Ethyl 2-[1-(4-chlorophenyl)-2,5-dioxo-1,2,3,5-tetrahydroimidazo[1′,2′:1,2]pyrimidino[5,4-*b*][1]benzofuran-3-yl]acetate

Yang-Gen Hu,[a]* Zheng-Rong Zhu[b] and Yu-Lu Chen[c]

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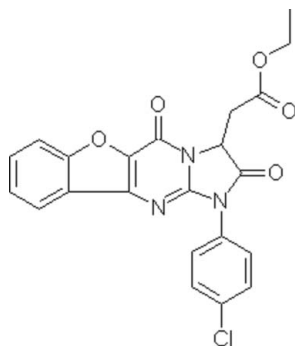Correspondence e-mail: huyangg111@yahoo.com.cn

The asymmetric unit of the title compound, $C_{22}H_{16}ClN_3O_5$, consists of two crystallographically independent molecules. The fused rings of the imidazo[1,2-*a*]benzo[4,5]furo[3,2-*d*]pyrimidine system are nearly coplanar and the chlorophenyl rings are twisted with respect to the two pyrimidinone ring systems by 71.00 (2) and 62.59 (2)°. The C atoms of the ethyl side chain are disordered and were refined using a split model. In the crystal structure, the molecules are connected *via* weak intra- and intermolecular C—H···O interactions are present. The ethyl group in one molecule is disordered over two positions, with site occupancy factors 0.55 and 0.45; in the other molecule only the methyl group is disordered over two positions, with site occupancy factors 0.6 and 0.4.

Related literature

For related literature on the synthesis and biological activity, see Moneam *et al.* (2004); Bodke & Sangapure (2003). For the crystal structures of other fused pyrimidinone derivatives, see: Hu *et al.* (2005, 2006, 2007).

Experimental

Crystal data

$C_{22}H_{16}ClN_3O_5$	γ = 110.085 (2)°
M_r = 437.83	V = 2068.0 (3) Å³
Triclinic, $P\bar{1}$	Z = 4
a = 11.9711 (11) Å	Mo $K\alpha$ radiation
b = 12.9079 (12) Å	μ = 0.23 mm⁻¹
c = 15.0957 (14) Å	T = 293 (2) K
α = 99.201 (2)°	0.30 × 0.20 × 0.20 mm
β = 102.486 (2)°	

Data collection

Bruker SMART 4K CCD area-detector diffractometer	19990 measured reflections
	8066 independent reflections
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2003)	5272 reflections with $I > 2\sigma(I)$
T_{min} = 0.936, T_{max} = 0.956	R_{int} = 0.101

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.056	4 restraints
$wR(F^2)$ = 0.161	H-atom parameters constrained
S = 0.99	$\Delta\rho_{max}$ = 0.31 e Å⁻³
8066 reflections	$\Delta\rho_{min}$ = −0.30 e Å⁻³
556 parameters	

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

D—H···A	D—H	H···A	D···A	D—H···A
C31—H31B···O9	0.97	2.48	3.069 (3)	119
C30—H30···O4	0.98	2.28	3.071 (3)	137
C4—H4···O1[i]	0.93	2.60	3.333 (3)	136
C2—H2···O10[ii]	0.93	2.49	3.399 (3)	165

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

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT-Plus* (Bruker, 2001); data reduction: *SAINT-Plus*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *PLATON* (Spek, 2003); software used to prepare material for publication: *SHELXTL* (Sheldrick, 2001).

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supporting information

Ethyl 2-[1-(4-chlorophenyl)-2,5-dioxo-1,2,3,5-tetrahydro-imidazo[1′,2′:1,2]pyrimidino[5,4-*b*][1]benzofuran-3-yl]acetate

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S1. Comment

The derivatives of benzofuropyrimidine play an important role in many natural products, antibiotics and drugs (Moneam *et al.*, 2004 and Bodke *et al.*, 2003). As a part of our ongoing investigations on the preparation of derivatives of heterocyclic compounds (Hu *et al.*, 2005, 2006, 2007)) we have synthesized and structurally characterized characterized the title compound. In the crystal structure of the title compound two crystallographically independent molecules are found in the asymmetric unit. All ring atoms of the imidazo[1,2-*a*]benzo[4,5]furo[3,2-*d*]pyrimidine system are essentially coplanar, with maximum deviations of -0.071 (1)Å and 0.092 (2))Å for O5 and C30. The benzene rings C1—C6 and C23—C28 are twisted with respect to the two pyrimidinone ring systems by 71.00 (2)° and 62.59 (2)°, respectively. In the crystal structure the molecules are connected *via* weak intramolecular and intermolecular C—H···O interactions.

S2. Experimental

Ethyl-3-((4-chlorophenylimino)methyleneamino)benzofuran-2-carboxylate (3 mmol) were dissolved in 5 ml of dichloro-methane. Afterwards diethyl-2-(aminomethyl)succinate(3 mmol) are added and the reaction mixture was stirred for 2 h. The solvent was removed under reduced pressure and 10 ml of anhydrous ethanol with several drops of EtONa dissolved in EtOH were added. The mixture was stirred for 6 h at room temperature. Afterwards the solution was concentrated under reduced pressure and the residue was recrystallized from ethanol to give 1.09 g of the title compound 1.09 g. Crystals suitable for single-crystal X-ray diffraction were obtained by slow evaporation of the solvent from a solution of the title compound in a mixture ethanol and dichloromethane (1:2 *v/v*)at room temperature.

S3. Refinement

All H-atoms were positioned with idealized geometry and refined isotropic (U_{iso}(H)= 1.5U_{eq}(C)for methyl H atoms and U_{iso}(H) =1.2U_{eq}(C) for all other H atoms) using a riding model with C—H = 0.93°, 0.97° and = 0.96 Å. The carbon atoms C11, C12 and *c*34 are disordered over two sites, and were refined using a split model with fixed s.o.f. of 0.55/0.45 for C11/C11′ and C12 C12′ and of 0.60/0.40 for C34/C34′, respectively.
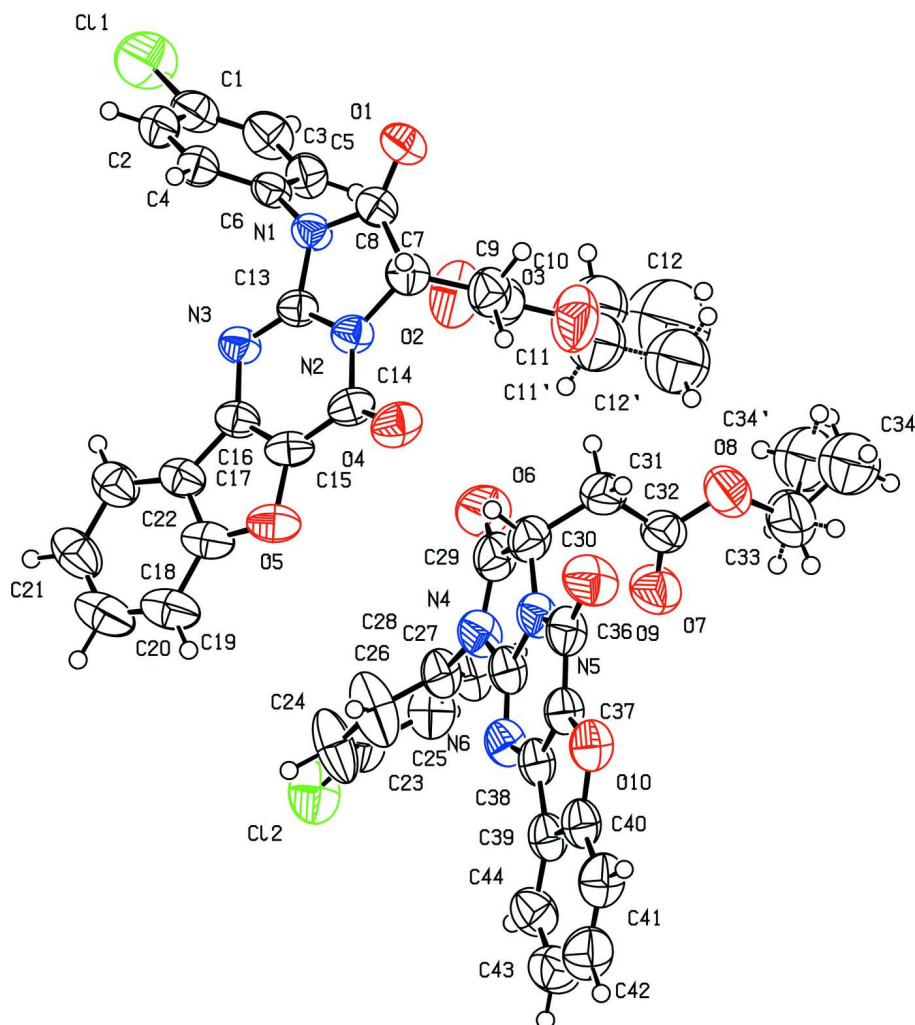
Figure 1

Molecular structure of the title compound with labelling and displacement ellipsoids drawn at the 50% probability level (The disordered C atoms with minor occupancy are only refined isotropic).

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Crystal data

$C_{22}H_{16}ClN_3O_5$	$Z = 4$
$M_r = 437.83$	$F(000) = 904$
Triclinic, $P\overline{1}$	$D_x = 1.406$ Mg m^{-3}
Hall symbol: -P 1	Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
$a = 11.9711$ (11) Å	Cell parameters from 5946 reflections
$b = 12.9079$ (12) Å	$\theta = 0.0$–$0.0°$
$c = 15.0957$ (14) Å	$\mu = 0.23$ mm^{-1}
$\alpha = 99.201$ (2)°	$T = 293$ K
$\beta = 102.486$ (2)°	Block, colorless
$\gamma = 110.085$ (2)°	$0.30 \times 0.20 \times 0.20$ mm
$V = 2068.0$ (3) Å^3	

Data collection

Bruker SMART 4K CCD area-detector diffractometer	19990 measured reflections
Radiation source: fine-focus sealed tube	8066 independent reflections
Graphite monochromator	5272 reflections with $I > 2\sigma(I)$
φ and ω scans	$R_{int} = 0.101$
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2003)	$\theta_{max} = 26.0°$, $\theta_{min} = 1.7°$
	$h = -14 \rightarrow 14$
	$k = -15 \rightarrow 15$
$T_{min} = 0.936$, $T_{max} = 0.956$	$l = -18 \rightarrow 18$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	
$R[F^2 > 2\sigma(F^2)] = 0.056$	Hydrogen site location: inferred from neighbouring sites
$wR(F^2) = 0.161$	
$S = 0.99$	H-atom parameters constrained
8066 reflections	$w = 1/[\sigma^2(F_o^2) + (0.0796P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
556 parameters	
4 restraints	$(\Delta/\sigma)_{max} = 0.001$
Primary atom site location: structure-invariant direct methods	$\Delta\rho_{max} = 0.31$ e Å^{-3}
	$\Delta\rho_{min} = -0.30$ e Å^{-3}

Special details

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

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 (Å^2)

	x	y	z	$U_{iso}*/U_{eq}$	Occ. (<1)
Cl1	1.31245 (7)	0.33661 (9)	1.33809 (5)	0.1148 (4)	
O1	1.04953 (14)	0.13649 (13)	0.87225 (11)	0.0619 (4)	
O2	0.9535 (2)	0.34943 (16)	0.87703 (14)	0.0966 (6)	
O3	0.9297 (3)	0.37601 (19)	0.73355 (16)	0.1144 (8)	
O4	0.57242 (14)	0.06236 (15)	0.74406 (11)	0.0699 (5)	
O5	0.45393 (13)	0.10771 (13)	0.89127 (12)	0.0662 (5)	
N1	0.93085 (15)	0.15775 (15)	0.96988 (11)	0.0491 (4)	
N2	0.75138 (15)	0.12384 (14)	0.86505 (11)	0.0501 (4)	
N3	0.76535 (15)	0.16729 (14)	1.02776 (12)	0.0499 (4)	
C1	1.1999 (2)	0.2852 (3)	1.23024 (16)	0.0674 (7)	
C2	1.1089 (2)	0.1778 (3)	1.20875 (17)	0.0701 (7)	
H2	1.1070	0.1341	1.2522	0.084*	
C3	1.2039 (2)	0.3521 (2)	1.16775 (18)	0.0702 (7)	
H3	1.2661	0.4249	1.1833	0.084*	
C4	1.0204 (2)	0.1351 (2)	1.12231 (16)	0.0593 (6)	
H4	0.9589	0.0621	1.1067	0.071*	

C5	1.1140 (2)	0.3096 (2)	1.08130 (16)	0.0597 (6)	
H5	1.1147	0.3539	1.0384	0.072*	
C6	1.02376 (18)	0.20104 (19)	1.05954 (14)	0.0478 (5)	
C7	0.9533 (2)	0.13632 (18)	0.88426 (15)	0.0508 (5)	
C8	0.8342 (2)	0.11147 (19)	0.80869 (14)	0.0544 (5)	
H8	0.8018	0.0320	0.7721	0.065*	
C9	0.8532 (2)	0.1886 (2)	0.74351 (16)	0.0681 (7)	
H9A	0.9018	0.1687	0.7051	0.082*	
H9B	0.7729	0.1755	0.7019	0.082*	
C10	0.9175 (3)	0.3124 (2)	0.7934 (2)	0.0736 (7)	
C11	1.0346 (8)	0.5111 (6)	0.7868 (6)	0.119 (2)*	0.55
H11A	1.0085	0.5404	0.8384	0.142*	0.55
H11B	1.1136	0.5068	0.8139	0.142*	0.55
C12	1.0553 (14)	0.5882 (12)	0.7364 (11)	0.245 (6)*	0.55
H12A	1.1141	0.6608	0.7765	0.367*	0.55
H12B	0.9786	0.5946	0.7087	0.367*	0.55
H12C	1.0881	0.5646	0.6878	0.367*	0.55
C11′	0.9661 (9)	0.4899 (7)	0.7637 (6)	0.110 (3)*	0.45
H11C	1.0471	0.5202	0.8100	0.132*	0.45
H11D	0.9082	0.5046	0.7949	0.132*	0.45
C12′	0.9739 (10)	0.5526 (9)	0.6900 (7)	0.133 (3)*	0.45
H12D	0.9923	0.6313	0.7167	0.199*	0.45
H12E	0.8960	0.5201	0.6414	0.199*	0.45
H12F	1.0386	0.5470	0.6639	0.199*	0.45
C13	0.81092 (18)	0.15222 (17)	0.95896 (14)	0.0466 (5)	
C14	0.6245 (2)	0.09868 (18)	0.82764 (16)	0.0542 (5)	
C15	0.57554 (19)	0.12095 (18)	0.90383 (17)	0.0554 (6)	
C16	0.64103 (18)	0.14966 (17)	0.99573 (15)	0.0501 (5)	
C17	0.5567 (2)	0.15575 (18)	1.04862 (17)	0.0561 (6)	
C18	0.4440 (2)	0.12948 (19)	0.9808 (2)	0.0629 (6)	
C19	0.3374 (2)	0.1262 (2)	1.0028 (3)	0.0780 (8)	
H19	0.2639	0.1101	0.9568	0.094*	
C20	0.3456 (3)	0.1477 (2)	1.0958 (3)	0.0878 (9)	
H20	0.2753	0.1462	1.1133	0.105*	
C21	0.4549 (3)	0.1721 (2)	1.1657 (2)	0.0806 (8)	
H21	0.4557	0.1847	1.2282	0.097*	
C22	0.5627 (2)	0.1776 (2)	1.14290 (19)	0.0680 (7)	
H22	0.6365	0.1955	1.1893	0.082*	
Cl2	0.33863 (8)	0.57533 (6)	1.01490 (5)	0.0883 (3)	
O6	0.60082 (18)	0.31763 (18)	0.75069 (12)	0.0853 (6)	
O7	0.51473 (18)	0.37899 (16)	0.54234 (14)	0.0860 (6)	
O8	0.6390 (2)	0.34225 (18)	0.46363 (15)	0.0954 (6)	
O9	0.30452 (17)	0.02466 (16)	0.42624 (12)	0.0829 (5)	
O10	0.05213 (15)	0.02539 (13)	0.36962 (10)	0.0639 (4)	
N4	0.39254 (18)	0.28569 (17)	0.69552 (12)	0.0623 (5)	
N5	0.33882 (17)	0.15994 (16)	0.55839 (11)	0.0565 (5)	
N6	0.18881 (18)	0.22927 (16)	0.59442 (12)	0.0577 (5)	
C23	0.3552 (2)	0.49233 (19)	0.92003 (15)	0.0602 (6)	

C24	0.2838 (3)	0.3782 (2)	0.8923 (2)	0.0984 (11)
H24	0.2273	0.3460	0.9238	0.118*
C25	0.4375 (2)	0.5398 (2)	0.87435 (18)	0.0711 (7)
H25	0.4863	0.6177	0.8940	0.085*
C26	0.2960 (3)	0.3113 (2)	0.8176 (2)	0.1000 (11)
H26	0.2471	0.2335	0.7982	0.120*
C27	0.4491 (2)	0.4730 (2)	0.79872 (17)	0.0660 (6)
H27	0.5040	0.5057	0.7662	0.079*
C28	0.3791 (2)	0.3579 (2)	0.77187 (14)	0.0583 (6)
C29	0.4996 (3)	0.2693 (2)	0.69287 (16)	0.0666 (6)
C30	0.4674 (2)	0.1778 (2)	0.60375 (15)	0.0630 (6)
H30	0.4684	0.1079	0.6207	0.076*
C31	0.5563 (2)	0.2119 (2)	0.54537 (17)	0.0671 (7)
H31A	0.6383	0.2207	0.5808	0.081*
H31B	0.5290	0.1509	0.4890	0.081*
C32	0.5654 (2)	0.3198 (2)	0.51802 (17)	0.0653 (6)
C33	0.6636 (4)	0.4482 (3)	0.4349 (3)	0.1105 (11)
H33A	0.5884	0.4439	0.3915	0.133*	0.60
H33B	0.6872	0.5108	0.4894	0.133*	0.60
H33C	0.6374	0.4331	0.3671	0.133*	0.40
H33D	0.6199	0.4907	0.4606	0.133*	0.40
C34	0.7589 (6)	0.4693 (6)	0.3917 (5)	0.121 (2)*	0.60
H34A	0.7734	0.5397	0.3737	0.181*	0.60
H34B	0.7350	0.4081	0.3371	0.181*	0.60
H34C	0.8337	0.4746	0.4349	0.181*	0.60
C34′	0.7928 (10)	0.5093 (10)	0.4709 (9)	0.131 (3)*	0.40
H34D	0.8153	0.5811	0.4547	0.197*	0.40
H34E	0.8344	0.4660	0.4447	0.197*	0.40
H34F	0.8171	0.5224	0.5380	0.197*	0.40
C35	0.2970 (2)	0.22450 (19)	0.61329 (14)	0.0548 (5)
C36	0.2644 (2)	0.0824 (2)	0.47202 (15)	0.0610 (6)
C37	0.1460 (2)	0.08853 (19)	0.45146 (14)	0.0554 (6)
C38	0.1131 (2)	0.15856 (18)	0.50819 (14)	0.0540 (5)
C39	−0.0124 (2)	0.14175 (19)	0.45958 (15)	0.0577 (6)
C40	−0.0446 (2)	0.0603 (2)	0.37588 (15)	0.0604 (6)
C41	−0.1572 (2)	0.0209 (2)	0.30865 (18)	0.0729 (7)
H41	−0.1760	−0.0336	0.2533	0.087*
C42	−0.2402 (3)	0.0656 (3)	0.3268 (2)	0.0835 (8)
H42	−0.3175	0.0410	0.2827	0.100*
C43	−0.2121 (3)	0.1473 (3)	0.4099 (2)	0.0848 (8)
H43	−0.2710	0.1757	0.4202	0.102*
C44	−0.0984 (2)	0.1863 (2)	0.47677 (18)	0.0721 (7)
H44	−0.0797	0.2411	0.5319	0.086*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0536 (9)	0.0706 (11)	0.0622 (10)	0.0248 (8)	0.0234 (8)	0.0090 (8)

O2	0.1407 (19)	0.0624 (12)	0.0714 (13)	0.0299 (12)	0.0240 (12)	0.0086 (10)
O3	0.192 (2)	0.0956 (16)	0.1072 (17)	0.0835 (17)	0.0784 (17)	0.0547 (14)
O4	0.0623 (10)	0.0782 (11)	0.0519 (10)	0.0264 (9)	−0.0050 (8)	0.0026 (8)
O5	0.0427 (9)	0.0667 (11)	0.0782 (12)	0.0215 (8)	0.0064 (8)	0.0044 (9)
N1	0.0384 (9)	0.0599 (11)	0.0444 (10)	0.0173 (8)	0.0102 (8)	0.0080 (8)
N2	0.0433 (10)	0.0540 (11)	0.0447 (10)	0.0175 (8)	0.0056 (8)	0.0043 (8)
N3	0.0424 (10)	0.0547 (11)	0.0487 (10)	0.0186 (8)	0.0116 (8)	0.0065 (8)
C1	0.0496 (14)	0.099 (2)	0.0466 (13)	0.0293 (15)	0.0102 (11)	0.0039 (13)
C2	0.0592 (15)	0.105 (2)	0.0593 (15)	0.0387 (16)	0.0222 (12)	0.0355 (15)
C3	0.0531 (14)	0.0731 (17)	0.0635 (16)	0.0109 (13)	0.0112 (12)	0.0005 (14)
C4	0.0471 (13)	0.0697 (15)	0.0615 (14)	0.0203 (11)	0.0161 (11)	0.0235 (12)
C5	0.0535 (13)	0.0648 (15)	0.0526 (13)	0.0149 (12)	0.0147 (11)	0.0125 (11)
C6	0.0362 (11)	0.0581 (13)	0.0473 (12)	0.0176 (10)	0.0132 (9)	0.0091 (10)
C7	0.0494 (13)	0.0468 (12)	0.0542 (13)	0.0175 (10)	0.0170 (10)	0.0082 (10)
C8	0.0577 (13)	0.0524 (13)	0.0455 (12)	0.0210 (11)	0.0109 (10)	−0.0003 (10)
C9	0.0752 (16)	0.0773 (17)	0.0474 (13)	0.0275 (14)	0.0158 (12)	0.0126 (12)
C10	0.094 (2)	0.0767 (18)	0.0711 (18)	0.0465 (16)	0.0363 (16)	0.0297 (16)
Cl1	0.0850 (5)	0.1759 (9)	0.0525 (4)	0.0461 (6)	−0.0078 (4)	−0.0043 (5)
Cl2	0.1290 (6)	0.0651 (4)	0.0637 (4)	0.0356 (4)	0.0326 (4)	−0.0032 (3)
C13	0.0412 (11)	0.0471 (12)	0.0471 (12)	0.0167 (9)	0.0087 (9)	0.0073 (9)
C14	0.0473 (13)	0.0504 (13)	0.0558 (14)	0.0199 (10)	0.0018 (11)	0.0054 (11)
C15	0.0397 (12)	0.0516 (13)	0.0667 (15)	0.0193 (10)	0.0049 (11)	0.0050 (11)
C16	0.0412 (12)	0.0434 (11)	0.0613 (14)	0.0152 (10)	0.0138 (10)	0.0065 (10)
C17	0.0496 (13)	0.0452 (12)	0.0741 (16)	0.0182 (10)	0.0226 (12)	0.0115 (11)
C18	0.0448 (13)	0.0472 (13)	0.0938 (19)	0.0186 (11)	0.0201 (13)	0.0095 (13)
C19	0.0520 (15)	0.0600 (16)	0.119 (3)	0.0214 (13)	0.0288 (16)	0.0111 (16)
C20	0.0663 (19)	0.0630 (17)	0.147 (3)	0.0259 (15)	0.058 (2)	0.0223 (18)
C21	0.091 (2)	0.0616 (16)	0.107 (2)	0.0325 (15)	0.0614 (19)	0.0195 (16)
C22	0.0696 (16)	0.0587 (15)	0.0837 (18)	0.0268 (13)	0.0360 (14)	0.0174 (13)
O6	0.0730 (12)	0.1010 (15)	0.0600 (11)	0.0287 (11)	0.0006 (10)	−0.0005 (10)
O7	0.1001 (14)	0.0830 (13)	0.0992 (14)	0.0507 (12)	0.0493 (12)	0.0276 (11)
O8	0.1111 (15)	0.1048 (15)	0.1158 (16)	0.0615 (13)	0.0718 (14)	0.0542 (13)
O9	0.0913 (13)	0.0927 (13)	0.0578 (10)	0.0419 (11)	0.0205 (9)	−0.0109 (9)
O10	0.0740 (11)	0.0617 (10)	0.0391 (8)	0.0171 (9)	0.0094 (7)	−0.0014 (7)
N4	0.0625 (12)	0.0733 (13)	0.0381 (10)	0.0208 (11)	0.0107 (9)	−0.0018 (9)
N5	0.0635 (12)	0.0642 (12)	0.0383 (9)	0.0241 (10)	0.0154 (9)	0.0052 (9)
N6	0.0643 (12)	0.0587 (12)	0.0380 (10)	0.0169 (10)	0.0137 (9)	−0.0016 (8)
C23	0.0785 (16)	0.0492 (13)	0.0424 (12)	0.0186 (12)	0.0139 (12)	0.0030 (10)
C24	0.138 (3)	0.0559 (16)	0.0803 (19)	−0.0014 (16)	0.071 (2)	−0.0027 (14)
C25	0.0845 (18)	0.0469 (13)	0.0637 (16)	0.0110 (13)	0.0172 (14)	0.0046 (12)
C26	0.136 (3)	0.0497 (15)	0.0815 (19)	−0.0077 (16)	0.062 (2)	−0.0109 (14)
C27	0.0678 (16)	0.0626 (15)	0.0563 (14)	0.0110 (13)	0.0219 (12)	0.0129 (12)
C28	0.0645 (14)	0.0604 (14)	0.0349 (11)	0.0135 (12)	0.0120 (10)	0.0003 (10)
C29	0.0718 (17)	0.0749 (17)	0.0451 (13)	0.0251 (14)	0.0111 (13)	0.0103 (12)
C30	0.0751 (16)	0.0696 (16)	0.0451 (12)	0.0333 (13)	0.0138 (12)	0.0102 (11)
C31	0.0692 (16)	0.0772 (17)	0.0634 (15)	0.0387 (14)	0.0210 (13)	0.0162 (13)
C32	0.0615 (15)	0.0785 (17)	0.0585 (14)	0.0319 (14)	0.0190 (12)	0.0128 (13)
C33	0.124 (3)	0.115 (3)	0.137 (3)	0.060 (2)	0.076 (3)	0.068 (2)

C35	0.0649 (15)	0.0541 (13)	0.0360 (11)	0.0154 (11)	0.0165 (10)	0.0022 (10)
C36	0.0791 (17)	0.0609 (14)	0.0376 (11)	0.0253 (13)	0.0172 (11)	0.0031 (10)
C37	0.0633 (14)	0.0549 (13)	0.0383 (11)	0.0169 (11)	0.0120 (10)	0.0041 (10)
C38	0.0638 (14)	0.0529 (13)	0.0378 (11)	0.0145 (11)	0.0176 (10)	0.0061 (10)
C39	0.0596 (14)	0.0564 (14)	0.0467 (12)	0.0116 (11)	0.0160 (11)	0.0098 (10)
C40	0.0661 (15)	0.0603 (14)	0.0457 (13)	0.0150 (12)	0.0168 (11)	0.0106 (11)
C41	0.0701 (17)	0.0689 (17)	0.0529 (14)	0.0080 (14)	0.0033 (13)	0.0072 (12)
C42	0.0649 (17)	0.091 (2)	0.0719 (18)	0.0135 (16)	0.0039 (14)	0.0195 (16)
C43	0.0637 (17)	0.098 (2)	0.090 (2)	0.0275 (16)	0.0249 (16)	0.0241 (18)
C44	0.0684 (17)	0.0766 (17)	0.0650 (16)	0.0225 (14)	0.0235 (14)	0.0090 (13)

Geometric parameters (Å, º)

Cl1—C1	1.732 (2)	Cl2—C23	1.737 (2)
O1—C7	1.204 (2)	O6—C29	1.212 (3)
O2—C10	1.199 (3)	O7—C32	1.188 (3)
O3—C10	1.313 (3)	O8—C32	1.322 (3)
O3—C11′	1.348 (8)	O8—C33	1.454 (4)
O3—C11	1.695 (8)	O9—C36	1.220 (3)
O4—C14	1.213 (3)	O10—C37	1.379 (2)
O5—C15	1.373 (2)	O10—C40	1.395 (3)
O5—C18	1.375 (3)	N4—C29	1.377 (3)
N1—C7	1.381 (3)	N4—C35	1.387 (3)
N1—C13	1.384 (2)	N4—C28	1.436 (3)
N1—C6	1.436 (2)	N5—C35	1.370 (3)
N2—C13	1.367 (3)	N5—C36	1.401 (3)
N2—C14	1.401 (3)	N5—C30	1.463 (3)
N2—C8	1.471 (3)	N6—C35	1.290 (3)
N3—C13	1.291 (3)	N6—C38	1.375 (3)
N3—C16	1.386 (3)	C23—C25	1.355 (3)
C1—C2	1.370 (4)	C23—C24	1.363 (3)
C1—C3	1.375 (4)	C24—C26	1.374 (4)
C2—C4	1.379 (3)	C24—H24	0.9300
C2—H2	0.9300	C25—C27	1.379 (3)
C3—C5	1.386 (3)	C25—H25	0.9300
C3—H3	0.9300	C26—C28	1.359 (4)
C4—C6	1.369 (3)	C26—H26	0.9300
C4—H4	0.9300	C27—C28	1.370 (3)
C5—C6	1.378 (3)	C27—H27	0.9300
C5—H5	0.9300	C29—C30	1.519 (3)
C7—C8	1.517 (3)	C30—C31	1.520 (3)
C8—C9	1.505 (3)	C30—H30	0.9800
C8—H8	0.9800	C31—C32	1.490 (4)
C9—C10	1.495 (4)	C31—H31A	0.9700
C9—H9A	0.9700	C31—H31B	0.9700
C9—H9B	0.9700	C33—C34	1.402 (7)
C11—C12	1.335 (13)	C33—C34′	1.405 (11)
C11—H11A	0.9700	C33—H33A	0.9700

C11—H11B	0.9700	C33—H33B	0.9700
C12—H12A	0.9600	C33—H33C	0.9700
C12—H12B	0.9600	C33—H33D	0.9700
C12—H12C	0.9600	C34—H34A	0.9600
C11′—C12′	1.478 (1)	C34—H34B	0.9600
C11′—H11C	0.9700	C34—H34C	0.9600
C11′—H11D	0.9700	C34′—H34D	0.9600
C12′—H12D	0.9600	C34′—H34E	0.9600
C12′—H12E	0.9600	C34′—H34F	0.9600
C12′—H12F	0.9600	C36—C37	1.417 (3)
C14—C15	1.432 (3)	C37—C38	1.359 (3)
C15—C16	1.357 (3)	C38—C39	1.446 (3)
C16—C17	1.430 (3)	C39—C44	1.391 (3)
C17—C22	1.387 (3)	C39—C40	1.392 (3)
C17—C18	1.400 (3)	C40—C41	1.371 (3)
C18—C19	1.375 (3)	C41—C42	1.361 (4)
C19—C20	1.361 (4)	C41—H41	0.9300
C19—H19	0.9300	C42—C43	1.396 (4)
C20—C21	1.392 (4)	C42—H42	0.9300
C20—H20	0.9300	C43—C44	1.377 (4)
C21—C22	1.387 (3)	C43—H43	0.9300
C21—H21	0.9300	C44—H44	0.9300
C22—H22	0.9300		
C10—O3—C11′	120.0 (4)	C35—N5—C30	111.52 (17)
C10—O3—C11	111.5 (4)	C36—N5—C30	124.84 (18)
C15—O5—C18	104.37 (17)	C35—N6—C38	111.59 (18)
C7—N1—C13	111.36 (17)	C25—C23—C24	120.5 (2)
C7—N1—C6	125.24 (16)	C25—C23—Cl2	120.77 (18)
C13—N1—C6	122.46 (16)	C24—C23—Cl2	118.7 (2)
C13—N2—C14	124.04 (18)	C23—C24—C26	119.5 (3)
C13—N2—C8	111.30 (16)	C23—C24—H24	120.3
C14—N2—C8	124.37 (17)	C26—C24—H24	120.3
C13—N3—C16	111.37 (17)	C23—C25—C27	120.1 (2)
C2—C1—C3	121.4 (2)	C23—C25—H25	119.9
C2—C1—Cl1	119.1 (2)	C27—C25—H25	119.9
C3—C1—Cl1	119.6 (2)	C28—C26—C24	120.5 (2)
C1—C2—C4	119.5 (2)	C28—C26—H26	119.8
C1—C2—H2	120.2	C24—C26—H26	119.8
C4—C2—H2	120.2	C28—C27—C25	119.4 (2)
C1—C3—C5	119.0 (2)	C28—C27—H27	120.3
C1—C3—H3	120.5	C25—C27—H27	120.3
C5—C3—H3	120.5	C26—C28—C27	120.0 (2)
C6—C4—C2	119.5 (2)	C26—C28—N4	119.4 (2)
C6—C4—H4	120.2	C27—C28—N4	120.6 (2)
C2—C4—H4	120.2	O6—C29—N4	126.9 (2)
C6—C5—C3	119.4 (2)	O6—C29—C30	125.9 (2)
C6—C5—H5	120.3	N4—C29—C30	107.2 (2)

C3—C5—H5	120.3	N5—C30—C29	101.64 (18)
C4—C6—C5	121.1 (2)	N5—C30—C31	114.95 (18)
C4—C6—N1	119.9 (2)	C29—C30—C31	113.2 (2)
C5—C6—N1	118.99 (19)	N5—C30—H30	108.9
O1—C7—N1	126.02 (19)	C29—C30—H30	108.9
O1—C7—C8	126.6 (2)	C31—C30—H30	108.9
N1—C7—C8	107.38 (17)	C32—C31—C30	113.7 (2)
N2—C8—C9	115.14 (18)	C32—C31—H31A	108.8
N2—C8—C7	101.66 (16)	C30—C31—H31A	108.8
C9—C8—C7	112.54 (19)	C32—C31—H31B	108.8
N2—C8—H8	109.1	C30—C31—H31B	108.8
C9—C8—H8	109.1	H31A—C31—H31B	107.7
C7—C8—H8	109.1	O7—C32—O8	124.1 (3)
C10—C9—C8	113.5 (2)	O7—C32—C31	124.9 (2)
C10—C9—H9A	108.9	O8—C32—C31	111.0 (2)
C8—C9—H9A	108.9	C34—C33—C34′	48.1 (5)
C10—C9—H9B	108.9	C34—C33—O8	111.2 (4)
C8—C9—H9B	108.9	C34′—C33—O8	105.2 (5)
H9A—C9—H9B	107.7	C34—C33—H33A	109.4
O2—C10—O3	124.1 (3)	C34′—C33—H33A	144.3
O2—C10—C9	124.8 (2)	O8—C33—H33A	109.4
O3—C10—C9	111.1 (3)	C34—C33—H33B	109.4
C12—C11—O3	119.2 (9)	C34′—C33—H33B	67.0
C12—C11—H11A	107.5	O8—C33—H33B	109.4
O3—C11—H11A	107.5	H33A—C33—H33B	108.0
C12—C11—H11B	107.5	C34—C33—H33C	64.0
O3—C11—H11B	107.5	C34′—C33—H33C	110.7
H11A—C11—H11B	107.0	O8—C33—H33C	110.7
C11—C12—H12A	109.5	H33A—C33—H33C	48.4
C11—C12—H12B	109.5	H33B—C33—H33C	138.7
H12A—C12—H12B	109.5	C34—C33—H33D	137.0
C11—C12—H12C	109.5	C34′—C33—H33D	110.7
H12A—C12—H12C	109.5	O8—C33—H33D	110.7
H12B—C12—H12C	109.5	H33A—C33—H33D	64.3
O3—C11′—C12′	115.4 (8)	H33B—C33—H33D	46.0
O3—C11′—H11C	108.4	H33C—C33—H33D	108.8
C12′—C11′—H11C	108.4	C33—C34—H34A	109.5
O3—C11′—H11D	108.4	H33C—C34—H34A	96.0
C12′—C11′—H11D	108.4	C33—C34—H34B	109.5
H11C—C11′—H11D	107.5	H33C—C34—H34B	78.0
C11′—C12′—H12D	109.5	H34A—C34—H34B	109.5
C11′—C12′—H12E	109.5	C33—C34—H34C	109.5
H12D—C12′—H12E	109.5	H33C—C34—H34C	148.2
C11′—C12′—H12F	109.5	H34A—C34—H34C	109.5
H12D—C12′—H12F	109.5	H34B—C34—H34C	109.5
H12E—C12′—H12F	109.5	C33—C34′—H34D	109.5
N3—C13—N2	127.63 (18)	C33—C34′—H34E	109.5
N3—C13—N1	124.03 (18)	H34D—C34′—H34E	109.5

N2—C13—N1	108.28 (17)	C33—C34′—H34F	109.5
O4—C14—N2	122.0 (2)	H34D—C34′—H34F	109.5
O4—C14—C15	129.9 (2)	H34E—C34′—H34F	109.5
N2—C14—C15	108.12 (18)	N6—C35—N5	127.52 (19)
C16—C15—O5	112.7 (2)	N6—C35—N4	124.65 (19)
C16—C15—C14	124.07 (19)	N5—C35—N4	107.8 (2)
O5—C15—C14	123.1 (2)	O9—C36—N5	121.5 (2)
C15—C16—N3	124.42 (19)	O9—C36—C37	129.9 (2)
C15—C16—C17	106.63 (19)	N5—C36—C37	108.56 (19)
N3—C16—C17	128.9 (2)	C38—C37—O10	112.6 (2)
C22—C17—C18	119.5 (2)	C38—C37—C36	124.3 (2)
C22—C17—C16	136.0 (2)	O10—C37—C36	123.08 (19)
C18—C17—C16	104.5 (2)	C37—C38—N6	124.4 (2)
C19—C18—O5	125.1 (2)	C37—C38—C39	106.41 (19)
C19—C18—C17	123.2 (3)	N6—C38—C39	129.2 (2)
O5—C18—C17	111.80 (19)	C44—C39—C40	118.8 (2)
C20—C19—C18	116.3 (3)	C44—C39—C38	136.1 (2)
C20—C19—H19	121.9	C40—C39—C38	105.1 (2)
C18—C19—H19	121.9	C41—C40—C39	123.6 (2)
C19—C20—C21	122.7 (3)	C41—C40—O10	125.0 (2)
C19—C20—H20	118.7	C39—C40—O10	111.4 (2)
C21—C20—H20	118.7	C42—C41—C40	116.7 (2)
C22—C21—C20	120.7 (3)	C42—C41—H41	121.6
C22—C21—H21	119.7	C40—C41—H41	121.6
C20—C21—H21	119.7	C41—C42—C43	121.7 (3)
C21—C22—C17	117.8 (3)	C41—C42—H42	119.2
C21—C22—H22	121.1	C43—C42—H42	119.2
C17—C22—H22	121.1	C44—C43—C42	121.0 (3)
C32—O8—C33	118.0 (2)	C44—C43—H43	119.5
C37—O10—C40	104.51 (16)	C42—C43—H43	119.5
C29—N4—C35	111.43 (18)	C43—C44—C39	118.2 (2)
C29—N4—C28	125.47 (19)	C43—C44—H44	120.9
C35—N4—C28	123.10 (19)	C39—C44—H44	120.9
C35—N5—C36	123.56 (19)		
C3—C1—C2—C4	0.9 (4)	C18—C17—C22—C21	0.1 (3)
Cl1—C1—C2—C4	−178.63 (18)	C16—C17—C22—C21	−178.2 (2)
C2—C1—C3—C5	−0.1 (4)	C25—C23—C24—C26	0.1 (5)
Cl1—C1—C3—C5	179.37 (17)	Cl2—C23—C24—C26	−179.8 (3)
C1—C2—C4—C6	−0.7 (3)	C24—C23—C25—C27	0.5 (4)
C1—C3—C5—C6	−0.7 (3)	Cl2—C23—C25—C27	−179.56 (19)
C2—C4—C6—C5	−0.1 (3)	C23—C24—C26—C28	0.5 (6)
C2—C4—C6—N1	−179.13 (19)	C23—C25—C27—C28	−1.7 (4)
C3—C5—C6—C4	0.9 (3)	C24—C26—C28—C27	−1.7 (5)
C3—C5—C6—N1	179.88 (19)	C24—C26—C28—N4	178.3 (3)
C7—N1—C6—C4	−115.8 (2)	C25—C27—C28—C26	2.3 (4)
C13—N1—C6—C4	76.3 (3)	C25—C27—C28—N4	−177.8 (2)
C7—N1—C6—C5	65.2 (3)	C29—N4—C28—C26	−118.3 (3)

C13—N1—C6—C5	−102.8 (2)	C35—N4—C28—C26	62.9 (3)
C13—N1—C7—O1	179.2 (2)	C29—N4—C28—C27	61.7 (3)
C6—N1—C7—O1	10.2 (3)	C35—N4—C28—C27	−117.1 (3)
C13—N1—C7—C8	−1.3 (2)	C35—N4—C29—O6	173.9 (3)
C6—N1—C7—C8	−170.42 (19)	C28—N4—C29—O6	−5.0 (4)
C13—N2—C8—C9	−121.6 (2)	C35—N4—C29—C30	−6.6 (3)
C14—N2—C8—C9	64.4 (3)	C28—N4—C29—C30	174.5 (2)
C13—N2—C8—C7	0.4 (2)	C35—N5—C30—C29	−3.2 (2)
C14—N2—C8—C7	−173.65 (18)	C36—N5—C30—C29	180.0 (2)
O1—C7—C8—N2	180.0 (2)	C35—N5—C30—C31	−125.9 (2)
N1—C7—C8—N2	0.6 (2)	C36—N5—C30—C31	57.3 (3)
O1—C7—C8—C9	−56.3 (3)	O6—C29—C30—N5	−174.7 (3)
N1—C7—C8—C9	124.3 (2)	N4—C29—C30—N5	5.8 (2)
N2—C8—C9—C10	64.4 (3)	O6—C29—C30—C31	−50.9 (3)
C7—C8—C9—C10	−51.5 (3)	N4—C29—C30—C31	129.6 (2)
C11′—O3—C10—O2	−11.2 (7)	N5—C30—C31—C32	59.3 (3)
C11—O3—C10—O2	15.3 (5)	C29—C30—C31—C32	−56.9 (3)
C11′—O3—C10—C9	168.8 (5)	C33—O8—C32—O7	2.6 (4)
C11—O3—C10—C9	−164.6 (4)	C33—O8—C32—C31	−176.6 (3)
C8—C9—C10—O2	1.2 (4)	C30—C31—C32—O7	3.9 (4)
C8—C9—C10—O3	−178.8 (2)	C30—C31—C32—O8	−176.9 (2)
C10—O3—C11—C12	−177.8 (9)	C32—O8—C33—C34	169.7 (4)
C11′—O3—C11—C12	−62.6 (14)	C32—O8—C33—C34′	119.3 (6)
C10—O3—C11′—C12′	−178.7 (6)	C38—N6—C35—N5	0.2 (3)
C11—O3—C11′—C12′	104.8 (16)	C38—N6—C35—N4	−179.5 (2)
C16—N3—C13—N2	0.7 (3)	C36—N5—C35—N6	−3.3 (4)
C16—N3—C13—N1	−176.20 (18)	C30—N5—C35—N6	179.8 (2)
C14—N2—C13—N3	−4.4 (3)	C36—N5—C35—N4	176.4 (2)
C8—N2—C13—N3	−178.5 (2)	C30—N5—C35—N4	−0.5 (3)
C14—N2—C13—N1	172.86 (17)	C29—N4—C35—N6	−175.7 (2)
C8—N2—C13—N1	−1.2 (2)	C28—N4—C35—N6	3.2 (4)
C7—N1—C13—N3	179.0 (2)	C29—N4—C35—N5	4.6 (3)
C6—N1—C13—N3	−11.6 (3)	C28—N4—C35—N5	−176.5 (2)
C7—N1—C13—N2	1.6 (2)	C35—N5—C36—O9	−178.3 (2)
C6—N1—C13—N2	171.02 (17)	C30—N5—C36—O9	−1.9 (4)
C13—N2—C14—O4	−172.1 (2)	C35—N5—C36—C37	3.1 (3)
C8—N2—C14—O4	1.2 (3)	C30—N5—C36—C37	179.5 (2)
C13—N2—C14—C15	6.6 (3)	C40—O10—C37—C38	0.9 (2)
C8—N2—C14—C15	179.83 (19)	C40—O10—C37—C36	−177.5 (2)
C18—O5—C15—C16	−0.1 (2)	O9—C36—C37—C38	−178.8 (3)
C18—O5—C15—C14	175.8 (2)	N5—C36—C37—C38	−0.4 (3)
O4—C14—C15—C16	172.2 (2)	O9—C36—C37—O10	−0.6 (4)
N2—C14—C15—C16	−6.3 (3)	N5—C36—C37—O10	177.80 (19)
O4—C14—C15—O5	−3.1 (4)	O10—C37—C38—N6	179.02 (19)
N2—C14—C15—O5	178.35 (18)	C36—C37—C38—N6	−2.6 (4)
O5—C15—C16—N3	179.52 (18)	O10—C37—C38—C39	−0.8 (3)
C14—C15—C16—N3	3.7 (3)	C36—C37—C38—C39	177.6 (2)
O5—C15—C16—C17	0.2 (2)	C35—N6—C38—C37	2.6 (3)

C14—C15—C16—C17	−175.6 (2)	C35—N6—C38—C39	−177.6 (2)
C13—N3—C16—C15	−0.4 (3)	C37—C38—C39—C44	−178.9 (3)
C13—N3—C16—C17	178.8 (2)	N6—C38—C39—C44	1.3 (4)
C15—C16—C17—C22	178.3 (3)	C37—C38—C39—C40	0.3 (2)
N3—C16—C17—C22	−1.0 (4)	N6—C38—C39—C40	−179.5 (2)
C15—C16—C17—C18	−0.2 (2)	C44—C39—C40—C41	0.0 (4)
N3—C16—C17—C18	−179.5 (2)	C38—C39—C40—C41	−179.4 (2)
C15—O5—C18—C19	−180.0 (2)	C44—C39—C40—O10	179.6 (2)
C15—O5—C18—C17	−0.1 (2)	C38—C39—C40—O10	0.2 (2)
C22—C17—C18—C19	1.3 (3)	C37—O10—C40—C41	178.9 (2)
C16—C17—C18—C19	−179.9 (2)	C37—O10—C40—C39	−0.7 (2)
C22—C17—C18—O5	−178.6 (2)	C39—C40—C41—C42	0.0 (4)
C16—C17—C18—O5	0.2 (2)	O10—C40—C41—C42	−179.6 (2)
O5—C18—C19—C20	178.5 (2)	C40—C41—C42—C43	−0.1 (4)
C17—C18—C19—C20	−1.4 (4)	C41—C42—C43—C44	0.3 (5)
C18—C19—C20—C21	0.1 (4)	C42—C43—C44—C39	−0.3 (4)
C19—C20—C21—C22	1.3 (4)	C40—C39—C44—C43	0.2 (4)
C20—C21—C22—C17	−1.4 (4)	C38—C39—C44—C43	179.3 (3)

Hydrogen-bond geometry (Å, º)

D—H⋯A	D—H	H⋯A	D⋯A	D—H⋯A
C31—H31B⋯O9	0.97	2.48	3.069 (3)	119
C30—H30⋯O4	0.98	2.28	3.071 (3)	137
C4—H4⋯O1[i]	0.93	2.60	3.333 (3)	136
C2—H2⋯O10[ii]	0.93	2.49	3.399 (3)	165

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