	171
C9—H9···Cl1^{iii}	1.00	2.72	3.6643 (14)	157
C17—H17···Cl1	0.95	2.70	3.6259 (15)	165

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