organic papers

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4',5',6',7'-Tetrachlorospiro[cyclohex-2-ene-1,2'-indan]-1',3'-dione

The title compound, $C_{14}H_8Cl_4O_2$, has been isolated following irradiation of a dichloromethane solution of *N*-acetyltetrachlorophthalimide and cyclohexene. The structure refinement is slightly compromised by the disorder over two positions of equal occupancy of a methylene group β to the spiro C atom.

Comment

The photochemistry of phthalimides has been extensively investigated over the past three decades and has yielded a rich diversity of molecular transformations, which have been reviewed by Kanaoka (1978), Coyle (1984) and Oelgemöller & Griesbeck (2002). Unsymmetrical 1,4-cycloaddition occurs across the benzo ring of *N*-benzoylphthalimide on irradiation in the presence of cyclohexene (McSweeney *et al.*, 2005), and similar 1,4-photocycloaddition occurs between *N*-ethyl-3,4,5,6-tetrachlorophthalimide and cyclohexene (Grimley *et al.*, 2005). The title compound, (I), and *rel*-(2*S*,7*R*)-8,9,11,12-tetrachlorotricyclo[6.2.2.0[2,7]]dodeca-9,11-diene-1,10-dicarboximide, (II), are products of the photochemical reaction between *N*-acetyl-3,4,5,6-tetrachlorophthalimide, (III), and cyclohexene in dichloromethane. Compound (II) is simply a further example of a compound resulting from 1,4-cycloaddition but (I), the structure of which is presented here, is more unusual.
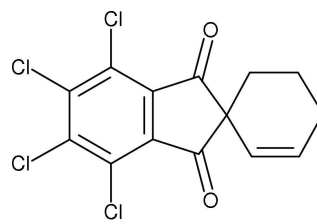
(I)
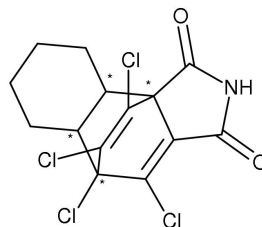
(II)

Formation of (I) most likely occurs *via* a sequence involving photoinitiated electron transfer/allylic proton transfer from cyclohexene to *N*-acetyltetrachlorophthalimide followed by radical coupling, to yield the corresponding cyclohexenyl carbinol. Subsequent thermal ring opening to give the corre-

sponding acyclic imido ketone, followed by enolization and ring closure with elimination of acetamide, would yield the observed dione (I).

The molecule of (I) is shown in Fig. 1. The majority of the bond lengths and angles are as expected for a molecule of this kind, but some data relating to particular features of the molecular geometry are given in Table 1.

In the course of refinement, the methylene group β to the spiro C atom was found to be disordered over two sites of equal occupancy, C13A and C13B. It is to this disorder, and possible limitations in modelling it, that the surprising variation in the length of the bonds to C13A and C13B and the unusual C13B—C14—C1 angle of 119.3 (6)° are attributed. Prolonged and ultimately wholly unsuccessful attempts were made to investigate an alternative model for the disorder in this part of the molecule, in which the entire cyclohexene ring was disordered over two orientations related by rotation through 180° about the vector between atoms C1 and C12. The relevant torsion angles in Table 1 show that atoms C13A and C13B each lie at the apex of a triangular flap which adopts two orientations, one tilted above and one below the plane of the other atoms in the cyclohexene ring. Other torsion angles in Table 1 reveal that the five-membered ring defined by C1–C3/C8/C9 is puckered, with pucker parameters (Cremer & Pople, 1975) of $Q_2 = 0.117$ (5) Å and $\varphi_2 = 348$ (2)° corresponding most closely to a twist conformation with twist about the C9—C1 bond.

The packing of the molecules of (I) in the unit cell (Fig. 2) brings about overlap of the rings defined by atoms C3–C8 related in pairs by crystallographic centres of symmetry (symmetry code: $1 - x, 1 - y, 1 - z$), in which the distance between the ring centroids, the perpendicular distance between the rings and the lateral displacement or slippage of the rings are 3.983, 3.624 and 1.652 Å, respectively. There are no other intermolecular contacts of any significance.

Experimental

Compound (I) was one of three products of the irradiation through Pyrex for 30 h of *N*-acetyl-3,4,5,6-tetrachlorophthalimide, (III), (3.08 g, 9.4 mmol) and cyclohexene (15.80 g, 192.7 mmol) in dichloromethane (300 ml). The solvents were removed under vacuum and the resulting mixture separated using a Chromatotron and a 4 mm plate. The eluent was a mixture of dichloromethane and light petroleum (b.p. 313–333 K) (2:98, increased stepwise to 60:40). This gave, in order of recovery from the plate, firstly compound (I), a yellow crystalline solid (15 mg, 5%), m.p. 401–404 K (from chloroform–light petroleum b.p. 363–373 K) [IR (ν_{max}, cm^{-1}): 2938 (aliphatic CH), 1749 (C=O), 1533 (C=C), 1284 and 1207; ^{1}H NMR (270 MHz, CDCl$_3$, δ, p.p.m.): 6.3 (1H, *t* of *d*, *J* = 9.9 Hz, *J* = 7.7 Hz), 5.3 (1H, *d*, *J* = 9.9 Hz) and 2.2–1.3 (6H, *m*, aliphatic H); ^{13}C NMR (CDCl$_3$, δ, p.p.m.): 197.1 (carbonyl C) 142.0, 135.6, 135.1, 131.1, 119.8 (unsaturated C), 56.7 (quaternary C), 28.7, 23.9 and 17.7 (aliphatic C)], secondly a mixture of a minor product and (III) as a brown oil (25 mg), thirdly unreacted (III) (2.80 g, 8.6 mmol), identified by comparison of its IR spectrum with that of an authentic sample, and finally the 1,4-adduct, *rel*-(2*S*,7*R*)-8,9,11,12-tetrachlorotricyclo-[6.2.2.0^{2,7}]dodeca-9,11-diene-1,10-dicarboximide, (II), (46 mg, 15%).
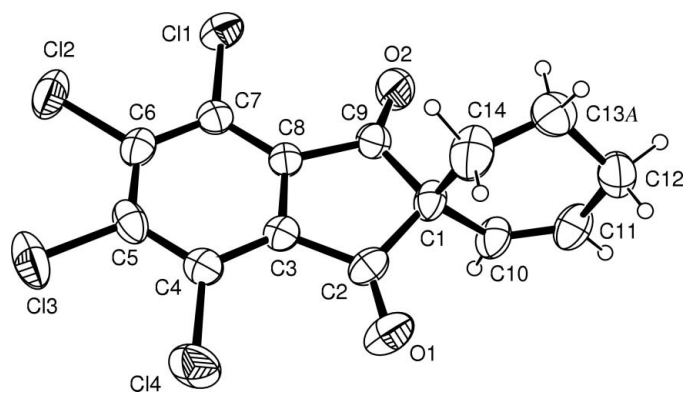

Figure 1
The molecular structure of (I). Displacement ellipsoids are drawn at the 30% probability level and H atoms are shown as small circles of arbitrary radii. The only component of the disorder noted elsewhere which is shown here is that which is compatible with C13A.
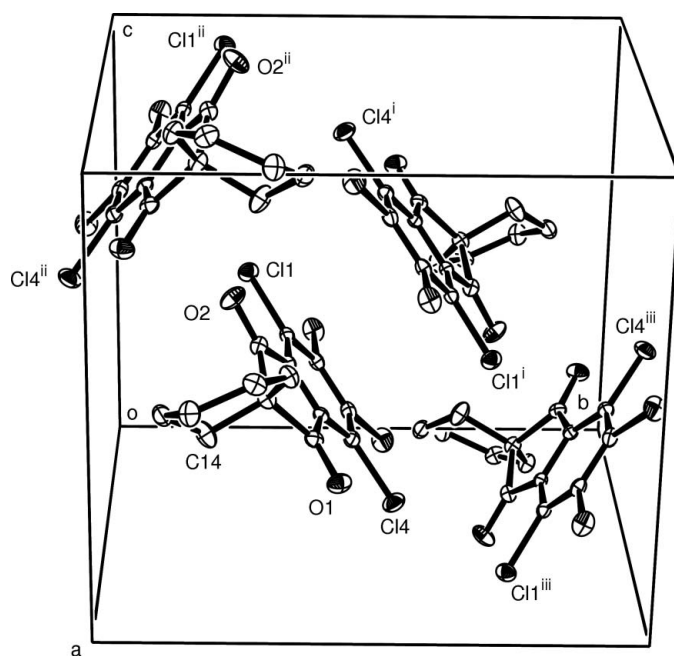
Figure 2
The unit-cell contents of (I). Displacement ellipsoids are drawn at the 10% probability level and H atoms have been omitted for clarity. Selected atoms are labelled. Atom C13A is the only representative of the disorder noted elsewhere which is shown here. [Symmetry codes (i) $1 - x, 1 - y, 1 - z$; (ii) $x, \frac{1}{2} - y, \frac{1}{2} + z$; (iii) $1 - x, \frac{1}{2} + y, \frac{1}{2} - z$.]

Crystal data

C$_{14}$H$_8$Cl$_4$O$_2$	$D_x = 1.662$ Mg m^{-3}
$M_r = 350.00$	Mo $K\alpha$ radiation
Monoclinic, $P2_1/c$	Cell parameters from 14
$a = 10.707$ (6) Å	reflections
$b = 12.103$ (8) Å	$\theta = 7.7$–10.6°
$c = 10.800$ (6) Å	$\mu = 0.84$ mm^{-1}
$\beta = 91.25$ (5)°	$T = 298$ (2) K
$V = 1399.2$ (14) Å^3	Block, yellow
$Z = 4$	$0.60 \times 0.60 \times 0.20$ mm

Data collection

Nicolet *P*3 four-circle diffractometer	1634 reflections with $I > 2\sigma(I)$
$\omega/2\theta$ scans	$\theta_{max} = 27.6°$
Absorption correction: ψ scan (North *et al.*, 1968)	$h = 0 \rightarrow 13$
	$k = 0 \rightarrow 15$
$T_{min} = 0.565$, $T_{max} = 0.845$	$l = -14 \rightarrow 14$
3222 measured reflections	2 standard reflections
3222 independent reflections	every 50 reflections
	intensity decay: none

Refinement

Refinement on F^2	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)] = 0.070$	$w = 1/[\sigma^2(F_o^2) + (0.102P)^2]$
$wR(F^2) = 0.198$	where $P = (F_o^2 + 2F_c^2)/3$
$S = 0.99$	$(\Delta/\sigma)_{max} < 0.001$
3222 reflections	$\Delta\rho_{max} = 0.35$ e Å^{-3}
190 parameters	$\Delta\rho_{min} = -0.31$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

C1—C10	1.521 (6)	C12—C13A	1.519 (8)
C1—C9	1.524 (6)	C12—C13B	1.495 (8)
C1—C14	1.525 (6)	C13A—C14	1.441 (8)
C1—C2	1.525 (6)	C13B—C14	1.449 (8)
C10—C1—C9	112.0 (4)	C14—C1—C2	108.4 (4)
C10—C1—C14	112.4 (4)	C14—C13A—C12	114.8 (7)
C9—C1—C14	108.0 (4)	C14—C13B—C12	115.8 (7)
C10—C1—C2	112.7 (4)	C13A—C14—C1	115.1 (6)
C9—C1—C2	102.8 (3)	C13B—C14—C1	119.3 (6)
C9—C1—C2—C3	−10.3 (4)	C1—C10—C11—C12	−2.4 (9)
C1—C2—C3—C8	5.1 (4)	C10—C11—C12—C13A	−16.1 (9)
C2—C3—C8—C9	2.8 (5)	C10—C11—C12—C13B	25.4 (9)
C3—C8—C9—C1	−9.6 (5)	C13A—C14—C1—C10	31.2 (8)
C8—C9—C1—C2	11.9 (4)	C13B—C14—C1—C10	−14.3 (9)
C14—C1—C10—C11	−4.3 (7)		

The atom of the cyclohexene ring β to the spiro atom, C1, is disordered over two sites of equal occupancy, C13*A* and C13*B*, which results in corresponding disorder and partial occupancy for the H atoms on these C atoms and also on the neighbouring atoms, C12 and C14. During refinement, bond distances involving C13*A* and C13*B* were restrained to 1.50 (1) Å. H atoms were placed in calculated positions, taking full account of the disorder noted above, with C—H set to 0.93 and 0.97 Å for alkene and methylene H atoms, respectively, and refined with a riding model, with $U_{iso}(H) = 1.2U_{eq}(C)$ in all cases.

Data collection: *Nicolet P3 Software* (Nicolet, 1980); cell refinement: *Nicolet P3 Software*; data reduction: *RDNIC* (Howie, 1980); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *SHELXL97* and *PLATON* (Spek, 2003).

References

Coyle, J. D. (1984). *Synthetic Organic Photochemistry*, edited by W. M. Horspool, pp. 259-284. New York: Plenum Press.
Cremer, D. & Pople, J. A. (1975). *J. Am. Chem. Soc.* **97**, 1354–1358.
Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
Grimley, F. M., O'Donnell, C., Pratt, A. C., Long, C. & Howie R. A. (2005). *Acta Cryst.* C**61**, o369–o372.
Howie, R. A. (1980). *RDNIC*. University of Aberdeen, Scotland.
Kanaoka, Y. (1978). *Acc. Chem. Res.* **11**, 407–413.
McSweeney, N., Pratt, A. C., Long, C. & Howie, R. A. (2005). *Acta Cryst.* E**61**, o547–o549.
Nicolet (1980). *Nicolet P3/R3 Data Collection Operator's Manual.* Nicolet XRD Corporation, Cupertino, California, USA.
North, A. C. T., Phillips, D. C. & Mathews, F. S. (1968). *Acta Cryst.* A**24**, 351–359.
Oelgemöller, M. & Griesbeck, A. G. (2002). *J. Photochem. Photobiol. C*, **3**, 109–127.
Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.
Spek, A. L. (2003). *J. Appl. Cryst.* **36**, 7–13.

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Crystal data

$C_{14}H_8Cl_4O_2$	$F(000) = 704$
$M_r = 350.00$	$D_x = 1.662$ Mg m^{-3}
Monoclinic, $P2_1/c$	Melting point = 401–404 K
Hall symbol: -P 2ybc	Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
$a = 10.707$ (6) Å	Cell parameters from 14 reflections
$b = 12.103$ (8) Å	$\theta = 7.7$–10.6°
$c = 10.800$ (6) Å	$\mu = 0.84$ mm^{-1}
$\beta = 91.25$ (5)°	$T = 298$ K
$V = 1399.2$ (14) Å^3	Block, yellow
$Z = 4$	$0.60 \times 0.60 \times 0.20$ mm

Data collection

Nicolet P3 four-circle diffractometer	3222 independent reflections
Radiation source: normal-focus sealed tube	1634 reflections with $I > 2\sigma(I)$
Graphite monochromator	$R_{int} = 0.000$
$\omega/2\theta$ scans	$\theta_{max} = 27.6°$, $\theta_{min} = 1.9°$
Absorption correction: ψ scan (North *et al.*, 1968)	$h = 0\rightarrow13$
	$k = 0\rightarrow15$
$T_{min} = 0.565$, $T_{max} = 0.845$	$l = -14\rightarrow14$
3222 measured reflections	2 standard reflections every 50 reflections
	intensity decay: none

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	
$R[F^2 > 2\sigma(F^2)] = 0.070$	Hydrogen site location: inferred from neighbouring sites
$wR(F^2) = 0.198$	
$S = 0.99$	H-atom parameters constrained
3222 reflections	$w = 1/[\sigma^2(F_o^2) + (0.102P)^2]$
190 parameters	where $P = (F_o^2 + 2F_c^2)/3$
4 restraints	$(\Delta/\sigma)_{max} < 0.001$
Primary atom site location: structure-invariant direct methods	$\Delta\rho_{max} = 0.35$ e Å^{-3}
	$\Delta\rho_{min} = -0.31$ e Å^{-3}

Special details

Experimental. Scan rates, dependent on prescan intensity (Ip), were in the range 58.6 (Ip>2500) to 5.33 (Ip<150) ° 2θ min^{-1}. Scan widths, dependent on 2θ, were in the range 2.4 to 2.7 ° 2θ. Stationary crystal, stationary counter background counts were taken on either side of the peak each for 25% of the total (peak plus background) count time.

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	0.42407 (12)	0.27220 (12)	0.56551 (12)	0.0807 (4)	
Cl2	0.26701 (12)	0.39876 (13)	0.36470 (16)	0.0964 (5)	
Cl3	0.39920 (16)	0.53456 (14)	0.16260 (14)	0.1076 (6)	
Cl4	0.68790 (16)	0.54769 (13)	0.15811 (12)	0.0962 (5)	
O1	0.9009 (3)	0.4407 (3)	0.3086 (3)	0.0906 (11)	
O2	0.7026 (3)	0.2471 (3)	0.6235 (4)	0.0966 (12)	
C1	0.8496 (4)	0.3105 (4)	0.4690 (4)	0.0613 (11)	
C2	0.8224 (4)	0.3959 (4)	0.3681 (4)	0.0603 (11)	
C3	0.6848 (4)	0.4098 (3)	0.3562 (4)	0.0512 (10)	
C4	0.6158 (5)	0.4693 (3)	0.2691 (4)	0.0601 (11)	
C5	0.4859 (5)	0.4653 (4)	0.2719 (4)	0.0669 (13)	
C6	0.4279 (4)	0.4035 (4)	0.3622 (4)	0.0603 (11)	
C7	0.4971 (4)	0.3466 (3)	0.4518 (4)	0.0547 (10)	
C8	0.6262 (4)	0.3494 (3)	0.4455 (3)	0.0493 (9)	
C9	0.7226 (4)	0.2951 (4)	0.5276 (4)	0.0618 (11)	
C10	0.9483 (4)	0.3491 (4)	0.5627 (4)	0.0704 (13)	
H10	0.9378	0.4171	0.6012	0.084*	
C11	1.0475 (5)	0.2905 (5)	0.5912 (5)	0.0806 (15)	
H11	1.1051	0.3211	0.6471	0.097*	
C12	1.0753 (5)	0.1795 (4)	0.5418 (5)	0.0883 (16)	
H12A	1.1062	0.1325	0.6085	0.106*	0.50
H12B	1.1401	0.1852	0.4807	0.106*	0.50
H12C	1.0471	0.1237	0.5993	0.106*	0.50
H12D	1.1649	0.1713	0.5337	0.106*	0.50
C13A	0.9592 (9)	0.1280 (8)	0.4827 (10)	0.082 (3)	0.50
H13A	0.9845	0.0660	0.4322	0.098*	0.50
H13B	0.9074	0.0992	0.5479	0.098*	0.50
C13B	1.0123 (9)	0.1623 (11)	0.4184 (8)	0.080 (3)	0.50
H13C	1.0615	0.1987	0.3560	0.096*	0.50
H13D	1.0126	0.0838	0.4001	0.096*	0.50
C14	0.8849 (5)	0.2020 (5)	0.4070 (6)	0.101 (2)	
H14A	0.8088	0.1640	0.3815	0.121*	0.50
H14B	0.9307	0.2187	0.3327	0.121*	0.50
H14C	0.8310	0.1449	0.4391	0.121*	0.50
H14D	0.8649	0.2091	0.3193	0.121*	0.50

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.0699 (8)	0.0889 (9)	0.0838 (8)	−0.0202 (7)	0.0105 (6)	0.0018 (7)
Cl2	0.0533 (7)	0.1046 (11)	0.1306 (13)	0.0062 (7)	−0.0184 (7)	−0.0185 (9)
Cl3	0.1207 (13)	0.1032 (12)	0.0969 (11)	0.0347 (9)	−0.0414 (9)	0.0129 (8)
Cl4	0.1232 (13)	0.1006 (11)	0.0647 (8)	−0.0101 (9)	0.0021 (8)	0.0265 (7)
O1	0.072 (2)	0.118 (3)	0.082 (2)	−0.032 (2)	0.0081 (19)	−0.001 (2)
O2	0.080 (2)	0.119 (3)	0.089 (3)	−0.012 (2)	−0.018 (2)	0.050 (2)
C1	0.049 (2)	0.068 (3)	0.066 (3)	0.001 (2)	−0.015 (2)	−0.012 (2)
C2	0.055 (3)	0.070 (3)	0.056 (3)	−0.014 (2)	0.000 (2)	−0.016 (2)
C3	0.059 (2)	0.051 (2)	0.043 (2)	−0.0055 (19)	−0.0059 (18)	−0.0110 (18)
C4	0.074 (3)	0.055 (3)	0.051 (2)	−0.006 (2)	−0.007 (2)	−0.005 (2)
C5	0.087 (4)	0.053 (3)	0.060 (3)	0.015 (2)	−0.024 (2)	−0.009 (2)
C6	0.054 (2)	0.055 (3)	0.071 (3)	0.001 (2)	−0.006 (2)	−0.020 (2)
C7	0.062 (3)	0.046 (2)	0.055 (2)	−0.0013 (19)	−0.001 (2)	−0.0098 (18)
C8	0.053 (2)	0.045 (2)	0.049 (2)	0.0008 (18)	−0.0028 (18)	−0.0078 (18)
C9	0.062 (3)	0.059 (3)	0.063 (3)	−0.004 (2)	−0.011 (2)	0.004 (2)
C10	0.057 (3)	0.071 (3)	0.083 (3)	−0.004 (2)	−0.018 (2)	−0.022 (3)
C11	0.060 (3)	0.104 (4)	0.077 (3)	−0.008 (3)	−0.014 (2)	−0.018 (3)
C12	0.074 (3)	0.082 (4)	0.108 (4)	0.009 (3)	−0.017 (3)	0.002 (3)
C13A	0.089 (9)	0.069 (7)	0.089 (8)	0.005 (6)	0.009 (7)	−0.016 (6)
C13B	0.086 (8)	0.090 (8)	0.063 (6)	0.019 (7)	−0.001 (6)	−0.007 (6)
C14	0.086 (4)	0.092 (4)	0.123 (5)	0.010 (3)	−0.025 (4)	−0.048 (4)

Geometric parameters (Å, º)

Cl1—C7	1.724 (4)	C10—H10	0.9300
Cl2—C6	1.724 (5)	C11—C12	1.479 (7)
Cl3—C5	1.706 (5)	C11—H11	0.9300
Cl4—C4	1.725 (5)	C12—C13A	1.519 (8)
O1—C2	1.199 (5)	C12—C13B	1.495 (8)
O2—C9	1.211 (5)	C12—H12A	0.9700
C1—C10	1.521 (6)	C12—H12B	0.9700
C1—C9	1.524 (6)	C12—H12C	0.9700
C1—C14	1.525 (6)	C12—H12D	0.9700
C1—C2	1.525 (6)	C13A—C14	1.441 (8)
C2—C3	1.485 (6)	C13A—H13A	0.9700
C3—C8	1.373 (5)	C13A—H13B	0.9700
C3—C4	1.386 (6)	C13B—C14	1.449 (8)
C4—C5	1.392 (7)	C13B—H13C	0.9700
C5—C6	1.386 (7)	C13B—H13D	0.9700
C6—C7	1.389 (6)	C14—H14A	0.9700
C7—C8	1.386 (6)	C14—H14B	0.9700
C8—C9	1.498 (6)	C14—H14C	0.9700
C10—C11	1.308 (7)	C14—H14D	0.9700
C10—C1—C9	112.0 (4)	C11—C12—C13A	110.8 (6)

C10—C1—C14	112.4 (4)	C11—C12—C13B	111.0 (6)
C9—C1—C14	108.0 (4)	C11—C12—H12A	109.5
C10—C1—C2	112.7 (4)	C13A—C12—H12A	109.5
C9—C1—C2	102.8 (3)	C11—C12—H12B	109.5
C14—C1—C2	108.4 (4)	C13A—C12—H12B	109.5
O1—C2—C3	127.4 (5)	H12A—C12—H12B	108.1
O1—C2—C1	124.4 (4)	C11—C12—H12C	109.4
C3—C2—C1	108.2 (4)	C13B—C12—H12C	109.4
C8—C3—C4	120.5 (4)	C11—C12—H12D	109.4
C8—C3—C2	110.3 (4)	C13B—C12—H12D	109.4
C4—C3—C2	129.1 (4)	H12C—C12—H12D	108.0
C3—C4—C5	119.1 (4)	C14—C13A—C12	114.8 (7)
C3—C4—Cl4	121.2 (4)	C14—C13A—H13A	108.6
C5—C4—Cl4	119.8 (4)	C12—C13A—H13A	108.6
C6—C5—C4	119.8 (4)	C14—C13A—H13B	108.6
C6—C5—Cl3	120.4 (4)	C12—C13A—H13B	108.6
C4—C5—Cl3	119.8 (4)	H13A—C13A—H13B	107.5
C5—C6—C7	121.2 (4)	C14—C13B—C12	115.8 (7)
C5—C6—Cl2	119.5 (4)	C14—C13B—H13C	108.3
C7—C6—Cl2	119.3 (4)	C12—C13B—H13C	108.3
C8—C7—C6	118.1 (4)	C14—C13B—H13D	108.3
C8—C7—Cl1	121.1 (3)	C12—C13B—H13D	108.3
C6—C7—Cl1	120.8 (4)	H13C—C13B—H13D	107.4
C3—C8—C7	121.3 (4)	C13A—C14—C1	115.1 (6)
C3—C8—C9	109.2 (4)	C13B—C14—C1	119.3 (6)
C7—C8—C9	129.4 (4)	C13A—C14—H14A	108.5
O2—C9—C8	125.8 (4)	C1—C14—H14A	108.5
O2—C9—C1	126.2 (4)	C13A—C14—H14B	108.5
C8—C9—C1	108.1 (4)	C1—C14—H14B	108.5
C11—C10—C1	122.6 (4)	H14A—C14—H14B	107.5
C11—C10—H10	118.7	C13B—C14—H14C	107.5
C1—C10—H10	118.7	C1—C14—H14C	107.5
C10—C11—C12	125.2 (5)	C13B—C14—H14D	107.5
C10—C11—H11	117.4	C1—C14—H14D	107.5
C12—C11—H11	117.4	H14C—C14—H14D	107.0
C9—C1—C2—C3	−10.3 (4)	C10—C11—C12—C13B	25.4 (9)
C1—C2—C3—C8	5.1 (4)	C11—C12—C13A—C14	42.8 (11)
C2—C3—C8—C9	2.8 (5)	C11—C12—C13B—C14	−42.1 (12)
C3—C8—C9—C1	−9.6 (5)	C12—C13A—C14—C1	−51.8 (11)
C8—C9—C1—C2	11.9 (4)	C12—C13B—C14—C1	38.6 (14)
C14—C1—C10—C11	−4.3 (7)	C13A—C14—C1—C10	31.2 (8)
C1—C10—C11—C12	−2.4 (9)	C13B—C14—C1—C10	−14.3 (9)
C10—C11—C12—C13A	−16.1 (9)		