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Crystal structures and hydrogen bonding in the anhydrous tryptaminium salts of the isomeric (2,4-dichlorophenoxy)acetic and (3,5-dichlorophenoxy)acetic acids

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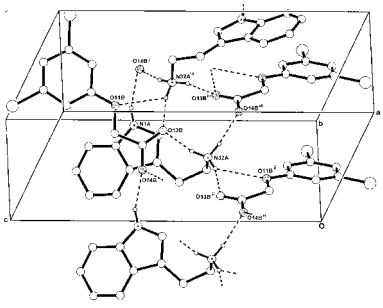
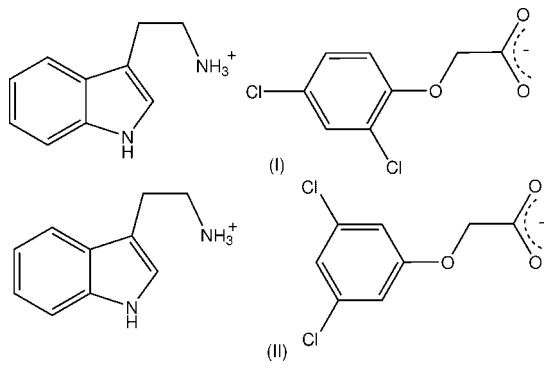
The anhydrous salts of 2-(1*H*-indol-3-yl)ethanamine (tryptamine) with isomeric (2,4-dichlorophenoxy)acetic acid (2,4-D) and (3,5-dichlorophenoxy)acetic (3,5-D), both $C_{10}H_{13}N_2^+\cdot C_8H_5Cl_2O_3^-$ [(I) and (II), respectively], have been determined and their one-dimensional hydrogen-bonded polymeric structures are described. In the crystal of (I), the aminium H atoms are involved in three separate inter-species N—H···O hydrogen-bonding interactions, two with carboxylate O-atom acceptors and the third in an asymmetric three-centre bidentate carboxylate *O,O′* chelate [graph set $R_1^2(4)$]. The indole H atom forms an N—H···$O_{carboxylate}$ hydrogen bond, extending the chain structure along the *b*-axis direction. In (II), two of the three aminium H atoms are also involved in N—H···$O_{carboxylate}$ hydrogen bonds similar to (I) but with the third, a three-centre asymmetric interaction with carboxylate and phenoxy O atoms is found [graph set $R_1^2(5)$]. The chain polymeric extension is also along *b*. There are no π–π ring interactions in either of the structures. The aminium side-chain conformations differ significantly between the two structures, reflecting the conformational ambivalence of the tryptaminium cation, as found also in the benzoate salts.

1. Chemical context

2-(1*H*-Indol-3-yl)ethanamine (tryptamine) is an alkaloid found in plants and fungi and is a possible intermediate in the biosynthetic pathway to the plant hormone indole-3-acetic acid (Takahashi, 1986). It is also found in trace amounts in the mammalian brain, possibly acting as a neuromodulator or neurotransmitter (Jones, 1982). As a relatively strong base (pK_a = 10.2), it readily forms salts with a number of organic acids. To investigate the modes of hydrogen-bonding interaction in crystals of the tryptaminium salts of ring-substituted phenoxyacetic acid analogues, the reaction of tryptamine with two isomeric homologues, the herbicidally active (2,4-dichlorophenoxy)acetic acid (2,4-D) (Zumdahl, 2010) and (3,5-dichlorophenoxy)acetic acid (3,5-D), gave the anhydrous salts, $C_{10}H_{13}N_2^+\cdot C_8H_5Cl_2O_3^-$, (I) and (II), respectively. Their structures and hydrogen-bonding modes are reported herein. The structure of the anhydrous salt with phenoxyacetic acid (Koshima *et al.*, 1999) represents the only reported example of a salt from this acid series. In that crystal, chirality was generated through hydrogen bonding, giving cation–anion units related along a 2_1 screw axes. A similar phenomenon was also observed in the tryptaminium 4-chlorobenzoate crystal (Koshima *et al.*, 2005).





2. Structural commentary

The asymmetric units of (I) and (II) comprise a tryptaminium cation (*A*) and either a 2,4-dichlorophenoxyacetate anion (*B*) (I) (Fig. 1) or a (3,5-dichlorophenoxy)acetate anion (II) (Fig. 2). Unlike a number of tryptaminium salts of benzoic acids in which the benzene rings in the cation and anion species are essentially parallel, giving π–π interactions, these planes in (I) and (II) are not so [dihedral angles = 74.1 (3) and 24.68 (17)°, respectively], giving no π–π interactive effects.

The alkylaminium side chains in the cations of (I) and (II) differ significantly, with the torsion angles C2*A*—C3*A*—C31*A*—C32*A* and C3*A*—C31*A*—C32*A*—C32*A*—N32*A* being −113.1 (5), 58.6 (5)° in (I), 7.3 (5) and in 75.7 (4)° (II), respectively. This variability is a standard feature in the structures of the known tryptaminium benzoate salts, which include the parent benzoate (Terakita *et al.*, 2004), 4-chlorobenzoate (Koshima *et al.*, 2005), 3,4-dimethoxybenzoate (Siripaisarnpipat & Larsen, 1987), 3,5-dinitro-2-hydroxybenzoate (Lynch *et al.*, 2015) and the pseudopolymorphic anhydrous, mono- and dihydrate 3,5-dinitrobenzoates salts (Lynch *et al.*, 2015). In the structure of tryptamine, determined from powder diffraction data (Nowell *et al.*, 2002), the corresponding angles are −89.4 (6) and 60.7 (6)°.

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1*A*—H1*A*···O13*B*^{i}	0.87 (4)	2.13 (5)	2.879 (6)	144 (6)
N32*A*—H34*A*···O14*B*^{ii}	0.89 (4)	1.89 (4)	2.782 (6)	175 (2)
N32*A*—H35*A*···O13*B*^{iii}	0.90 (4)	2.10 (5)	2.817 (6)	137 (4)
N32*A*—H36*A*···O13*B*	0.89 (3)	2.57 (4)	3.231 (6)	132 (4)
N32*A*—H36*A*···O14*B*	0.89 (3)	1.94 (4)	2.816 (6)	171 (5)

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

In (I) the phenoxyacetate side chain of the 2,4-D anion is significantly rotated out of the benzene plane [defining torsion angle C1*B*—O11*B*—C12*B*—C13*B* = 81.2 (6)°], similar to that of the parent acid which also has the *synclinal* side chain conformation (torsion angle 90±30°) (comparative torsion angle = 75.2°; Smith *et al.*, 1976). However, in the potassium salt (Kennard *et al.*, 1983) and the ammonium salt (Liu *et al.*, 2009) (both hemihydrates), the *antiperiplanar* (180±30°) conformation is found. The 3,5-D anion in (II) adopts the *antiperiplanar* conformation with the defining C1*B*—O1*B*—C12*B*—C13*B* torsion angle = −166.5 (3). The structure of the parent acid is not known but the equivalent angle in the ammonium salt is −171.35 (15)° (Smith, 2015) but in the 2:1 adduct of 3,5-D with 4,4′-bipyridine (Lynch *et al.*, 2003), the angle is −71.6 (3)° (*synclinal*).

3. Supramolecular features

In the crystal structures of (I) and (II), one-dimensional hydrogen-bonded structures involving N—H···O$_{carboxylate}$ interactions are found. However, the hydrogen-bonding patterns differ significantly. In the crystal of (I), the three aminium H atoms give different inter-species interactions, two with single carboxylate O-atom acceptors (O13*B*^{iii}, O14*B*^{ii}) and third giving a three-centre *O,O′* chelate with carboxylate O atoms (O13, O14) [graph set $R_1^2(4)$] (Table 1). The indole H

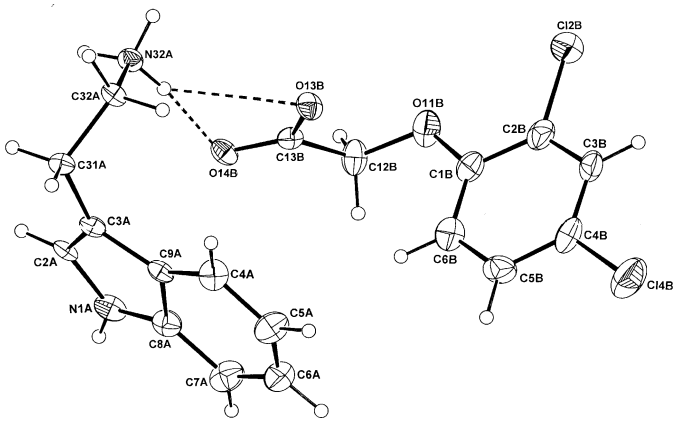


Figure 1
The atom-numbering scheme and the molecular conformation of the TRYP^{+} cation (*A*) and the 2,4-D^{-} anion (*B*) in (I) with displacement ellipsoids drawn at the 40% probability level. The cation–anion hydrogen bonds are shown as dashed lines.

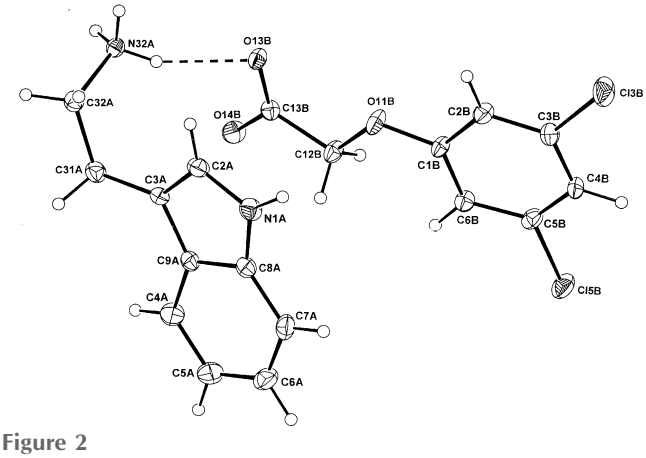


Figure 2
The atom-numbering scheme and the molecular conformation of the TRYP^{+} cation (*A*) and the 3,5-D^{-} anion (*B*) in (II) with displacement ellipsoids drawn at the 40% probability level. The cation–anion hydrogen bond is shown as a dashed line.

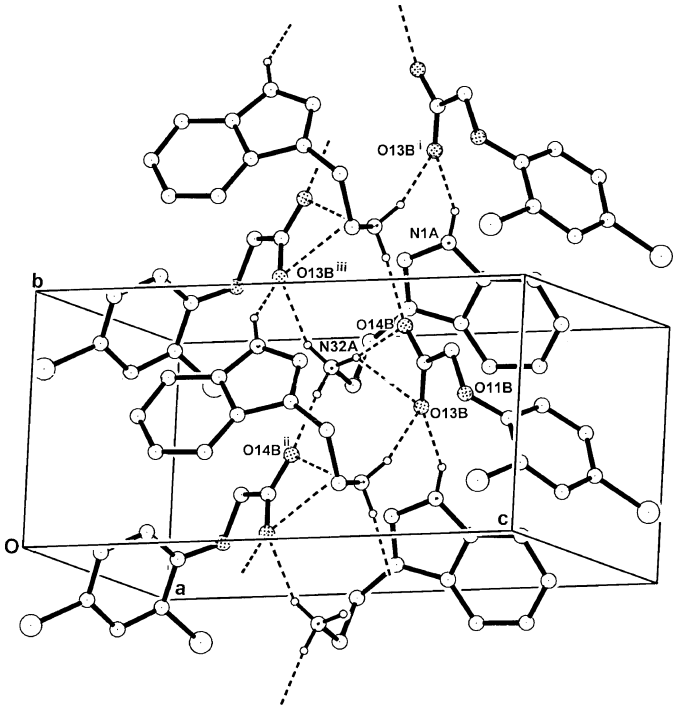


Figure 3
The one-dimensional hydrogen-bonded polymeric structure of (I) extending along [010], with non-associative H atoms omitted. For symmetry codes, see Table 1.

atom gives an N—H···$O_{carboxylate}$ hydrogen bond, extending the chain structure down the [010] axis (Fig. 3). In the crystal of (II), as with (I), two of the three aminium N—H···O interactions are with single carboxylate O atoms [(O13*B*, O14*B*^{iii}) but the third differs in that it forms a three-centre asymmetric interaction with carboxylate and phenoxy O atoms of the anion (O13*B*^{ii}, O11*B*^{ii}) [graph set $R_1^2(4)$] (Table 2).

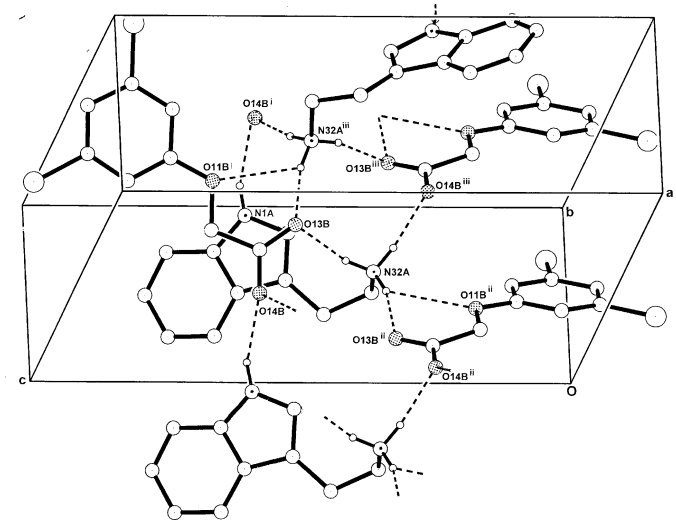


Figure 4
The one-dimensional hydrogen-bonded polymeric structure of (II) extending along [010], with non-associative H-atoms omitted. For symmetry codes, see Table 2.

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1*A*—H1*A*···O14*B*^{i}	0.87 (4)	2.04 (4)	2.838 (4)	152 (4)
N32*A*—H34*A*···O13*B*	0.87 (2)	2.05 (3)	2.875 (4)	160 (4)
N32*A*—H35*A*···O11*B*^{ii}	0.89 (3)	2.60 (4)	3.160 (4)	122 (3)
N32*A*—H35*A*···O13*B*^{ii}	0.89 (3)	1.87 (3)	2.739 (4)	164 (4)
N32*A*—H36*A*···O14*B*^{iii}	0.89 (4)	1.90 (4)	2.775 (4)	170 (4)
C2*A*—H2*A*···O13*B*^{iii}	0.95	2.55	3.495 (4)	177

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

The chain polymeric N1—H··· O14*B* extension is also along [010] (Fig. 4).

The present pair of structures of salts of tryptamine with isomeric (2,4-dichlorophenoxy)acetic acid and (3,5-dichlorophenoxy)acetic acid provide examples which further reflect the conformational ambivalence of the cationic alkylaminium side chain of the tryptamine cation, shown also in the benzoate salts.

4. Synthesis and crystallization

The title compounds (I) and (II) were prepared by warming together for 2 min, solutions containing equimolar quantities of (2,4-dichlorophenoxy)acetic acid (2,4-D) or (3,5-dichlorophenoxy)acetic acid (3,5-D) (138 mg) with 100 mg of tryptamine in ethanol. Room temperature evaporation of the solutions gave in both cases, colourless needles of (I) and (II) from which specimens were cleaved for the X-ray analyses.

5. Refinement details

Crystal data, data collection and structure refinement details are given in Table 3. Hydrogen atoms were placed in calculated positions [C—$H_{aromatic}$ = 0.95 Å or C—$H_{methylene}$ = 0.99 Å] and were allowed to ride in the refinements, with U_{iso}(H) = 1.2U_{eq}(C). The aminium H atoms were located in difference-Fourier analyses and were allowed to refine with bond length restraints [*d*(N—H = 0.88 (2) Å], and with U_{iso}(H) = 1.2U_{eq}(N). Although possibly not of relevance in these crystals involving achiral molecules, the Flack absolute structure factors (Flack, 1983) were determined as 0.01 (7) for (II) (2232 Friedel pairs) and 0.45 (15) for (I) (1619 Friedel pairs), in the case of (I) suggesting possible racemic twinning. No indication of conventional twinning was found with the crystals of either isomer.

Acknowledgements

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Table 3
Experimental details.

	(I)	(II)
Crystal data		
Chemical formula	$C_{10}H_{13}N_2^+\cdot C_8H_5Cl_2O_3^-$	$C_{10}H_{13}N_2^+\cdot C_8H_5Cl_2O_3^-$
M_r	381.25	381.24
Crystal system, space group	Monoclinic, $P2_1$	Monoclinic, $P2_1$
Temperature (K)	200	200
a, b, c (Å)	8.9818 (11), 6.8899 (7), 14.6850 (15)	9.5154 (8), 6.1951 (5), 15.3646 (9)
β (°)	93.565 (9)	102.579 (7)
V (Å^3)	907.00 (17)	883.99 (12)
Z	2	2
Radiation type	Mo $K\alpha$	Mo $K\alpha$
μ (mm^{-1})	0.38	0.39
Crystal size (mm)	0.50 × 0.15 × 0.05	0.50 × 0.12 × 0.06
Data collection		
Diffractometer	Oxford Diffraction Gemini-S CCD-detector	Oxford Diffraction Gemini-S CCD-detector
Absorption correction	Multi-scan (*CrysAlis PRO*; Agilent, 2013)	Multi-scan (*CrysAlis PRO*; Agilent, 2013)
T_{min}, T_{max}	0.940, 0.990	0.872, 0.980
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	3991, 2896, 2299	3845, 2800, 2451
R_{int}	0.035	0.027
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.617	0.617
Refinement		
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.068, 0.187, 1.06	0.042, 0.105, 1.08
No. of reflections	2896	2800
No. of parameters	226	238
No. of restraints	1	5
H-atom treatment	H atoms treated by a mixture of independent and constrained refinement	H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.37, −0.26	0.22, −0.24
Absolute structure	Flack (1983)	Flack (1983)
Absolute structure parameter	0.45 (15)	0.01 (7)

Computer programs: *CrysAlis PRO* (Agilent, 2013), *SIR92* (Altomare *et al.*, 1993), *SHELX97* (Sheldrick, 2008) within *WinGX* (Farrugia, 2012) and *PLATON* (Spek, 2009).

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supporting information

Acta Cryst. (2015). E**71**, 671-674 [doi:10.1107/S205698901500907X]

Crystal structures and hydrogen bonding in the anhydrous tryptaminium salts of the isomeric (2,4-dichlorophenoxy)acetic and (3,5-dichlorophenoxy)acetic acids

Graham Smith and Daniel E. Lynch

Computing details

For both compounds, data collection: *CrysAlis PRO* (Agilent, 2013); cell refinement: *CrysAlis PRO* (Agilent, 2013); data reduction: *CrysAlis PRO* (Agilent, 2013); program(s) used to solve structure: *SIR92* (Altomare *et al.*, 1993). Program(s) used to refine structure: *SHELX97* (Sheldrick, 2008) within *WinGX* (Farrugia, 2012) for (I); *SHELXL97* (Sheldrick, 2008) within *WinGX* (Farrugia, 2012) for (II). For both compounds, molecular graphics: *PLATON* (Spek, 2009); software used to prepare material for publication: *PLATON* (Spek, 2009).

(I) 2-(1*H*-Indol-3-yl)ethanaminium (2,4-dichlorophenoxy)acetate

Crystal data

$C_{10}H_{13}N_2^+ \cdot C_8H_5Cl_2O_3^-$
M_r = 381.25
Monoclinic, $P2_1$
Hall symbol: P 2yb
a = 8.9818 (11) Å
b = 6.8899 (7) Å
c = 14.6850 (15) Å
β = 93.565 (9)°
V = 907.00 (17) Å^3
Z = 2
$F(000)$ = 396
D_x = 1.396 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 940 reflections
θ = 3.8–24.0°
μ = 0.38 mm^{-1}
T = 200 K
Needle, colourless
0.50 × 0.15 × 0.05 mm

Data collection

Oxford Diffraction Gemini-S CCD-detector diffractometer
Radiation source: Enhance (Mo) X-ray source
Graphite monochromator
Detector resolution: 16.077 pixels mm^{-1}
ω scans
Absorption correction: multi-scan (*CrysAlis PRO*; Agilent, 2013)
T_{min} = 0.940, T_{max} = 0.990
3991 measured reflections
2896 independent reflections
2299 reflections with $I > 2\sigma(I)$
R_{int} = 0.035
θ_{max} = 26.0°, θ_{min} = 3.3°
h = −10→11
k = −7→8
l = −17→18

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.068
$wR(F^2)$ = 0.187
S = 1.06
2896 reflections
226 parameters
1 restraint
Primary atom site location: structure-invariant direct methods

Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0918P)^2 + 0.3461P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.37$ e Å^{-3}
$\Delta\rho_{min} = -0.26$ e Å^{-3}
Absolute structure: Flack (1983)
Absolute structure parameter: 0.45 (15)

Special details

Geometry. Bond distances, angles etc. have been calculated using the rounded fractional coordinates. All su's are estimated from the variances of the (full) variance-covariance matrix. The cell esds are taken into account in the estimation of distances, angles and torsion angles

Refinement. Refinement of F^2 against ALL reflections. The weighted R-factor wR and goodness of fit S are based on F^2, conventional R-factors R are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > 2\text{sigma}(F^2)$ is used only for calculating R-factors(gt) etc. and is not relevant to the choice of reflections for refinement. R-factors based on F^2 are statistically about twice as large as those based on F, and R- factors based on ALL data will be even larger.

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

	x	*y*	*z*	U_{iso}*/U_{eq}
Cl2B	0.28249 (19)	0.2639 (3)	0.84021 (11)	0.0625 (6)
Cl4B	0.8117 (2)	0.2352 (4)	1.03185 (12)	0.0817 (8)
O11B	0.3858 (5)	0.6285 (7)	0.7750 (3)	0.0565 (16)
O13B	0.5619 (4)	0.6213 (5)	0.6335 (3)	0.0378 (12)
O14B	0.5207 (4)	0.9327 (5)	0.6033 (3)	0.0346 (11)
C1B	0.4889 (7)	0.5483 (10)	0.8340 (4)	0.0444 (19)
C2B	0.4569 (7)	0.3677 (10)	0.8717 (4)	0.047 (2)
C3B	0.5495 (7)	0.2673 (11)	0.9322 (3)	0.049 (2)
C4B	0.6859 (8)	0.3564 (12)	0.9564 (4)	0.057 (3)
C5B	0.7253 (7)	0.5320 (11)	0.9216 (4)	0.051 (2)
C6B	0.6281 (8)	0.6256 (11)	0.8585 (4)	0.055 (2)
C12B	0.4160 (7)	0.8064 (9)	0.7333 (4)	0.045 (2)
C13B	0.5129 (5)	0.7839 (8)	0.6498 (3)	0.0290 (17)
N1A	0.7673 (5)	1.3071 (7)	0.6100 (3)	0.0397 (16)
N32A	0.6027 (5)	0.7972 (7)	0.4326 (3)	0.0317 (14)
C2A	0.7670 (5)	1.2445 (7)	0.5215 (3)	0.0262 (14)
C3A	0.8329 (5)	1.0707 (7)	0.5166 (3)	0.0274 (16)
C4A	0.9521 (6)	0.8575 (9)	0.6506 (4)	0.0410 (17)
C5A	0.9790 (7)	0.8583 (11)	0.7434 (4)	0.052 (2)
C6A	0.9345 (8)	1.0100 (12)	0.7973 (5)	0.060 (3)
C7A	0.8638 (7)	1.1725 (11)	0.7595 (4)	0.055 (2)
C8A	0.8359 (6)	1.1754 (9)	0.6647 (4)	0.0388 (19)
C9A	0.8793 (5)	1.0155 (8)	0.6100 (4)	0.0296 (16)
C31A	0.8527 (6)	0.9501 (7)	0.4333 (3)	0.0304 (17)
C32A	0.7656 (5)	0.7611 (8)	0.4290 (3)	0.0296 (16)
H3B	0.52290	0.14500	0.95620	0.0580*
H5B	0.81840	0.58950	0.94030	0.0620*
H6B	0.65760	0.74420	0.83190	0.0660*
H12B	0.46840	0.89250	0.77880	0.0550*

H13B	0.32040	0.86940	0.71320	0.0550*
H1A	0.743 (7)	1.423 (5)	0.627 (4)	0.0480*
H2A	0.72550	1.31490	0.47040	0.0310*
H4A	0.98260	0.75130	0.61500	0.0490*
H5A	1.02980	0.75130	0.77180	0.0620*
H6A	0.95280	1.00250	0.86160	0.0720*
H7A	0.83540	1.27770	0.79650	0.0660*
H30B	0.96010	0.91970	0.43050	0.0360*
H31B	0.82230	1.02830	0.37880	0.0360*
H32B	0.78460	0.69190	0.37180	0.0360*
H33B	0.80010	0.67720	0.48090	0.0360*
H34A	0.559 (6)	0.684 (5)	0.419 (3)	0.0380*
H35A	0.581 (6)	0.883 (6)	0.388 (3)	0.0380*
H36A	0.581 (6)	0.828 (8)	0.4889 (19)	0.0380*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl2B	0.0639 (11)	0.0664 (11)	0.0572 (10)	−0.0152 (10)	0.0030 (7)	−0.0014 (9)
Cl4B	0.0779 (13)	0.1142 (19)	0.0512 (10)	0.0072 (13)	−0.0110 (8)	0.0160 (11)
O11B	0.055 (3)	0.064 (3)	0.051 (2)	0.004 (2)	0.007 (2)	0.017 (2)
O13B	0.044 (2)	0.024 (2)	0.045 (2)	0.0037 (17)	−0.0007 (17)	0.0043 (16)
O14B	0.0250 (19)	0.029 (2)	0.050 (2)	−0.0007 (16)	0.0030 (15)	0.0017 (17)
C1B	0.047 (3)	0.055 (4)	0.032 (3)	0.005 (3)	0.009 (2)	−0.002 (3)
C2B	0.063 (4)	0.050 (4)	0.029 (3)	0.004 (3)	0.009 (3)	−0.005 (3)
C3B	0.069 (4)	0.049 (4)	0.029 (3)	−0.005 (3)	0.011 (2)	0.004 (3)
C4B	0.069 (5)	0.074 (5)	0.028 (3)	0.007 (4)	0.012 (3)	0.005 (3)
C5B	0.036 (3)	0.064 (5)	0.054 (4)	−0.010 (3)	0.004 (3)	−0.008 (4)
C6B	0.059 (4)	0.059 (4)	0.047 (4)	0.000 (4)	0.008 (3)	0.004 (3)
C12B	0.048 (4)	0.042 (4)	0.047 (3)	0.016 (3)	0.010 (3)	0.015 (3)
C13B	0.028 (3)	0.025 (3)	0.033 (3)	0.006 (2)	−0.0060 (18)	−0.002 (2)
N1A	0.031 (2)	0.028 (3)	0.060 (3)	0.004 (2)	0.003 (2)	−0.009 (2)
N32A	0.024 (2)	0.035 (3)	0.036 (2)	−0.003 (2)	0.0010 (17)	0.000 (2)
C2A	0.019 (2)	0.016 (2)	0.043 (3)	−0.004 (2)	−0.0016 (18)	0.001 (2)
C3A	0.018 (2)	0.023 (3)	0.041 (3)	0.000 (2)	0.0008 (19)	−0.004 (2)
C4A	0.030 (3)	0.043 (3)	0.050 (3)	0.013 (3)	0.003 (2)	0.003 (3)
C5A	0.042 (3)	0.062 (4)	0.050 (4)	0.013 (3)	−0.008 (3)	0.000 (3)
C6A	0.068 (5)	0.072 (5)	0.041 (4)	−0.016 (4)	0.003 (3)	−0.002 (4)
C7A	0.045 (4)	0.075 (5)	0.045 (3)	0.003 (4)	0.009 (3)	−0.012 (3)
C8A	0.027 (3)	0.044 (4)	0.046 (3)	−0.002 (3)	0.008 (2)	−0.005 (3)
C9A	0.020 (2)	0.027 (3)	0.042 (3)	−0.005 (2)	0.0047 (19)	−0.003 (2)
C31A	0.026 (3)	0.030 (3)	0.035 (3)	−0.002 (2)	0.001 (2)	−0.004 (2)
C32A	0.025 (2)	0.023 (3)	0.041 (3)	−0.001 (2)	0.0029 (19)	−0.001 (2)

Geometric parameters (Å, º)

Cl2B—C2B	1.758 (7)	C6B—H6B	0.9500
Cl4B—C4B	1.745 (7)	C12B—H13B	0.9900

O11B—C1B	1.347 (8)	C12B—H12B	0.9900
O11B—C12B	1.404 (8)	C2A—C3A	1.340 (7)
O13B—C13B	1.233 (6)	C3A—C9A	1.459 (7)
O14B—C13B	1.236 (6)	C3A—C31A	1.499 (6)
N1A—C8A	1.337 (8)	C4A—C5A	1.369 (8)
N1A—C2A	1.369 (6)	C4A—C9A	1.386 (8)
N32A—C32A	1.488 (6)	C5A—C6A	1.385 (10)
N1A—H1A	0.87 (4)	C6A—C7A	1.386 (11)
N32A—H34A	0.89 (4)	C7A—C8A	1.399 (8)
N32A—H36A	0.89 (3)	C8A—C9A	1.432 (8)
N32A—H35A	0.90 (4)	C31A—C32A	1.518 (7)
C1B—C2B	1.399 (9)	C2A—H2A	0.9500
C1B—C6B	1.386 (10)	C4A—H4A	0.9500
C2B—C3B	1.367 (9)	C5A—H5A	0.9500
C3B—C4B	1.396 (10)	C6A—H6A	0.9500
C4B—C5B	1.369 (11)	C7A—H7A	0.9500
C5B—C6B	1.391 (9)	C31A—H30B	0.9900
C12B—C13B	1.555 (7)	C31A—H31B	0.9900
C3B—H3B	0.9500	C32A—H32B	0.9900
C5B—H5B	0.9500	C32A—H33B	0.9900

C1B—O11B—C12B	119.7 (5)	N1A—C2A—C3A	111.0 (4)
C2A—N1A—C8A	109.3 (5)	C2A—C3A—C9A	106.5 (4)
C8A—N1A—H1A	125 (4)	C2A—C3A—C31A	127.9 (4)
C2A—N1A—H1A	125 (4)	C9A—C3A—C31A	125.6 (4)
C32A—N32A—H35A	105 (3)	C5A—C4A—C9A	118.3 (6)
H34A—N32A—H36A	107 (5)	C4A—C5A—C6A	122.2 (7)
C32A—N32A—H36A	110 (3)	C5A—C6A—C7A	121.5 (6)
H34A—N32A—H35A	110 (4)	C6A—C7A—C8A	117.3 (6)
C32A—N32A—H34A	105 (3)	N1A—C8A—C9A	108.5 (5)
H35A—N32A—H36A	118 (4)	C7A—C8A—C9A	120.6 (6)
O11B—C1B—C2B	118.0 (6)	N1A—C8A—C7A	130.9 (6)
C2B—C1B—C6B	116.3 (6)	C3A—C9A—C4A	135.2 (5)
O11B—C1B—C6B	125.6 (6)	C3A—C9A—C8A	104.8 (5)
Cl2B—C2B—C1B	117.4 (5)	C4A—C9A—C8A	120.1 (5)
C1B—C2B—C3B	125.2 (6)	C3A—C31A—C32A	115.0 (4)
Cl2B—C2B—C3B	117.5 (5)	N32A—C32A—C31A	111.1 (4)
C2B—C3B—C4B	115.6 (6)	N1A—C2A—H2A	125.00
Cl4B—C4B—C5B	119.2 (5)	C3A—C2A—H2A	125.00
Cl4B—C4B—C3B	118.3 (6)	C5A—C4A—H4A	121.00
C3B—C4B—C5B	122.5 (6)	C9A—C4A—H4A	121.00
C4B—C5B—C6B	119.5 (6)	C4A—C5A—H5A	119.00
C1B—C6B—C5B	120.9 (7)	C6A—C5A—H5A	119.00
O11B—C12B—C13B	112.9 (5)	C5A—C6A—H6A	119.00
O13B—C13B—O14B	127.8 (5)	C7A—C6A—H6A	119.00
O13B—C13B—C12B	117.9 (5)	C6A—C7A—H7A	121.00
O14B—C13B—C12B	114.0 (5)	C8A—C7A—H7A	121.00
C2B—C3B—H3B	122.00	C3A—C31A—H30B	108.00

C4B—C3B—H3B	122.00	C3A—C31A—H31B	109.00
C6B—C5B—H5B	120.00	C32A—C31A—H30B	109.00
C4B—C5B—H5B	120.00	C32A—C31A—H31B	109.00
C1B—C6B—H6B	120.00	H30B—C31A—H31B	108.00
C5B—C6B—H6B	120.00	N32A—C32A—H32B	109.00
O11B—C12B—H13B	109.00	N32A—C32A—H33B	109.00
C13B—C12B—H13B	109.00	C31A—C32A—H32B	109.00
H12B—C12B—H13B	108.00	C31A—C32A—H33B	109.00
C13B—C12B—H12B	109.00	H32B—C32A—H33B	108.00
O11B—C12B—H12B	109.00		

C12B—O11B—C1B—C2B	−177.8 (5)	N1A—C2A—C3A—C9A	0.3 (5)
C12B—O11B—C1B—C6B	−0.5 (9)	N1A—C2A—C3A—C31A	178.5 (5)
C1B—O11B—C12B—C13B	81.2 (6)	C2A—C3A—C9A—C4A	−179.6 (6)
C8A—N1A—C2A—C3A	0.9 (6)	C2A—C3A—C9A—C8A	−1.3 (5)
C2A—N1A—C8A—C7A	−179.8 (6)	C31A—C3A—C9A—C4A	2.1 (9)
C2A—N1A—C8A—C9A	−1.7 (6)	C31A—C3A—C9A—C8A	−179.6 (5)
C6B—C1B—C2B—C3B	2.3 (10)	C2A—C3A—C31A—C32A	−113.1 (5)
O11B—C1B—C6B—C5B	178.8 (6)	C9A—C3A—C31A—C32A	64.8 (6)
C2B—C1B—C6B—C5B	−3.8 (9)	C9A—C4A—C5A—C6A	0.7 (9)
O11B—C1B—C2B—C3B	179.9 (6)	C5A—C4A—C9A—C3A	179.0 (6)
C6B—C1B—C2B—Cl2B	−178.9 (5)	C5A—C4A—C9A—C8A	0.8 (8)
O11B—C1B—C2B—Cl2B	−1.3 (8)	C4A—C5A—C6A—C7A	−1.9 (11)
Cl2B—C2B—C3B—C4B	−179.1 (5)	C5A—C6A—C7A—C8A	1.5 (10)
C1B—C2B—C3B—C4B	−0.2 (9)	C6A—C7A—C8A—N1A	178.0 (6)
C2B—C3B—C4B—Cl4B	−178.9 (5)	C6A—C7A—C8A—C9A	0.1 (9)
C2B—C3B—C4B—C5B	−0.4 (9)	N1A—C8A—C9A—C3A	1.8 (6)
C3B—C4B—C5B—C6B	−1.1 (10)	N1A—C8A—C9A—C4A	−179.5 (5)
Cl4B—C4B—C5B—C6B	177.3 (5)	C7A—C8A—C9A—C3A	−179.9 (5)
C4B—C5B—C6B—C1B	3.4 (10)	C7A—C8A—C9A—C4A	−1.2 (8)
O11B—C12B—C13B—O13B	−5.6 (7)	C3A—C31A—C32A—N32A	58.6 (5)
O11B—C12B—C13B—O14B	168.9 (5)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1*A*—H1*A*···O13*B*^{i}	0.87 (4)	2.13 (5)	2.879 (6)	144 (6)
N32*A*—H34*A*···O14*B*^{ii}	0.89 (4)	1.89 (4)	2.782 (6)	175 (2)
N32*A*—H35*A*···O13*B*^{iii}	0.90 (4)	2.10 (5)	2.817 (6)	137 (4)
N32*A*—H36*A*···O13*B*	0.89 (3)	2.57 (4)	3.231 (6)	132 (4)
N32*A*—H36*A*···O14*B*	0.89 (3)	1.94 (4)	2.816 (6)	171 (5)
C2*A*—H2*A*···O14*B*^{iii}	0.95	2.53	3.336 (6)	142

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

(II) 2-(1*H*-Indol-3-yl)ethanaminium (3,5-dichlorophenoxy)acetate

Crystal data

$C_{10}H_{13}N_2^+ \cdot C_8H_5Cl_2O_3^-$
M_r = 381.24
Monoclinic, $P2_1$
Hall symbol: P 2yb
a = 9.5154 (8) Å
b = 6.1951 (5) Å
c = 15.3646 (9) Å
β = 102.579 (7)°
V = 883.99 (12) Å^3
Z = 2
$F(000)$ = 396
D_x = 1.432 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 1140 reflections
θ = 3.9–28.2°
μ = 0.39 mm^{-1}
T = 200 K
Needle, colourless
0.50 × 0.12 × 0.06 mm

Data collection

Oxford Diffraction Gemini-S CCD-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
Detector resolution: 16.077 pixels mm^{-1}
ω scans
Absorption correction: multi-scan (*CrysAlis PRO*; Agilent, 2013)
T_{min} = 0.872, T_{max} = 0.980
3845 measured reflections
2800 independent reflections
2451 reflections with $I > 2\sigma(I)$
R_{int} = 0.027
θ_{max} = 26.0°, θ_{min} = 3.1°
h = −11→7
k = −7→7
l = −18→18

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.042
$wR(F^2)$ = 0.105
S = 1.08
2800 reflections
238 parameters
5 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0513P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.22 e Å^{-3}
$\Delta\rho_{min}$ = −0.24 e Å^{-3}
Absolute structure: Flack (1983)
Absolute structure parameter: 0.01 (7)

Special details

Geometry. Bond distances, angles etc. have been calculated using the rounded fractional coordinates. All su's are estimated from the variances of the (full) variance-covariance matrix. The cell esds are taken into account in the estimation of distances, angles and torsion angles

Refinement. Refinement of F^2 against ALL reflections. The weighted R-factor wR and goodness of fit S are based on F^2, conventional R-factors R are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > 2$sigma(F^2) is used only for calculating R-factors(gt) etc. and is not relevant to the choice of reflections for refinement. R-factors based on F^2 are statistically about twice as large as those based on F, and R- factors based on ALL data will be even larger.

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

	x	*y*	*z*	U_{iso}*/U_{eq}
Cl3B	0.88773 (13)	1.0762 (2)	0.94414 (6)	0.0556 (4)
Cl5B	0.61601 (10)	0.46571 (17)	1.09183 (5)	0.0421 (3)
O11B	0.6022 (3)	0.4935 (4)	0.75745 (13)	0.0337 (8)

O13B	0.5666 (3)	0.2827 (4)	0.60040 (14)	0.0269 (7)
O14B	0.4608 (3)	0.0059 (4)	0.65216 (15)	0.0353 (8)
C1B	0.6435 (3)	0.5773 (6)	0.84115 (19)	0.0266 (10)
C2B	0.7290 (4)	0.7600 (6)	0.8496 (2)	0.0304 (10)
C3B	0.7776 (4)	0.8496 (6)	0.9324 (2)	0.0301 (11)
C4B	0.7436 (4)	0.7632 (6)	1.0088 (2)	0.0306 (10)
C5B	0.6601 (4)	0.5807 (7)	0.99749 (19)	0.0298 (10)
C6B	0.6062 (3)	0.4868 (6)	0.91596 (19)	0.0279 (10)
C12B	0.5268 (4)	0.2927 (6)	0.7495 (2)	0.0297 (11)
C13B	0.5178 (3)	0.1909 (5)	0.6593 (2)	0.0243 (10)
N1A	0.2443 (3)	0.6837 (5)	0.6332 (2)	0.0339 (10)
N32A	0.3860 (3)	0.2111 (5)	0.42680 (18)	0.0238 (8)
C2A	0.2536 (3)	0.5745 (7)	0.5567 (2)	0.0314 (11)
C3A	0.1828 (3)	0.3819 (6)	0.5518 (2)	0.0238 (10)
C4A	0.0375 (4)	0.2219 (7)	0.6612 (2)	0.0366 (11)
C5A	−0.0092 (4)	0.2656 (7)	0.7385 (2)	0.0435 (14)
C6A	0.0307 (4)	0.4575 (8)	0.7856 (2)	0.0420 (13)
C7A	0.1174 (4)	0.6077 (7)	0.7582 (2)	0.0366 (11)
C8A	0.1640 (3)	0.5635 (6)	0.6792 (2)	0.0293 (10)
C9A	0.1237 (3)	0.3729 (6)	0.6306 (2)	0.0258 (10)
C31A	0.1548 (4)	0.2190 (6)	0.4786 (2)	0.0307 (11)
C32A	0.2298 (3)	0.2630 (6)	0.4028 (2)	0.0290 (10)
H2B	0.75380	0.82280	0.79850	0.0360*
H4B	0.77660	0.82720	1.06580	0.0370*
H6B	0.54530	0.36380	0.91080	0.0340*
H12B	0.42830	0.31680	0.75890	0.0350*
H13B	0.57670	0.19270	0.79650	0.0350*
H1A	0.291 (4)	0.803 (5)	0.649 (3)	0.0530*
H2A	0.30260	0.62610	0.51320	0.0380*
H4A	0.01130	0.09110	0.62960	0.0440*
H5A	−0.06890	0.16460	0.75980	0.0530*
H6A	−0.00350	0.48420	0.83830	0.0500*
H7A	0.14490	0.73640	0.79120	0.0440*
H30A	0.04970	0.21190	0.45400	0.0370*
H31A	0.18580	0.07560	0.50430	0.0370*
H32A	0.18350	0.17590	0.35030	0.0350*
H33A	0.21760	0.41720	0.38590	0.0350*
H34A	0.420 (4)	0.237 (7)	0.4830 (14)	0.0530*
H35A	0.407 (4)	0.080 (4)	0.409 (3)	0.0530*
H36A	0.438 (4)	0.292 (7)	0.398 (3)	0.0530*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl3B	0.0674 (7)	0.0572 (7)	0.0408 (5)	−0.0354 (6)	0.0090 (5)	−0.0097 (5)
Cl5B	0.0558 (6)	0.0482 (6)	0.0235 (4)	−0.0057 (5)	0.0112 (4)	0.0014 (4)
O11B	0.0576 (15)	0.0254 (14)	0.0187 (10)	−0.0111 (13)	0.0099 (10)	−0.0035 (10)
O13B	0.0352 (13)	0.0241 (13)	0.0215 (11)	−0.0037 (11)	0.0061 (10)	−0.0029 (10)

O14B	0.0435 (14)	0.0235 (15)	0.0423 (13)	−0.0097 (12)	0.0168 (11)	−0.0078 (11)
C1B	0.0358 (18)	0.0236 (18)	0.0205 (15)	−0.0006 (16)	0.0066 (14)	−0.0050 (15)
C2B	0.0355 (18)	0.032 (2)	0.0243 (16)	−0.0005 (17)	0.0081 (15)	0.0001 (16)
C3B	0.0319 (19)	0.027 (2)	0.0300 (17)	−0.0034 (16)	0.0038 (16)	−0.0043 (16)
C4B	0.0289 (17)	0.037 (2)	0.0240 (16)	−0.0029 (16)	0.0015 (14)	−0.0103 (16)
C5B	0.0341 (18)	0.034 (2)	0.0215 (15)	0.0066 (18)	0.0066 (13)	0.0039 (16)
C6B	0.0336 (17)	0.0249 (19)	0.0251 (15)	−0.0020 (16)	0.0059 (14)	0.0008 (15)
C12B	0.042 (2)	0.024 (2)	0.0236 (16)	−0.0046 (17)	0.0085 (15)	−0.0044 (15)
C13B	0.0266 (17)	0.0200 (19)	0.0257 (15)	−0.0023 (16)	0.0041 (14)	0.0012 (15)
N1A	0.0349 (17)	0.0248 (17)	0.0427 (16)	−0.0066 (14)	0.0099 (14)	−0.0069 (15)
N32A	0.0264 (14)	0.0230 (17)	0.0216 (12)	0.0016 (13)	0.0044 (12)	0.0005 (13)
C2A	0.0261 (17)	0.033 (2)	0.0366 (18)	−0.0040 (17)	0.0103 (14)	−0.0023 (17)
C3A	0.0202 (16)	0.0249 (19)	0.0268 (16)	−0.0021 (14)	0.0064 (14)	0.0008 (15)
C4A	0.041 (2)	0.034 (2)	0.0362 (18)	−0.0090 (19)	0.0115 (17)	0.0043 (17)
C5A	0.044 (2)	0.052 (3)	0.038 (2)	−0.008 (2)	0.0163 (19)	0.007 (2)
C6A	0.040 (2)	0.061 (3)	0.0260 (16)	0.009 (2)	0.0092 (16)	0.004 (2)
C7A	0.0336 (19)	0.044 (2)	0.0280 (17)	0.0066 (18)	−0.0026 (15)	−0.0081 (18)
C8A	0.0212 (16)	0.035 (2)	0.0297 (17)	0.0061 (16)	0.0010 (14)	0.0006 (17)
C9A	0.0213 (16)	0.028 (2)	0.0262 (16)	0.0012 (15)	0.0012 (14)	0.0018 (15)
C31A	0.0281 (17)	0.029 (2)	0.0348 (18)	−0.0045 (16)	0.0062 (15)	−0.0042 (16)
C32A	0.0267 (17)	0.032 (2)	0.0269 (17)	0.0044 (16)	0.0027 (15)	−0.0013 (16)

Geometric parameters (Å, º)

Cl3B—C3B	1.738 (4)	C6B—H6B	0.9500
Cl5B—C5B	1.746 (3)	C12B—H13B	0.9900
O11B—C1B	1.363 (4)	C12B—H12B	0.9900
O11B—C12B	1.428 (5)	C2A—C3A	1.364 (5)
O13B—C13B	1.241 (4)	C3A—C9A	1.443 (4)
O14B—C13B	1.263 (4)	C3A—C31A	1.491 (5)
N1A—C8A	1.369 (4)	C4A—C5A	1.383 (5)
N1A—C2A	1.376 (5)	C4A—C9A	1.392 (5)
N32A—C32A	1.487 (4)	C5A—C6A	1.401 (6)
N1A—H1A	0.87 (4)	C6A—C7A	1.369 (6)
N32A—H34A	0.87 (2)	C7A—C8A	1.407 (4)
N32A—H36A	0.89 (4)	C8A—C9A	1.405 (5)
N32A—H35A	0.89 (3)	C31A—C32A	1.517 (5)
C1B—C2B	1.383 (5)	C2A—H2A	0.9500
C1B—C6B	1.393 (4)	C4A—H4A	0.9500
C2B—C3B	1.373 (4)	C5A—H5A	0.9500
C3B—C4B	1.391 (5)	C6A—H6A	0.9500
C4B—C5B	1.371 (6)	C7A—H7A	0.9500
C5B—C6B	1.375 (4)	C31A—H30A	0.9900
C12B—C13B	1.508 (4)	C31A—H31A	0.9900
C2B—H2B	0.9500	C32A—H32A	0.9900
C4B—H4B	0.9500	C32A—H33A	0.9900
C1B—O11B—C12B	116.7 (2)	N1A—C2A—C3A	110.8 (3)

C2A—N1A—C8A	108.7 (3)	C2A—C3A—C9A	105.4 (3)
C8A—N1A—H1A	129 (3)	C2A—C3A—C31A	129.7 (3)
C2A—N1A—H1A	122 (3)	C9A—C3A—C31A	124.6 (3)
C32A—N32A—H35A	114 (3)	C5A—C4A—C9A	118.8 (4)
H34A—N32A—H36A	105 (4)	C4A—C5A—C6A	120.6 (4)
C32A—N32A—H36A	113 (3)	C5A—C6A—C7A	122.1 (3)
H34A—N32A—H35A	114 (4)	C6A—C7A—C8A	117.2 (4)
C32A—N32A—H34A	110 (3)	N1A—C8A—C9A	107.5 (3)
H35A—N32A—H36A	100 (4)	C7A—C8A—C9A	121.4 (3)
O11B—C1B—C2B	116.3 (3)	N1A—C8A—C7A	131.0 (3)
C2B—C1B—C6B	120.2 (3)	C3A—C9A—C4A	132.5 (3)
O11B—C1B—C6B	123.5 (3)	C3A—C9A—C8A	107.6 (3)
C1B—C2B—C3B	119.4 (3)	C4A—C9A—C8A	119.9 (3)
C2B—C3B—C4B	122.2 (3)	C3A—C31A—C32A	114.9 (3)
Cl3B—C3B—C4B	118.0 (3)	N32A—C32A—C31A	112.5 (3)
Cl3B—C3B—C2B	119.8 (3)	N1A—C2A—H2A	125.00
C3B—C4B—C5B	116.4 (3)	C3A—C2A—H2A	125.00
Cl5B—C5B—C4B	117.9 (2)	C5A—C4A—H4A	121.00
Cl5B—C5B—C6B	118.3 (3)	C9A—C4A—H4A	121.00
C4B—C5B—C6B	123.7 (3)	C4A—C5A—H5A	120.00
C1B—C6B—C5B	118.0 (3)	C6A—C5A—H5A	120.00
O11B—C12B—C13B	111.7 (3)	C5A—C6A—H6A	119.00
O13B—C13B—O14B	125.1 (3)	C7A—C6A—H6A	119.00
O13B—C13B—C12B	121.5 (3)	C6A—C7A—H7A	121.00
O14B—C13B—C12B	113.4 (3)	C8A—C7A—H7A	121.00
C1B—C2B—H2B	120.00	C3A—C31A—H30A	109.00
C3B—C2B—H2B	120.00	C3A—C31A—H31A	109.00
C5B—C4B—H4B	122.00	C32A—C31A—H30A	109.00
C3B—C4B—H4B	122.00	C32A—C31A—H31A	109.00
C1B—C6B—H6B	121.00	H30A—C31A—H31A	107.00
C5B—C6B—H6B	121.00	N32A—C32A—H32A	109.00
O11B—C12B—H13B	109.00	N32A—C32A—H33A	109.00
C13B—C12B—H13B	109.00	C31A—C32A—H32A	109.00
H12B—C12B—H13B	108.00	C31A—C32A—H33A	109.00
C13B—C12B—H12B	109.00	H32A—C32A—H33A	108.00
O11B—C12B—H12B	109.00		

C12B—O11B—C1B—C2B	173.6 (3)	N1A—C2A—C3A—C9A	0.8 (4)
C12B—O11B—C1B—C6B	−5.2 (5)	N1A—C2A—C3A—C31A	174.8 (3)
C1B—O11B—C12B—C13B	−166.5 (3)	C2A—C3A—C9A—C4A	177.5 (4)
C2A—N1A—C8A—C7A	−176.4 (3)	C2A—C3A—C9A—C8A	−0.3 (4)
C2A—N1A—C8A—C9A	0.8 (4)	C31A—C3A—C9A—C4A	3.1 (6)
C8A—N1A—C2A—C3A	−1.0 (4)	C31A—C3A—C9A—C8A	−174.8 (3)
O11B—C1B—C6B—C5B	177.0 (3)	C2A—C3A—C31A—C32A	7.3 (5)
C2B—C1B—C6B—C5B	−1.8 (5)	C9A—C3A—C31A—C32A	−179.7 (3)
O11B—C1B—C2B—C3B	−178.3 (3)	C9A—C4A—C5A—C6A	−0.6 (6)
C6B—C1B—C2B—C3B	0.6 (5)	C5A—C4A—C9A—C3A	−176.3 (3)
C1B—C2B—C3B—Cl3B	178.8 (3)	C5A—C4A—C9A—C8A	1.3 (5)

C1B—C2B—C3B—C4B	−0.1 (6)	C4A—C5A—C6A—C7A	−0.5 (6)
Cl3B—C3B—C4B—C5B	−178.1 (3)	C5A—C6A—C7A—C8A	0.9 (6)
C2B—C3B—C4B—C5B	0.9 (6)	C6A—C7A—C8A—N1A	176.6 (4)
C3B—C4B—C5B—C6B	−2.3 (6)	C6A—C7A—C8A—C9A	−0.2 (5)
C3B—C4B—C5B—Cl5B	179.5 (3)	N1A—C8A—C9A—C3A	−0.3 (4)
Cl5B—C5B—C6B—C1B	−179.1 (3)	N1A—C8A—C9A—C4A	−178.4 (3)
C4B—C5B—C6B—C1B	2.7 (6)	C7A—C8A—C9A—C3A	177.2 (3)
O11B—C12B—C13B—O13B	−2.9 (5)	C7A—C8A—C9A—C4A	−0.9 (5)
O11B—C12B—C13B—O14B	175.3 (3)	C3A—C31A—C32A—N32A	75.7 (4)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1*A*—H1*A*···O14*B*^{i}	0.87 (4)	2.04 (4)	2.838 (4)	152 (4)
N32*A*—H34*A*···O13*B*	0.87 (2)	2.05 (3)	2.875 (4)	160 (4)
N32*A*—H35*A*···O11*B*^{ii}	0.89 (3)	2.60 (4)	3.160 (4)	122 (3)
N32*A*—H35*A*···O13*B*^{ii}	0.89 (3)	1.87 (3)	2.739 (4)	164 (4)
N32*A*—H36*A*···O14*B*^{iii}	0.89 (4)	1.90 (4)	2.775 (4)	170 (4)
C2*A*—H2*A*···O13*B*^{iii}	0.95	2.55	3.495 (4)	177

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