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Substituted clonidine derivatives. I. 3,5-Dichloro-4-(imidazolidin-2-ylideneamino)benzonitrile and 3,5-dichloro-4-(1,3-diisobutyrylimidazolidin-2-ylideneamino)benzonitrile

E. M. Elssfah, K. Chinnakali, H.-K. Fun, I. W. Mathison, E. K. Gan, M. Zubaid, T. W. Sam and K. S. Khoo

Abstract

In the title compounds, $C_{10}H_8C_{12}N_4$, (I), and $C_{18}H_{20}C_{12}N_4O_2$, (II), the dihedral angle between the imidazolidine and phenyl rings are 67.7 (1) and 70.34 (9)°, respectively. In (I), the imidazolidine ring adopts a half-chair conformation, whereas in (II), it is in a flattened envelope conformation. In (I), the glide related molecules are linked by N2—H2A···N1(x, 1/2 − y, z − 1/2) hydrogen bonds with an N···N distance of 2.848 (4)Å to form an infinite chain along the c axis with the phenyl rings stacked at a perpendicular distance of 3.633 (5) Å. In (II), a weak C—H···N intramolecular hydrogen bond exists between C11 and N1, with a C···N distance of 2.902 (4) Å.

Comment

Clonidine, an imidazolidine derivative is one of the centrally acting antihypertensive drugs which act primarily *via* α-adrenoceptors in the brain. Quantitative structure-activity studies suggest that the hypertensive activity of this molecule is governed by distinct steric and electronic characteristics (van Zwieten *et al.*, 1983). In recent years a large number of imidazolidine derivatives were synthesized in an effort to obtain a more active molecule, but none of them were found to be more potent than clonidine itself. Recently we have also prepared some imidazolidine derivatives and they were subjected to pharmacological studies. The title compounds, (I) and (II), from a class of such compounds studied contain a strongly electron withdrawing –CN group at the *para* position of the phenyl ring. Both the compounds were found to be inactive against hypertension. The *x*-ray structure determination studies were carried out in order to elucidate the molecular conformation.

The N—C distances observed in (I) are comparable with those observed in the clonidine moiety (Byre *et al.*, 1976; Cody & DeTitta, 1979; Carpy *et al.*, 1986) indicating delocalization of electron cloud around C7. In (II), the N1—C7 distance shows a double bond character and as a result, the N2—C7 and N3—C7 bonds are elongated. The N1—C1—C2 angle in (I) and C1—N1—C7 in (II) are widened compared to clonidine. The imidazolidine ring of (I) adopts a half-chair conformation and that of (II) is in a flattened envelope conformation with C8 deviating from the mean plane by 0.203 (3) Å. The mean planes through the phenyl and imidazolidine rings form dihedral angles of 67.7 (1) and 70.34 (9)° for (I) and (II), respectively. In (II) one of the isobutryl chain is twisted more compared to other due to steric interactions (C7—N2—C14—O2 11.9 (4)° and C7—N3—C10—O1 178.0 (3)°).

In the crystal of (I), the glide related molecules are linked by N2—H2A···N1(x, 1/2 − y, z − 1/2) hydrogen bonds with an N···N distance of 2.848 (4)Å to form an infinite chain along the c axis with the phenyl rings stacked at a perpendicular distance of 3.633 (5) Å. In (II), a weak C—H·N intramolecular hydrogen bond exists between C11 and N1 with a C···N distance of 2.898 (4) Å.

CIF access

Experimental

p-Aminobenzonitrile was converted to 2,6-dichloro-4-aminobenzonitrile with a mixture of concentrated hydrochloric acid and hydrogen peroxide. The product was in turn reacted with the formylating agent from formic acid and acetic anhydride to give the formate. Dehydration and chlorination of the amide with a mixture of thionyl chloride and sulfuryl chloride gave the dichloroimine derivative which on reaction with anhydrous ethylenediamine gave 2-(4-cyano- 2,6-dichlorophenylimino)imidazolidine, (I). Recrystallization from ethyl acetate gave the crystals used in this study.

2-(4-Cyano-2,6-dichlorophenylimino)imidazolidine was next reacted with an excess of isobutyryl anhydride in isobutyric acid for 12 h at 60°C. The reaction was quenched with aqueous ammonia and extracted with ether. Evaporation of the ether extract gave a mixture from which 2-(4-cyano-2,6- dichlorophenylimino) −1,3-diisobutyimidazolidine, (II), was isolated by flash column chromatography on silica gel with toluene: ethyl acetae (7:3) as eluant. Final recrystallization from ethyl acetate gave plate-like crystals.

Refinement

The data collection for (I) was covered over a hemisphere of reciprocal space by a combination of three sets of exposures; each set had a different φ angle (0, 88 and 180°) for the crystal and each exposure of 30 s covered 0.3° in ω. The crystal-to-detector distance was 4 cm and the detector swing angle was −35°. Coverage of the unique set is over 99% complete. Crystal decay was monitored by repeating thirty initial frames at the end of data collection and analysing the duplicate reflections, and was found to be negligible. For refinement only reflections having 2θ<55° were used (only few observed reflections for 2θ>55°)

Computing details

Data collection: *SMART* (Siemens, 1996) for (IUC9900066-1); *XSCANS* (Siemens, 1994) for (IUC9900066-2). Cell refinement: *SAINT* (Siemens, 1996) for (IUC9900066-1); *XSCANS* (Siemens, 1994) for (IUC9900066-2). Data reduction: *SAINT* (Siemens, 1996) for (IUC9900066-1); *XSCANS* (Siemens, 1994) for (IUC9900066-2). Program(s) used to solve structure: *SHELXS97* (Sheldrick, 1990) for (IUC9900066-1); *SHELXTL* (Sheldrick, 1997a) for (IUC9900066-2). Program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997b) for (IUC9900066-1); *SHELXTL* (Sheldrick, 1997a) for (IUC9900066-2). Molecular graphics: *SHELXL97* (Sheldrick, 1997b) for (IUC9900066-1); *SHELXTL* (Sheldrick, 1997a) for (IUC9900066-2). Software used to prepare material for publication: *SHELXTL* (Sheldrick, 1997a) and *PARST* (Nardelli, 1995) for (IUC9900066-1); *SHELXTL* (Sheldrick, 1997a) *PARST* (Nardelli, 1995) for (IUC9900066-2)

2-(4-cyano-2,6-dichlorophenylimino)imidazolidine

Crystal data

$C_{10}H_8Cl_2N_4$	$V = 1123.69\ (8)$ Å^3
$M_r = 255.10$	$Z = 4$
Monoclinic, $P2_1/c$	Mo $K\alpha$
$a = 10.3746\ (4)$ Å	$\mu = 0.55$ mm^{-1}
$b = 13.7857\ (5)$ Å	$T = 293\ (2)$ K

c = 7.9098 (4) Å
β = 96.637 (2)°
0.32 × 0.14 × 0.08 mm

Data collection

Siemens SMART CCD area-detector diffractometer
2569 independent reflections
Absorption correction: empirical (using intensity measurements) (SADABS; Siemens, 1996)
1316 reflections with $I > 2\sigma(I)$
T_{min} = 0.82, T_{max} = 0.96
R_{int} = 0.079
7186 measured reflections

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.068
145 parameters
$wR(F^2)$ = 0.158
H-atom parameters constrained
S = 1.04
$\Delta\rho_{max}$ = 0.34 e Å^{-3}
2569 reflections
$\Delta\rho_{min}$ = −0.27 e Å^{-3}

Table 1
Selected geometric parameters (Å)

N1—C7	1.316 (5)	N3—C7	1.349 (5)
N1—C1	1.379 (4)	N3—C9	1.460 (5)
N2—C7	1.333 (5)	N4—C10	1.134 (5)
N2—C8	1.443 (5)		

2-(4-cyano-2,6-dichlorophenylimino)-1,3-diisobutyrylimidazolidine

Crystal data

$C_{18}H_{20}Cl_2N_4O_2$
V = 1944.9 (4) Å^3
M_r = 395.28
Z = 4
Monoclinic, $P2_1/c$
Mo $K\alpha$
a = 9.8442 (13) Å
μ = 0.35 mm^{-1}
b = 18.997 (3) Å
T = 293 (2) K
c = 10.9399 (12) Å
0.54 × 0.40 × 0.20 mm
β = 108.079 (10)°

Data collection

Siemens P4 diffractometer
1840 reflections with $I > 2\sigma(I)$
Absorption correction: empirical (using intensity measurements) (XSCANS; Siemens, 1994)
R_{int} = 0.037
T_{min} = 0.832, T_{max} = 0.933
3 standard reflections
5509 measured reflections
every 97 reflections

4424 independent reflections	intensity decay: <3%

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.044$	4424 reflections
$wR(F^2) = 0.077$	240 parameters
$S = 0.96$	H-atom parameters constrained

Table 2
Selected geometric parameters (Å)

O1—C10	1.210 (3)	N2—C8	1.475 (3)
O2—C14	1.213 (3)	N3—C10	1.407 (3)
N1—C7	1.258 (3)	N3—C7	1.408 (3)
N1—C1	1.389 (3)	N3—C9	1.467 (3)
N2—C7	1.400 (3)	N4—C18	1.146 (3)
N2—C14	1.400 (3)		

Acknowledgements

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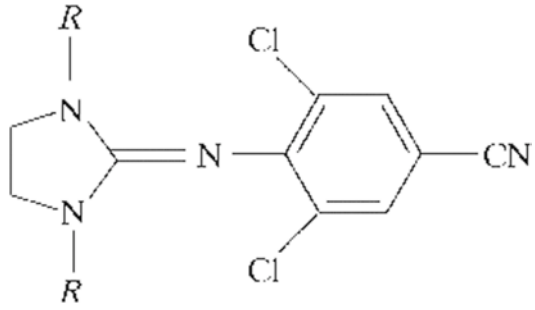
Scheme 1



supplementary materials

2-(4-cyano-2,6-dichlorophenylimino)imidazolidine

Crystal data

$C_{10}H_8Cl_2N_4$	F_{000} = 520
M_r = 255.10	D_x = 1.508 Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation λ = 0.71073 Å
a = 10.3746 (4) Å	Cell parameters from 1846 reflections
b = 13.7857 (5) Å	θ = 3.0–33.0º
c = 7.9098 (4) Å	μ = 0.55 mm^{-1}
β = 96.637 (2)º	T = 293 (2) K
V = 1123.69 (8) Å^3	Plate, colourless
Z = 4	0.32 × 0.14 × 0.08 mm

Data collection

Siemens SMART CCD area-detector diffractometer	2569 independent reflections
Radiation source: fine-focus sealed tube	1316 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.079
Detector resolution: 8.33 pixels mm^{-1}	θ_{max} = 27.5º
T = 293(2) K	θ_{min} = 3.0º
ω scans	h = −13→13
Absorption correction: empirical (using intensity measurements) (SADABS; Siemens, 1996)	k = 0→17
T_{min} = 0.82, T_{max} = 0.96	l = 0→10
7186 measured reflections	

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)]$ = 0.068	H-atom parameters constrained
$wR(F^2)$ = 0.158	$w = 1/[\sigma^2(F_o^2) + (0.058P)^2 + 0.149P]$ where $P = (F_o^2 + 2F_c^2)/3$
S = 1.04	$(\Delta/\sigma)_{max}$ = <0.001
2569 reflections	$\Delta\rho_{max}$ = 0.34 e Å^{-3}
145 parameters	$\Delta\rho_{min}$ = −0.27 e Å^{-3}
Primary atom site location: structure-invariant direct methods	Extinction correction: none

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.

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 (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
Cl1	0.85523 (10)	0.06145 (7)	0.91589 (15)	0.0559 (4)
Cl2	0.76736 (11)	0.44854 (8)	0.93052 (16)	0.0612 (4)
N1	0.7010 (3)	0.2430 (2)	0.9836 (4)	0.0396 (8)
N2	0.5918 (3)	0.1569 (2)	0.7479 (4)	0.0454 (9)
H2A	0.6380	0.1764	0.6710	0.054*
N3	0.5158 (3)	0.1477 (2)	0.9936 (5)	0.0494 (9)
H3A	0.5140	0.1512	1.1019	0.059*
N4	1.2936 (4)	0.3263 (4)	0.7301 (7)	0.0938 (16)
C1	0.8165 (3)	0.2560 (3)	0.9159 (5)	0.0336 (9)
C2	0.9017 (4)	0.1809 (3)	0.8831 (5)	0.0377 (9)
C3	1.0221 (4)	0.1973 (3)	0.8332 (5)	0.0479 (11)
H3B	1.0747	0.1452	0.8114	0.057*
C4	1.0653 (4)	0.2910 (3)	0.8152 (5)	0.0482 (11)
C5	0.9858 (4)	0.3687 (3)	0.8463 (5)	0.0459 (11)
H5A	1.0138	0.4321	0.8342	0.055*
C6	0.8650 (4)	0.3504 (3)	0.8953 (5)	0.0384 (10)
C7	0.6107 (4)	0.1849 (3)	0.9101 (5)	0.0351 (9)
C8	0.4848 (4)	0.0899 (3)	0.7165 (6)	0.0581 (13)
H8A	0.5154	0.0241	0.7043	0.070*
H8B	0.4280	0.1077	0.6152	0.070*
C9	0.4165 (4)	0.1007 (3)	0.8755 (6)	0.0539 (12)
H9A	0.3396	0.1408	0.8541	0.065*
H9B	0.3925	0.0380	0.9181	0.065*
C10	1.1928 (5)	0.3108 (4)	0.7656 (6)	0.0624 (14)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.0500 (7)	0.0411 (6)	0.0770 (9)	0.0065 (5)	0.0093 (6)	−0.0011 (6)
Cl2	0.0529 (7)	0.0450 (6)	0.0887 (10)	0.0096 (5)	0.0213 (6)	0.0045 (6)
N1	0.0306 (17)	0.049 (2)	0.042 (2)	−0.0030 (15)	0.0132 (15)	−0.0091 (16)
N2	0.041 (2)	0.061 (2)	0.035 (2)	−0.0187 (16)	0.0089 (15)	0.0017 (17)

N3	0.047 (2)	0.058 (2)	0.047 (2)	−0.0089 (17)	0.0236 (17)	−0.0002 (18)
N4	0.050 (3)	0.102 (4)	0.138 (5)	0.010 (2)	0.046 (3)	0.028 (3)
C1	0.0288 (19)	0.045 (2)	0.027 (2)	0.0020 (16)	0.0053 (16)	−0.0001 (17)
C2	0.031 (2)	0.042 (2)	0.041 (2)	0.0002 (17)	0.0065 (18)	0.0009 (19)
C3	0.039 (2)	0.058 (3)	0.047 (3)	0.014 (2)	0.008 (2)	0.001 (2)
C4	0.034 (2)	0.064 (3)	0.047 (3)	0.003 (2)	0.010 (2)	0.003 (2)
C5	0.038 (2)	0.053 (3)	0.047 (3)	−0.002 (2)	0.006 (2)	0.010 (2)
C6	0.030 (2)	0.043 (2)	0.043 (3)	0.0053 (17)	0.0052 (18)	−0.0001 (19)
C7	0.032 (2)	0.037 (2)	0.038 (3)	0.0022 (16)	0.0099 (18)	0.0048 (19)
C8	0.050 (3)	0.067 (3)	0.057 (3)	−0.018 (2)	0.009 (2)	−0.005 (2)
C9	0.038 (2)	0.053 (3)	0.072 (3)	−0.008 (2)	0.012 (2)	0.007 (2)
C10	0.044 (3)	0.070 (3)	0.077 (4)	0.006 (2)	0.023 (3)	0.017 (3)

Geometric parameters (Å, °)

Cl1—C2	1.743 (4)	C1—C2	1.405 (5)
Cl2—C6	1.732 (4)	C1—C6	1.411 (5)
N1—C7	1.316 (5)	C2—C3	1.371 (5)
N1—C1	1.379 (4)	C3—C4	1.380 (6)
N2—C7	1.333 (5)	C4—C5	1.390 (5)
N2—C8	1.443 (5)	C4—C10	1.449 (6)
N3—C7	1.349 (5)	C5—C6	1.377 (5)
N3—C9	1.460 (5)	C8—C9	1.521 (6)
N4—C10	1.134 (5)		
C7—N1—C1	120.9 (3)	C5—C4—C10	118.8 (4)
C7—N2—C8	112.1 (3)	C6—C5—C4	119.1 (4)
C7—N3—C9	110.9 (3)	C5—C6—C1	123.3 (4)
N1—C1—C2	124.7 (3)	C5—C6—Cl2	118.1 (3)
N1—C1—C6	120.1 (3)	C1—C6—Cl2	118.6 (3)
C2—C1—C6	114.7 (3)	N1—C7—N2	128.3 (4)
C3—C2—C1	123.0 (4)	N1—C7—N3	122.6 (4)
C3—C2—Cl1	118.4 (3)	N2—C7—N3	109.1 (3)
C1—C2—Cl1	118.5 (3)	N2—C8—C9	102.6 (3)
C2—C3—C4	120.1 (4)	N3—C9—C8	102.2 (3)
C3—C4—C5	119.8 (4)	N4—C10—C4	178.6 (6)
C3—C4—C10	121.4 (4)		
C7—N1—C1—C2	−56.3 (5)	C2—C1—C6—C5	0.3 (5)
C7—N1—C1—C6	132.1 (4)	N1—C1—C6—Cl2	−8.2 (5)
N1—C1—C2—C3	−172.7 (4)	C2—C1—C6—Cl2	179.4 (3)
C6—C1—C2—C3	−0.7 (5)	C1—N1—C7—N2	−23.6 (6)
N1—C1—C2—Cl1	4.5 (5)	C1—N1—C7—N3	159.9 (4)
C6—C1—C2—Cl1	176.6 (3)	C8—N2—C7—N1	178.1 (4)
C1—C2—C3—C4	0.9 (6)	C8—N2—C7—N3	−5.1 (5)
Cl1—C2—C3—C4	−176.4 (3)	C9—N3—C7—N1	169.7 (3)
C2—C3—C4—C5	−0.6 (6)	C9—N3—C7—N2	−7.4 (5)
C2—C3—C4—C10	178.9 (4)	C7—N2—C8—C9	14.4 (5)
C3—C4—C5—C6	0.2 (6)	C7—N3—C9—C8	15.6 (5)
C10—C4—C5—C6	−179.3 (4)	N2—C8—C9—N3	−17.1 (4)
C4—C5—C6—C1	0.0 (6)	C3—C4—C10—N4	−91 (23)

C4—C5—C6—Cl2	−179.2 (3)	C5—C4—C10—N4	89 (23)
N1—C1—C6—C5	172.7 (4)		

2-(4-cyano-2,6-dichlorophenylimino)-1,3-diisobutyrylimidazolidine

Crystal data

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c = 10.9399 (12) Å	μ = 0.35 mm^{-1}
β = 108.079 (10)º	T = 293 (2) K
V = 1944.9 (4) Å^3	Parallepiped, colourless
Z = 4	0.54 × 0.40 × 0.20 mm

Data collection

Siemens P4 diffractometer	R_{int} = 0.037
Radiation source: fine-focus sealed tube	θ_{max} = 27.5º
Monochromator: graphite	θ_{min} = 2.1º
T = 293(2) K	h = −12→12
θ/2θ scans	k = 0→24
Absorption correction: empirical (using intensity measurements) (XSCANS; Siemens, 1994)	l = 0→14
T_{min} = 0.832, T_{max} = 0.933	3 standard reflections
5509 measured reflections	every 97 reflections
4424 independent reflections	intensity decay: <3%
1840 reflections with $I > 2\sigma(I)$	

Refinement

Refinement on F^2	Primary atom site location: structure-invariant direct methods
Least-squares matrix: full	Secondary atom site location: difference Fourier map
$R[F^2 > 2\sigma(F^2)]$ = 0.044	Hydrogen site location: difference Fourier map
$wR(F^2)$ = 0.077	H-atom parameters constrained
S = 0.96	$w = 1/[\sigma^2(F_o^2) + (0.0407P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
4424 reflections	$(\Delta/\sigma)_{max}$ = 0.001
240 parameters	Extinction correction: none

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.

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 (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
Cl1	0.59928 (9)	0.09802 (4)	0.37202 (8)	0.0667 (2)
Cl2	0.91374 (8)	0.15872 (4)	0.05877 (7)	0.0639 (2)
O1	0.8934 (2)	−0.13827 (11)	0.1240 (2)	0.0816 (7)
O2	0.9517 (2)	0.13486 (11)	0.45516 (17)	0.0633 (6)
N1	0.7882 (2)	0.06348 (11)	0.2169 (2)	0.0464 (6)
N2	1.0393 (2)	0.05941 (11)	0.3376 (2)	0.0470 (6)
N3	0.9336 (2)	−0.03480 (11)	0.2222 (2)	0.0519 (6)
N4	0.5715 (3)	0.40350 (14)	0.1851 (3)	0.0764 (8)
C1	0.7576 (3)	0.13465 (13)	0.2206 (2)	0.0422 (6)
C2	0.6623 (3)	0.15764 (14)	0.2833 (2)	0.0426 (6)
C3	0.6165 (3)	0.22651 (14)	0.2758 (2)	0.0471 (7)
H3A	0.5537	0.2407	0.3193	0.057*
C4	0.6650 (3)	0.27490 (13)	0.2025 (3)	0.0464 (7)
C5	0.7587 (3)	0.25370 (13)	0.1386 (3)	0.0474 (7)
H5A	0.7917	0.2859	0.0903	0.057*
C6	0.8029 (3)	0.18462 (14)	0.1467 (2)	0.0432 (7)
C7	0.9084 (3)	0.03411 (13)	0.2574 (2)	0.0438 (6)
C8	1.1572 (3)	0.01116 (16)	0.3393 (4)	0.0791 (10)
H8A	1.2175	0.0025	0.4266	0.095*
H8B	1.2153	0.0304	0.2901	0.095*
C9	1.0838 (3)	−0.05517 (14)	0.2785 (3)	0.0613 (8)
H9A	1.1230	−0.0719	0.2127	0.074*
H9B	1.0939	−0.0918	0.3424	0.074*
C10	0.8406 (3)	−0.08272 (15)	0.1388 (3)	0.0554 (8)
C11	0.6887 (3)	−0.06412 (15)	0.0715 (3)	0.0643 (9)
H11A	0.6550	−0.0322	0.1263	0.077*
C12	0.6808 (5)	−0.0265 (2)	−0.0527 (3)	0.1153 (15)
H12A	0.7437	0.0134	−0.0343	0.173*
H12B	0.5846	−0.0109	−0.0938	0.173*
H12C	0.7090	−0.0582	−0.1088	0.173*
C13	0.5949 (4)	−0.12970 (17)	0.0451 (3)	0.0914 (12)
H13A	0.6065	−0.1544	0.1242	0.137*
H13B	0.6225	−0.1598	−0.0136	0.137*

H13C	0.4967	−0.1162	0.0081	0.137*
C14	1.0546 (3)	0.11376 (15)	0.4274 (3)	0.0497 (7)
C15	1.2012 (3)	0.14533 (15)	0.4795 (3)	0.0561 (8)
H15A	1.2702	0.1073	0.5121	0.067*
C16	1.2412 (4)	0.18390 (18)	0.3736 (3)	0.0820 (11)
H16A	1.2354	0.1520	0.3041	0.123*
H16B	1.3369	0.2016	0.4071	0.123*
H16C	1.1764	0.2224	0.3428	0.123*
C17	1.2086 (4)	0.19638 (18)	0.5905 (3)	0.0800 (10)
H17B	1.1828	0.1719	0.6568	0.120*
H17C	1.1435	0.2347	0.5591	0.120*
H17A	1.3040	0.2144	0.6247	0.120*
C18	0.6138 (3)	0.34689 (16)	0.1941 (3)	0.0546 (7)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.0654 (5)	0.0702 (5)	0.0731 (5)	0.0094 (4)	0.0341 (4)	0.0187 (4)
Cl2	0.0686 (5)	0.0664 (5)	0.0694 (5)	0.0077 (4)	0.0397 (4)	−0.0006 (4)
O1	0.0808 (16)	0.0566 (13)	0.1144 (19)	0.0066 (12)	0.0404 (15)	−0.0179 (13)
O2	0.0465 (12)	0.0906 (15)	0.0503 (12)	0.0069 (11)	0.0115 (10)	−0.0141 (11)
N1	0.0404 (14)	0.0438 (12)	0.0559 (15)	0.0042 (11)	0.0162 (12)	−0.0032 (11)
N2	0.0382 (13)	0.0435 (12)	0.0585 (14)	0.0058 (11)	0.0140 (12)	0.0019 (11)
N3	0.0520 (15)	0.0409 (12)	0.0652 (15)	0.0113 (12)	0.0218 (13)	0.0030 (12)
N4	0.079 (2)	0.0551 (16)	0.096 (2)	0.0129 (15)	0.0279 (17)	−0.0018 (15)
C1	0.0375 (15)	0.0446 (15)	0.0392 (15)	0.0090 (12)	0.0042 (13)	−0.0045 (12)
C2	0.0350 (15)	0.0521 (15)	0.0398 (14)	0.0029 (13)	0.0101 (12)	0.0027 (13)
C3	0.0405 (16)	0.0515 (17)	0.0477 (16)	0.0091 (14)	0.0113 (14)	−0.0072 (14)
C4	0.0453 (17)	0.0396 (15)	0.0497 (16)	0.0013 (13)	0.0080 (14)	−0.0064 (13)
C5	0.0465 (17)	0.0475 (16)	0.0484 (16)	0.0040 (14)	0.0152 (14)	−0.0027 (13)
C6	0.0394 (16)	0.0506 (16)	0.0416 (15)	0.0037 (13)	0.0154 (13)	−0.0063 (13)
C7	0.0479 (18)	0.0403 (15)	0.0492 (16)	−0.0001 (14)	0.0239 (15)	0.0020 (13)
C8	0.053 (2)	0.070 (2)	0.109 (3)	0.0151 (18)	0.017 (2)	−0.013 (2)
C9	0.0522 (19)	0.0469 (16)	0.091 (2)	0.0126 (15)	0.0307 (18)	0.0038 (16)
C10	0.066 (2)	0.0450 (17)	0.065 (2)	0.0048 (16)	0.0351 (17)	−0.0039 (15)
C11	0.074 (2)	0.0518 (17)	0.0577 (19)	0.0093 (17)	0.0067 (18)	−0.0149 (15)
C12	0.155 (4)	0.103 (3)	0.073 (2)	0.000 (3)	0.015 (3)	0.019 (2)
C13	0.084 (3)	0.081 (2)	0.095 (3)	−0.009 (2)	0.006 (2)	−0.018 (2)
C14	0.0469 (18)	0.0574 (17)	0.0412 (15)	0.0142 (15)	0.0086 (14)	0.0079 (14)
C15	0.0437 (18)	0.0552 (17)	0.0613 (19)	0.0065 (14)	0.0046 (16)	−0.0001 (15)
C16	0.087 (3)	0.075 (2)	0.095 (3)	−0.018 (2)	0.044 (2)	−0.012 (2)
C17	0.066 (2)	0.098 (3)	0.062 (2)	−0.006 (2)	0.0005 (18)	−0.013 (2)
C18	0.0497 (18)	0.0581 (18)	0.0548 (18)	−0.0010 (16)	0.0145 (15)	−0.0030 (15)

Geometric parameters (Å, °)

Cl1—C2	1.727 (3)	C1—C6	1.406 (3)
Cl2—C6	1.735 (3)	C2—C3	1.378 (3)
O1—C10	1.210 (3)	C3—C4	1.398 (4)

O2—C14	1.213 (3)	C4—C5	1.379 (4)
N1—C7	1.258 (3)	C4—C18	1.451 (4)
N1—C1	1.389 (3)	C5—C6	1.377 (3)
N2—C7	1.400 (3)	C8—C9	1.501 (4)
N2—C14	1.400 (3)	C10—C11	1.490 (4)
N2—C8	1.475 (3)	C11—C12	1.517 (4)
N3—C10	1.407 (3)	C11—C13	1.524 (4)
N3—C7	1.408 (3)	C14—C15	1.502 (4)
N3—C9	1.467 (3)	C15—C16	1.522 (4)
N4—C18	1.146 (3)	C15—C17	1.538 (4)
C1—C2	1.393 (3)		
C7—N1—C1	127.6 (2)	C1—C6—Cl2	119.6 (2)
C7—N2—C14	124.8 (2)	N1—C7—N2	130.6 (2)
C7—N2—C8	111.2 (2)	N1—C7—N3	122.8 (3)
C14—N2—C8	122.4 (2)	N2—C7—N3	106.5 (2)
C10—N3—C7	130.1 (2)	N2—C8—C9	104.3 (2)
C10—N3—C9	118.0 (2)	N3—C9—C8	104.4 (2)
C7—N3—C9	111.8 (2)	O1—C10—N3	115.5 (3)
N1—C1—C2	120.3 (2)	O1—C10—C11	123.2 (3)
N1—C1—C6	122.0 (2)	N3—C10—C11	121.2 (2)
C2—C1—C6	117.0 (2)	C10—C11—C12	108.6 (3)
C3—C2—C1	121.7 (2)	C10—C11—C13	110.9 (2)
C3—C2—Cl1	119.3 (2)	C12—C11—C13	110.8 (3)
C1—C2—Cl1	119.0 (2)	O2—C14—N2	120.1 (3)
C2—C3—C4	119.7 (2)	O2—C14—C15	123.5 (3)
C5—C4—C3	120.0 (2)	N2—C14—C15	116.3 (2)
C5—C4—C18	121.1 (3)	C14—C15—C16	110.4 (2)
C3—C4—C18	118.9 (3)	C14—C15—C17	110.8 (3)
C6—C5—C4	119.5 (3)	C16—C15—C17	109.7 (2)
C5—C6—C1	122.0 (2)	N4—C18—C4	178.7 (3)
C5—C6—Cl2	118.4 (2)		
C7—N1—C1—C2	−121.9 (3)	C9—N3—C7—N1	177.6 (3)
C7—N1—C1—C6	67.9 (4)	C10—N3—C7—N2	−176.9 (2)
N1—C1—C2—C3	−172.1 (2)	C9—N3—C7—N2	−0.8 (3)
C6—C1—C2—C3	−1.4 (4)	C7—N2—C8—C9	−13.5 (3)
N1—C1—C2—Cl1	7.6 (3)	C14—N2—C8—C9	153.1 (3)
C6—C1—C2—Cl1	178.30 (18)	C10—N3—C9—C8	169.3 (3)
C1—C2—C3—C4	0.7 (4)	C7—N3—C9—C8	−7.4 (3)
Cl1—C2—C3—C4	−178.94 (19)	N2—C8—C9—N3	12.0 (3)
C2—C3—C4—C5	−0.3 (4)	C7—N3—C10—O1	178.0 (3)
C2—C3—C4—C18	179.0 (2)	C9—N3—C10—O1	2.1 (4)
C3—C4—C5—C6	0.5 (4)	C7—N3—C10—C11	−0.3 (4)
C18—C4—C5—C6	−178.7 (3)	C9—N3—C10—C11	−176.2 (3)
C4—C5—C6—C1	−1.2 (4)	O1—C10—C11—C12	−91.5 (4)
C4—C5—C6—Cl2	176.88 (19)	N3—C10—C11—C12	86.7 (3)
N1—C1—C6—C5	172.2 (3)	O1—C10—C11—C13	30.5 (4)
C2—C1—C6—C5	1.6 (4)	N3—C10—C11—C13	−151.3 (3)
N1—C1—C6—Cl2	−5.9 (3)	C7—N2—C14—O2	11.9 (4)

C2—C1—C6—Cl2	−176.45 (19)	C8—N2—C14—O2	−152.7 (3)
C1—N1—C7—N2	13.7 (5)	C7—N2—C14—C15	−165.0 (2)
C1—N1—C7—N3	−164.3 (2)	C8—N2—C14—C15	30.3 (4)
C14—N2—C7—N1	24.7 (4)	O2—C14—C15—C16	−110.4 (3)
C8—N2—C7—N1	−169.1 (3)	N2—C14—C15—C16	66.5 (3)
C14—N2—C7—N3	−157.1 (2)	O2—C14—C15—C17	11.4 (4)
C8—N2—C7—N3	9.1 (3)	N2—C14—C15—C17	−171.8 (2)
C10—N3—C7—N1	1.5 (4)		