CRYSTALLOGRAPHIC COMMUNICATIONS

OPEN ACCESS

ISSN 2056-9890

Crystal structure of 1,7,8,9-tetrachloro-4-(3,5-dichlorobenzyl)-10,10-dimethoxy-4-azatricyclo[5.2.1.$0^{2,6}$]dec-8-ene-3,5-dione

He Liu,[a] Jia-liang Zhong,[b]* Wen-xia Sun,[b] Yan-qing Gong[c] and Li-hong Liu[a]

[a]Beijing Chao-Yang Hospital, Capital Medical University, Beijing 100020, People's Republic of China, [b]Shanghai Institute of Pharmaceutical Industry, Shanghai 200040, People's Republic of China, and [c]State Key Laboratory of Bio-Organic & Natural Products Chmemistry, Shanghai Institute of Organic Chemistry, CAS., Shanghai 200032, People's Republic of China. *Correspondence e-mail: zhong.jialiang@sipi.com.cn

Received 13 November 2014; accepted 26 November 2014

Edited by V. V. Chernyshev, Moscow State University, Russia

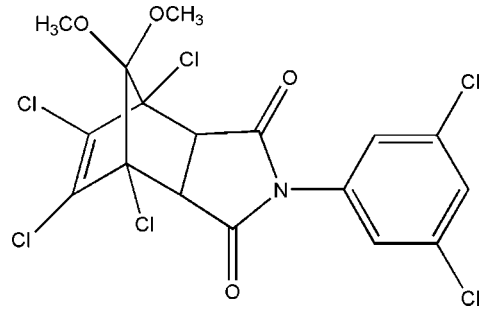
In the title compound, $C_{17}H_{11}Cl_6NO_4$, the configuration of the cycloalkene skeleton is *endo,cis*. The benzene ring is twisted by 58.94 (8)° from the attached pyrrolidine ring. Two carbonyl groups play a key role in the crystal packing. A short intermolecular C···O distance of 3.017 (3) Å reveals that one carbonyl group is involved in dipole–dipole interactions, which link two adjacent enantiomers into an inversion dimer. Another carbonyl group provides an acceptor for the weak intermolecular C—H···O hydrogen bonds which link these dimers into layers parallel to (011).

Keywords: crystal structure; tricyclo[5.2.1.$0^{2,6}$]dec-8-ene-3,5-dione; biological activity; cycloalkene skeleton; dipole–dipole interactions; hydrogen bonding.

CCDC reference: 1036270

1. Related literature

For related crystal structures, see: Shan *et al.* (2012); Kossakowski *et al.* (2009). For the biological activity of related compounds, see: Kossakowski *et al.* (2006, 2008); Struga *et al.* (2007).



2. Experimental

2.1. Crystal data

$C_{17}H_{11}Cl_6NO_4$
M_r = 505.97
Triclinic, $P\overline{1}$
a = 8.9905 (18) Å
b = 11.351 (2) Å
c = 11.482 (2) Å
α = 119.52 (3)°
β = 94.51 (3)°
γ = 90.23 (3)°
V = 1015.2 (4) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.87 mm^{-1}
T = 296 K
0.25 × 0.20 × 0.15 mm

2.2. Data collection

Bruker APEXII CCD diffractometer
10037 measured reflections
4611 independent reflections
3865 reflections with $I > 2\sigma(I)$
R_{int} = 0.036

2.3. Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.043
$wR(F^2)$ = 0.111
S = 1.05
4611 reflections
254 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.70 e Å^{-3}
$\Delta\rho_{min}$ = −0.64 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
C14—H14A···O2^{i}	0.93	2.57	3.265 (4)	132
C18—H18A···O2^{ii}	0.96	2.45	3.260 (4)	141

Symmetry codes: (i) $-x+2, -y+3, -z+1$; (ii) $-x+1, -y+2, -z$.

Data collection: *APEX2* (Bruker, 2009); cell refinement: *SAINT* (Bruker, 2009); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXTL*.

Acknowledgements

We gratefully acknowledge financial support from the National Natural Sciencce Foundation of China (No. 81072530).

Supporting information for this paper is available from the IUCr electronic archives (Reference: CV5477).

References

Bruker (2009). *APEX2* and *SAINT*. Bruker AXS Inc., Madison, Wisconsin, USA.

Kossakowski, J., Bielenica, A., Mirosław, B., Kozioł, A. E., Dybała, I. & Struga, M. (2008). *Molecules*, **13**, 1570–1583.

Kossakowski, J., Pakosinska-Parys, M., Struga, M., Dybala, I., Koziol, A. E., La Colla, P., Marongiu, L. E., Ibba, C., Collu, D. & Loddo, R. (2009). *Molecules*, **14**, 5189–5202.

Kossakowski, J., Wojciechowkai, A. & Kozio, A. E. (2006). *Acta Pol. Pharm. Drug Res.* **63**, 261–264.

Shan, W.-Z., Gu, S.-J. & Liu, H. (2012). *Chin. J. Struct. Chem.* **31**, 1524–1527.

Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.

Struga, M., Kossakowski, J., Kedzierska, E., Fidecka, S. & Stefanska, J. (2007). *Chem. Pharm. Bull.* **55**, 796–799.

supporting information

Acta Cryst. (2015). E**71**, o14–o15 [doi:10.1107/S2056989014025961]

Crystal structure of 1,7,8,9-tetrachloro-4-(3,5-dichlorobenzyl)-10,10-dimethoxy-4-azatricyclo[5.2.1.$0^{2,6}$]dec-8-ene-3,5-dione

He Liu, Jia-liang Zhong, Wen-xia Sun, Yan-qing Gong and Li-hong Liu

S1. Comment

The title compound, (I) (Fig. 1), was synthesized from *N*-(3′,5′-dichlorobenzyl)maleimide and 5,5-dimethoxy-1,2,3,4-tetrachlorocydopentadiene. The fused pyrrolidine ring systems are frequently encountered structural units in many synthetically challenging and biologically active alkaloids. The interest of constructing skeletons of this type was further enlightened by the recent disclosure that the rigid arylcyclo analogues having azatricyclo ring systems show anti-HIV-1, anti-cancer, antiviral, and antibacterial activities (Kossakowski *et al.*, 2006).

In (I), the configuration of the cycloalkene skeleton is *endo, cis*. The dihedral angle of pyrrolidine ring and benzene ring is 58.94 (8)°. Two carbonyl groups play a key role in the crystal packing (Fig. 2). One carbonyl group is involved in dipole-dipole interactions, with C3···O1(-*x*+2, -*y*+2, -*z*) distance of 3.017 (3) Å, which link two adjacent enantiomers into inversion dimers. The other carbonyl group provides an acceptor for two weak intermolecular C—H···O hydrogen bonds (Table 1). These intermoleclular interactions link these dimers into layers parallel to (011).

S2. Experimental

N-(3,5-Dichlorobenzyl)maleimide (2.44 g, 10 mmol) and 5,5-dimethoxy-1,2,3,4-tetrachlorocydopentadiene (2.63 g, 10 mmol) were dissolved in anhydrous toluene (100 ml). Then, the solution was refluxed for 8 h. After the solvent was removed under reduced pressure, the residue was dissolved in ether (150 ml), washed with water and brine, dried over anhydrous sodium sulfate, and concentrated to dryness. The product was purified by flash-chromatography (petroleum ether/ethyl acetate, 6:1) and the title compound was isolated as a white solid (4.16 g, 82%), with melting point between 104 and 106°C.

The crystals appropriate for X-ray data collection were obtained from acetone solution at room temperature after four days.

S3. Refinement

All H atoms were placed in geometically idealized positions and constrained to ride on their parent atoms with C—H distances of 0.93 Å (0.98 for alicylic CH) for aromatic ring CH, and U_{iso}(H) = 1.2–1.5 U_{eq}(C).

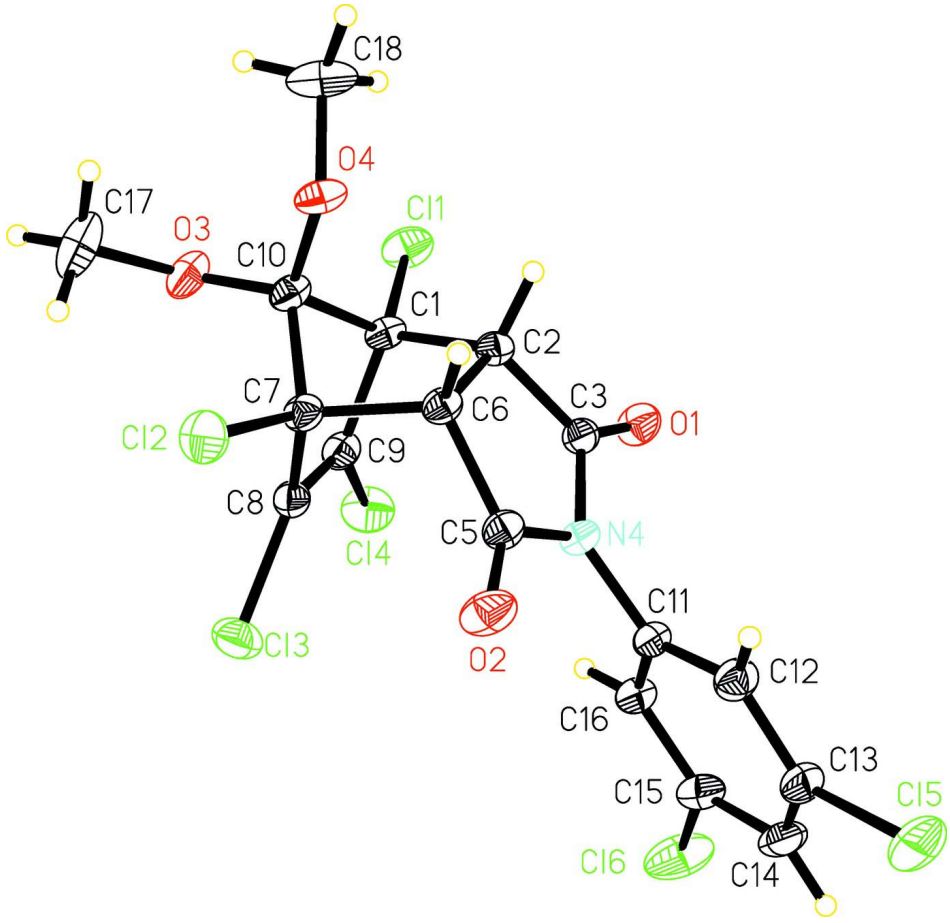


Figure 1

View of (I) showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level.

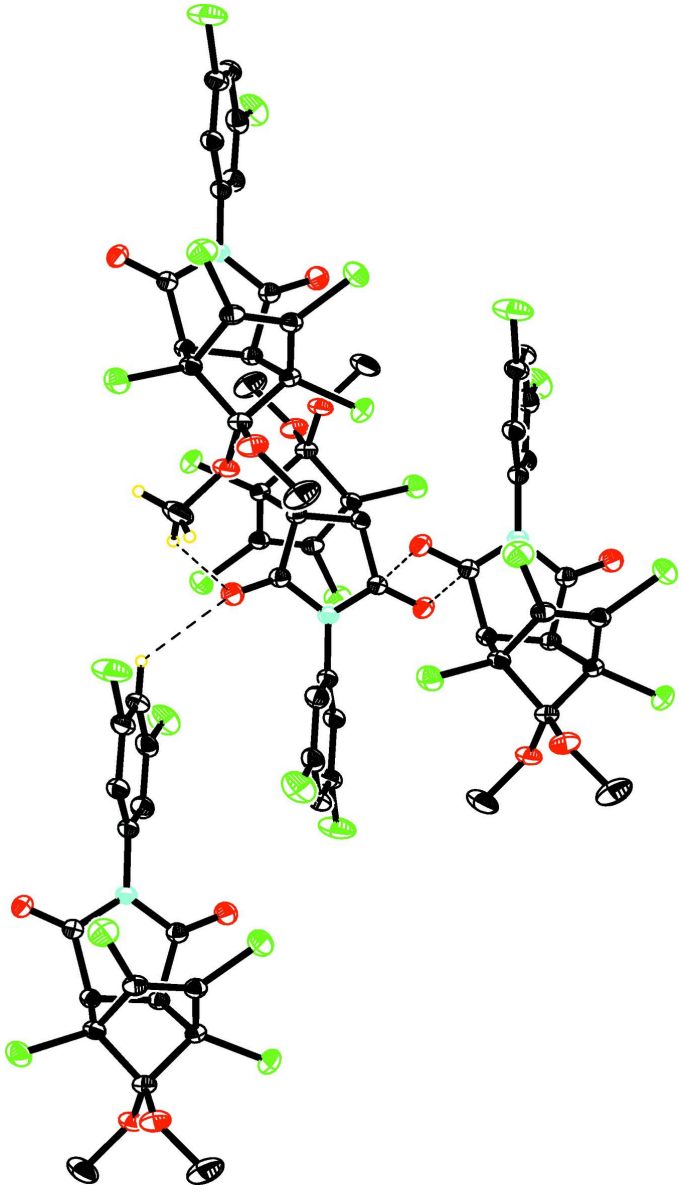


Figure 2

A portion of the crystal packing viewed approximately along [01-1]. The dipole-dipole and intermolecular C—H···O interactions are shown by dashed lines. H atoms not involved in C—H···O interactions are omitted for clarity.

1,7,8,9-Tetrachloro-4-(3,5-dichlorobenzyl)-10,10-dimethoxy-4-azatricyclo[5.2.1.$0^{2,6}$]dec-8-ene-3,5-dione

Crystal data

$C_{17}H_{11}Cl_6NO_4$
M_r = 505.97
Triclinic, $P\overline{1}$
Hall symbol: -P 1
a = 8.9905 (18) Å
b = 11.351 (2) Å
c = 11.482 (2) Å
α = 119.52 (3)°
β = 94.51 (3)°
γ = 90.23 (3)°
V = 1015.2 (4) Å^3
Z = 2
F(000) = 508
D_x = 1.655 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 4035 reflections
θ = 3.0–27.3°
μ = 0.87 mm^{-1}

T = 296 K
Column, colourless

0.25 × 0.20 × 0.15 mm

Data collection

Bruker APEXII CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
φ and ω scans
10037 measured reflections
4611 independent reflections

3865 reflections with $I > 2\sigma(I)$
R_{int} = 0.036
θ_{max} = 27.5°, θ_{min} = 3.0°
h = −10→11
k = −14→14
l = −14→14

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.043
$wR(F^2)$ = 0.111
S = 1.05
4611 reflections
254 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
$w = 1/[\sigma^2(F_o^2) + (0.0412P)^2 + 0.6429P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.70 e Å^{-3}
$\Delta\rho_{min}$ = −0.64 e Å^{-3}
Extinction correction: *SHELXL97* (Sheldrick, 2008), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.041 (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.

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 $F^2 > \sigma(F^2)$ 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}
Cl1	0.86211 (7)	0.64648 (6)	0.01529 (7)	0.04952 (18)
Cl2	0.42071 (6)	1.01968 (7)	0.22921 (7)	0.04994 (18)
Cl3	0.68066 (8)	1.06565 (7)	0.47167 (6)	0.05378 (19)
Cl4	0.95541 (7)	0.84784 (7)	0.33571 (7)	0.05465 (19)
Cl5	1.13965 (11)	1.59013 (7)	0.35138 (9)	0.0729 (3)
Cl6	1.36383 (11)	1.31140 (9)	0.58471 (9)	0.0808 (3)
O1	1.08016 (17)	0.94674 (16)	0.09979 (18)	0.0439 (4)
O2	0.72222 (19)	1.24926 (16)	0.29413 (19)	0.0498 (4)
O3	0.54476 (19)	0.70159 (18)	0.11801 (19)	0.0484 (4)
O4	0.53317 (18)	0.77943 (16)	−0.03513 (15)	0.0412 (4)
N4	0.92584 (19)	1.11941 (17)	0.21831 (17)	0.0314 (4)
C1	0.7734 (2)	0.8000 (2)	0.1025 (2)	0.0314 (4)
C2	0.8098 (2)	0.9081 (2)	0.0613 (2)	0.0298 (4)

H2A	0.8017	0.8681	−0.0367	0.036*
C3	0.9573 (2)	0.9867 (2)	0.1245 (2)	0.0314 (4)
C5	0.7731 (2)	1.1424 (2)	0.2225 (2)	0.0330 (4)
C6	0.6890 (2)	1.0109 (2)	0.1241 (2)	0.0299 (4)
H6A	0.6270	1.0203	0.0553	0.036*
C7	0.5955 (2)	0.9495 (2)	0.1918 (2)	0.0323 (4)
C8	0.6949 (2)	0.9539 (2)	0.3068 (2)	0.0335 (4)
C9	0.8003 (2)	0.8677 (2)	0.2542 (2)	0.0343 (5)
C10	0.5982 (2)	0.7951 (2)	0.0854 (2)	0.0340 (5)
C11	1.0373 (2)	1.2261 (2)	0.2981 (2)	0.0333 (4)
C12	1.0362 (3)	1.3406 (2)	0.2843 (2)	0.0409 (5)
H12A	0.9684	1.3467	0.2222	0.049*
C13	1.1394 (3)	1.4455 (2)	0.3660 (3)	0.0449 (6)
C14	1.2412 (3)	1.4386 (2)	0.4583 (3)	0.0472 (6)
H14A	1.3094	1.5104	0.5128	0.057*
C15	1.2391 (3)	1.3220 (2)	0.4674 (2)	0.0438 (5)
C16	1.1374 (2)	1.2143 (2)	0.3883 (2)	0.0371 (5)
H16A	1.1369	1.1365	0.3960	0.044*
C17	0.3854 (3)	0.6908 (4)	0.1208 (4)	0.0770 (10)
H17A	0.3617	0.6226	0.1436	0.115*
H17B	0.3366	0.6663	0.0339	0.115*
H17C	0.3516	0.7763	0.1866	0.115*
C18	0.5309 (4)	0.6462 (3)	−0.1513 (3)	0.0659 (9)
H18A	0.4837	0.6481	−0.2282	0.099*
H18B	0.4761	0.5833	−0.1356	0.099*
H18C	0.6314	0.6183	−0.1673	0.099*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.0530 (4)	0.0289 (3)	0.0557 (4)	0.0074 (2)	−0.0005 (3)	0.0132 (3)
Cl2	0.0286 (3)	0.0601 (4)	0.0557 (4)	0.0069 (2)	0.0054 (3)	0.0243 (3)
Cl3	0.0559 (4)	0.0620 (4)	0.0311 (3)	0.0005 (3)	0.0030 (3)	0.0138 (3)
Cl4	0.0498 (4)	0.0656 (4)	0.0509 (4)	0.0087 (3)	−0.0132 (3)	0.0330 (3)
Cl5	0.1059 (7)	0.0302 (3)	0.0815 (5)	−0.0056 (3)	0.0087 (5)	0.0269 (3)
Cl6	0.0848 (6)	0.0607 (5)	0.0737 (5)	−0.0117 (4)	−0.0449 (4)	0.0228 (4)
O1	0.0299 (8)	0.0397 (9)	0.0530 (10)	0.0028 (6)	0.0050 (7)	0.0157 (8)
O2	0.0422 (9)	0.0303 (8)	0.0605 (11)	0.0061 (7)	0.0023 (8)	0.0103 (8)
O3	0.0439 (9)	0.0454 (10)	0.0604 (11)	−0.0151 (7)	−0.0078 (8)	0.0314 (9)
O4	0.0420 (9)	0.0352 (8)	0.0356 (8)	−0.0046 (6)	−0.0142 (7)	0.0117 (7)
N4	0.0293 (9)	0.0271 (8)	0.0332 (9)	−0.0022 (6)	−0.0018 (7)	0.0121 (7)
C1	0.0332 (10)	0.0242 (9)	0.0327 (10)	−0.0013 (7)	−0.0050 (8)	0.0120 (8)
C2	0.0312 (10)	0.0291 (10)	0.0269 (9)	0.0003 (8)	−0.0012 (8)	0.0127 (8)
C3	0.0327 (11)	0.0302 (10)	0.0304 (10)	−0.0008 (8)	0.0009 (8)	0.0147 (8)
C5	0.0340 (11)	0.0291 (10)	0.0357 (10)	0.0007 (8)	−0.0008 (9)	0.0163 (9)
C6	0.0289 (10)	0.0287 (10)	0.0308 (10)	−0.0007 (7)	−0.0033 (8)	0.0147 (8)
C7	0.0259 (10)	0.0343 (11)	0.0328 (10)	−0.0009 (8)	−0.0019 (8)	0.0143 (9)
C8	0.0329 (11)	0.0382 (11)	0.0295 (10)	−0.0052 (8)	−0.0013 (8)	0.0175 (9)

C9	0.0345 (11)	0.0368 (11)	0.0354 (11)	−0.0046 (8)	−0.0063 (9)	0.0222 (9)
C10	0.0326 (11)	0.0315 (10)	0.0352 (10)	−0.0053 (8)	−0.0068 (9)	0.0159 (9)
C11	0.0337 (11)	0.0279 (10)	0.0329 (10)	−0.0038 (8)	0.0024 (8)	0.0109 (8)
C12	0.0456 (13)	0.0327 (11)	0.0433 (12)	0.0004 (9)	0.0026 (10)	0.0183 (10)
C13	0.0556 (15)	0.0258 (10)	0.0477 (13)	−0.0016 (9)	0.0114 (12)	0.0128 (10)
C14	0.0459 (14)	0.0309 (11)	0.0450 (13)	−0.0068 (9)	0.0004 (11)	0.0041 (10)
C15	0.0429 (13)	0.0364 (12)	0.0381 (11)	−0.0010 (9)	−0.0052 (10)	0.0089 (10)
C16	0.0394 (12)	0.0309 (11)	0.0360 (11)	−0.0017 (8)	−0.0003 (9)	0.0134 (9)
C17	0.0500 (17)	0.078 (2)	0.113 (3)	−0.0235 (16)	0.0000 (18)	0.056 (2)
C18	0.076 (2)	0.0465 (16)	0.0449 (15)	0.0048 (14)	−0.0211 (14)	0.0028 (13)

Geometric parameters (Å, º)

Cl1—C1	1.758 (2)	C5—C6	1.507 (3)
Cl2—C7	1.752 (2)	C6—C7	1.558 (3)
Cl3—C8	1.698 (2)	C6—H6A	0.9800
Cl4—C9	1.696 (2)	C7—C8	1.514 (3)
Cl5—C13	1.730 (2)	C7—C10	1.568 (3)
Cl6—C15	1.735 (3)	C8—C9	1.318 (3)
O1—C3	1.197 (3)	C11—C16	1.375 (3)
O2—C5	1.199 (3)	C11—C12	1.385 (3)
O3—C10	1.383 (3)	C12—C13	1.383 (3)
O3—C17	1.442 (3)	C12—H12A	0.9300
O4—C10	1.385 (3)	C13—C14	1.377 (4)
O4—C18	1.440 (3)	C14—C15	1.379 (4)
N4—C5	1.399 (3)	C14—H14A	0.9300
N4—C3	1.400 (3)	C15—C16	1.386 (3)
N4—C11	1.436 (3)	C16—H16A	0.9300
C1—C9	1.515 (3)	C17—H17A	0.9600
C1—C2	1.558 (3)	C17—H17B	0.9600
C1—C10	1.569 (3)	C17—H17C	0.9600
C2—C3	1.511 (3)	C18—H18A	0.9600
C2—C6	1.538 (3)	C18—H18B	0.9600
C2—H2A	0.9800	C18—H18C	0.9600
C10—O3—C17	117.0 (2)	C8—C9—Cl4	127.64 (18)
C10—O4—C18	117.17 (19)	C1—C9—Cl4	123.58 (17)
C5—N4—C3	113.59 (17)	O3—C10—O4	113.93 (18)
C5—N4—C11	121.92 (17)	O3—C10—C7	118.24 (19)
C3—N4—C11	124.39 (18)	O4—C10—C7	106.81 (17)
C9—C1—C2	107.61 (16)	O3—C10—C1	108.32 (17)
C9—C1—C10	100.14 (17)	O4—C10—C1	116.35 (18)
C2—C1—C10	100.16 (16)	C7—C10—C1	91.53 (15)
C9—C1—Cl1	114.51 (15)	C16—C11—C12	122.2 (2)
C2—C1—Cl1	114.82 (15)	C16—C11—N4	119.76 (19)
C10—C1—Cl1	117.67 (14)	C12—C11—N4	118.0 (2)
C3—C2—C6	105.59 (16)	C13—C12—C11	117.7 (2)
C3—C2—C1	114.53 (17)	C13—C12—H12A	121.2

C6—C2—C1	102.43 (16)	C11—C12—H12A	121.2
C3—C2—H2A	111.3	C14—C13—C12	122.2 (2)
C6—C2—H2A	111.3	C14—C13—Cl5	119.21 (19)
C1—C2—H2A	111.3	C12—C13—Cl5	118.6 (2)
O1—C3—N4	124.78 (19)	C13—C14—C15	118.0 (2)
O1—C3—C2	127.70 (19)	C13—C14—H14A	121.0
N4—C3—C2	107.52 (17)	C15—C14—H14A	121.0
O2—C5—N4	124.4 (2)	C14—C15—C16	122.0 (2)
O2—C5—C6	127.7 (2)	C14—C15—Cl6	119.06 (19)
N4—C5—C6	107.93 (17)	C16—C15—Cl6	118.9 (2)
C5—C6—C2	105.31 (16)	C11—C16—C15	117.9 (2)
C5—C6—C7	113.74 (17)	C11—C16—H16A	121.0
C2—C6—C7	103.90 (16)	C15—C16—H16A	121.0
C5—C6—H6A	111.2	O3—C17—H17A	109.5
C2—C6—H6A	111.2	O3—C17—H17B	109.5
C7—C6—H6A	111.2	H17A—C17—H17B	109.5
C8—C7—C6	107.50 (16)	O3—C17—H17C	109.5
C8—C7—C10	100.17 (17)	H17A—C17—H17C	109.5
C6—C7—C10	99.80 (16)	H17B—C17—H17C	109.5
C8—C7—Cl2	116.77 (15)	O4—C18—H18A	109.5
C6—C7—Cl2	112.98 (15)	O4—C18—H18B	109.5
C10—C7—Cl2	117.57 (15)	H18A—C18—H18B	109.5
C9—C8—C7	107.49 (18)	O4—C18—H18C	109.5
C9—C8—Cl3	127.66 (18)	H18A—C18—H18C	109.5
C7—C8—Cl3	124.45 (16)	H18B—C18—H18C	109.5
C8—C9—C1	108.25 (19)		

C9—C1—C2—C3	48.4 (2)	Cl1—C1—C9—C8	−160.38 (16)
C10—C1—C2—C3	152.57 (17)	C2—C1—C9—Cl4	−101.47 (19)
Cl1—C1—C2—C3	−80.38 (19)	C10—C1—C9—Cl4	154.36 (16)
C9—C1—C2—C6	−65.4 (2)	Cl1—C1—C9—Cl4	27.5 (2)
C10—C1—C2—C6	38.79 (18)	C17—O3—C10—O4	−54.4 (3)
Cl1—C1—C2—C6	165.84 (13)	C17—O3—C10—C7	72.3 (3)
C5—N4—C3—O1	176.3 (2)	C17—O3—C10—C1	174.4 (2)
C11—N4—C3—O1	−0.1 (3)	C18—O4—C10—O3	−51.2 (3)
C5—N4—C3—C2	−2.6 (2)	C18—O4—C10—C7	176.3 (2)
C11—N4—C3—C2	−178.97 (18)	C18—O4—C10—C1	75.9 (3)
C6—C2—C3—O1	−176.5 (2)	C8—C7—C10—O3	60.5 (2)
C1—C2—C3—O1	71.6 (3)	C6—C7—C10—O3	170.41 (18)
C6—C2—C3—N4	2.3 (2)	Cl2—C7—C10—O3	−67.1 (2)
C1—C2—C3—N4	−109.57 (19)	C8—C7—C10—O4	−169.50 (16)
C3—N4—C5—O2	−178.8 (2)	C6—C7—C10—O4	−59.55 (19)
C11—N4—C5—O2	−2.4 (3)	Cl2—C7—C10—O4	62.9 (2)
C3—N4—C5—C6	1.8 (2)	C8—C7—C10—C1	−51.33 (17)
C11—N4—C5—C6	178.21 (18)	C6—C7—C10—C1	58.61 (17)
O2—C5—C6—C2	−179.5 (2)	Cl2—C7—C10—C1	−178.91 (15)
N4—C5—C6—C2	−0.1 (2)	C9—C1—C10—O3	−70.1 (2)
O2—C5—C6—C7	−66.4 (3)	C2—C1—C10—O3	179.76 (16)

N4—C5—C6—C7	112.96 (19)	Cl1—C1—C10—O3	54.6 (2)
C3—C2—C6—C5	−1.3 (2)	C9—C1—C10—O4	160.04 (18)
C1—C2—C6—C5	118.88 (17)	C2—C1—C10—O4	49.9 (2)
C3—C2—C6—C7	−121.17 (17)	Cl1—C1—C10—O4	−75.2 (2)
C1—C2—C6—C7	−0.97 (19)	C9—C1—C10—C7	50.40 (18)
C5—C6—C7—C8	−47.1 (2)	C2—C1—C10—C7	−59.74 (17)
C2—C6—C7—C8	66.9 (2)	Cl1—C1—C10—C7	175.13 (15)
C5—C6—C7—C10	−151.10 (17)	C5—N4—C11—C16	121.4 (2)
C2—C6—C7—C10	−37.14 (18)	C3—N4—C11—C16	−62.5 (3)
C5—C6—C7—Cl2	83.22 (19)	C5—N4—C11—C12	−56.3 (3)
C2—C6—C7—Cl2	−162.83 (13)	C3—N4—C11—C12	119.8 (2)
C6—C7—C8—C9	−68.0 (2)	C16—C11—C12—C13	−0.6 (3)
C10—C7—C8—C9	35.8 (2)	N4—C11—C12—C13	177.0 (2)
Cl2—C7—C8—C9	163.88 (16)	C11—C12—C13—C14	0.2 (4)
C6—C7—C8—Cl3	105.23 (19)	C11—C12—C13—Cl5	−179.64 (17)
C10—C7—C8—Cl3	−151.01 (16)	C12—C13—C14—C15	0.4 (4)
Cl2—C7—C8—Cl3	−22.9 (2)	Cl5—C13—C14—C15	−179.70 (19)
C7—C8—C9—C1	−1.3 (2)	C13—C14—C15—C16	−0.8 (4)
Cl3—C8—C9—C1	−174.27 (16)	C13—C14—C15—Cl6	−178.90 (19)
C7—C8—C9—Cl4	170.36 (16)	C12—C11—C16—C15	0.3 (3)
Cl3—C8—C9—Cl4	−2.6 (3)	N4—C11—C16—C15	−177.3 (2)
C2—C1—C9—C8	70.7 (2)	C14—C15—C16—C11	0.4 (4)
C10—C1—C9—C8	−33.5 (2)	Cl6—C15—C16—C11	178.55 (17)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C14—H14*A*···O2^{i}	0.93	2.57	3.265 (4)	132
C18—H18*A*···O2^{ii}	0.96	2.45	3.260 (4)	141

Symmetry codes: (i) −*x*+2, −*y*+3, −*z*+1; (ii) −*x*+1, −*y*+2, −*z*.