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5-Amino-1-(2-chloronicotinoyl)-3-trifluoromethyl-1*H*-1,2,4-triazole: hydrogen-bonded sheets of alternating $R_2^2(8)$ and $R_6^6(36)$ rings

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Key indicators

Single-crystal X-ray study
T = 120 K
Mean σ(C–C) = 0.003 Å
R factor = 0.040
wR factor = 0.094
Data-to-parameter ratio = 15.0

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

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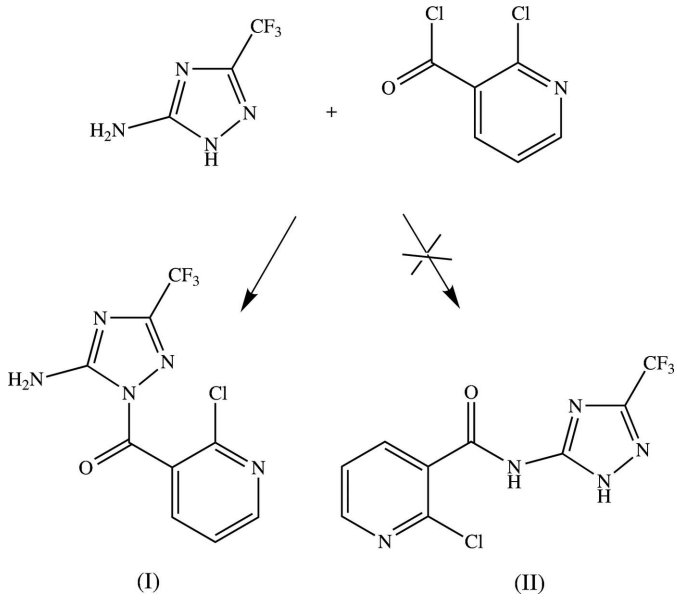
The molecules of the title compound, $C_9H_5ClF_3N_5O$, are linked by two independent N—H···N hydrogen bonds into sheets containing alternating $R_2^2(8)$ and $R_6^6(36)$ rings.

Comment

We have recently reported the molecular and supramolecular structures of a number of *N*-aryl-2-chloronicotinamides obtained from the reactions between 2-chloronicotinoyl chloride and substituted anilines (de Souza *et al.*, 2005; Cuffini *et al.*, 2006). In a continuation of this study, we now report the structure of the title compound, (I), obtained from the reaction between 2-chloronicotinoyl chloride and 5-amino-3-trifluoromethyl-1*H*-1,2,4-triazole. The formation of (I) was unexpected, as reaction at the exocyclic amino group was expected to yield the isomeric compound, (II) (see scheme).



(I) (II)

The carbonyl group of (I) is almost coplanar with the triazole ring (Fig. 1, Table 1) and this is possibly controlled by the intramolecular N—H···O hydrogen bond (Table 2). On the other hand, the pyridyl ring is rotated significantly out of this plane. The bond distances in the triazole ring provide evidence for strong bond fixation within this ring.

The molecules of compound (I) are linked by two independent N—H···N hydrogen bonds (Table 2) into sheets, whose formation can readily be analysed in terms of two simple substructures, each utilizing just one hydrogen bond. One substructure is finite and zero-dimensional, while the other is one-dimensional.

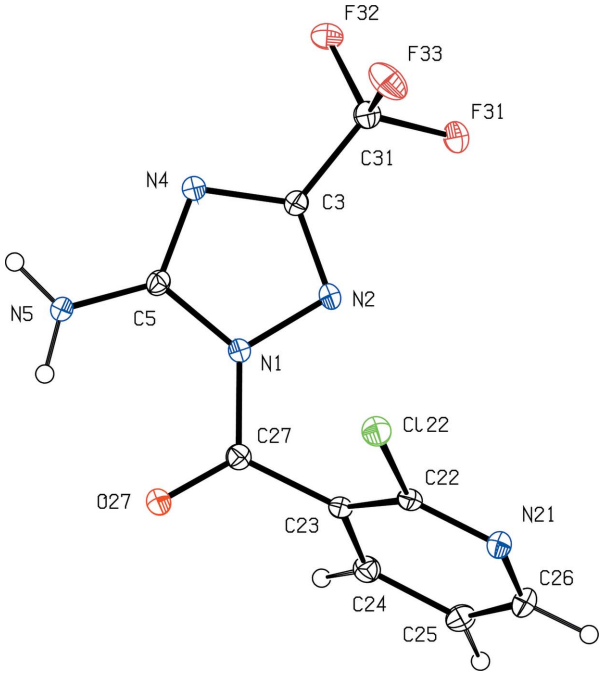


Figure 1
The molecular structure of compound (I), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level.

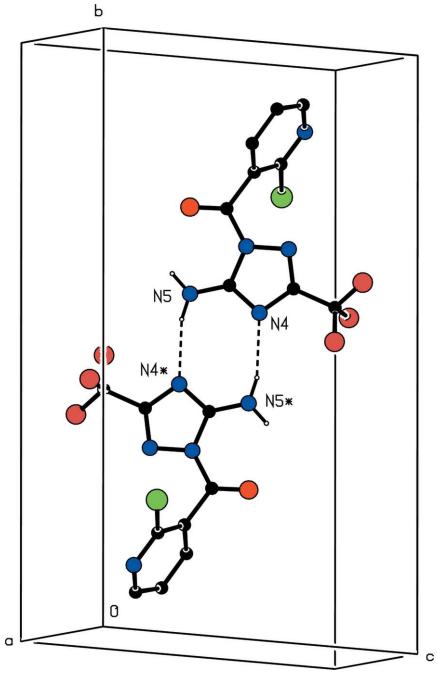


Figure 2
Part of the crystal structure of compound (I), showing the formation of the hydrogen-bonded $R_2^2(8)$ dimer centred at $(\frac{1}{2}, \frac{1}{2}, \frac{1}{2})$. For the sake of clarity, H atoms bonded to C atoms have been omitted. Atoms marked with an asterisk (*) are at the symmetry position $(1-x, 1-y, 1-z)$.

The finite substructure is formed from paired hydrogen bonds. Amino atom N5 in the molecule at (x, y, z) acts as hydrogen-bond donor, *via* atom H5*B*, to the triazole ring atom N4 in the molecule at $(1-x, 1-y, 1-z)$, so forming by inversion an $R_2^2(8)$ (Bernstein *et al.*, 1995) dimer centred at $(\frac{1}{2}, \frac{1}{2}, \frac{1}{2})$ (Fig. 2). This dimer can conveniently be regarded as the basic building block in the sheet structure.

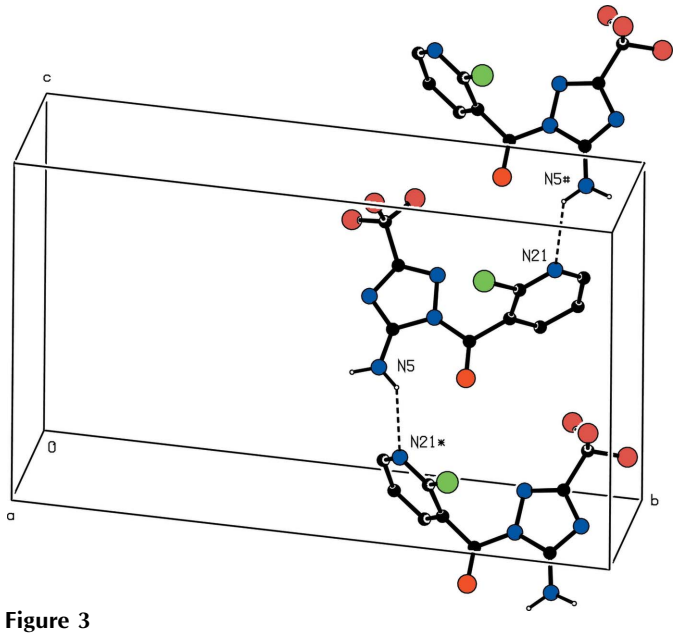


Figure 3
Part of the crystal structure of compound (I), showing the formation of a hydrogen-bonded $C(8)$ chain along $[10\bar{1}]$. For the sake of clarity, H atoms bonded to C atoms have been omitted. Atoms marked with an asterisk (*) or a hash (#) are at the symmetry positions $(\frac{1}{2}+x, \frac{3}{2}-y, -\frac{1}{2}+z)$ and $(-\frac{1}{2}+x, \frac{3}{2}-y, \frac{1}{2}+z)$ respectively.

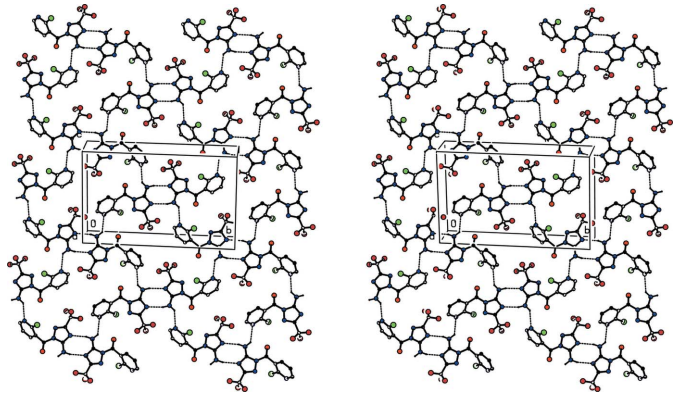


Figure 4
A stereoscopic view of part of the crystal structure of compound (I), showing the formation of a sheet of alternating $R_2^2(8)$ and $R_6^6(36)$ rings parallel to (101). For the sake of clarity, H atoms bonded to C atoms have been omitted.

In the second substructure, amino atom N5 acts as hydrogen-bond donor, *via* atom H5*A*, to pyridyl ring atom N21 in the molecule at $(\frac{1}{2}+x, \frac{3}{2}-y, -\frac{1}{2}+z)$, so forming a $C(8)$ chain running parallel to the $[10\bar{1}]$ direction and generated by the *c*-glide plane at $y=\frac{3}{4}$ (Fig. 3). This chain motif directly links the $R_2^2(8)$ dimer unit centred at $(\frac{1}{2}, \frac{1}{2}, \frac{1}{2})$ to the four dimers centred at (0, 0, 1), (0, 1, 1), (1, 0, 0) and (1, 1, 0), thereby generating a sheet of alternating $R_2^2(8)$ and $R_6^6(36)$ rings parallel to (101) (Fig. 4).

There are no direction-specific interactions between adjacent sheets. In particular, C—H$\cdots\pi$ hydrogen bonds and π–π stacking interactions are absent.

Experimental

A mixture of 2-chloronicotinoyl chloride (0.88 g, 5 mmol) and 5-amino-3-trifluoromethyl-1*H*-1,2,4-triazole (0.76 g, 5 mmol) (Lopyrev & Rakhmatulina, 1983) in 1,2-dichloroethane (15 ml) was heated under reflux for 1 h. The mixture was then cooled and the solvent removed under reduced pressure. The resulting solid product, (I), was recrystallized from ethyl acetate to give crystals suitable for single-crystal X-ray diffraction.

Crystal data

$C_9H_5ClF_3N_5O$
M_r = 291.63
Monoclinic, $P2_1/n$
a = 4.64770 (10) Å
b = 19.7414 (10) Å
c = 12.3721 (5) Å
β = 91.147 (3)°
V = 1134.94 (8) Å^3
Z = 4
D_x = 1.707 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 0.38 mm^{-1}
T = 120 (2) K
Needle, colourless
0.26 × 0.06 × 0.05 mm

Data collection

Bruker Nonius KappaCCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2003)
T_{min} = 0.927, T_{max} = 0.981
14659 measured reflections
2588 independent reflections
1923 reflections with $I > 2\sigma(I)$
R_{int} = 0.049
θ_{max} = 27.6°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.040
$wR(F^2)$ = 0.094
S = 1.04
2588 reflections
172 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0386P)^2 + 0.619P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.25 e Å^{-3}
$\Delta\rho_{min}$ = −0.33 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

N1—N2	1.391 (2)	N1—C27	1.401 (2)
N2—C3	1.305 (2)	C27—O27	1.209 (2)
C3—N4	1.364 (2)	C3—C31	1.488 (3)
N4—C5	1.328 (2)	C5—N5	1.324 (2)
C5—N1	1.392 (2)		
N2—N1—C27—O27	−178.39 (17)	N1—C27—C23—C22	70.4 (2)
N2—N1—C27—C23	0.1 (3)		

Table 2
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
N5—H5A···O27	0.88	2.25	2.836 (2)	124
N5—H5A···N21^{i}	0.88	2.40	3.053 (2)	131
N5—H5B···N4^{ii}	0.88	2.13	2.985 (2)	163

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

All H atoms were located in difference maps and then treated as riding atoms, with C—H = 0.95 Å and N—H = 0.88 Å, and with U_{iso}(H) = 1.2U_{eq}(C,N).

Data collection: *COLLECT* (Nonius, 1999); cell refinement: *DENZO* (Otwinowski & Minor, 1997) and *COLLECT*; data reduction: *DENZO* and *COLLECT*; program(s) used to solve structure: *OSCAIL* (McArdle, 2003) and *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *OSCAIL* and *SHELXL97* (Sheldrick, 1997); molecular graphics: *PLATON* (Spek, 2003); software used to prepare material for publication: *SHELXL97* and *PRPKAPPA* (Ferguson, 1999).

The X-ray data were collected at the EPSRC X-Ray Crystallographic Service, University of Southampton, UK; the authors thank the staff of the Service for all their help and advice. JLW thanks CNPq and FAPERJ for financial support.

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supporting information

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Crystal data

$C_9H_5ClF_3N_5O$
$M_r = 291.63$
Monoclinic, $P2_1/n$
Hall symbol: -P 2yn
$a = 4.6477$ (1) Å
$b = 19.7414$ (10) Å
$c = 12.3721$ (5) Å
$\beta = 91.147$ (3)°
$V = 1134.94$ (8) Å^3
$Z = 4$
$F(000) = 584$
$D_x = 1.707$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 2588 reflections
$\theta = 3.9$–27.6°
$\mu = 0.38$ mm^{-1}
$T = 120$ K
Needle, colourless
0.26 × 0.06 × 0.05 mm

Data collection

Bruker Nonius KappaCCD area-detector diffractometer
Radiation source: Bruker Nonius FR591 rotating anode
Graphite monochromator
Detector resolution: 9.091 pixels mm^{-1}
φ and ω scans
Absorption correction: multi-scan (SADABS; Sheldrick, 2003)
$T_{min} = 0.927$, $T_{max} = 0.981$
14659 measured reflections
2588 independent reflections
1923 reflections with $I > 2\sigma(I)$
$R_{int} = 0.049$
$\theta_{max} = 27.6°$, $\theta_{min} = 3.9°$
$h = -5\rightarrow6$
$k = -24\rightarrow25$
$l = -16\rightarrow16$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.040$
$wR(F^2) = 0.094$
$S = 1.04$
2588 reflections
172 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0386P)^2 + 0.619P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.25$ e Å^{-3}
$\Delta\rho_{min} = -0.33$ e Å^{-3}

Special details

Experimental. IR (KBr disk, ν, cm^{-1}): 3368, 3304, 3213, 3156, 1655, 1581, 1567, 1446, 1408, 1383, 1332, 1202, 1139, 1080, 985, 812, 759, 730, 657, 556, 503.

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

	x	*y*	*z*	U_{iso}*/U_{eq}
N1	0.3230 (3)	0.66804 (8)	0.53946 (13)	0.0196 (4)
N2	0.1330 (3)	0.66249 (8)	0.62443 (13)	0.0204 (4)
C3	0.1366 (4)	0.59720 (10)	0.64084 (15)	0.0188 (4)
C31	−0.0261 (4)	0.56599 (10)	0.73046 (17)	0.0229 (5)
F31	−0.2485 (3)	0.60360 (6)	0.76044 (10)	0.0316 (3)
F32	−0.1271 (3)	0.50499 (6)	0.70283 (11)	0.0346 (3)
F33	0.1419 (3)	0.55681 (7)	0.81869 (10)	0.0391 (4)
N4	0.3061 (3)	0.55840 (8)	0.57685 (13)	0.0198 (4)
C5	0.4244 (4)	0.60400 (10)	0.51248 (15)	0.0183 (4)
N5	0.6132 (4)	0.59136 (9)	0.43635 (13)	0.0229 (4)
C27	0.4050 (4)	0.73123 (10)	0.49861 (15)	0.0189 (4)
O27	0.5656 (3)	0.73437 (7)	0.42315 (11)	0.0236 (3)
N21	0.2498 (3)	0.86511 (9)	0.70620 (13)	0.0227 (4)
C22	0.3523 (4)	0.81141 (10)	0.65678 (15)	0.0184 (4)
Cl22	0.61338 (10)	0.76614 (3)	0.72787 (4)	0.02480 (15)
C23	0.2760 (4)	0.79157 (10)	0.55210 (15)	0.0177 (4)
C24	0.0833 (4)	0.83207 (10)	0.49519 (17)	0.0222 (4)
C25	−0.0272 (4)	0.88902 (11)	0.54533 (17)	0.0265 (5)
C26	0.0579 (4)	0.90288 (11)	0.65022 (18)	0.0262 (5)
H5A	0.6809	0.6248	0.3972	0.027*
H5B	0.6709	0.5496	0.4249	0.027*
H24	0.0276	0.8211	0.4230	0.027*
H25	−0.1591	0.9179	0.5080	0.032*
H26	−0.0232	0.9411	0.6848	0.031*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.0229 (9)	0.0167 (9)	0.0194 (9)	0.0009 (7)	0.0067 (7)	0.0003 (7)
N2	0.0220 (8)	0.0207 (9)	0.0188 (9)	0.0003 (7)	0.0074 (7)	−0.0007 (7)
C3	0.0205 (10)	0.0172 (10)	0.0186 (10)	0.0004 (8)	0.0010 (8)	−0.0012 (8)
C31	0.0215 (10)	0.0225 (11)	0.0249 (11)	0.0006 (8)	0.0024 (8)	0.0004 (9)
F31	0.0292 (7)	0.0296 (7)	0.0365 (7)	0.0042 (5)	0.0157 (5)	0.0004 (6)
F32	0.0414 (8)	0.0215 (7)	0.0415 (8)	−0.0087 (6)	0.0152 (6)	0.0003 (6)
F33	0.0351 (7)	0.0578 (10)	0.0243 (7)	−0.0024 (6)	0.0001 (5)	0.0129 (6)
N4	0.0228 (9)	0.0172 (9)	0.0195 (8)	0.0004 (7)	0.0046 (7)	0.0001 (7)
C5	0.0207 (10)	0.0168 (10)	0.0173 (10)	0.0002 (8)	−0.0006 (8)	−0.0029 (8)
N5	0.0307 (9)	0.0159 (9)	0.0224 (9)	0.0025 (7)	0.0096 (7)	−0.0003 (7)
C27	0.0192 (9)	0.0197 (10)	0.0177 (10)	−0.0008 (8)	−0.0005 (8)	−0.0009 (8)
O27	0.0277 (8)	0.0211 (8)	0.0222 (7)	−0.0017 (6)	0.0076 (6)	−0.0015 (6)
N21	0.0258 (9)	0.0199 (9)	0.0225 (9)	−0.0001 (7)	0.0048 (7)	−0.0027 (7)
C22	0.0190 (9)	0.0165 (10)	0.0197 (10)	−0.0015 (8)	0.0030 (8)	0.0026 (8)
Cl22	0.0241 (3)	0.0272 (3)	0.0230 (3)	0.0032 (2)	−0.00125 (19)	0.0024 (2)
C23	0.0188 (9)	0.0148 (10)	0.0196 (10)	−0.0023 (8)	0.0044 (8)	0.0007 (8)
C24	0.0250 (10)	0.0221 (11)	0.0196 (10)	−0.0009 (8)	0.0008 (8)	0.0007 (8)

C25	0.0293 (11)	0.0211 (11)	0.0290 (12)	0.0066 (9)	0.0018 (9)	0.0048 (9)
C26	0.0291 (11)	0.0200 (11)	0.0298 (12)	0.0053 (9)	0.0059 (9)	−0.0033 (9)

Geometric parameters (Å, º)

N1—N2	1.391 (2)	N5—H5B	0.88
N2—C3	1.305 (2)	C27—C23	1.494 (3)
C3—N4	1.364 (2)	N21—C22	1.318 (3)
N4—C5	1.328 (2)	N21—C26	1.344 (3)
C5—N1	1.392 (2)	C22—C23	1.392 (3)
N1—C27	1.401 (2)	C22—Cl22	1.7328 (19)
C27—O27	1.209 (2)	C23—C24	1.383 (3)
C3—C31	1.488 (3)	C24—C25	1.388 (3)
C31—F31	1.331 (2)	C24—H24	0.95
C31—F32	1.334 (2)	C25—C26	1.377 (3)
C31—F33	1.342 (2)	C25—H25	0.95
C5—N5	1.324 (2)	C26—H26	0.95
N5—H5A	0.88		
N2—N1—C5	109.43 (15)	O27—C27—N1	120.05 (18)
N2—N1—C27	121.60 (15)	O27—C27—C23	124.11 (18)
C5—N1—C27	128.76 (16)	N1—C27—C23	115.82 (16)
C3—N2—N1	100.91 (15)	C22—N21—C26	116.71 (17)
N2—C3—N4	118.04 (17)	N21—C22—C23	124.78 (18)
N2—C3—C31	121.34 (17)	N21—C22—Cl22	115.82 (15)
N4—C3—C31	120.54 (18)	C23—C22—Cl22	119.32 (15)
F31—C31—F32	107.68 (16)	C24—C23—C22	117.46 (18)
F31—C31—F33	106.92 (16)	C24—C23—C27	119.80 (17)
F32—C31—F33	106.41 (17)	C22—C23—C27	122.66 (17)
F31—C31—C3	112.67 (17)	C23—C24—C25	118.87 (19)
F32—C31—C3	111.36 (17)	C23—C24—H24	120.6
F33—C31—C3	111.47 (16)	C25—C24—H24	120.6
C5—N4—C3	102.61 (16)	C26—C25—C24	118.71 (19)
N5—C5—N4	125.95 (18)	C26—C25—H25	120.6
N5—C5—N1	125.04 (18)	C24—C25—H25	120.6
N4—C5—N1	109.00 (16)	N21—C26—C25	123.43 (19)
C5—N5—H5A	120.0	N21—C26—H26	118.3
C5—N5—H5B	120.0	C25—C26—H26	118.3
H5A—N5—H5B	120.0		
C5—N1—N2—C3	0.0 (2)	C5—N1—C27—O27	7.3 (3)
C27—N1—N2—C3	−175.24 (17)	N2—N1—C27—C23	0.1 (3)
N1—N2—C3—N4	−0.1 (2)	C5—N1—C27—C23	−174.18 (18)
N1—N2—C3—C31	176.58 (17)	C26—N21—C22—C23	0.0 (3)
N2—C3—C31—F31	24.8 (3)	C26—N21—C22—Cl22	176.68 (14)
N4—C3—C31—F31	−158.62 (17)	N21—C22—C23—C24	1.5 (3)
N2—C3—C31—F32	145.91 (18)	Cl22—C22—C23—C24	−175.11 (14)
N4—C3—C31—F32	−37.5 (2)	N21—C22—C23—C27	178.22 (18)

N2—C3—C31—F33	−95.4 (2)	Cl22—C22—C23—C27	1.6 (3)
N4—C3—C31—F33	81.2 (2)	O27—C27—C23—C24	65.5 (3)
N2—C3—N4—C5	0.1 (2)	N1—C27—C23—C24	−112.9 (2)
C31—C3—N4—C5	−176.59 (17)	O27—C27—C23—C22	−111.2 (2)
C3—N4—C5—N5	178.57 (19)	N1—C27—C23—C22	70.4 (2)
C3—N4—C5—N1	−0.1 (2)	C22—C23—C24—C25	−1.2 (3)
N2—N1—C5—N5	−178.62 (17)	C27—C23—C24—C25	−178.06 (18)
C27—N1—C5—N5	−3.8 (3)	C23—C24—C25—C26	−0.4 (3)
N2—N1—C5—N4	0.0 (2)	C22—N21—C26—C25	−1.8 (3)
C27—N1—C5—N4	174.86 (18)	C24—C25—C26—N21	2.0 (3)
N2—N1—C27—O27	−178.39 (17)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N5—H5*A*···O27	0.88	2.25	2.836 (2)	124
N5—H5*A*···N21^{i}	0.88	2.40	3.053 (2)	131
N5—H5*B*···N4^{ii}	0.88	2.13	2.985 (2)	163

Symmetry codes: (i) *x*+1/2, −*y*+3/2, *z*−1/2; (ii) −*x*+1, −*y*+1, −*z*+1.