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3-(2-Chloropyridin-3-yl)quinazoline-2,4(1*H*,3*H*)-dione chloroform monosolvate

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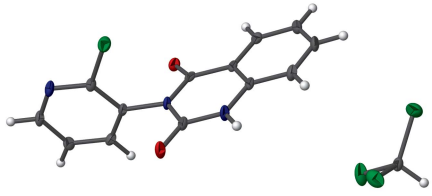
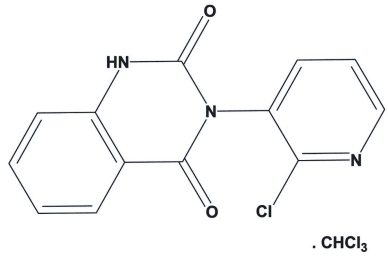
Structural data: full structural data are available from iucrdata.iucr.org

The solvated title compound, $C_{13}H_8ClN_3O_2\cdot CHCl_3$, is a product of a condensation reaction between 2-amino-*N*-(2-chloropyridin-3-yl)benzamide and phosgene. The presence of the chlorine substituent in the pyridine ring forces the latter to adopt a nearly perpendicular orientation relative to the planar quinazoline ring (r.m.s. deviation = 0.04 Å), the two ring systems being inclined to one another by 84.28 (9)°. In the crystal, molecules are linked by pairs of N—H···O hydrogen bonds, forming inversion dimers with an $R_2^2(8)$ ring motif. The dimers are linked by C—H···O hydrogen bonds, forming ribbons propagating along the *a*-axis direction. The chloroform solvent molecules are linked to the organic molecule by C—H···N hydrogen bonds.

3D view



Chemical scheme



Structure description

The title compound results from our ongoing research aimed at the development of subtype-selective ligands for muscarinic receptors (Tahtaoui *et al.*, 2004; Mohr *et al.*, 2010). It was isolated as a side-product in the course of the synthesis of AFDX-type allosteric modulators of muscarinic M2 receptors (Mohr *et al.*, 2004). Specifically, the incomplete condensation between ethyl 2-aminobenzoate and 3-amino-2-chloropyridine (Holzgrabe & Heller, 2003) gave the ring-opened 2-amino-*N*-(2-chloropyridin-3-yl)-benzamide (**1**) in 23% yield. This compound, (**1**), was subjected to condensation with phosgene giving the title compound (**2**) in 85% yield. This two-step approach is of general interest for the synthesis of differently substituted (1*H*,3*H*)-quinazoline-2,4-diones.

The molecular structure of the title compound (**2**), which crystallized as a chloroform monosolvate, is shown in Fig. 1. The pyridine ring (N1/C1–C5) is nearly perpendicular to the planar quinazoline ring (N2/N3/C6–C13; r.m.s. deviation = 0.04 Å), making a dihedral angle of 84.28 (9)°.

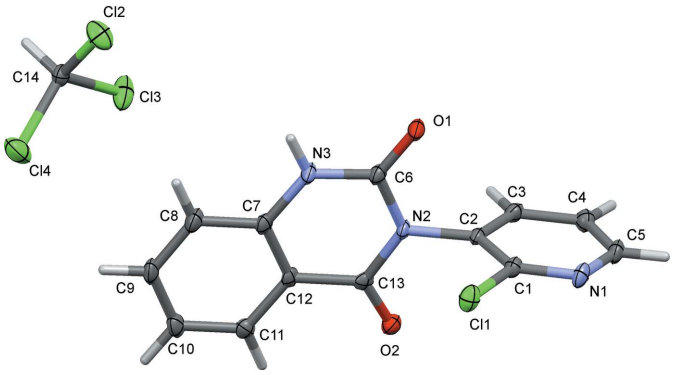


Figure 1
The molecular structure of the solvated title compound, (**2**)·$CHCl_3$, with the atom labelling and displacement ellipsoids drawn at the 50% probability level.

In the crystal, molecules are linked by pairs of N—H···O hydrogen bonds forming inversion dimers, with an $R_2^2(8)$ ring motif (Fig. 2 and Table 1). The chloroform solvate molecules are linked to the organic molecule by C—H···N hydrogen bonds, and the dimers are linked by C—H···O hydrogen bonds, forming ribbons propagating along the *a*-axis direction (Fig. 2 and Table 1).

Synthesis and crystallization

2-Amino-*N*-(2-chloropyridin-3-yl)benzamide (1).

3-Amino-2-chloropyridine (2.57 g, 20.0 mmol), ethyl 2-aminobenzoate (3.39 g, 20.5 mmol) and KO^{t}Bu (7.29 g, 65.0 mmol) were suspended in dry 1,4-dioxane (100 ml) under argon. The mixture was heated by microwaves (gradient of heating: 2 min to 333 K; holding time: 10 min at 333 K; gradient of heating: 3 min from 333–373 K; holding time: 2.5 h at 373 K). After cooling to 298 K, the solution was treated with 1 *M* NaH_2PO_4 (60 ml) and stirred for 30 min. The dioxane was evaporated *in vacuo* and the residue was treated with 50 ml water. The solid obtained was filtered and dried. The product (**1**) was then purified by silica chromatography

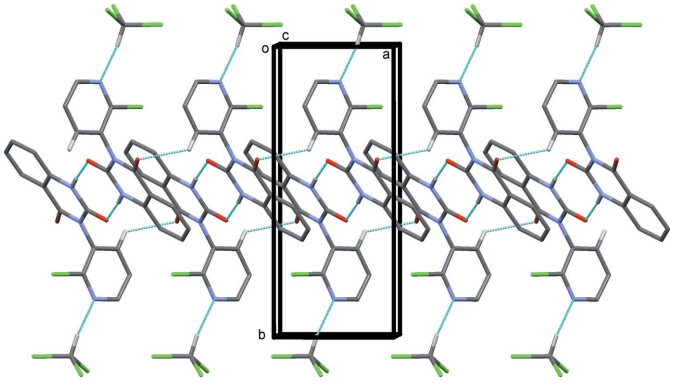


Figure 2
A view along the *c* axis of the crystal packing of the solvated title compound, (**2**)·$CHCl_3$. The hydrogen bonds are shown as dashed lines (see Table 1). For clarity, only the H atoms involved in hydrogen bonding have been included.

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N3—H3···O1^{i}	0.88	1.91	2.791 (3)	175
C14—H14···N1^{ii}	1.00	2.39	3.200 (3)	137
C3—H3*A*···O2^{iii}	0.95	2.48	3.123 (3)	125

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

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{13}H_8ClN_3O_2·CHCl_3$
M_r	393.04
Crystal system, space group	Monoclinic, $P2_1/c$
Temperature (K)	100
a, *b*, *c* (Å)	5.6382 (11), 13.622 (3), 20.662 (4)
β (°)	92.289 (6)
V (Å^3)	1585.7 (5)
Z	4
Radiation type	Mo $K\alpha$
μ (mm^{-1})	0.76
Crystal size (mm)	0.59 × 0.32 × 0.26
Data collection	
Diffractometer	Bruker APEXII CCD
Absorption correction	Multi-scan (*SADABS*; Bruker, 2013)
T_{min}, T_{max}	0.656, 0.980
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	15463, 3370, 2796
R_{int}	0.054
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.634
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, *S*	0.040, 0.109, 1.08
No. of reflections	3370
No. of parameters	208
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.45, −0.49

Computer programs: *APEX2* and *SAINT* (Bruker, 2013), *olex2.solve* (Bourhis *et al.*, 2015), *SHELXL2014* (Sheldrick, 2015), *Mercury* (Macrae *et al.*, 2008), *OLEX2* (Dolomanov *et al.*, 2009), *SHELXL2014* (Sheldrick, 2015), *enCIFer* (Allen *et al.*, 2004) and *publCIF* (Westrip (2010).

(ethyl acetate/petroleum ether 1:1, R_f = 0.78), giving a pale-yellow solid (yield: 1.16 g, 23.3%; m.p. 477.3 K). ^{1}H NMR (400 MHz, DMSO-d_6) δ 9.89 (1*H*, *br*, N—H), 8.29 (1*H*, *dd*, *J* = 4.7, 1.8 Hz), 8.07 (1*H*, *dd*, *J* = 7.9, 1.8 Hz), 7.74 (1*H*, *dd*, *J* = 8.1, 1.5 Hz), 7.49 (1*H*, *dd*, *J* = 7.9, 4.7 Hz), 7.24 (1*H*, *ddd*, *J* = 8.4, 7.1, 1.5 Hz), 6.79 (1*H*, *dd*, *J* = 8.4, 1.2 Hz), 6.62 (1*H*, *ddd*, *J* = 8.1, 7.1, 1.2 Hz), 6.47 (2*H*, *br*, NH_2). ^{13}C NMR (100 MHz, DMSO-d_6) δ 168.27 (C═O), 150.73, 146.71 (C—Cl), 146.68 (CH), 136.96 (C), 133.22 (C), 132.82 (C), 129.25 (C), 123.85 (CH), 117.22 (C), 115.35 (C), 113.86 (C). IR (ATR, cm^{-1}): 3433 (NH), 3330, 3286 (NH_2), 3073 (CH), 1644 (C═O amide), 1616, 1578, 1569, 1503, 1486, 1391, 802, 743, 735. MS (ESI): *m*/*z* (%): 248.2 (*M*+1).

3-(2-Chloropyridin-3-yl)quinazoline-2,4-(1*H*,3*H*)dione (2).

Compound (**1**) (4.22 g, 20.0 mmol) and Hueunig's base (7.0 ml, 40.0 mmol) were dissolved in dry 1,4-dioxane (150 ml) under argon. A solution of 20% phosgene in toluene (18.5 ml, 35.0 mmol) was added dropwise over 30 min. The solution was heated using microwaves (gradient of heating: 3 min to 358 K; holding time: 2 h at 358 K). After cooling to 298 K, the

mixture was quenched with 1.0 *M* NaH_2PO_4 (100 ml) and stirred for 1 h at room temperature. The dioxane was evaporated and the solid obtained was filtered by suction and dried over P_4O_{10}, giving a white solid (yield: 4.68 g, 85.5%; m.p. 510.6 K). The product was recrystallized from chloroform giving colourless block-like crystals of the title compound (**2**). ^{1}H NMR (400 MHz, $CDCl_3$) δ 10.52 (1*H*, *br*, N—H), 8.56 (1*H*, *dd*, *J* = 4.8, 1.8 Hz), 8.15 (1*H*, *dd*, *J* = 7.9, 1.1 Hz), 7.76 (1*H*, *dd*, *J* = 7.8, 1.8 Hz), 7.61 (1*H*, *ddd*, *J* = 8.1, 7.0, 1.1 Hz), 7.46 (1*H*, *dd*, *J* = 7.8, 4.8 Hz), 7.27 (1*H*, *ddd*, *J* = 7.9, 7.0, 1.0 Hz), 7.02 (1*H*, *dd*, *J* = 8.1, 1.0 Hz). ^{13}C NMR (100 MHz, $CDCl_3$) δ 161.58 (C═O), 150.95 (C═O), 150.40 (C—Cl), 149.94 (CH), 139.64 (C), 138.87 (C), 135.94 (C), 129.94 (C), 128.73 (C), 123.99 (C), 123.39 (CH), 115.70 (C), 114.34 (C). IR (ATR, cm^{-1}): 3348 (NH), 3072 (CH), 1680 (C═O), 1730 (C═O), 1580, 734. MS (ESI): *m*/*z* (%): 274.6 (*M*+1).

Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2.

Acknowledgements

The authors thank Andreas Lorbach and Todd B. Marder (Institute of Inorganic Chemistry, Wuerzburg University) for the data collection and structure solution. We appreciate the financial support provided to NSR by the Deutscher Akademischer Austauschdienst (DAAD).

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full crystallographic data

IUCrData (2017). **2**, x170580 [https://doi.org/10.1107/S2414314617005806]

3-(2-Chloropyridin-3-yl)quinazoline-2,4(1*H*,3*H*)-dione chloroform monosolvate

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3-(2-Chloropyridin-3-yl)quinazoline-2,4(1*H*,3*H*)-dione chloroform monosolvate

Crystal data

$C_{13}H_8ClN_3O_2·CHCl_3$
M_r = 393.04
Monoclinic, $P2_1/c$
a = 5.6382 (11) Å
b = 13.622 (3) Å
c = 20.662 (4) Å
β = 92.289 (6)°
V = 1585.7 (5) Å^3
Z = 4
$F(000)$ = 792
D_x = 1.646 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 3659 reflections
θ = 2.5–26.4°
μ = 0.76 mm^{-1}
T = 100 K
Block, colourless
0.59 × 0.32 × 0.26 mm

Data collection

Bruker APEXII CCD diffractometer
φ and ω scans
Absorption correction: multi-scan (SADABS; Bruker, 2013)
T_{min} = 0.656, T_{max} = 0.980
15463 measured reflections
3370 independent reflections
2796 reflections with $I > 2\sigma(I)$
R_{int} = 0.054
θ_{max} = 26.8°, θ_{min} = 1.8°
h = −7→7
k = −17→16
l = −26→26

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.040
$wR(F^2)$ = 0.109
S = 1.08
3370 reflections
208 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
$w = 1/[\sigma^2(F_o^2) + (0.0529P)^2 + 0.6894P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.45 e Å^{-3}
$\Delta\rho_{min}$ = −0.49 e Å^{-3}

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

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

	x	*y*	*z*	U_{iso}*/U_{eq}
Cl1	0.12965 (11)	0.78921 (4)	0.58061 (3)	0.02149 (16)
Cl2	0.36470 (11)	0.13451 (5)	0.50999 (3)	0.02813 (17)
Cl3	0.40964 (14)	0.12297 (5)	0.64924 (3)	0.03261 (19)
Cl4	−0.03576 (12)	0.07201 (5)	0.58295 (4)	0.03620 (19)
O2	0.1523 (3)	0.60895 (12)	0.73418 (7)	0.0176 (4)
O1	0.5724 (3)	0.60283 (12)	0.55085 (8)	0.0213 (4)
N2	0.3498 (3)	0.60913 (13)	0.64027 (9)	0.0136 (4)
N1	0.4692 (4)	0.87525 (14)	0.64811 (9)	0.0186 (4)
N3	0.2842 (4)	0.48850 (14)	0.56200 (9)	0.0172 (4)
H3	0.3204	0.4603	0.5254	0.021*
C10	−0.2711 (4)	0.36988 (17)	0.66329 (12)	0.0217 (5)
H10	−0.3989	0.3427	0.6859	0.026*
C11	−0.1409 (4)	0.44692 (16)	0.69008 (11)	0.0172 (5)
H11	−0.1781	0.4724	0.7313	0.021*
C5	0.6601 (4)	0.87655 (17)	0.68915 (11)	0.0191 (5)
H5	0.7306	0.9381	0.6997	0.023*
C6	0.4117 (4)	0.56808 (16)	0.58157 (10)	0.0158 (5)
C4	0.7592 (4)	0.79289 (17)	0.71677 (11)	0.0186 (5)
H4	0.8932	0.7968	0.7460	0.022*
C12	0.0464 (4)	0.48717 (16)	0.65602 (10)	0.0139 (4)
C9	−0.2150 (4)	0.33221 (17)	0.60333 (12)	0.0226 (5)
H9	−0.3059	0.2795	0.5853	0.027*
C3	0.6576 (4)	0.70314 (16)	0.70056 (11)	0.0168 (5)
H3A	0.7219	0.6441	0.7184	0.020*
C8	−0.0304 (5)	0.36989 (17)	0.56961 (12)	0.0209 (5)
H8	0.0080	0.3428	0.5290	0.025*
C7	0.1001 (4)	0.44849 (16)	0.59596 (11)	0.0156 (5)
C13	0.1806 (4)	0.57113 (16)	0.68188 (10)	0.0137 (4)
C2	0.4630 (4)	0.70030 (16)	0.65844 (10)	0.0135 (4)
C1	0.3755 (4)	0.78838 (16)	0.63368 (10)	0.0154 (5)
C14	0.2739 (4)	0.07115 (17)	0.57913 (11)	0.0181 (5)
H14	0.3274	0.0014	0.5759	0.022*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.0248 (3)	0.0162 (3)	0.0229 (3)	0.0005 (2)	−0.0065 (2)	0.0010 (2)
Cl2	0.0266 (3)	0.0344 (4)	0.0234 (3)	−0.0062 (3)	0.0005 (2)	0.0095 (2)
Cl3	0.0510 (5)	0.0250 (3)	0.0219 (3)	−0.0152 (3)	0.0019 (3)	−0.0036 (2)
Cl4	0.0211 (3)	0.0288 (4)	0.0594 (5)	−0.0018 (3)	0.0103 (3)	0.0094 (3)
O2	0.0216 (9)	0.0148 (8)	0.0166 (8)	−0.0002 (7)	0.0025 (6)	−0.0021 (6)
O1	0.0281 (10)	0.0146 (8)	0.0218 (9)	−0.0078 (7)	0.0100 (7)	−0.0063 (6)
N2	0.0179 (10)	0.0071 (9)	0.0159 (9)	−0.0027 (7)	0.0020 (7)	−0.0021 (7)
N1	0.0277 (11)	0.0089 (9)	0.0191 (10)	−0.0023 (8)	0.0004 (8)	0.0001 (7)
N3	0.0237 (11)	0.0113 (9)	0.0168 (9)	−0.0052 (8)	0.0040 (8)	−0.0045 (7)

C10	0.0199 (13)	0.0121 (12)	0.0331 (14)	−0.0036 (10)	0.0026 (10)	0.0036 (9)
C11	0.0175 (12)	0.0120 (11)	0.0222 (12)	0.0021 (9)	0.0019 (9)	0.0026 (9)
C5	0.0266 (13)	0.0110 (11)	0.0197 (12)	−0.0048 (10)	0.0023 (9)	−0.0029 (8)
C6	0.0203 (12)	0.0096 (11)	0.0175 (11)	−0.0009 (9)	0.0018 (9)	−0.0006 (8)
C4	0.0192 (12)	0.0172 (12)	0.0194 (12)	−0.0025 (10)	−0.0010 (9)	−0.0026 (9)
C12	0.0153 (11)	0.0074 (10)	0.0189 (11)	0.0017 (9)	−0.0008 (8)	0.0022 (8)
C9	0.0228 (13)	0.0104 (11)	0.0344 (14)	−0.0053 (10)	−0.0006 (10)	−0.0018 (10)
C3	0.0206 (12)	0.0111 (11)	0.0190 (11)	0.0020 (9)	0.0035 (9)	−0.0009 (8)
C8	0.0274 (13)	0.0115 (11)	0.0239 (12)	−0.0021 (10)	0.0010 (10)	−0.0032 (9)
C7	0.0183 (12)	0.0081 (10)	0.0203 (11)	0.0006 (9)	0.0003 (9)	0.0014 (8)
C13	0.0150 (11)	0.0098 (10)	0.0165 (11)	0.0043 (9)	0.0009 (8)	0.0021 (8)
C2	0.0172 (11)	0.0089 (10)	0.0148 (10)	−0.0018 (9)	0.0053 (8)	−0.0020 (8)
C1	0.0197 (12)	0.0113 (11)	0.0152 (11)	−0.0011 (9)	0.0009 (8)	−0.0004 (8)
C14	0.0210 (12)	0.0113 (11)	0.0221 (12)	−0.0012 (10)	0.0024 (9)	−0.0004 (9)

Geometric parameters (Å, º)

Cl1—C1	1.733 (2)	C11—H11	0.9500
Cl2—C14	1.763 (2)	C11—C12	1.404 (3)
Cl3—C14	1.759 (2)	C5—H5	0.9500
Cl4—C14	1.751 (2)	C5—C4	1.383 (3)
O2—C13	1.214 (3)	C4—H4	0.9500
O1—C6	1.222 (3)	C4—C3	1.385 (3)
N2—C6	1.393 (3)	C12—C7	1.392 (3)
N2—C13	1.408 (3)	C12—C13	1.461 (3)
N2—C2	1.439 (3)	C9—H9	0.9500
N1—C5	1.343 (3)	C9—C8	1.375 (4)
N1—C1	1.325 (3)	C3—H3A	0.9500
N3—H3	0.8800	C3—C2	1.373 (3)
N3—C6	1.354 (3)	C8—H8	0.9500
N3—C7	1.387 (3)	C8—C7	1.397 (3)
C10—H10	0.9500	C2—C1	1.387 (3)
C10—C11	1.383 (3)	C14—H14	1.0000
C10—C9	1.389 (3)		
C6—N2—C13	125.71 (19)	C8—C9—C10	121.2 (2)
C6—N2—C2	116.71 (18)	C8—C9—H9	119.4
C13—N2—C2	117.53 (18)	C4—C3—H3A	120.3
C1—N1—C5	117.1 (2)	C2—C3—C4	119.3 (2)
C6—N3—H3	117.9	C2—C3—H3A	120.3
C6—N3—C7	124.24 (19)	C9—C8—H8	120.5
C7—N3—H3	117.9	C9—C8—C7	119.1 (2)
C11—C10—H10	120.0	C7—C8—H8	120.5
C11—C10—C9	120.1 (2)	N3—C7—C12	119.7 (2)
C9—C10—H10	120.0	N3—C7—C8	119.8 (2)
C10—C11—H11	120.2	C12—C7—C8	120.5 (2)
C10—C11—C12	119.6 (2)	O2—C13—N2	120.3 (2)
C12—C11—H11	120.2	O2—C13—C12	125.0 (2)

N1—C5—H5	118.3	N2—C13—C12	114.72 (18)
N1—C5—C4	123.4 (2)	C3—C2—N2	121.6 (2)
C4—C5—H5	118.3	C3—C2—C1	118.2 (2)
O1—C6—N2	120.9 (2)	C1—C2—N2	120.2 (2)
O1—C6—N3	123.4 (2)	N1—C1—Cl1	115.99 (17)
N3—C6—N2	115.66 (19)	N1—C1—C2	123.8 (2)
C5—C4—H4	120.9	C2—C1—Cl1	120.18 (17)
C5—C4—C3	118.1 (2)	Cl2—C14—H14	108.3
C3—C4—H4	120.9	Cl3—C14—Cl2	109.87 (12)
C11—C12—C13	120.8 (2)	Cl3—C14—H14	108.3
C7—C12—C11	119.6 (2)	Cl4—C14—Cl2	110.84 (13)
C7—C12—C13	119.6 (2)	Cl4—C14—Cl3	111.24 (13)
C10—C9—H9	119.4	Cl4—C14—H14	108.3
N2—C2—C1—Cl1	−0.1 (3)	C4—C3—C2—C1	0.0 (3)
N2—C2—C1—N1	179.9 (2)	C9—C10—C11—C12	0.6 (4)
N1—C5—C4—C3	0.8 (4)	C9—C8—C7—N3	−178.8 (2)
C10—C11—C12—C7	−0.5 (3)	C9—C8—C7—C12	1.0 (4)
C10—C11—C12—C13	177.5 (2)	C3—C2—C1—Cl1	−179.73 (17)
C10—C9—C8—C7	−1.0 (4)	C3—C2—C1—N1	0.2 (3)
C11—C10—C9—C8	0.2 (4)	C7—N3—C6—O1	−179.5 (2)
C11—C12—C7—N3	179.6 (2)	C7—N3—C6—N2	0.9 (3)
C11—C12—C7—C8	−0.3 (3)	C7—C12—C13—O2	−178.2 (2)
C11—C12—C13—O2	3.9 (3)	C7—C12—C13—N2	3.1 (3)
C11—C12—C13—N2	−174.84 (19)	C13—N2—C6—O1	−175.0 (2)
C5—N1—C1—Cl1	−179.98 (17)	C13—N2—C6—N3	4.6 (3)
C5—N1—C1—C2	0.1 (3)	C13—N2—C2—C3	84.3 (3)
C5—C4—C3—C2	−0.5 (3)	C13—N2—C2—C1	−95.3 (2)
C6—N2—C13—O2	174.7 (2)	C13—C12—C7—N3	1.6 (3)
C6—N2—C13—C12	−6.5 (3)	C13—C12—C7—C8	−178.3 (2)
C6—N2—C2—C3	−98.0 (3)	C2—N2—C6—O1	7.4 (3)
C6—N2—C2—C1	82.4 (3)	C2—N2—C6—N3	−172.91 (19)
C6—N3—C7—C12	−3.9 (3)	C2—N2—C13—O2	−7.8 (3)
C6—N3—C7—C8	176.0 (2)	C2—N2—C13—C12	171.01 (19)
C4—C3—C2—N2	−179.6 (2)	C1—N1—C5—C4	−0.6 (4)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N3—H3···O1^{i}	0.88	1.91	2.791 (3)	175
C14—H14···N1^{ii}	1.00	2.39	3.200 (3)	137
C3—H3*A*···O2^{iii}	0.95	2.48	3.123 (3)	125

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