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Crystal structures of 4-phenylpiperazin-1-ium 6-chloro-5-ethyl-2,4-dioxopyrimidin-1-ide and 4-phenylpiperazin-1-ium 6-chloro-5-isopropyl-2,4-dioxopyrimidin-1-ide

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Crystal structures of 4-phenylpiperazin-1-ium 6-chloro-5-ethyl-2,4-dioxopyrimidin-1-ide and 4-phenylpiperazin-1-ium 6-chloro-5-isopropyl-2,4-dioxopyrimidin-1-ide

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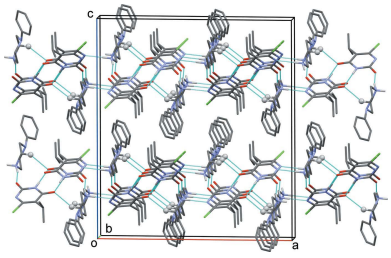
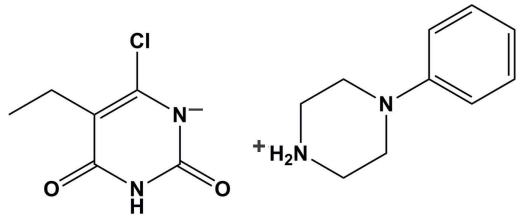
The title molecular salts, $C_{10}H_{15}N_2^+\cdot C_6H_6ClN_2O_2^-$, (I), and $C_{10}H_{15}N_2^+\cdot C_7H_8ClN_2O_2^-$, (II), consist of 4-phenylpiperazin-1-ium cations with a 6-chloro-5-ethyl-2,4-dioxopyrimidin-1-ide anion in (I) and a 6-chloro-5-isopropyl-2,4-dioxopyrimidin-1-ide anion in (II). Salt (I) crystallizes with two independent cations and anions in the asymmetric unit. In the crystal structures of both salts, the ions are linked *via* N—H···O and N—H···N hydrogen bonds, forming sheets which are parallel to (100) in (I) and to (001) in (II). In (I), the sheets are linked *via* C—H···Cl hydrogen bonds, forming a three-dimensional framework.

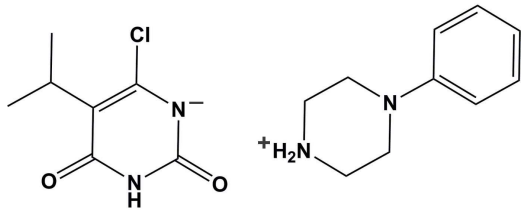
1. Chemical context

2,4-Dioxopyrimidine derivatives (uracils) and their related analogues are known for their diverse chemotherapeutic activities including anticancer activity (Ghoshal & Jacob, 1997; Spáčilová *et al.*, 2007; Blokhina *et al.*, 1972), anti-HIV activity (Tanaka *et al.*, 1995; El-Emam *et al.*, 2004) and antibacterial activity (Al-Turkistani *et al.*, 2011). In addition, the piperazine nucleus constitutes the core pharmacophore of several biologically active compounds which display antiviral (Romero *et al.*, 1994, 1996), anticancer (Fytas *et al.*, 2015; Kamal *et al.*, 2015; Arnatt *et al.*, 2014), antitubercular and antibacterial (Nagesh *et al.*, 2014; Peng *et al.*, 2015; Kapić *et al.*, 2011; Wang *et al.*, 2014) and central nervous system activities (Bender *et al.*, 2014; Bali *et al.*, 2010).





(I)



(II)

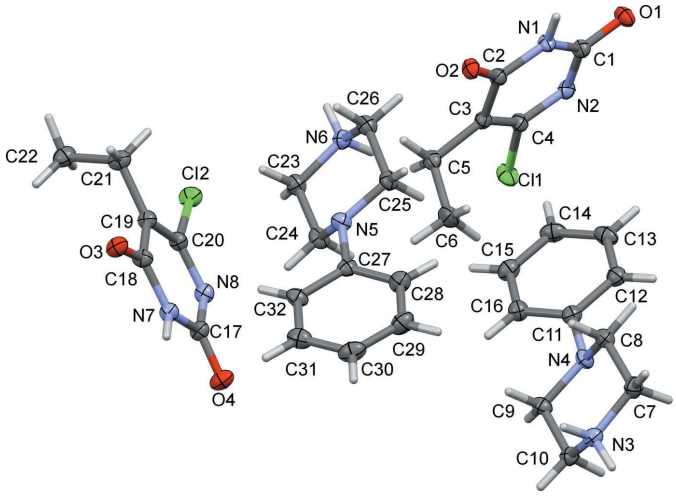


Figure 1
The molecular structure of compound (I), showing the atom labelling. Displacement ellipsoids are drawn at the 50% probability level.

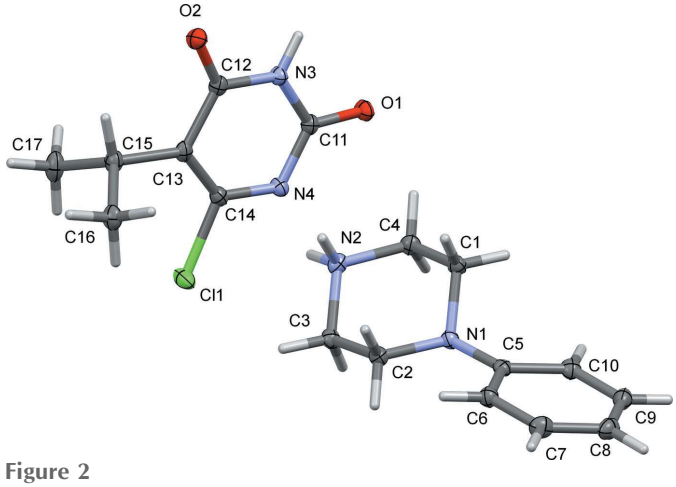


Figure 2
The molecular structure of compound (II), showing the atom labelling. Displacement ellipsoids are drawn at the 50% probability level.

As a result of the relative acidity of 2,4-dioxopyrimidines (Kurinovich & Lee, 2002; Jang *et al.*, 2001; Nguyen *et al.*, 1998), the title piperazinium salts were isolated as minor byproducts during the reaction of 1-phenylpiperazine with 5-alkyl-6-chlorouracils (Al-Turkistani *et al.*, 2011). In a continuation of our interest in the structures of piperazinium salts (Al-Omary *et al.*, 2014), we report herein on the isolation and crystal structures of these two new piperazinium salts, (I) and (II).

2. Structural commentary

The molecular structures of the title salts (I) and (II) are illustrated in Figs. 1 and 2, respectively. Compound (I) crystallizes with two independent 4-phenylpiperazin-1-ium cations (*A* and *B*) and two independent 6-chloro-5-ethyl-2, 4-dioxopyrimidin-1-ide anions (*C* and *D*) in the asymmetric unit. In both compounds, the piperazine rings adopt a distorted chair conformation with a positively charged quaternary N atom **[OK? Or does quaternary mean attached to four C atoms, not a protonated N atom?]**. In compound (I), the mean plane of the piperazine ring makes a dihedral angle of 34.8 (2)° with the attached phenyl ring in cation *A*, and 39.7 (2)° in cation *B*. The equivalent dihedral angle is 39.61 (9)° in the cation of compound (II). In the uracil anions, the pyrimidine rings are almost planar with r.m.s. deviations of 0.008 Å in both anions (*C* and *D*) of compound (I), and 0.024 Å in compound (II).

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···O2^{i}	0.86	2.00	2.859 (4)	173
N3—H3*A*···O1^{ii}	0.89	2.83	3.465 (4)	129
N6—H6*A*···O4^{iii}	0.89	1.81	2.681 (5)	165
N7—H7···O3^{iv}	0.86	2.02	2.873 (4)	174
N3—H3*A*···N2^{ii}	0.89	1.92	2.808 (4)	174
N6—H6*B*···N8^{v}	0.89	1.92	2.798 (5)	169
C10—H10*B*···O2^{vi}	0.97	2.46	3.355 (5)	154
C26—H26*A*···O3^{vii}	0.97	2.58	3.444 (5)	147
C16—H16···Cl2^{viii}	0.93	2.80	3.462 (4)	129

Symmetry codes: (i) $-x+1, -y-1, -z+2$; (ii) $x, y+1, z$; (iii) $x, y-1, z$; (iv) $-x+2, -y+1, -z+2$; (v) $-x+2, y-\frac{1}{2}, -z+\frac{3}{2}$; (vi) $x, -y+\frac{1}{2}, z-\frac{1}{2}$; (vii) $-x+2, -y, -z+2$; (viii) $-x+2, y+\frac{1}{2}, -z+\frac{3}{2}$.

3. Supramolecular features

In the crystal of (I), two tetranuclear units are formed, involving cation *A* and anion *B*, and cation *C* and anion *D* **[this nomenclature is totally inconsistent with lines 167 and 168, where the cations are called A and B]**, *via* N—H···O and C—H···O hydrogen bonds. These units are linked *via* N—H···N hydrogen bonds, forming separate *A*/*B* and *C*/*D* sheets parallel to the *bc* plane (Table 1 and Fig. 3). The sheets are linked *via* C—H···Cl hydrogen bonds, forming a three-dimensional framework (Fig. 3 and Table 1).

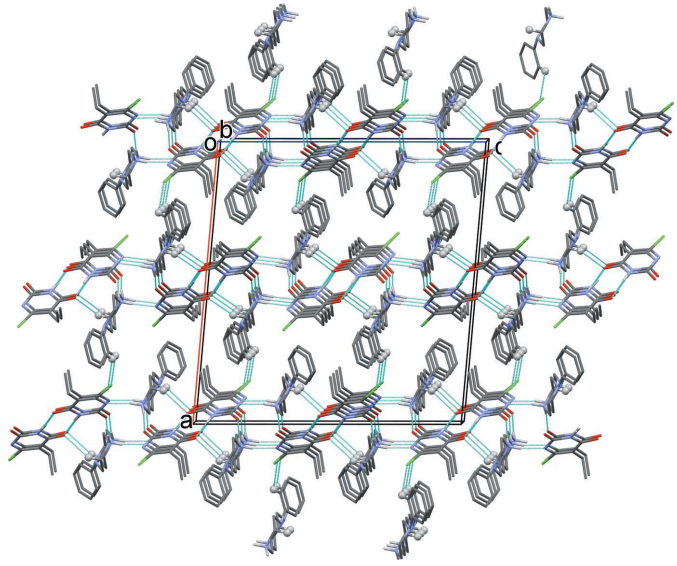


Figure 3
The crystal packing of compound (I), viewed along the *b* axis, showing the **most relevant [OK?]** hydrogen bonding (dashed lines; see Table 1).

Files: e/su5162/su5162.3d e/su5162/su5162.sgml **SU5162** GO IU-1510/42(15)7 159/32(13)7 (77) SU5162 PROOFS E:GO:2015:71:1:0:0–0

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

D—H···A	D—H	H···A	D···A	D—H···A
N2—H2*N*···N4	0.89	1.93	2.813 (2)	174
N2—H3*N*···O1^{i}	0.89	1.84	2.705 (2)	164
N3—H3···O2^{ii}	0.86	1.98	2.834 (2)	174
C3—H3*A*···O2^{iii}	0.97	2.54	3.394 (2)	147

Symmetry codes: (i) $-x+1, y-\frac{1}{2}, -z+\frac{1}{2}$; (ii) $-x+\frac{1}{2}, -y+\frac{5}{2}, -z+\frac{1}{2}$; (iii) $x+\frac{1}{2}, -y+2, z$.

In the crystal of (II), the cation and anion are linked by N—H···O and C—H···O hydrogen bonds, forming chains extending along the *b*-axis direction. The chains are linked *via* N—H···N hydrogen bonds, forming sheets lying parallel to the *ac* plane (Table 2 and Fig. 4).

4. Database survey

A search of the Cambridge Structural Database (Version 5.36, last update November 2014; Groom & Allen, 2014) for the anion 6-chloro-5-ethyl-2,4-dioxopyrimidin-1-ide, present in compound (I), gave no hits, while for the anion 6-chloro-5-isopropyl-2,4-dioxopyrimidin-1-ide, present in compound (II), one hit was obtained, with the cation 4-(2-methoxyphenyl)piperazin-1-ium (Al-Omary *et al.*, 2014).

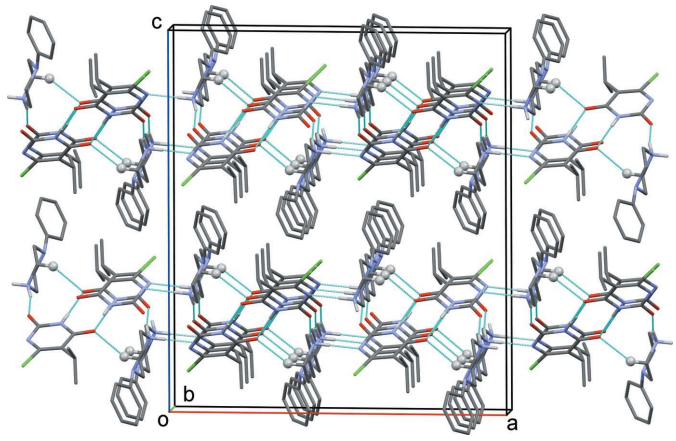


Figure 4
The crystal packing of compound (II), viewed along the *b* axis, showing the **most relevant [OK?]** hydrogen bonding (dashed lines; see Table 2).

5. Synthesis and crystallization

Compound (I): A mixture of 6-chloro-5-ethyluracil (349 mg, 2.0 mmol), 1-phenylpiperazine (325 mg, 2.0 mmol) and anhydrous potassium carbonate (276 mg, 2.0 mmol), in ethanol (8 ml), was heated under reflux for 6 h. On cooling, the precipitate, thus formed was separated by filtration to yield 306 mg (51%) of 5-ethyl-6-(4-phenyl-1-piperazinyl)uracil. The filtrate was concentrated by vacuum distillation to 5 ml and allowed to stand at room temperature overnight to yield

Table 3
Experimental details.

	(I)	(II)
Crystal data		
Chemical formula	$C_{10}H_{15}N_2^+\cdot C_6H_6ClN_2O_2^-$	$C_{10}H_{15}N_2^+\cdot C_7H_8ClN_2O_2^-$
M_r	336.82	350.84
Crystal system, space group	Monoclinic, $P2_1/c$	Monoclinic, $I2/a$
Temperature (K)	293	101
a, b, c (Å)	21.676 (1), 7.6446 (5), 20.5444 (8)	20.5012 (3), 7.4565 (1), 23.1414 (3)
β (°)	95.065 (5)	90.639 (1)
V (Å^3)	3391.0 (3)	3537.34 (8)
Z	8	8
Radiation type	Cu $K\alpha$	Cu $K\alpha$
μ (mm^{-1})	2.12	2.05
Crystal size (mm)	0.17 × 0.08 × 0.06	0.34 × 0.13 × 0.09
Data collection		
Diffractometer	Agilent Xcalibur Ruby Gemini	Agilent Xcalibur Ruby Gemini
Absorption correction	Multi-scan (*CrysAlis PRO*; Agilent, 2014)	Multi-scan (*CrysAlis PRO*; Agilent, 2014)
T_{min}, T_{max}	0.809, 0.880	0.760, 0.828
No. of measured, independent and observed $[I > 2\sigma(I)]$ reflections	32461, 6532, 3596	13174, 3396, 2926
R_{int}	0.135	0.069
$(\sin\theta/\lambda)_{max}$ (Å^{-1})	0.612	0.612
Refinement		
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.066, 0.185, 1.01	0.044, 0.122, 1.03
No. of reflections	6457	3346
No. of parameters	415	217
H-atom treatment	H-atom parameters constrained	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.42, −0.36	0.55, −0.56

Computer programs: *CrysAlis PRO* (Agilent, 2014), *SIR2011* (Burla *et al.*, 2012), *Mercury* (Macrae *et al.*, 2008), *SHELXL2014* (Sheldrick, 2015), *PLATON* (Spek, 2009) and *publCIF* (Westrip, 2010).

compound (I) as colourless crystals (m.p.: 459–461 K). ^{1}H NMR (DMSO-*d*6, 500.13 MHz): δ 0.93 (*t*, 3H, CH_3, J = 7.0 Hz), 2.35 (*q*, 2H, CH_2), 3.25 (*s*, 4H, piperazine-H), 3.45 (*s*, 4H, piperazine-H), 6.83–6.95 (*m*, 3H, Ar—H), 7.21 (*d*, 2H, Ar—H, J = 6.6 Hz), 8.15–8.17 (*m*, 2H, NH_2), 10.83 (*s*, 1H, NH). ^{13}C NMR (DMSO-*d*6, 125.76 MHz): δ 13.80 (CH_3), 19.55 (CH_2), 44.18, 47.86 (piperazine-C), 116.32, 119.62, 128.44, 150.70 (Ar—C), 108.88, 153.90, 155.94, 164.80 (pyrimidine-C),

Compound (II): 6-Chloro-5-isopropyluracil (377 mg, 2.0 mmol), 1-phenylpiperazine (325 mg, 2.0 mmol) and anhydrous potassium carbonate (276 mg, 2.0 mmol), in ethanol (8 ml), was heated under reflux for 6 h. On cooling, the precipitate thus formed was separated by filtration to yield 566 mg (90%) of 5-isopropyl-6-(4-phenyl-1-piperazinyl)uracil. The filtrate was concentrated by vacuum distillation to 5 ml and allowed to stand at room temperature overnight to yield compound (II) as colourless crystals (m.p.: 473–475 K). ^{1}H NMR (DMSO-d_6, 500.13 MHz): δ 1.20 (*d*, 6H, CH_3, J = 7.8 Hz), 2.52–2.56 (*m*, 1H, CH), 3.18 (*s*, 4H, piperazine-H), 3.24 (*s*, 4H, piperazine-H), 6.88–7.02 (*m*, 3H, Ar—H), 7.20–7.22 (*m*, 2H, Ar—H), 8.04–8.08 (*m*, 2H, NH_2), 11.02 (*s*, 1H, NH). ^{13}C NMR (DMSO-d_6, 125.76 MHz): δ 19.98 (CH_3), 27.0 (CH), 44.50, 47.98 (piperazine-C), 116.16, 119.80, 129.04, 150.0 (Ar—C), 110.82, 152.30, 154.04, 164.06 (pyrimidine-C).

6. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 3. The H atoms were included in calculated positions and treated as riding atoms: N—H = 0.86–0.90 Å, C—H = 0.95–1.00 Å with U_{iso}(H) = 1.5U_{eq}(C) for methyl H atoms and 1.2U_{eq}(N,C) for other H atoms.

Acknowledgements

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supporting information

Crystal structures of 4-phenylpiperazin-1-ium 6-chloro-5-ethyl-2,4-dioxopyrimidin-1-ide and 4-phenylpiperazin-1-ium 6-chloro-5-isopropyl-2,4-dioxopyrimidin-1-ide

Monirah A. Al-Alshaikh, Ali A. El-Emam,* Omar A. Al-Deeb, Mohammed S. M. Abdelbaky and Santiago Garcia-Granda*

Computing details

For both compounds, data collection: *CrysAlis PRO* (Agilent, 2014); cell refinement: *CrysAlis PRO* (Agilent, 2014); data reduction: *CrysAlis PRO* (Agilent, 2014); program(s) used to solve structure: *SIR2011* (Burla *et al.*, 2012); program(s) used to refine structure: *SHELXL2014* (Sheldrick, 2015); molecular graphics: *Mercury* (Macrae *et al.*, 2008); software used to prepare material for publication: *SHELXL2014* (Sheldrick, 2015), *PLATON* (Spek, 2009) and *publCIF* (Westrip, 2010).

(I) 4-Phenylpiperazin-1-ium 6-chloro-5-ethyl-2,4-dioxopyrimidin-1-ide

Crystal data

$C_{10}H_{15}N_2^+ \cdot C_6H_6ClN_2O_2^-$
M_r = 336.82
Monoclinic, $P2_1/c$
a = 21.676 (1) Å
b = 7.6446 (5) Å
c = 20.5444 (8) Å
β = 95.065 (5)°
V = 3391.0 (3) Å^3
Z = 8
$F(000)$ = 1424
D_x = 1.319 Mg m^{-3}
Cu $K\alpha$ radiation, λ = 1.54184 Å
Cell parameters from 2418 reflections
θ = 4.1–70.3°
μ = 2.12 mm^{-1}
T = 293 K
Prism, colourless
0.17 × 0.08 × 0.06 mm

Data collection

Agilent Xcalibur Ruby Gemini diffractometer
Radiation source: Enhance (Cu) X-ray Source
Detector resolution: 10.2673 pixels mm^{-1}
ω scans
Absorption correction: multi-scan (*CrysAlis PRO*; Agilent, 2014)
T_{min} = 0.809, T_{max} = 0.880
32461 measured reflections
6532 independent reflections
3596 reflections with $I > 2\sigma(I)$
R_{int} = 0.135
θ_{max} = 70.7°, θ_{min} = 4.1°
h = −26→26
k = −9→9
l = −21→25

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.066
$wR(F^2)$ = 0.185
S = 1.01
6457 reflections
415 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

$w = 1/[\sigma^2(F_o^2) + (0.0563P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.42$ e Å^{-3}
$\Delta\rho_{min} = -0.36$ e Å^{-3}

Special details

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

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

	x	*y*	*z*	U_{iso}*/U_{eq}
Cl2	1.13235 (5)	0.04012 (14)	0.82294 (5)	0.0408 (3)
Cl1	0.65035 (5)	−0.09582 (15)	0.82219 (5)	0.0437 (3)
O1	0.51594 (14)	−0.6011 (4)	0.82514 (13)	0.0381 (7)
O2	0.55114 (13)	−0.3192 (4)	1.02030 (13)	0.0344 (7)
O3	1.04166 (14)	0.2995 (4)	1.02077 (13)	0.0378 (7)
N1	0.53525 (15)	−0.4583 (4)	0.92238 (15)	0.0288 (8)
H1	0.5119	−0.5322	0.9401	0.035*
O4	1.02351 (14)	0.5927 (4)	0.82725 (14)	0.0415 (8)
N7	1.03409 (15)	0.4421 (4)	0.92325 (15)	0.0306 (8)
H7	1.0140	0.5236	0.9410	0.037*
N2	0.57984 (15)	−0.3662 (4)	0.82841 (15)	0.0301 (8)
N8	1.07502 (16)	0.3338 (5)	0.82954 (16)	0.0330 (8)
N3	0.56236 (15)	0.6236 (4)	0.69138 (16)	0.0320 (8)
H3A	0.5695	0.6194	0.7347	0.038*
H3B	0.5338	0.7053	0.6817	0.038*
N4	0.64335 (15)	0.3661 (5)	0.64533 (16)	0.0330 (8)
N5	0.86014 (15)	0.0992 (4)	0.85634 (16)	0.0325 (8)
N6	0.93121 (15)	−0.1748 (4)	0.80703 (16)	0.0337 (8)
H6A	0.9565	−0.2655	0.8153	0.040*
H6B	0.9245	−0.1637	0.7639	0.040*
C1	0.54296 (19)	−0.4798 (5)	0.85679 (19)	0.0313 (9)
C18	1.05419 (19)	0.3021 (5)	0.9626 (2)	0.0328 (10)
C17	1.04364 (19)	0.4618 (6)	0.85811 (19)	0.0313 (9)
C4	0.60638 (18)	−0.2374 (5)	0.8663 (2)	0.0315 (9)
C2	0.56213 (18)	−0.3272 (5)	0.9617 (2)	0.0308 (9)
C20	1.09503 (18)	0.1988 (5)	0.8674 (2)	0.0309 (9)
C11	0.68606 (18)	0.2301 (5)	0.6354 (2)	0.0311 (9)
C9	0.6682 (2)	0.5311 (5)	0.6731 (2)	0.0345 (10)
H9A	0.7051	0.5634	0.6524	0.041*
H9B	0.6796	0.5166	0.7195	0.041*
C27	0.82314 (18)	0.2433 (5)	0.8709 (2)	0.0315 (9)
C3	0.60166 (18)	−0.2074 (5)	0.93082 (19)	0.0301 (9)
C25	0.82936 (19)	−0.0520 (5)	0.8253 (2)	0.0341 (10)
H25A	0.8201	−0.0296	0.7789	0.041*

H25B	0.7907	−0.0736	0.8443	0.041*
C19	1.08842 (19)	0.1695 (5)	0.93181 (19)	0.0316 (9)
C23	0.96153 (19)	−0.0141 (6)	0.8346 (2)	0.0357 (10)
H23A	0.9982	0.0104	0.8123	0.043*
H23B	0.9742	−0.0317	0.8806	0.043*
C21	1.1136 (2)	0.0116 (6)	0.9694 (2)	0.0377 (10)
H21A	1.1044	−0.0924	0.9433	0.045*
H21B	1.0928	0.0007	1.0091	0.045*
C28	0.7616 (2)	0.2637 (6)	0.8454 (2)	0.0397 (11)
H28	0.7448	0.1857	0.8139	0.048*
C10	0.62039 (19)	0.6734 (5)	0.6625 (2)	0.0336 (10)
H10A	0.6365	0.7809	0.6825	0.040*
H10B	0.6116	0.6941	0.6161	0.040*
C5	0.6318 (2)	−0.0569 (5)	0.9687 (2)	0.0359 (10)
H5A	0.6721	−0.0353	0.9533	0.043*
H5B	0.6381	−0.0893	1.0145	0.043*
C7	0.53832 (19)	0.4524 (6)	0.6672 (2)	0.0378 (10)
H7A	0.5248	0.4610	0.6211	0.045*
H7B	0.5029	0.4196	0.6903	0.045*
C16	0.74788 (19)	0.2396 (6)	0.6587 (2)	0.0357 (10)
H16	0.7618	0.3334	0.6849	0.043*
C15	0.7893 (2)	0.1105 (6)	0.6436 (2)	0.0412 (11)
H15	0.8308	0.1192	0.6592	0.049*
C26	0.87103 (19)	−0.2105 (6)	0.8348 (2)	0.0371 (10)
H26A	0.8784	−0.2368	0.8811	0.044*
H26B	0.8511	−0.3110	0.8132	0.044*
C12	0.6660 (2)	0.0859 (6)	0.5972 (2)	0.0369 (10)
H12	0.6246	0.0765	0.5812	0.044*
C8	0.58805 (18)	0.3138 (5)	0.6776 (2)	0.0344 (10)
H8A	0.5991	0.2979	0.7240	0.041*
H8B	0.5724	0.2033	0.6597	0.041*
C32	0.8473 (2)	0.3663 (5)	0.9167 (2)	0.0374 (10)
H32	0.8884	0.3573	0.9337	0.045*
C24	0.91766 (19)	0.1391 (6)	0.8266 (2)	0.0362 (10)
H24A	0.9370	0.2417	0.8473	0.043*
H24B	0.9083	0.1645	0.7805	0.043*
C29	0.7254 (2)	0.3984 (7)	0.8662 (2)	0.0480 (12)
H29	0.6843	0.4089	0.8493	0.058*
C31	0.8113 (2)	0.4999 (6)	0.9369 (2)	0.0442 (12)
H31	0.8282	0.5796	0.9677	0.053*
C13	0.7077 (2)	−0.0424 (6)	0.5832 (2)	0.0423 (11)
H13	0.6938	−0.1384	0.5582	0.051*
C22	1.1830 (2)	0.0208 (6)	0.9874 (2)	0.0433 (11)
H22A	1.1963	−0.0826	1.0111	0.065*
H22B	1.2040	0.0288	0.9483	0.065*
H22C	1.1924	0.1219	1.0141	0.065*
C14	0.7692 (2)	−0.0311 (6)	0.6054 (2)	0.0450 (12)
H14	0.7970	−0.1173	0.5948	0.054*

C30	0.7503 (2)	0.5173 (6)	0.9120 (2)	0.0487 (13)
H30	0.7261	0.6082	0.9259	0.058*
C6	0.5943 (2)	0.1094 (6)	0.9627 (2)	0.0529 (13)
H6C	0.6158	0.2002	0.9878	0.079*
H6D	0.5887	0.1440	0.9177	0.079*
H6E	0.5546	0.0899	0.9788	0.079*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl2	0.0470 (6)	0.0405 (6)	0.0359 (6)	0.0097 (5)	0.0099 (5)	−0.0029 (5)
Cl1	0.0503 (7)	0.0474 (7)	0.0347 (6)	−0.0174 (5)	0.0101 (5)	0.0010 (5)
O1	0.0488 (18)	0.0360 (17)	0.0301 (16)	−0.0110 (14)	0.0063 (14)	−0.0051 (13)
O2	0.0396 (17)	0.0410 (17)	0.0226 (15)	−0.0075 (13)	0.0030 (13)	0.0008 (12)
O3	0.0446 (18)	0.0400 (18)	0.0299 (16)	0.0085 (14)	0.0087 (14)	0.0022 (13)
N1	0.0337 (18)	0.0299 (18)	0.0231 (17)	−0.0075 (15)	0.0036 (14)	0.0018 (14)
O4	0.0462 (19)	0.0444 (19)	0.0349 (17)	0.0130 (15)	0.0087 (14)	0.0079 (14)
N7	0.0323 (18)	0.033 (2)	0.0266 (18)	0.0058 (15)	0.0057 (15)	−0.0034 (15)
N2	0.0301 (18)	0.036 (2)	0.0244 (17)	−0.0008 (15)	0.0033 (14)	0.0004 (15)
N8	0.0331 (19)	0.036 (2)	0.0305 (19)	0.0011 (16)	0.0053 (15)	−0.0007 (16)
N3	0.0349 (19)	0.0336 (19)	0.0280 (18)	0.0064 (15)	0.0052 (15)	−0.0001 (15)
N4	0.0304 (19)	0.033 (2)	0.0355 (19)	−0.0054 (15)	0.0046 (15)	−0.0023 (16)
N5	0.0297 (18)	0.0307 (19)	0.038 (2)	0.0003 (15)	0.0089 (16)	−0.0023 (16)
N6	0.038 (2)	0.035 (2)	0.0287 (19)	0.0004 (16)	0.0042 (16)	−0.0002 (15)
C1	0.032 (2)	0.033 (2)	0.028 (2)	−0.0028 (18)	0.0043 (18)	0.0009 (18)
C18	0.035 (2)	0.032 (2)	0.032 (2)	−0.0007 (18)	0.0079 (19)	0.0012 (18)
C17	0.032 (2)	0.035 (2)	0.028 (2)	0.0040 (18)	0.0048 (18)	0.0007 (18)
C4	0.027 (2)	0.035 (2)	0.033 (2)	−0.0055 (17)	0.0018 (18)	0.0043 (18)
C2	0.029 (2)	0.031 (2)	0.032 (2)	0.0002 (17)	0.0011 (18)	0.0027 (18)
C20	0.028 (2)	0.033 (2)	0.032 (2)	0.0001 (18)	0.0054 (18)	−0.0055 (18)
C11	0.029 (2)	0.033 (2)	0.032 (2)	0.0018 (18)	0.0075 (18)	0.0018 (18)
C9	0.039 (2)	0.029 (2)	0.036 (2)	−0.0052 (19)	0.009 (2)	−0.0006 (19)
C27	0.031 (2)	0.033 (2)	0.031 (2)	−0.0006 (18)	0.0070 (18)	0.0058 (18)
C3	0.031 (2)	0.033 (2)	0.026 (2)	−0.0006 (17)	−0.0008 (17)	0.0012 (17)
C25	0.032 (2)	0.038 (2)	0.032 (2)	−0.0057 (19)	0.0044 (18)	0.0012 (19)
C19	0.034 (2)	0.032 (2)	0.029 (2)	0.0009 (18)	0.0051 (18)	0.0000 (18)
C23	0.031 (2)	0.039 (3)	0.038 (2)	−0.0023 (19)	0.0063 (19)	−0.006 (2)
C21	0.042 (3)	0.038 (2)	0.035 (2)	0.006 (2)	0.010 (2)	0.004 (2)
C28	0.039 (2)	0.044 (3)	0.037 (3)	0.004 (2)	0.006 (2)	−0.001 (2)
C10	0.041 (2)	0.033 (2)	0.027 (2)	0.0013 (19)	0.0050 (19)	0.0011 (18)
C5	0.041 (2)	0.039 (3)	0.028 (2)	−0.009 (2)	0.0027 (19)	0.0014 (19)
C7	0.031 (2)	0.044 (3)	0.038 (2)	0.004 (2)	0.0009 (19)	−0.003 (2)
C16	0.034 (2)	0.038 (2)	0.036 (2)	0.0002 (19)	0.0054 (19)	−0.0014 (19)
C15	0.036 (2)	0.045 (3)	0.043 (3)	0.006 (2)	0.008 (2)	0.004 (2)
C26	0.038 (2)	0.039 (3)	0.034 (2)	−0.006 (2)	0.007 (2)	−0.0022 (19)
C12	0.040 (2)	0.037 (2)	0.034 (2)	0.000 (2)	0.0024 (19)	0.0026 (19)
C8	0.035 (2)	0.031 (2)	0.038 (2)	−0.0043 (19)	0.0045 (19)	−0.0033 (19)
C32	0.039 (2)	0.033 (2)	0.041 (3)	−0.0047 (19)	0.011 (2)	−0.002 (2)

C24	0.032 (2)	0.041 (3)	0.037 (2)	−0.0063 (19)	0.0111 (19)	−0.002 (2)
C29	0.044 (3)	0.055 (3)	0.045 (3)	0.015 (2)	0.006 (2)	0.005 (2)
C31	0.055 (3)	0.037 (3)	0.043 (3)	−0.001 (2)	0.017 (2)	−0.003 (2)
C13	0.058 (3)	0.034 (2)	0.037 (3)	−0.004 (2)	0.014 (2)	−0.006 (2)
C22	0.042 (3)	0.046 (3)	0.043 (3)	0.005 (2)	0.007 (2)	0.009 (2)
C14	0.053 (3)	0.037 (3)	0.047 (3)	0.010 (2)	0.017 (2)	0.002 (2)
C30	0.056 (3)	0.045 (3)	0.047 (3)	0.017 (2)	0.019 (3)	0.005 (2)
C6	0.065 (3)	0.040 (3)	0.051 (3)	0.002 (2)	−0.008 (3)	−0.010 (2)

Geometric parameters (Å, º)

Cl2—C20	1.758 (4)	C19—C21	1.509 (6)
Cl1—C4	1.747 (4)	C23—C24	1.508 (6)
O1—C1	1.249 (5)	C23—H23A	0.9700
O2—C2	1.248 (5)	C23—H23B	0.9700
O3—C18	1.249 (5)	C21—C22	1.517 (6)
N1—C1	1.382 (5)	C21—H21A	0.9700
N1—C2	1.384 (5)	C21—H21B	0.9700
N1—H1	0.8600	C28—C29	1.386 (6)
O4—C17	1.243 (5)	C28—H28	0.9300
N7—C17	1.380 (5)	C10—H10A	0.9700
N7—C18	1.388 (5)	C10—H10B	0.9700
N7—H7	0.8600	C5—C6	1.508 (6)
N2—C1	1.347 (5)	C5—H5A	0.9700
N2—C4	1.351 (5)	C5—H5B	0.9700
N8—C20	1.342 (5)	C7—C8	1.513 (6)
N8—C17	1.354 (5)	C7—H7A	0.9700
N3—C7	1.478 (5)	C7—H7B	0.9700
N3—C10	1.487 (5)	C16—C15	1.388 (6)
N3—H3A	0.8900	C16—H16	0.9300
N3—H3B	0.8900	C15—C14	1.385 (7)
N4—C11	1.419 (5)	C15—H15	0.9300
N4—C9	1.467 (5)	C26—H26A	0.9700
N4—C8	1.475 (5)	C26—H26B	0.9700
N5—C27	1.410 (5)	C12—C13	1.380 (6)
N5—C25	1.454 (5)	C12—H12	0.9300
N5—C24	1.468 (5)	C8—H8A	0.9700
N6—C23	1.482 (5)	C8—H8B	0.9700
N6—C26	1.495 (5)	C32—C31	1.371 (6)
N6—H6A	0.8900	C32—H32	0.9300
N6—H6B	0.8900	C24—H24A	0.9700
C18—C19	1.436 (6)	C24—H24B	0.9700
C4—C3	1.359 (6)	C29—C30	1.383 (7)
C2—C3	1.440 (6)	C29—H29	0.9300
C20—C19	1.362 (6)	C31—C30	1.383 (7)
C11—C16	1.385 (6)	C31—H31	0.9300
C11—C12	1.400 (6)	C13—C14	1.374 (7)
C9—C10	1.505 (6)	C13—H13	0.9300

218	C9—H9A	0.9700	C22—H22A	0.9600
219	C9—H9B	0.9700	C22—H22B	0.9600
220	C27—C28	1.398 (6)	C22—H22C	0.9600
221	C27—C32	1.399 (6)	C14—H14	0.9300
222	C3—C5	1.505 (6)	C30—H30	0.9300
223	C25—C26	1.514 (6)	C6—H6C	0.9600
224	C25—H25A	0.9700	C6—H6D	0.9600
225	C25—H25B	0.9700	C6—H6E	0.9600
226				
227	C1—N1—C2	125.1 (3)	H21A—C21—H21B	107.8
228	C1—N1—H1	117.5	C29—C28—C27	121.0 (5)
229	C2—N1—H1	117.5	C29—C28—H28	119.5
230	C17—N7—C18	125.6 (3)	C27—C28—H28	119.5
231	C17—N7—H7	117.2	N3—C10—C9	110.7 (3)
232	C18—N7—H7	117.2	N3—C10—H10A	109.5
233	C1—N2—C4	117.3 (3)	C9—C10—H10A	109.5
234	C20—N8—C17	117.0 (3)	N3—C10—H10B	109.5
235	C7—N3—C10	112.2 (3)	C9—C10—H10B	109.5
236	C7—N3—H3A	109.2	H10A—C10—H10B	108.1
237	C10—N3—H3A	109.2	C3—C5—C6	113.3 (4)
238	C7—N3—H3B	109.2	C3—C5—H5A	108.9
239	C10—N3—H3B	109.2	C6—C5—H5A	108.9
240	H3A—N3—H3B	107.9	C3—C5—H5B	108.9
241	C11—N4—C9	117.6 (3)	C6—C5—H5B	108.9
242	C11—N4—C8	115.6 (3)	H5A—C5—H5B	107.7
243	C9—N4—C8	110.1 (3)	N3—C7—C8	110.3 (3)
244	C27—N5—C25	117.8 (3)	N3—C7—H7A	109.6
245	C27—N5—C24	116.5 (3)	C8—C7—H7A	109.6
246	C25—N5—C24	110.9 (3)	N3—C7—H7B	109.6
247	C23—N6—C26	112.2 (3)	C8—C7—H7B	109.6
248	C23—N6—H6A	109.2	H7A—C7—H7B	108.1
249	C26—N6—H6A	109.2	C11—C16—C15	120.8 (4)
250	C23—N6—H6B	109.2	C11—C16—H16	119.6
251	C26—N6—H6B	109.2	C15—C16—H16	119.6
252	H6A—N6—H6B	107.9	C14—C15—C16	120.3 (4)
253	O1—C1—N2	121.5 (4)	C14—C15—H15	119.8
254	O1—C1—N1	120.3 (4)	C16—C15—H15	119.8
255	N2—C1—N1	118.2 (4)	N6—C26—C25	109.6 (3)
256	O3—C18—N7	119.1 (4)	N6—C26—H26A	109.8
257	O3—C18—C19	125.1 (4)	C25—C26—H26A	109.8
258	N7—C18—C19	115.8 (3)	N6—C26—H26B	109.8
259	O4—C17—N8	121.8 (4)	C25—C26—H26B	109.8
260	O4—C17—N7	120.4 (4)	H26A—C26—H26B	108.2
261	N8—C17—N7	117.8 (4)	C13—C12—C11	120.0 (4)
262	N2—C4—C3	128.3 (4)	C13—C12—H12	120.0
263	N2—C4—Cl1	112.1 (3)	C11—C12—H12	120.0
264	C3—C4—Cl1	119.5 (3)	N4—C8—C7	110.1 (3)
265	O2—C2—N1	119.5 (4)	N4—C8—H8A	109.6

266	O2—C2—C3	124.5 (4)	C7—C8—H8A	109.6
267	N1—C2—C3	116.1 (3)	N4—C8—H8B	109.6
268	N8—C20—C19	129.3 (4)	C7—C8—H8B	109.6
269	N8—C20—Cl2	111.7 (3)	H8A—C8—H8B	108.2
270	C19—C20—Cl2	119.0 (3)	C31—C32—C27	121.1 (4)
271	C16—C11—C12	118.5 (4)	C31—C32—H32	119.5
272	C16—C11—N4	122.4 (4)	C27—C32—H32	119.5
273	C12—C11—N4	118.9 (4)	N5—C24—C23	110.1 (3)
274	N4—C9—C10	109.9 (4)	N5—C24—H24A	109.6
275	N4—C9—H9A	109.7	C23—C24—H24A	109.6
276	C10—C9—H9A	109.7	N5—C24—H24B	109.6
277	N4—C9—H9B	109.7	C23—C24—H24B	109.6
278	C10—C9—H9B	109.7	H24A—C24—H24B	108.1
279	H9A—C9—H9B	108.2	C30—C29—C28	120.1 (5)
280	C28—C27—C32	117.7 (4)	C30—C29—H29	120.0
281	C28—C27—N5	123.4 (4)	C28—C29—H29	120.0
282	C32—C27—N5	118.8 (4)	C32—C31—C30	120.7 (5)
283	C4—C3—C2	115.0 (4)	C32—C31—H31	119.6
284	C4—C3—C5	124.6 (4)	C30—C31—H31	119.6
285	C2—C3—C5	120.4 (3)	C14—C13—C12	121.3 (4)
286	N5—C25—C26	109.5 (4)	C14—C13—H13	119.3
287	N5—C25—H25A	109.8	C12—C13—H13	119.3
288	C26—C25—H25A	109.8	C21—C22—H22A	109.5
289	N5—C25—H25B	109.8	C21—C22—H22B	109.5
290	C26—C25—H25B	109.8	H22A—C22—H22B	109.5
291	H25A—C25—H25B	108.2	C21—C22—H22C	109.5
292	C20—C19—C18	114.5 (4)	H22A—C22—H22C	109.5
293	C20—C19—C21	124.4 (4)	H22B—C22—H22C	109.5
294	C18—C19—C21	121.1 (4)	C13—C14—C15	119.0 (4)
295	N6—C23—C24	110.4 (4)	C13—C14—H14	120.5
296	N6—C23—H23A	109.6	C15—C14—H14	120.5
297	C24—C23—H23A	109.6	C29—C30—C31	119.4 (4)
298	N6—C23—H23B	109.6	C29—C30—H30	120.3
299	C24—C23—H23B	109.6	C31—C30—H30	120.3
300	H23A—C23—H23B	108.1	C5—C6—H6C	109.5
301	C19—C21—C22	113.2 (4)	C5—C6—H6D	109.5
302	C19—C21—H21A	108.9	H6C—C6—H6D	109.5
303	C22—C21—H21A	108.9	C5—C6—H6E	109.5
304	C19—C21—H21B	108.9	H6C—C6—H6E	109.5
305	C22—C21—H21B	108.9	H6D—C6—H6E	109.5
306				
307	C4—N2—C1—O1	179.0 (4)	N8—C20—C19—C21	−179.6 (4)
308	C4—N2—C1—N1	−0.6 (6)	Cl2—C20—C19—C21	2.8 (6)
309	C2—N1—C1—O1	−178.9 (4)	O3—C18—C19—C20	178.5 (4)
310	C2—N1—C1—N2	0.7 (6)	N7—C18—C19—C20	−1.4 (6)
311	C17—N7—C18—O3	−179.1 (4)	O3—C18—C19—C21	−0.9 (7)
312	C17—N7—C18—C19	0.9 (6)	N7—C18—C19—C21	179.2 (4)
313	C20—N8—C17—O4	179.6 (4)	C26—N6—C23—C24	−53.9 (5)

C20—N8—C17—N7	−0.8 (6)	C20—C19—C21—C22	76.2 (6)
C18—N7—C17—O4	179.9 (4)	C18—C19—C21—C22	−104.4 (5)
C18—N7—C17—N8	0.3 (6)	C32—C27—C28—C29	−1.8 (6)
C1—N2—C4—C3	1.1 (7)	N5—C27—C28—C29	173.5 (4)
C1—N2—C4—Cl1	−177.7 (3)	C7—N3—C10—C9	−54.1 (4)
C1—N1—C2—O2	179.0 (4)	N4—C9—C10—N3	56.9 (4)
C1—N1—C2—C3	−1.0 (6)	C4—C3—C5—C6	84.3 (5)
C17—N8—C20—C19	0.1 (7)	C2—C3—C5—C6	−92.5 (5)
C17—N8—C20—Cl2	177.9 (3)	C10—N3—C7—C8	53.7 (4)
C9—N4—C11—C16	8.8 (6)	C12—C11—C16—C15	1.2 (6)
C8—N4—C11—C16	−124.1 (4)	N4—C11—C16—C15	−174.7 (4)
C9—N4—C11—C12	−167.1 (4)	C11—C16—C15—C14	−0.7 (7)
C8—N4—C11—C12	60.0 (5)	C23—N6—C26—C25	54.9 (5)
C11—N4—C9—C10	164.1 (3)	N5—C25—C26—N6	−58.0 (4)
C8—N4—C9—C10	−60.6 (4)	C16—C11—C12—C13	−0.4 (6)
C25—N5—C27—C28	−13.7 (6)	N4—C11—C12—C13	175.6 (4)
C24—N5—C27—C28	121.7 (4)	C11—N4—C8—C7	−163.1 (4)
C25—N5—C27—C32	161.5 (4)	C9—N4—C8—C7	60.6 (4)
C24—N5—C27—C32	−63.1 (5)	N3—C7—C8—N4	−56.5 (4)
N2—C4—C3—C2	−1.4 (7)	C28—C27—C32—C31	1.5 (6)
Cl1—C4—C3—C2	177.3 (3)	N5—C27—C32—C31	−174.0 (4)
N2—C4—C3—C5	−178.3 (4)	C27—N5—C24—C23	161.1 (4)
Cl1—C4—C3—C5	0.4 (6)	C25—N5—C24—C23	−60.5 (5)
O2—C2—C3—C4	−178.8 (4)	N6—C23—C24—N5	55.5 (4)
N1—C2—C3—C4	1.2 (6)	C27—C28—C29—C30	1.2 (7)
O2—C2—C3—C5	−1.7 (6)	C27—C32—C31—C30	−0.6 (7)
N1—C2—C3—C5	178.3 (4)	C11—C12—C13—C14	−0.8 (7)
C27—N5—C25—C26	−160.5 (3)	C12—C13—C14—C15	1.3 (7)
C24—N5—C25—C26	61.7 (4)	C16—C15—C14—C13	−0.5 (7)
N8—C20—C19—C18	1.1 (7)	C28—C29—C30—C31	−0.3 (7)
Cl2—C20—C19—C18	−176.5 (3)	C32—C31—C30—C29	0.0 (7)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···O2^{i}	0.86	2.00	2.859 (4)	173
N3—H3*A*···O1^{ii}	0.89	2.83	3.465 (4)	129
N6—H6*A*···O4^{iii}	0.89	1.81	2.681 (5)	165
N7—H7···O3^{iv}	0.86	2.02	2.873 (4)	174
N3—H3*A*···N2^{ii}	0.89	1.92	2.808 (4)	174
N6—H6*B*···N8^{v}	0.89	1.92	2.798 (5)	169
C10—H10*B*···O2^{vi}	0.97	2.46	3.355 (5)	154
C26—H26*A*···O3^{vii}	0.97	2.58	3.444 (5)	147
C16—H16···Cl2^{viii}	0.93	2.80	3.462 (4)	129

Symmetry codes: (i) −*x*+1, −*y*−1, −*z*+2; (ii) *x*, *y*+1, *z*; (iii) *x*, *y*−1, *z*; (iv) −*x*+2, −*y*+1, −*z*+2; (v) −*x*+2, *y*−1/2, −*z*+3/2; (vi) *x*, −*y*+1/2, *z*−1/2; (vii) −*x*+2, −*y*, −*z*+2; (viii) −*x*+2, *y*+1/2, −*z*+3/2.

(II) 4-Phenylpiperazin-1-ium 6-chloro-5-isopropyl-2,4-dioxopyrimidin-1-ide

Crystal data

$C_{10}H_{15}N_2^+ \cdot C_7H_8ClN_2O_2^-$
$M_r = 350.84$
Monoclinic, $I2/a$
$a = 20.5012$ (3) Å
$b = 7.4565$ (1) Å
$c = 23.1414$ (3) Å
$\beta = 90.639$ (1)°
$V = 3537.34$ (8) Å^3
$Z = 8$
$F(000) = 1488$
$D_x = 1.318$ Mg m^{-3}
Cu $K\alpha$ radiation, $\lambda = 1.5418$ Å
Cell parameters from 6927 reflections
$\theta = 3.8$–$70.0°$
$\mu = 2.05$ mm^{-1}
$T = 101$ K
Prism, colourless
0.34 × 0.13 × 0.09 mm

Data collection

Agilent Xcalibur Ruby Gemini diffractometer
Detector resolution: 10.2673 pixels mm^{-1}
ω scans
Absorption correction: multi-scan (*CrysAlis PRO*; Agilent, 2014)
$T_{min} = 0.760$, $T_{max} = 0.828$
13174 measured reflections
3396 independent reflections
2926 reflections with $I > 2\sigma(I)$
$R_{int} = 0.069$
$\theta_{max} = 70.6°$, $\theta_{min} = 3.8°$
$h = -24 \rightarrow 25$
$k = -9 \rightarrow 7$
$l = -27 \rightarrow 28$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.044$
$wR(F^2) = 0.122$
$S = 1.03$
3346 reflections
217 parameters
0 restraints
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0585P)^2 + 3.6877P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.55$ e Å^{-3}
$\Delta\rho_{min} = -0.56$ e Å^{-3}

Special details

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

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
Cl1	0.43825 (2)	0.80800 (7)	0.38729 (2)	0.03420 (17)
O1	0.42855 (6)	1.32956 (18)	0.26398 (6)	0.0286 (3)
O2	0.23184 (6)	1.07264 (19)	0.29852 (6)	0.0288 (3)
N3	0.33106 (7)	1.1946 (2)	0.28125 (6)	0.0227 (3)
H3	0.3124	1.2711	0.2587	0.027*
N4	0.42771 (7)	1.0889 (2)	0.32365 (6)	0.0228 (3)
N2	0.56397 (7)	1.1049 (2)	0.31180 (6)	0.0246 (3)
H3N	0.5734	1.0238	0.2849	0.029*
H2N	0.5208	1.1087	0.3152	0.029*
C11	0.39765 (9)	1.2092 (2)	0.28906 (7)	0.0228 (4)

N1	0.60877 (7)	1.3622 (2)	0.39443 (6)	0.0230 (3)
C2	0.58171 (9)	1.1911 (2)	0.41332 (8)	0.0237 (4)
H2A	0.5352	1.2036	0.4195	0.028*
H2B	0.6020	1.1556	0.4496	0.028*
C4	0.58796 (10)	1.2834 (3)	0.29310 (8)	0.0271 (4)
H4A	0.5649	1.3201	0.2582	0.032*
H4B	0.6341	1.2760	0.2845	0.032*
C14	0.39071 (9)	0.9583 (2)	0.34646 (7)	0.0236 (4)
C12	0.29194 (9)	1.0666 (3)	0.30679 (7)	0.0232 (4)
C6	0.58544 (9)	1.4868 (3)	0.49109 (8)	0.0273 (4)
H6	0.5581	1.3906	0.4992	0.033*
C13	0.32496 (9)	0.9324 (2)	0.34113 (7)	0.0231 (4)
C3	0.59378 (9)	1.0490 (3)	0.36799 (8)	0.0245 (4)
H3A	0.6403	1.0318	0.3634	0.029*
H3B	0.5750	0.9361	0.3802	0.029*
C10	0.65511 (9)	1.6458 (3)	0.42609 (8)	0.0272 (4)
H10	0.6749	1.6571	0.3903	0.033*
C15	0.28407 (10)	0.7802 (3)	0.36508 (8)	0.0286 (4)
H15	0.2393	0.8063	0.3523	0.034*
C5	0.61534 (9)	1.4980 (2)	0.43717 (8)	0.0224 (4)
C8	0.63624 (10)	1.7627 (3)	0.52142 (9)	0.0341 (5)
H8	0.6436	1.8500	0.5494	0.041*
C1	0.57729 (9)	1.4207 (3)	0.34016 (7)	0.0249 (4)
H1A	0.5953	1.5352	0.3284	0.030*
H1B	0.5309	1.4365	0.3462	0.030*
C7	0.59623 (10)	1.6182 (3)	0.53262 (8)	0.0332 (5)
H7	0.5763	1.6088	0.5684	0.040*
C9	0.66531 (10)	1.7757 (3)	0.46790 (9)	0.0320 (5)
H9	0.6921	1.8731	0.4599	0.038*
C16	0.28114 (10)	0.7739 (3)	0.43025 (8)	0.0310 (4)
H16A	0.2708	0.8909	0.4448	0.047*
H16B	0.2481	0.6902	0.4418	0.047*
H16C	0.3227	0.7365	0.4456	0.047*
C17	0.30027 (12)	0.6004 (3)	0.33815 (9)	0.0394 (5)
H17A	0.3015	0.6125	0.2969	0.059*
H17B	0.3421	0.5605	0.3522	0.059*
H17C	0.2675	0.5143	0.3483	0.059*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.0307 (3)	0.0306 (3)	0.0413 (3)	0.00185 (19)	0.0001 (2)	0.0141 (2)
O1	0.0225 (7)	0.0278 (7)	0.0355 (7)	−0.0043 (6)	0.0020 (5)	0.0107 (6)
O2	0.0210 (6)	0.0341 (8)	0.0313 (7)	−0.0042 (6)	0.0017 (5)	0.0053 (6)
N3	0.0210 (7)	0.0233 (8)	0.0238 (7)	−0.0013 (6)	0.0012 (6)	0.0032 (6)
N4	0.0233 (7)	0.0211 (8)	0.0239 (7)	0.0003 (6)	0.0022 (6)	0.0025 (6)
N2	0.0227 (7)	0.0256 (8)	0.0255 (7)	0.0009 (7)	0.0023 (6)	−0.0055 (6)
C11	0.0238 (9)	0.0216 (9)	0.0231 (8)	−0.0010 (7)	0.0033 (7)	−0.0021 (7)

N1	0.0261 (8)	0.0203 (8)	0.0225 (7)	0.0005 (6)	0.0007 (6)	0.0002 (6)
C2	0.0254 (9)	0.0221 (9)	0.0235 (8)	0.0009 (7)	0.0020 (7)	0.0010 (7)
C4	0.0302 (10)	0.0284 (10)	0.0226 (9)	−0.0025 (8)	0.0023 (7)	−0.0003 (8)
C14	0.0295 (9)	0.0207 (9)	0.0206 (8)	0.0023 (8)	0.0024 (7)	−0.0001 (7)
C12	0.0255 (9)	0.0244 (9)	0.0198 (8)	−0.0036 (8)	0.0043 (7)	−0.0030 (7)
C6	0.0300 (10)	0.0248 (10)	0.0273 (9)	−0.0002 (8)	0.0003 (8)	−0.0004 (8)
C13	0.0287 (9)	0.0209 (9)	0.0198 (8)	−0.0031 (8)	0.0042 (7)	−0.0022 (7)
C3	0.0231 (9)	0.0221 (9)	0.0284 (9)	0.0017 (7)	0.0017 (7)	−0.0005 (7)
C10	0.0241 (9)	0.0254 (10)	0.0322 (10)	0.0019 (8)	0.0017 (7)	−0.0005 (8)
C15	0.0317 (10)	0.0262 (10)	0.0279 (9)	−0.0059 (8)	0.0039 (8)	0.0007 (8)
C5	0.0201 (8)	0.0211 (9)	0.0260 (9)	0.0053 (7)	−0.0028 (7)	−0.0015 (7)
C8	0.0376 (11)	0.0290 (11)	0.0354 (10)	0.0050 (9)	−0.0084 (9)	−0.0106 (9)
C1	0.0284 (9)	0.0236 (9)	0.0227 (8)	0.0002 (8)	0.0006 (7)	0.0016 (7)
C7	0.0385 (11)	0.0350 (11)	0.0260 (9)	0.0066 (9)	0.0012 (8)	−0.0053 (8)
C9	0.0273 (10)	0.0241 (10)	0.0445 (11)	−0.0005 (8)	−0.0056 (8)	−0.0034 (9)
C16	0.0318 (10)	0.0306 (11)	0.0309 (10)	−0.0049 (9)	0.0092 (8)	0.0003 (8)
C17	0.0492 (13)	0.0299 (11)	0.0396 (11)	−0.0130 (10)	0.0139 (10)	−0.0048 (9)

Geometric parameters (Å, º)

Cl1—C14	1.7544 (18)	C6—C7	1.389 (3)
O1—C11	1.246 (2)	C6—C5	1.399 (3)
O2—C12	1.246 (2)	C6—H6	0.9300
N3—C11	1.379 (2)	C13—C15	1.519 (3)
N3—C12	1.383 (2)	C3—H3A	0.9700
N3—H3	0.8600	C3—H3B	0.9700
N4—N4	0.000 (5)	C10—C9	1.383 (3)
N4—C14	1.346 (2)	C10—C5	1.397 (3)
N4—C11	1.347 (2)	C10—H10	0.9300
N2—C4	1.485 (2)	C15—C16	1.511 (3)
N2—C3	1.490 (2)	C15—C17	1.517 (3)
N2—H3N	0.8900	C15—H15	0.9800
N2—H2N	0.8900	C8—C7	1.380 (3)
C11—N4	1.347 (2)	C8—C9	1.384 (3)
N1—C5	1.421 (2)	C8—H8	0.9300
N1—C2	1.460 (2)	C1—H1A	0.9700
N1—C1	1.472 (2)	C1—H1B	0.9700
C2—C3	1.513 (2)	C7—H7	0.9300
C2—H2A	0.9700	C9—H9	0.9300
C2—H2B	0.9700	C16—H16A	0.9600
C4—C1	1.512 (3)	C16—H16B	0.9600
C4—H4A	0.9700	C16—H16C	0.9600
C4—H4B	0.9700	C17—H17A	0.9600
C14—N4	1.346 (2)	C17—H17B	0.9600
C14—C13	1.366 (3)	C17—H17C	0.9600
C12—C13	1.442 (3)		
C11—N3—C12	125.17 (16)	C12—C13—C15	117.51 (16)

C11—N3—H3	117.4		N2—C3—C2	109.92 (15)
C12—N3—H3	117.4		N2—C3—H3A	109.7
N4—N4—C14	0 (10)		C2—C3—H3A	109.7
N4—N4—C11	0 (10)		N2—C3—H3B	109.7
C14—N4—C11	117.31 (15)		C2—C3—H3B	109.7
C4—N2—C3	111.77 (14)		H3A—C3—H3B	108.2
C4—N2—H3N	109.3		C9—C10—C5	120.54 (18)
C3—N2—H3N	109.3		C9—C10—H10	119.7
C4—N2—H2N	109.3		C5—C10—H10	119.7
C3—N2—H2N	109.3		C16—C15—C17	113.20 (18)
H3N—N2—H2N	107.9		C16—C15—C13	114.57 (16)
O1—C11—N4	121.67 (16)		C17—C15—C13	112.72 (16)
O1—C11—N4	121.67 (16)		C16—C15—H15	105.1
N4—C11—N4	0.00 (14)		C17—C15—H15	105.1
O1—C11—N3	120.23 (17)		C13—C15—H15	105.1
N4—C11—N3	118.10 (16)		C10—C5—C6	118.26 (17)
N4—C11—N3	118.10 (16)		C10—C5—N1	119.06 (16)
C5—N1—C2	116.59 (14)		C6—C5—N1	122.64 (17)
C5—N1—C1	114.83 (15)		C7—C8—C9	119.04 (19)
C2—N1—C1	110.50 (14)		C7—C8—H8	120.5
N1—C2—C3	109.78 (14)		C9—C8—H8	120.5
N1—C2—H2A	109.7		N1—C1—C4	110.37 (15)
C3—C2—H2A	109.7		N1—C1—H1A	109.6
N1—C2—H2B	109.7		C4—C1—H1A	109.6
C3—C2—H2B	109.7		N1—C1—H1B	109.6
H2A—C2—H2B	108.2		C4—C1—H1B	109.6
N2—C4—C1	110.25 (15)		H1A—C1—H1B	108.1
N2—C4—H4A	109.6		C8—C7—C6	120.75 (19)
C1—C4—H4A	109.6		C8—C7—H7	119.6
N2—C4—H4B	109.6		C6—C7—H7	119.6
C1—C4—H4B	109.6		C10—C9—C8	120.9 (2)
H4A—C4—H4B	108.1		C10—C9—H9	119.5
N4—C14—N4	0.00 (17)		C8—C9—H9	119.5
N4—C14—C13	128.81 (17)		C15—C16—H16A	109.5
N4—C14—C13	128.81 (17)		C15—C16—H16B	109.5
N4—C14—Cl1	111.18 (13)		H16A—C16—H16B	109.5
N4—C14—Cl1	111.18 (13)		C15—C16—H16C	109.5
C13—C14—Cl1	120.01 (14)		H16A—C16—H16C	109.5
O2—C12—N3	119.11 (17)		H16B—C16—H16C	109.5
O2—C12—C13	124.56 (17)		C15—C17—H17A	109.5
N3—C12—C13	116.32 (16)		C15—C17—H17B	109.5
C7—C6—C5	120.48 (19)		H17A—C17—H17B	109.5
C7—C6—H6	119.8		C15—C17—H17C	109.5
C5—C6—H6	119.8		H17A—C17—H17C	109.5
C14—C13—C12	114.11 (16)		H17B—C17—H17C	109.5
C14—C13—C15	128.36 (17)			
N4—N4—C11—O1	0.0 (5)		N3—C12—C13—C14	4.0 (2)

C14—N4—C11—O1	−176.64 (16)	O2—C12—C13—C15	4.8 (3)
C14—N4—C11—N4	0 (100)	N3—C12—C13—C15	−174.41 (15)
N4—N4—C11—N3	0.0 (4)	C4—N2—C3—C2	55.49 (19)
C14—N4—C11—N3	2.5 (2)	N1—C2—C3—N2	−58.11 (19)
C12—N3—C11—O1	179.96 (16)	C14—C13—C15—C16	65.7 (3)
C12—N3—C11—N4	0.8 (3)	C12—C13—C15—C16	−116.20 (19)
C12—N3—C11—N4	0.8 (3)	C14—C13—C15—C17	−65.7 (3)
C5—N1—C2—C3	−165.61 (15)	C12—C13—C15—C17	112.4 (2)
C1—N1—C2—C3	60.88 (19)	C9—C10—C5—C6	1.1 (3)
C3—N2—C4—C1	−54.5 (2)	C9—C10—C5—N1	−176.58 (17)
C11—N4—C14—N4	0 (100)	C7—C6—C5—C10	−1.1 (3)
N4—N4—C14—C13	0.0 (3)	C7—C6—C5—N1	176.42 (17)
C11—N4—C14—C13	−2.5 (3)	C2—N1—C5—C10	164.11 (16)
N4—N4—C14—Cl1	0.0 (3)	C1—N1—C5—C10	−64.4 (2)
C11—N4—C14—Cl1	177.09 (13)	C2—N1—C5—C6	−13.4 (2)
C11—N3—C12—O2	176.56 (16)	C1—N1—C5—C6	118.11 (19)
C11—N3—C12—C13	−4.2 (3)	C5—N1—C1—C4	165.63 (15)
N4—C14—C13—C12	−0.9 (3)	C2—N1—C1—C4	−59.98 (19)
N4—C14—C13—C12	−0.9 (3)	N2—C4—C1—N1	56.0 (2)
Cl1—C14—C13—C12	179.55 (12)	C9—C8—C7—C6	0.4 (3)
N4—C14—C13—C15	177.27 (17)	C5—C6—C7—C8	0.4 (3)
N4—C14—C13—C15	177.27 (17)	C5—C10—C9—C8	−0.3 (3)
Cl1—C14—C13—C15	−2.3 (3)	C7—C8—C9—C10	−0.5 (3)
O2—C12—C13—C14	−176.82 (17)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H2*N*···N4	0.89	1.93	2.813 (2)	174
N2—H3*N*···O1^{i}	0.89	1.84	2.705 (2)	164
N3—H3···O2^{ii}	0.86	1.98	2.834 (2)	174
C3—H3*A*···O2^{iii}	0.97	2.54	3.394 (2)	147

Symmetry codes: (i) −*x*+1, *y*−1/2, −*z*+1/2; (ii) −*x*+1/2, −*y*+5/2, −*z*+1/2; (iii) *x*+1/2, −*y*+2, *z*.

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