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N-(3,5-Dichloro-4-hydroxyphenyl)acetamide

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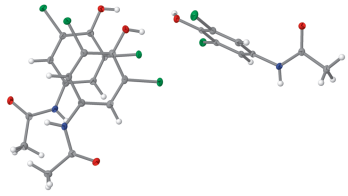
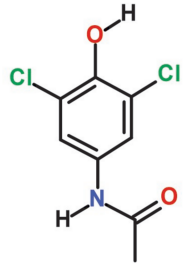
The title compound, $C_8H_7Cl_2NO_2$, crystallizes in the triclinic space group $P\overline{1}$ with three molecules in the asymmetric unit. Two of them are essentially planar, with mean deviations of 13 non-hydrogen atoms of 0.029 and 0.030 Å, and differ only in the conformation of the O—H hydrogen atom. The third molecule is quite nonplanar, with the CCNO acetamide plane forming a dihedral angle of 67.56 (5)° with the remainder of the molecule. In the extended structure, the two almost planar molecules form antiparallel hydrogen-bonded chains through N—H···O interactions of the acetamide substituents. The N—H group of the nonplanar molecule donates a bifurcated hydrogen bond to the O—H group and a Cl substituent of one of the planar molecules. The O—H groups of all molecules form intermolecular hydrogen bonds to other O—H groups or carbonyl O atoms. Together, the hydrogen bonds generate a three-dimensional network.

3D view



Chemical scheme



Structure description

The title compound, $C_8H_7Cl_2NO_2$ (**I**), is one of the two well characterized chlorination products formed when acetaminophen [*N*-(4-hydroxyphenyl)acetamide, $C_8H_9NO_2$], also known as paracetamol, reacts with hypochlorous acid–hypochlorite ($HOCl/^{-}OCl$; $pK_a \simeq 7.5$) under mildly oxidative, near-neutral pH conditions (Bedner & MacCrehan, 2006). Ring-chlorinated products of this type have been detected when wastewater and surface water samples spiked with environmentally relevant concentrations of acetaminophen were subjected to chlorine-based disinfection (Cao *et al.*, 2016; Kolpin *et al.*, 2002; Paíga *et al.*, 2025). Although the trichlorinated derivative of acetaminophen does not form under these conditions, the mono- and dichloro-substituted products are typically accompanied by *p*-benzoquinone imine, *p*-benzoquinone, and several high-molecular-weight species with *m*/*z* values between 320 and 610 (Bedner & MacCrehan, 2006; Glassmeyer & Shoemaker, 2005; Li *et al.*, 2022). These transformation products, particularly *p*-benzoquinone imine and *p*-benzoquinone, are generally perceived to possess greater toxicological potency, prompting the adoption of combined and advanced oxidation processes

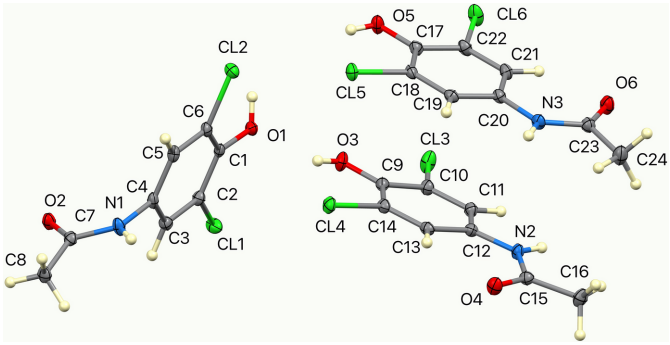


Figure 1
The molecular structure of (**I**), shown with displacement ellipsoids at the 50% probability level.

for their efficient removal and detoxification in treated wastewaters (Dahlin & Nelson, 1982; Postigo & Richardson, 2014; Qutob *et al.*, 2022; Phong Vo *et al.*, 2019).

Similar to those described for HOCl/$^{-}$OCl-mediated oxidations (Bedner & MacCrehan, 2006), the myeloperoxidase–H_2O_2–Cl^{-}-acetaminophen system may generate various chlorinated products, including the title compound (Van Zyl *et al.*, 1989). While *N*-(3,5-dichloro-4-hydroxyphenyl)acetamide may serve as a biomarker of chlorination, the compound itself could also pose a significant toxicological concern. Based on linear free-energy relationships and Hammett substituent principles, pK_a of (**I**) is predicted to be approximately 2.0–2.3 units lower than that of acetaminophen (p$K_a \simeq 9.5$), since each chlorine substituent in the *ortho* position to the phenolic –OH typically lowers the pK_a by about 1.0–1.2 units through strong inductive (–I) effects and stabilization of the phenoxide anion (Hansch *et al.*, 1991; Perrin *et al.*, 1981). Accordingly, with an expected pK_a in the range of 7.2–7.5, the title compound falls squarely within the classical 'uncoupler window' (pK_a 4–8; Heytler & Prichard, 1962). Thus, at physiological pH (7.4), roughly half of the molecules would be deprotonated and half protonated; the likely membrane-permeable properties of (**I**) would therefore favor its behavior as a protonophoric uncoupler, capable of dissipating the mitochondrial protonmotive force that drives ATP synthesis from ADP and inorganic phosphate.

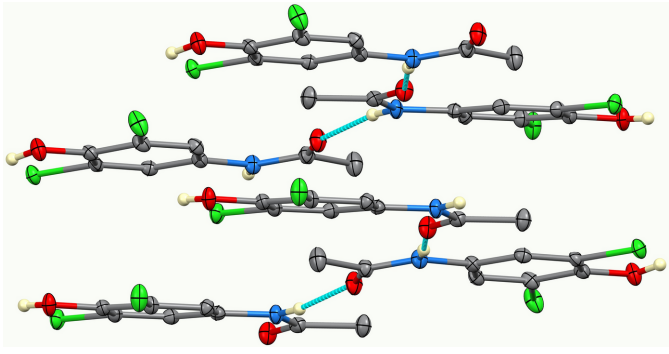


Figure 2
Intermolecular hydrogen-bonding network in the crystal structure of (**I**).

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

D—H···A	D—H	H···A	D···A	D—H···A
O1—H10*H*···O2^{i}	0.83 (2)	1.94 (2)	2.6776 (18)	148 (2)
N1—H1*N*···Cl3^{ii}	0.84 (2)	2.78 (2)	3.3925 (17)	131 (2)
N1—H1*N*···O3^{ii}	0.84 (2)	2.60 (2)	3.401 (2)	161 (2)
C8—H8*C*···O1^{ii}	0.98	2.60	3.521 (2)	157
O3—H30*H*···O1	0.81 (2)	2.03 (2)	2.7602 (18)	149 (3)
O3—H30*H*···Cl4	0.81 (2)	2.59 (2)	3.0528 (14)	118 (2)
N2—H2*N*···O6^{iii}	0.84 (2)	2.09 (2)	2.920 (2)	172 (2)
C13—H13···O4	0.95	2.26	2.866 (2)	121
O5—H50*H*···O2^{i}	0.81 (2)	2.22 (2)	2.9546 (18)	150 (3)
O5—H50*H*···Cl5	0.81 (2)	2.51 (2)	2.9949 (14)	119 (2)
N3—H3*N*···O4^{iv}	0.84 (2)	2.08 (2)	2.913 (2)	176 (2)
C21—H21···O6	0.95	2.24	2.853 (2)	121
C24—H24*A*···O4^{iv}	0.98	2.59	3.464 (2)	149

Symmetry codes: (i) $-x, -y+1, -z$; (ii) $x, y-1, z$; (iii) $-x+1, -y+2, -z+1$; (iv) $-x+1, -y+1, -z+1$.

In essence, the stabilization of the phenoxide anion by the two Cl substituents through inductive and intramolecular hydrogen-bonding effects, together with their likely enhanced lipophilicity and minimal steric hindrance, could render this compound an effective mitochondrial uncoupler. To provide an unambiguous structural basis for these chemical and biological considerations, single crystals of (**I**) were grown from aqueous solution and analyzed by single-crystal X-ray diffraction.

Compound (**I**) crystallizes in the triclinic space group $P\overline{1}$ with three independent molecules in the asymmetric unit (Fig. 1). Two of these molecules, containing atoms N2 and N3, are essentially planar, with mean deviations of their 13 non-hydrogen atoms of 0.029 and 0.030 Å, respectively, and are nearly parallel, forming a dihedral angle of 7.51 (4)°. They differ only in the conformation of the OH hydrogen atom (C14—C9—O3—H30H = 3.09°; C18—C17—O5—H50H = −6.9°). The third molecule containing atom N1 is distinctly nonplanar, with the C7/C8/N1/O2 acetamide plane forming a dihedral angle of 67.56 (5)° with the remainder of the molecule. In the three molecules, the C—Cl distances range from 1.7197 (17) to 1.7381 (18) Å (mean 1.7311 Å), and the C—OH distances range from 1.351 (2) to 1.359 (2) Å (mean 1.354 Å).

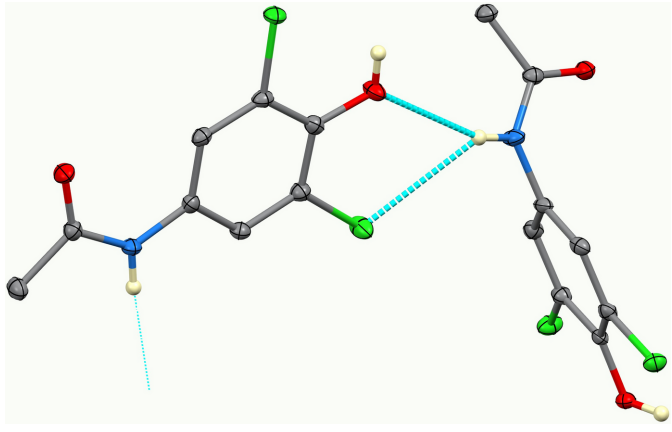


Figure 3
Detail of the bifurcated O—H···(O,Cl) hydrogen-bonding motif observed in the crystal packing of (**I**).

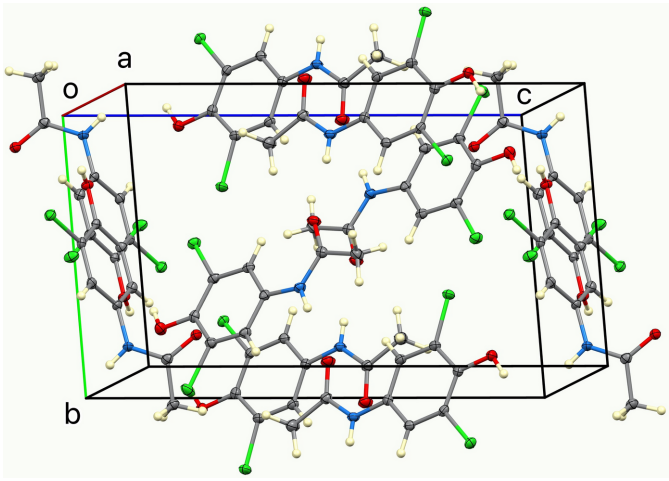


Figure 4
View of the unit cell of (**I**) along the *a*-axis direction.

In the extended structure of (**I**), the molecules are linked by numerous hydrogen bonds (Table 1). Among these, the two planar molecules form antiparallel hydrogen-bonded chains through N—H···O interactions of the acetamide substituents. The third, nonplanar molecule links adjacent chains *via* additional bifurcated O—H···(O,Cl) interactions, generating a three-dimensional hydrogen-bonded network that consolidates the crystal packing (Fig. 2). Figs. 3 and 4 show, respectively, selected bifurcated hydrogen-bonding motifs and a view of the unit cell along the *a*-axis direction.

Synthesis and crystallization

The title compound was synthesized by acetylation of 4-amino-2,6-phenol (CAS 5930–28-9; purity: 97%) using acetic anhydride in acetic acid solvent: 1.78 g (10 mmol) of 4-amino-2,6-phenol in 10 ml of glacial acetic was allowed to react with 1.23 g (12 mmol) of acetic anhydride for 24–48 h at room temperature. The reaction mixture was stirred continuously during the reaction. In the end, the mixture was dried under vacuum, and the residue was purified by recrystallization once from aqueous solution. Single crystals of (**I**) in the form of colorless needles were grown in water by slow cooling of a hot and nearly saturated solution.

Refinement

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

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_8H_7Cl_2NO_2$
M_r	220.05
Crystal system, space group	Triclinic, $P\overline{1}$
Temperature (K)	100
a, b, c (Å)	9.6844 (14), 9.8884 (16), 14.976 (3)
α, β, γ (°)	85.181 (10), 76.43 (1), 74.769 (7)
V (Å^3)	1344.8 (4)
Z	6
Radiation type	Cu $K\alpha$
μ (mm^{-1})	6.24
Crystal size (mm)	0.20 × 0.15 × 0.01
Data collection	
Diffractometer	Bruker D8 Venture DUO with Photon III C14
Absorption correction	Multi-scan (*SADABS*; Krause *et al.*, 2015)
T_{min}, T_{max}	0.625, 0.940
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	31306, 5786, 5206
R_{int}	0.053
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.640
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.032, 0.089, 1.06
No. of reflections	5786
No. of parameters	373
No. of restraints	6
H-atom treatment	H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.55, −0.39

Computer programs: *APEX5* and *SAINT* (Bruker, 2016), *SHELXT2018/2* (Sheldrick, 2015*a*), *SHELXL2019/1* (Sheldrick, 2015*b*) and *Mercury* (Macrae *et al.*, 2020).

Funding information

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full crystallographic data

IUCrData (2026). **11**, x260075 [https://doi.org/10.1107/S2414314626000751]

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Crystal data

$C_8H_7Cl_2NO_2$
M_r = 220.05
Triclinic, $P\overline{1}$
a = 9.6844 (14) Å
b = 9.8884 (16) Å
c = 14.976 (3) Å
α = 85.181 (10)°
β = 76.43 (1)°
γ = 74.769 (7)°
V = 1344.8 (4) Å^3
Z = 6
$F(000)$ = 672
D_x = 1.630 Mg m^{-3}
Cu $K\alpha$ radiation, λ = 1.54184 Å
Cell parameters from 9884 reflections
θ = 3.0–79.7°
μ = 6.24 mm^{-1}
T = 100 K
Plate, colourless
0.20 × 0.15 × 0.01 mm

Data collection

Bruker D8 Venture DUO with Photon III C14 diffractometer
Radiation source: IμS 3.0 microfocus
φ and ω scans
Absorption correction: multi-scan (SADABS; Krause *et al.*, 2015)
T_{min} = 0.625, T_{max} = 0.940
31306 measured reflections
5786 independent reflections
5206 reflections with $I > 2\sigma(I)$
R_{int} = 0.053
θ_{max} = 80.5°, θ_{min} = 3.0°
h = −12→12
k = −12→12
l = −18→19

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.032
$wR(F^2)$ = 0.089
S = 1.06
5786 reflections
373 parameters
6 restraints
Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0476P)^2 + 0.6745P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.55 e Å^{-3}
$\Delta\rho_{min}$ = −0.39 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.

Refinement. All non-H atoms were refined anisotropically. H atoms were treated by a mixed independent and riding model.

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

	x	*y*	*z*	U_{iso}*/U_{eq}
Cl1	0.35134 (4)	0.53881 (4)	−0.05637 (3)	0.01871 (10)
Cl2	−0.13004 (4)	0.52183 (4)	0.20588 (3)	0.01605 (10)
O1	0.09880 (13)	0.65297 (12)	0.08713 (8)	0.0140 (2)
H10H	0.0124 (19)	0.696 (2)	0.1091 (16)	0.021*
O2	0.13322 (14)	0.12695 (13)	−0.12814 (8)	0.0169 (3)
N1	0.17559 (17)	0.08975 (15)	0.01623 (10)	0.0161 (3)
H1N	0.204 (3)	0.029 (2)	0.0552 (15)	0.019*
C1	0.11189 (18)	0.51671 (17)	0.07256 (11)	0.0124 (3)
C2	0.23029 (18)	0.44736 (18)	0.00553 (12)	0.0141 (3)
C3	0.25114 (19)	0.30765 (18)	−0.01319 (12)	0.0151 (3)
H3	0.332077	0.262650	−0.059234	0.018*
C4	0.15275 (19)	0.23420 (17)	0.03591 (12)	0.0143 (3)
C5	0.03595 (19)	0.29868 (18)	0.10440 (11)	0.0142 (3)
H5	−0.030199	0.247607	0.138824	0.017*
C6	0.01692 (19)	0.43888 (18)	0.12201 (11)	0.0134 (3)
C7	0.17477 (18)	0.04592 (18)	−0.06649 (12)	0.0143 (3)
C8	0.2294 (2)	−0.10968 (18)	−0.07887 (12)	0.0171 (3)
H8A	0.329501	−0.141133	−0.068846	0.026*
H8B	0.229353	−0.131997	−0.141389	0.026*
H8C	0.165136	−0.157433	−0.034474	0.026*
Cl3	0.40021 (5)	1.01733 (5)	0.16640 (3)	0.02288 (11)
Cl4	0.33207 (4)	0.48954 (4)	0.21640 (3)	0.01722 (10)
O3	0.28764 (15)	0.78676 (14)	0.13405 (9)	0.0186 (3)
H30H	0.254 (3)	0.722 (2)	0.1269 (18)	0.028*
O4	0.65977 (15)	0.44791 (13)	0.43476 (9)	0.0194 (3)
N2	0.63076 (16)	0.68011 (15)	0.39607 (10)	0.0150 (3)
H2N	0.652 (3)	0.754 (2)	0.4057 (16)	0.018*
C9	0.37032 (18)	0.75224 (18)	0.19782 (11)	0.0147 (3)
C10	0.43264 (19)	0.85323 (18)	0.22059 (12)	0.0151 (3)
C11	0.51803 (18)	0.82759 (18)	0.28510 (12)	0.0144 (3)
H11	0.558929	0.898799	0.298694	0.017*
C12	0.54439 (18)	0.69736 (18)	0.33037 (12)	0.0136 (3)
C13	0.48653 (18)	0.59321 (18)	0.30858 (12)	0.0142 (3)
H13	0.504517	0.503491	0.338150	0.017*
C14	0.40183 (18)	0.62223 (18)	0.24281 (12)	0.0144 (3)
C15	0.68395 (18)	0.56115 (18)	0.44264 (12)	0.0146 (3)
C16	0.7786 (2)	0.57841 (19)	0.50564 (13)	0.0194 (4)
H16A	0.781445	0.676874	0.504637	0.029*
H16B	0.878336	0.519833	0.484830	0.029*
H16C	0.737581	0.549727	0.568375	0.029*
Cl5	−0.00475 (5)	0.72743 (4)	0.31367 (3)	0.01681 (10)
Cl6	0.03404 (6)	1.25666 (4)	0.32909 (3)	0.02303 (11)
O5	−0.05222 (15)	1.03136 (13)	0.25812 (9)	0.0190 (3)
H50H	−0.069 (3)	0.965 (2)	0.2380 (18)	0.028*
O6	0.32528 (15)	1.05309 (13)	0.56203 (10)	0.0208 (3)

N3	0.28449 (16)	0.85184 (15)	0.52262 (10)	0.0152 (3)
H3N	0.298 (3)	0.766 (2)	0.5329 (16)	0.018*
C17	0.02825 (19)	0.98609 (18)	0.32290 (11)	0.0149 (3)
C18	0.06138 (19)	0.84906 (18)	0.35663 (12)	0.0141 (3)
C19	0.14531 (19)	0.80609 (18)	0.42180 (12)	0.0141 (3)
H19	0.166334	0.710941	0.442481	0.017*
C20	0.19856 (18)	0.90281 (18)	0.45676 (11)	0.0136 (3)
C21	0.16516 (19)	1.04206 (18)	0.42647 (12)	0.0150 (3)
H21	0.200021	1.109642	0.450257	0.018*
C22	0.0804 (2)	1.08122 (18)	0.36118 (12)	0.0160 (3)
C23	0.34095 (19)	0.92514 (19)	0.57035 (12)	0.0162 (3)
C24	0.4274 (2)	0.8378 (2)	0.63615 (14)	0.0233 (4)
H24A	0.427483	0.739182	0.632890	0.035*
H24B	0.382235	0.870236	0.698858	0.035*
H24C	0.528572	0.847223	0.619395	0.035*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.0173 (2)	0.0159 (2)	0.0224 (2)	−0.00726 (15)	0.00099 (15)	−0.00318 (15)
Cl2	0.01757 (19)	0.01529 (19)	0.01441 (19)	−0.00439 (15)	−0.00074 (15)	−0.00249 (14)
O1	0.0149 (6)	0.0111 (6)	0.0170 (6)	−0.0038 (5)	−0.0042 (5)	−0.0025 (4)
O2	0.0232 (6)	0.0122 (6)	0.0165 (6)	−0.0030 (5)	−0.0086 (5)	−0.0007 (5)
N1	0.0260 (8)	0.0093 (7)	0.0143 (7)	−0.0043 (6)	−0.0075 (6)	0.0002 (5)
C1	0.0144 (8)	0.0112 (8)	0.0143 (8)	−0.0035 (6)	−0.0077 (6)	−0.0007 (6)
C2	0.0154 (8)	0.0135 (8)	0.0152 (8)	−0.0060 (6)	−0.0047 (6)	0.0008 (6)
C3	0.0167 (8)	0.0148 (8)	0.0138 (8)	−0.0034 (6)	−0.0040 (6)	−0.0012 (6)
C4	0.0209 (8)	0.0103 (8)	0.0145 (8)	−0.0041 (6)	−0.0088 (7)	−0.0011 (6)
C5	0.0184 (8)	0.0144 (8)	0.0131 (8)	−0.0076 (6)	−0.0065 (6)	0.0011 (6)
C6	0.0162 (8)	0.0150 (8)	0.0102 (7)	−0.0040 (6)	−0.0049 (6)	−0.0009 (6)
C7	0.0146 (8)	0.0140 (8)	0.0152 (8)	−0.0056 (6)	−0.0028 (6)	−0.0012 (6)
C8	0.0216 (9)	0.0125 (8)	0.0190 (8)	−0.0052 (7)	−0.0062 (7)	−0.0019 (6)
Cl3	0.0330 (2)	0.0166 (2)	0.0271 (2)	−0.01274 (18)	−0.01799 (19)	0.00836 (16)
Cl4	0.0192 (2)	0.0145 (2)	0.0218 (2)	−0.00792 (15)	−0.00755 (16)	−0.00149 (15)
O3	0.0243 (7)	0.0177 (6)	0.0196 (6)	−0.0108 (5)	−0.0112 (5)	0.0022 (5)
O4	0.0244 (7)	0.0116 (6)	0.0251 (7)	−0.0050 (5)	−0.0113 (5)	0.0020 (5)
N2	0.0179 (7)	0.0115 (7)	0.0182 (7)	−0.0054 (6)	−0.0073 (6)	−0.0010 (6)
C9	0.0143 (8)	0.0170 (8)	0.0134 (8)	−0.0048 (6)	−0.0031 (6)	−0.0021 (6)
C10	0.0168 (8)	0.0130 (8)	0.0164 (8)	−0.0048 (6)	−0.0045 (6)	0.0003 (6)
C11	0.0151 (8)	0.0133 (8)	0.0163 (8)	−0.0052 (6)	−0.0042 (6)	−0.0012 (6)
C12	0.0126 (7)	0.0129 (8)	0.0147 (8)	−0.0020 (6)	−0.0028 (6)	−0.0020 (6)
C13	0.0141 (8)	0.0115 (8)	0.0164 (8)	−0.0037 (6)	−0.0015 (6)	−0.0013 (6)
C14	0.0143 (8)	0.0129 (8)	0.0170 (8)	−0.0056 (6)	−0.0016 (6)	−0.0029 (6)
C15	0.0145 (8)	0.0139 (8)	0.0152 (8)	−0.0034 (6)	−0.0031 (6)	−0.0001 (6)
C16	0.0224 (9)	0.0170 (9)	0.0221 (9)	−0.0062 (7)	−0.0116 (7)	0.0039 (7)
Cl5	0.0224 (2)	0.01470 (19)	0.01636 (19)	−0.00743 (15)	−0.00643 (15)	−0.00220 (14)
Cl6	0.0402 (3)	0.01053 (19)	0.0213 (2)	−0.00473 (17)	−0.01516 (19)	0.00195 (15)
O5	0.0283 (7)	0.0134 (6)	0.0187 (6)	−0.0043 (5)	−0.0130 (5)	−0.0001 (5)

O6	0.0262 (7)	0.0128 (6)	0.0278 (7)	−0.0062 (5)	−0.0127 (6)	−0.0017 (5)
N3	0.0190 (7)	0.0089 (7)	0.0190 (7)	−0.0031 (6)	−0.0074 (6)	−0.0001 (5)
C17	0.0173 (8)	0.0153 (8)	0.0119 (7)	−0.0031 (7)	−0.0034 (6)	−0.0014 (6)
C18	0.0161 (8)	0.0142 (8)	0.0127 (7)	−0.0055 (6)	−0.0011 (6)	−0.0039 (6)
C19	0.0154 (8)	0.0114 (8)	0.0146 (8)	−0.0029 (6)	−0.0019 (6)	−0.0007 (6)
C20	0.0157 (8)	0.0134 (8)	0.0118 (7)	−0.0036 (6)	−0.0029 (6)	−0.0009 (6)
C21	0.0193 (8)	0.0122 (8)	0.0142 (8)	−0.0052 (6)	−0.0033 (6)	−0.0016 (6)
C22	0.0219 (8)	0.0109 (8)	0.0147 (8)	−0.0035 (7)	−0.0038 (7)	−0.0005 (6)
C23	0.0174 (8)	0.0147 (8)	0.0175 (8)	−0.0040 (7)	−0.0052 (7)	−0.0018 (6)
C24	0.0298 (10)	0.0171 (9)	0.0274 (10)	−0.0048 (8)	−0.0158 (8)	−0.0007 (7)

Geometric parameters (Å, º)

Cl1—C2	1.7197 (17)	C10—C11	1.380 (2)
Cl2—C6	1.7311 (17)	C11—C12	1.395 (2)
O1—C1	1.351 (2)	C11—H11	0.9500
O1—H10H	0.833 (16)	C12—C13	1.388 (2)
O2—C7	1.240 (2)	C13—C14	1.391 (2)
N1—C7	1.349 (2)	C13—H13	0.9500
N1—C4	1.432 (2)	C15—C16	1.508 (2)
N1—H1N	0.838 (18)	C16—H16A	0.9800
C1—C6	1.394 (2)	C16—H16B	0.9800
C1—C2	1.398 (2)	C16—H16C	0.9800
C2—C3	1.386 (2)	Cl5—C18	1.7317 (17)
C3—C4	1.386 (2)	Cl6—C22	1.7303 (18)
C3—H3	0.9500	O5—C17	1.359 (2)
C4—C5	1.387 (2)	O5—H50H	0.812 (17)
C5—C6	1.389 (2)	O6—C23	1.233 (2)
C5—H5	0.9500	N3—C23	1.347 (2)
C7—C8	1.502 (2)	N3—C20	1.414 (2)
C8—H8A	0.9800	N3—H3N	0.835 (18)
C8—H8B	0.9800	C17—C18	1.388 (2)
C8—H8C	0.9800	C17—C22	1.395 (2)
Cl3—C10	1.7381 (18)	C18—C19	1.384 (2)
Cl4—C14	1.7357 (17)	C19—C20	1.387 (2)
O3—C9	1.353 (2)	C19—H19	0.9500
O3—H30H	0.814 (17)	C20—C21	1.391 (2)
O4—C15	1.223 (2)	C21—C22	1.387 (3)
N2—C15	1.359 (2)	C21—H21	0.9500
N2—C12	1.407 (2)	C23—C24	1.511 (3)
N2—H2N	0.836 (18)	C24—H24A	0.9800
C9—C14	1.395 (2)	C24—H24B	0.9800
C9—C10	1.396 (2)	C24—H24C	0.9800
C1—O1—H10H	112.4 (17)	C12—C13—C14	118.97 (16)
C7—N1—C4	123.14 (15)	C12—C13—H13	120.5
C7—N1—H1N	118.3 (17)	C14—C13—H13	120.5
C4—N1—H1N	117.9 (17)	C13—C14—C9	123.16 (16)

O1—C1—C6	124.18 (15)	C13—C14—Cl4	117.92 (13)
O1—C1—C2	118.36 (15)	C9—C14—Cl4	118.92 (14)
C6—C1—C2	117.44 (15)	O4—C15—N2	123.95 (16)
C3—C2—C1	121.68 (16)	O4—C15—C16	121.55 (16)
C3—C2—Cl1	119.26 (13)	N2—C15—C16	114.50 (15)
C1—C2—Cl1	119.05 (13)	C15—C16—H16A	109.5
C2—C3—C4	119.35 (16)	C15—C16—H16B	109.5
C2—C3—H3	120.3	H16A—C16—H16B	109.5
C4—C3—H3	120.3	C15—C16—H16C	109.5
C3—C4—C5	120.55 (15)	H16A—C16—H16C	109.5
C3—C4—N1	118.90 (16)	H16B—C16—H16C	109.5
C5—C4—N1	120.54 (16)	C17—O5—H50H	109.9 (19)
C4—C5—C6	119.17 (16)	C23—N3—C20	128.15 (15)
C4—C5—H5	120.4	C23—N3—H3N	118.5 (16)
C6—C5—H5	120.4	C20—N3—H3N	113.3 (16)
C5—C6—C1	121.77 (15)	O5—C17—C18	124.46 (16)
C5—C6—Cl2	119.79 (13)	O5—C17—C22	119.62 (15)
C1—C6—Cl2	118.43 (13)	C18—C17—C22	115.91 (16)
O2—C7—N1	123.09 (16)	C19—C18—C17	122.96 (16)
O2—C7—C8	122.14 (15)	C19—C18—Cl5	119.16 (13)
N1—C7—C8	114.77 (15)	C17—C18—Cl5	117.88 (13)
C7—C8—H8A	109.5	C18—C19—C20	119.59 (16)
C7—C8—H8B	109.5	C18—C19—H19	120.2
H8A—C8—H8B	109.5	C20—C19—H19	120.2
C7—C8—H8C	109.5	C19—C20—C21	119.39 (16)
H8A—C8—H8C	109.5	C19—C20—N3	116.74 (15)
H8B—C8—H8C	109.5	C21—C20—N3	123.87 (16)
C9—O3—H30H	110.7 (19)	C22—C21—C20	119.35 (16)
C15—N2—C12	128.27 (15)	C22—C21—H21	120.3
C15—N2—H2N	118.2 (16)	C20—C21—H21	120.3
C12—N2—H2N	113.5 (16)	C21—C22—C17	122.76 (16)
O3—C9—C14	125.72 (15)	C21—C22—Cl6	118.03 (13)
O3—C9—C10	118.25 (15)	C17—C22—Cl6	119.18 (14)
C14—C9—C10	116.03 (16)	O6—C23—N3	123.84 (17)
C11—C10—C9	122.28 (16)	O6—C23—C24	121.59 (16)
C11—C10—Cl3	118.96 (13)	N3—C23—C24	114.58 (16)
C9—C10—Cl3	118.76 (14)	C23—C24—H24A	109.5
C10—C11—C12	120.13 (16)	C23—C24—H24B	109.5
C10—C11—H11	119.9	H24A—C24—H24B	109.5
C12—C11—H11	119.9	C23—C24—H24C	109.5
C13—C12—C11	119.41 (16)	H24A—C24—H24C	109.5
C13—C12—N2	123.90 (16)	H24B—C24—H24C	109.5
C11—C12—N2	116.70 (15)		

O1—C1—C2—C3	179.89 (15)	C11—C12—C13—C14	−0.9 (2)
C6—C1—C2—C3	1.7 (3)	N2—C12—C13—C14	179.62 (16)
O1—C1—C2—Cl1	−1.1 (2)	C12—C13—C14—C9	−0.7 (3)
C6—C1—C2—Cl1	−179.35 (13)	C12—C13—C14—Cl4	179.54 (13)

C1—C2—C3—C4	−0.3 (3)	O3—C9—C14—C13	−179.34 (16)
Cl1—C2—C3—C4	−179.28 (13)	C10—C9—C14—C13	1.7 (2)
C2—C3—C4—C5	−1.2 (3)	O3—C9—C14—Cl4	0.4 (2)
C2—C3—C4—N1	−179.79 (16)	C10—C9—C14—Cl4	−178.52 (13)
C7—N1—C4—C3	−61.8 (2)	C12—N2—C15—O4	−1.8 (3)
C7—N1—C4—C5	119.65 (19)	C12—N2—C15—C16	177.56 (16)
C3—C4—C5—C6	1.3 (3)	O5—C17—C18—C19	179.13 (16)
N1—C4—C5—C6	179.84 (15)	C22—C17—C18—C19	−2.3 (3)
C4—C5—C6—C1	0.1 (3)	O5—C17—C18—Cl5	−0.2 (2)
C4—C5—C6—Cl2	178.90 (13)	C22—C17—C18—Cl5	178.36 (13)
O1—C1—C6—C5	−179.70 (15)	C17—C18—C19—C20	0.7 (3)
C2—C1—C6—C5	−1.6 (2)	Cl5—C18—C19—C20	−179.88 (13)
O1—C1—C6—Cl2	1.5 (2)	C18—C19—C20—C21	0.8 (2)
C2—C1—C6—Cl2	179.64 (13)	C18—C19—C20—N3	−179.53 (15)
C4—N1—C7—O2	−10.9 (3)	C23—N3—C20—C19	−175.73 (17)
C4—N1—C7—C8	168.63 (16)	C23—N3—C20—C21	3.9 (3)
O3—C9—C10—C11	179.73 (16)	C19—C20—C21—C22	−0.6 (3)
C14—C9—C10—C11	−1.3 (3)	N3—C20—C21—C22	179.69 (16)
O3—C9—C10—Cl3	0.2 (2)	C20—C21—C22—C17	−1.0 (3)
C14—C9—C10—Cl3	179.25 (13)	C20—C21—C22—Cl6	177.07 (13)
C9—C10—C11—C12	−0.2 (3)	O5—C17—C22—C21	−178.93 (16)
Cl3—C10—C11—C12	179.27 (13)	C18—C17—C22—C21	2.4 (3)
C10—C11—C12—C13	1.3 (3)	O5—C17—C22—Cl6	3.0 (2)
C10—C11—C12—N2	−179.14 (15)	C18—C17—C22—Cl6	−175.66 (13)
C15—N2—C12—C13	6.5 (3)	C20—N3—C23—O6	−0.6 (3)
C15—N2—C12—C11	−172.98 (16)	C20—N3—C23—C24	179.64 (17)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H10*H*···O2^{i}	0.83 (2)	1.94 (2)	2.6776 (18)	148 (2)
N1—H1*N*···Cl3^{ii}	0.84 (2)	2.78 (2)	3.3925 (17)	131 (2)
N1—H1*N*···O3^{ii}	0.84 (2)	2.60 (2)	3.401 (2)	161 (2)
C8—H8*C*···O1^{ii}	0.98	2.60	3.521 (2)	157
O3—H30*H*···O1	0.81 (2)	2.03 (2)	2.7602 (18)	149 (3)
O3—H30*H*···Cl4	0.81 (2)	2.59 (2)	3.0528 (14)	118 (2)
N2—H2*N*···O6^{iii}	0.84 (2)	2.09 (2)	2.920 (2)	172 (2)
C13—H13···O4	0.95	2.26	2.866 (2)	121
O5—H50*H*···O2^{i}	0.81 (2)	2.22 (2)	2.9546 (18)	150 (3)
O5—H50*H*···Cl5	0.81 (2)	2.51 (2)	2.9949 (14)	119 (2)
N3—H3*N*···O4^{iv}	0.84 (2)	2.08 (2)	2.913 (2)	176 (2)
C21—H21···O6	0.95	2.24	2.853 (2)	121
C24—H24*A*···O4^{iv}	0.98	2.59	3.464 (2)	149

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