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A chalcone showing positional disorder, two related diarylcyclohexenones showing enantiomeric disorder and a related hydroxyterphenyl, all derived from simple carbonyl precursors

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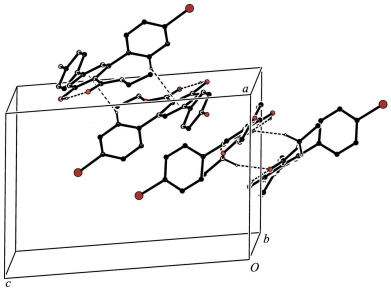
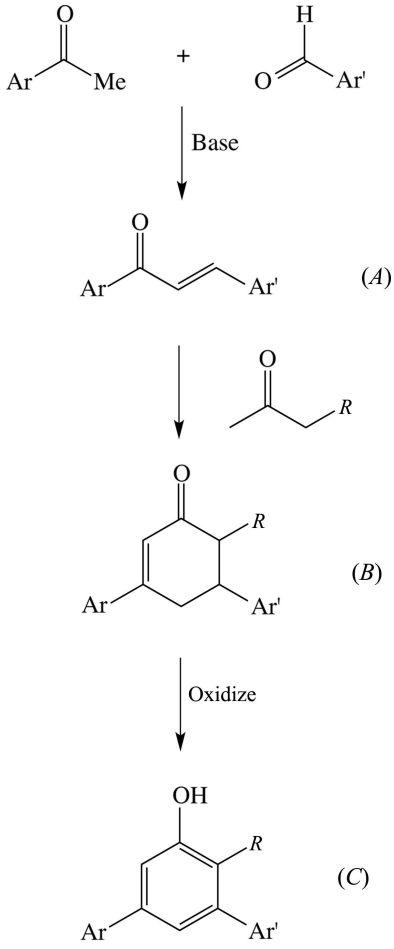
Four compounds are reported, all of which lie along a versatile reaction pathway which leads from simple carbonyl compounds to terphenyls. (2*E*)-1-(2,4-Dichlorophenyl)-3- [4-(prop-1-en-2-yl)phenyl]prop-2-en-1-one, $C_{18}H_{14}Cl_2O$, (I), prepared from 4-(prop-1-en-2-yl)benzaldehyde and 2,4-dichloroacetophenone, exhibits disorder over two sets of atomic sites having occupancies of 0.664 (6) and 0.336 (6). The related chalcone (2*E*)-3-(4-chlorophenyl)-1-(4-fluorophenyl)-prop-2-en-1-one reacts with acetone to produce (5*RS*)-3-(4-chlorophenyl)-5-[4-(propan-2-yl)phenyl]cyclohex-2-en-1-one, $C_{21}H_{21}ClO$, (II), which exhibits enantiomeric disorder with occupancies at the reference site of 0.662 (4) and 0.338 (4) for the (5*R*) and (5*S*) forms; the same chalcone reacts with methyl 3-oxobutanoate to give methyl (1*RS*,6*SR*)-4-(4-chlorophenyl)-6-[4-(propan-2-yl)phenyl]-2-oxocyclohex-3-ene-1-carboxylate, $C_{23}H_{23}ClO_3$, (III), where the reference site contains both (1*R*,6*S*) and (1*S*,6*R*) forms with occupancies of 0.923 (3) and 0.077 (3), respectively. Oxidation, using 2,3-dichloro-5,6-dicyano-1,4-benzoquinone, of ethyl (1*RS*,6*SR*)-6-(4-bromophenyl)-4-(4-fluorophenyl)-2-oxocyclohex-3-ene-1-carboxylate, prepared in a similar manner to (II) and (III), produces ethyl 4″-bromo-4-fluoro-5′-hydroxy-1,1′:3′,1″-terphenyl-4′-carboxylate, $C_{21}H_{16}BrFO_3$, (IV), which crystallizes with Z' = 2 in the space group $P\overline{1}$. There are no significant intermolecular interactions in the structures of compounds (I) and (II), but for the major disorder component of compound (III), the molecules are linked into sheets by a combination of C—H···O and C—H···π(arene) hydrogen bonds. The two independent molecules of compound (IV) form two different centrosymmetric dimers, one built from inversion-related pairs of C—H···O hydrogen bonds and the other from inversion-related pairs of C—H···π(arene) hydrogen bonds. Comparisons are made with related compounds.



1. Introduction

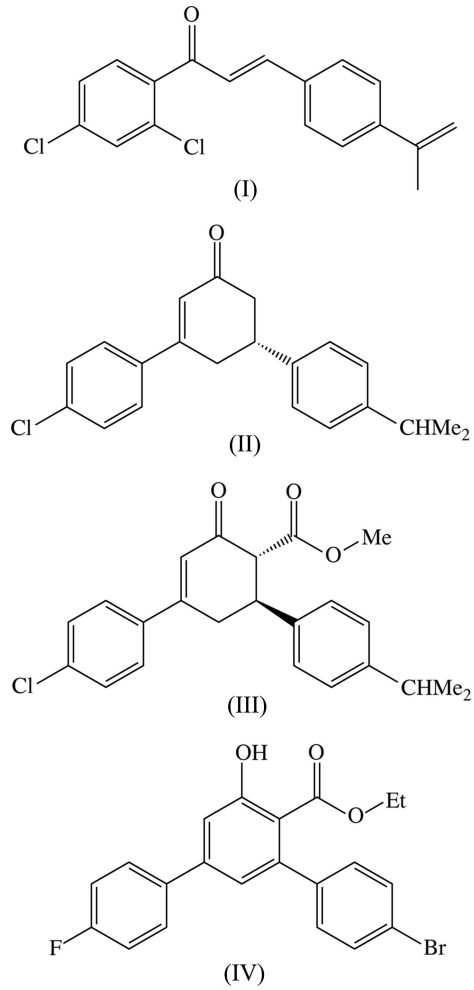
Terphenyls exhibit a wide range of biological activity (Liu, 2007), including anticoagulant and antithrombotic activity, as well as cytotoxic and neuroprotective activity, and they also act as potent immunosuppresants. A convenient and versatile synthesis of 1,1′:3′,1″-terphenyls has now been developed based on 1,3-diarylprop-2-en-1-ones (chalcones) of type (*A*) (see Scheme 1) as the key intermediate. Compounds of type (*A*) are readily synthesized by base-catalysed condensation of acyl arenes with aryl aldehydes, and they react with com-

pounds containing activated methylene groups in a two-step process involving both Michael addition and a condensation reaction to form substituted cyclohexenones of type (*B*) (Scheme 1). Application of a two-electron oxidant to compounds of type (*B*) leads to the loss of two H atoms from the central ring, and the subsequent aromatization of this ring



Scheme 1

generates a substituted terphenyl of type (*C*). This sequence thus represents a simple route to these terphenyls, based on straightforward reaction chemistry using only simple precursor compounds, and it permits the development of considerable structural diversity involving the various substituents. The general value of this reaction sequence is enhanced by the applicability of the cyclohexenone intermediates (*B*) as building blocks for the synthesis of a wide range of heterocyclic systems (Padmavathi *et al.*, 2001; Sridharan *et al.*, 2005; Chandrika *et al.*, 2008; Sapnakumari *et al.*, 2014), as well as by their antibacterial, antifungal and anti-inflammatory actions (Tanaka *et al.*, 1997; Hiromichi *et al.*, 2002; Abdel-Latif *et al.*, 2014). We report here the synthesis and the molecular and supramolecular structures of representative examples of each of compound types (*A*), (*B*) and (*C*), namely (2*E*)-1-(2,4-dichlorophenyl)-3-[4-(prop-1-en-2-yl)phenyl]prop-2-en-1-one, (I) (Scheme 2), as an example of the type (*A*) intermediate, (5*RS*)-3-(4-chlorophenyl)-5-[4-(propan-2-yl)phenyl]cyclohex-2-en-1-one, (II), and methyl (1*RS*,6*SR*)-4-(4-chlorophenyl)-6-[4-(propan-2-yl)phenyl]-2-oxocyclohex-3-ene-1-carboxylate, compound (III), as two examples of the type (*B*) intermediate and ethyl 4″-bromo-4-fluoro-5′-hydroxy-1,1′:3′,1″-terphenyl-4′-carboxylate, (IV), as an example of the type (*C*) product. Thus, compound (I) was prepared using the condensation reaction between 4-(prop-1-en-2-yl)benzaldehyde and 2,4-dichloroacetophenone, compounds (II) and (III) were prepared using the cycloaddition reactions of (2*E*)-3-(4-chlorophenyl)-1-(4-fluorophenyl)prop-2-en-1-one with acetone and methyl 3-oxobutanoate, respectively, and compound (IV) was prepared by the oxidation of ethyl 6-(4-bromophenyl)-4-(4-fluorophenyl)-2-oxocyclohex-3-ene-1-carboxylate using 2,3-dichloro-5,6-dicyano-1,4-benzoquinone.



Scheme 2

2. Experimental

2.1. Synthesis and crystallization

For the synthesis of compound (I), aqueous sodium hydroxide solution (10% *w*/*v*, 15 ml) was added to a mixture of 4-(prop-1-en-2-yl)benzaldehyde (0.01 mol) and 2,4-dichloroacetophenone (0.01 mol), and the mixture was stirred at 275 K for 3 h. The resulting product, (I), was collected by filtration and recrystallized from ethanol (m.p. 341–343 K). Crystals suitable for single-crystal X-ray diffraction were grown by

slow evaporation, at ambient temperature and in the presence of air, of a solution in *N*,*N*-dimethylformamide.

For the synthesis of compound (II), a mixture of (2*E*)-3-(4-chlorophenyl)-1-(4-fluorophenyl)prop-2-en-1-one (0.01 mol), prepared in a manner analogous to the foregoing, acetone (0.01 mol) and dry ethanol (25 ml) was heated under reflux for 8 h in the presence of a catalytic quantity of sodium hydroxide. The reaction mixture was cooled to ambient temperature and the resulting product, (II), was collected by filtration. Crystals suitable for single-crystal X-ray diffraction were grown by slow evaporation, at ambient temperature and in the presence of air, of a solution in methanol (m.p. 389–391 K).

Crystals of compound (III) were prepared in a manner similar to those for (II), but using methyl 3-oxobutanoate (0.01 mol) in place of acetone (m.p. 431–433 K).

For the synthesis of compound (IV), a mixture of ethyl 6-(4-bromophenyl)-4-(4-fluorophenyl)-2-oxocyclohex-3-ene-1-carboxylate (0.01 mol), prepared as described previously (Kant *et al.*, 2012), and 2,3-dichloro-5,6-dicyano-1,4-benzoquinone (0.02 mol) in ethyl 3-oxobutanoate (25 ml) was heated under reflux for 14 h. The reaction mixture was cooled to ambient temperature and exhaustively extracted with diethyl ether; the combined extracts were then washed with dilute aqueous and sodium hydrogen carbonate solution, and dried over anhydrous sodium sulfate. The solvent was removed under reduced pressure and the resulting product, (IV), was crystallized by slow evaporation, at ambient temperature and in the presence of air, of a solution in ethanol (m.p. 363–365 K).

2.2. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 1. The H atoms in compound (IV) and those in the major disorder components of compounds (I)–(III) were all located in difference maps. H atoms bonded to C atoms in these components were then treated as riding atoms, with C—H = 0.93 (aromatic and alkenyl), 0.96 (CH_3), 0.97 (CH_2) or 0.98 Å (aliphatic CH), and with $U_{iso}(H) = kU_{eq}(C)$, where $k = 1.5$ for the methyl groups and 1.2 for all other H atoms bonded to C atoms. For the minor disorder components in compounds (I)–(III), the H atoms were included in calculated positions on exactly the same basis as for the major components. In the final refinements for compounds (I)–(III), the methyl groups were not permitted to rotate; when such rotation was permitted, these methyl groups underwent continuous libration, possibly as a result of the presence of two disorder components having similar atomic coordinates, such that the refinements attempted under these conditions did not converge. For the H atoms bonded to O atoms in compound (IV), the atomic coordinates were refined, with $U_{iso}(H) = 1.5U_{eq}(O)$, giving O—H distances of 0.85 (5) Å in molecule 1 and 0.85 (4) Å in molecule 2. It was apparent from an early stage in the refinement of compound (I) that the molecule was disordered over two sets of atomic sites having unequal occupancies. For the minor component, the bonded distances and the one-angle nonbonded distances were restrained to be identical to the corresponding distances in the major components, subject to experimental uncertainties of 0.005 and 0.01 Å, respectively; in addition, the anisotropic displacement parameters of pairs of corresponding atoms occupying essentially the same regions of physical space were constrained to be identical. On this basis, the occupancies of the two disorder components refined to 0.664 (6) and 0.336 (6). Compounds (II) and (III) likewise exhibit whole-molecule disorder but in these two cases the disorder does not merely involve a minor translation between the two components, but now the disorder is enantiomeric disorder. Thus, in compound (II), the major disorder component at the reference site has the (5*R*) configuration and the minor component has the (5*S*) configuration; in compound (III), the major disorder component has the (1*R*,6*S*) configuration and the minor component has the (1*S*,6*R*) configuration. For each of (II) and (III), the disorder was handled in essentially the same way as for compound (I), giving site occupancies of 0.662 (4) and 0.338 (4) in (II), and 0.923 (3) and 0.077 (3) in (III). Compound (IV) crystallizes with $Z' = 2$, but a search for possible additional crystallographic symmetry found none. For compound (II), a rather high value of K was found in the analysis of variance for the group of very weak reflections having $F_c/F_c(\max)$ in the range $0 < F_c/F_c(\max) < 0.004$. As there appeared to be no experimental justification for the removal of these weak reflections from the data set, they were retained.

3. Results and discussion

Compound (I) (Fig. 1) exhibits disorder over two sets of atomic sites with occupancies of 0.664 (6) and 0.336 (6), but the molecular conformations of the two disorder components are very similar (Fig. 1) and we comment in detail only on that of the major component. The central spacer unit of the major component, between atoms C111 and C131, is effectively planar, as indicated by the relevant torsion angles (Table 2); the maximum deviation from the plane through these five C atoms is only 0.041 (14) Å for atom C131, with an r.m.s. deviation of 0.038 Å. In addition, the nonchlorinated aryl ring (C131–C116) is nearly coplanar with the spacer unit, making with it a dihedral angle of only 3 (2)°. On the other hand, the chlorinated aryl ring (C111–C136) makes a dihedral angle of 48.6 (16)° with the spacer unit, possibly influenced by the

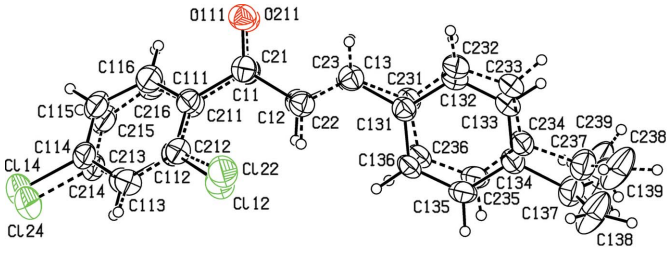


Figure 1
The molecular structure of compound (I), showing the atom-labelling scheme and the two disorder components, whose occupancies are 0.664 (6) (solid bonds) and 0.336 (6) (broken bonds). Displacement ellipsoids are drawn at the 30% probability level.

Table 1
Experimental details.

	(I)	(II)	(III)	(IV)
Crystal data				
Chemical formula	$C_{18}H_{14}Cl_2O$	$C_{21}H_{21}ClO$	$C_{23}H_{23}ClO_3$	$C_{21}H_{16}BrFO_3$
M_r	317.19	324.83	382.86	415.24
Crystal system, space group	Orthorhombic, *Pbca*	Monoclinic, $P2_1/n$	Monoclinic, $P2_1/c$	Triclinic, $P\overline{1}$
Temperature (K)	296	293	296	296
a, *b*, *c* (Å)	13.4314 (4), 8.2405 (4), 29.9252 (9)	6.6259 (8), 8.6868 (11), 30.472 (4)	17.4871 (11), 11.5900 (8), 10.0167 (7)	10.0725 (3), 12.5793 (3), 14.9438 (4)
α, β, γ (°)	90, 90, 90	90, 91.765 (4), 90	90, 95.300 (2), 90	104.332 (1), 92.467 (1), 94.565 (1)
V (Å^3)	3312.2 (2)	1753.1 (4)	2021.5 (2)	1824.84 (9)
Z	8	4	4	4
Radiation type	Mo $K\alpha$	Mo $K\alpha$	Mo $K\alpha$	Mo $K\alpha$
μ (mm^{-1})	0.39	0.22	0.21	2.28
Crystal size (mm)	0.40 × 0.30 × 0.15	0.19 × 0.15 × 0.11	0.26 × 0.19 × 0.15	0.45 × 0.45 × 0.45
Data collection				
Diffractometer	Bruker APEXII CCD diffractometer	Bruker APEXII CCD diffractometer	Bruker APEXII CCD diffractometer	Bruker APEXII CCD diffractometer
Absorption correction	–	Multi-scan (*SADABS*; Sheldrick, 2003)	Multi-scan (*SADABS*; Sheldrick, 2003)	Multi-scan (*SADABS*; Sheldrick, 2003)
T_{min}, T_{max}	–	0.663, 0.976	0.834, 0.969	0.215, 0.358
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	17929, 3450, 2028	21442, 3272, 1931	20398, 4991, 3514	34603, 8396, 5320
R_{int}	0.033	0.082	0.041	0.034
$(\sin\theta/\lambda)_{max}$ (Å^{-1})	0.629	0.607	0.668	0.651
Refinement				
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.058, 0.206, 1.04	0.059, 0.138, 1.03	0.059, 0.161, 1.04	0.043, 0.118, 1.03
No. of reflections	3450	3272	4991	8396
No. of parameters	254	279	326	478
No. of restraints	52	60	70	0
H-atom treatment	H-atom parameters constrained	H-atom parameters constrained	H-atom parameters constrained	H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.46, −0.31	0.31, −0.18	0.40, −0.34	0.62, −0.71

Computer programs: *APEX2* (Bruker, 2007), *SAINT* (Bruker, 2007), *SHELXS97* (Sheldrick, 2008), *SHELXL2014* (Sheldrick, 2015) and *PLATON* (Spek, 2009).

Table 2
Selected torsion angles (°) for (I).

C111—C11—C12—C13	−177 (2)	C211—C21—C22—C23	−171 (4)
C11—C12—C13—C131	174 (2)	C21—C22—C23—C231	−156 (5)
C12—C11—C111—C112	51 (3)	C22—C21—C211—C212	42 (7)
C12—C13—C131—C132	176 (3)	C22—C23—C231—C232	−179 (5)

contact between atoms H12 and Cl12. The dihedral angle between the isopropenyl group containing atom C137 and the adjacent aryl ring is 83.8 (11)°. The molecules of compound (I) thus exhibit no internal symmetry and so are chiral, although, in principal at least, all of the non-H atoms could lie on a mirror plane; the centrosymmetric space group accommodates equal numbers of the two conformational enantiomers.

The molecule of compound (II) (Fig. 2) contains a stereogenic centre at position 5 of the cyclohexenone ring and the reference molecule was selected as one having the *R* configuration at this site; the centrosymmetric space group confirms that the compound crystallizes as a racemic mixture of the (5*R*) and (5*S*) forms. However, the compound exhibits enantiomeric disorder such that, when averaged over the crystal selected for data collection, the reference site contains a fraction of 0.662 (4) of molecules having the (5*R*) configuration and a fraction of 0.338 (4) of molecules having the (5*S*) configuration (Fig. 2). For the major component, the ring-

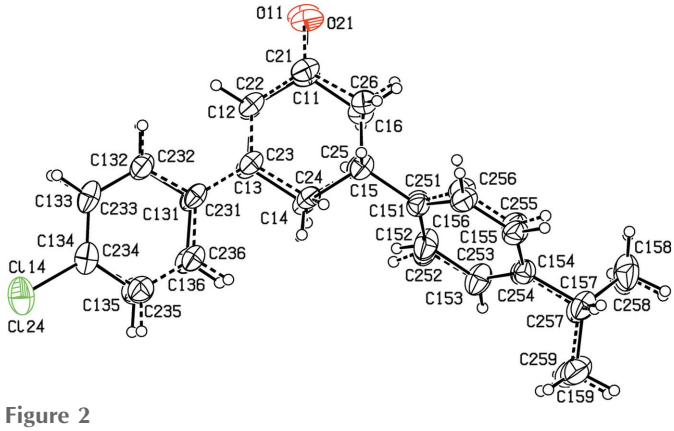


Figure 2
The molecular structure of compound (II), showing the atom-labelling scheme and the enantiomeric disorder, where the (5*R*) and (5*S*) disorder components have occupancies of 0.662 (4) (solid bonds) and 0.338 (4) (broken bonds). Displacement ellipsoids are drawn at the 30% probability level.

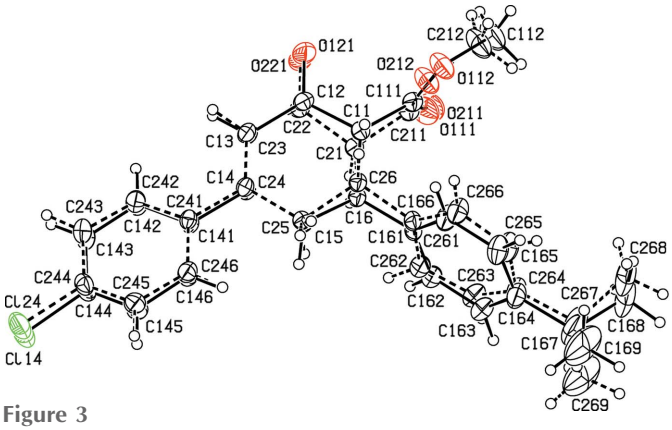


Figure 3
The molecular structure of compound (III), showing the atom-labelling scheme and the enantiomeric disorder, where the (1*R*,6*S*) and (1*S*,6*R*) disorder components have occupancies of 0.923 (3) (solid bonds) and 0.077 (3) (broken bonds). Displacement ellipsoids are drawn at the 30% probability level.

puckering parameters (Cremer & Pople, 1975) calculated for the atom sequence C11–C12–C13–C14–C15–C16 are Q = 0.494 (11) Å, θ = 125 (2)° and φ = 76 (3)°, indicating an approximate envelope conformation, for which the idealized values for the ring-puckering angles are $\theta = 54.7°$ and $\varphi = 60k°$, where k represents an integer (Boeyens, 1978); the fold of the envelope is across the C14···C16 line. The corresponding ring-puckering parameters for the minor component, calculated for the atom sequence C21–C22–C23–C24–C25–C26, are Q = 0.49 (2) Å, $\theta = 57$ (5)° and $\varphi = 258$ (5)°, where the values of the puckering angles confirm the change of enantiomorph.

There are two stereogenic centres in the molecule of compound (III) at positions 1 and 6 of the cyclohexenone ring; the reference molecule was selected as one having the *R* configuration at position 1 and, on this basis, the configuration at position 6 is *S*. As in compound (II), the centrosymmetric space group confirms that compound (III) crystallizes as a racemic mixture of the (1*R*,6*S*) and (1*S*,6*R*) forms. Because of the different numbering of the cyclohexenone rings in compounds (II) and (III), consistent with the systematic names of these compounds, together with the specification of *R* stereochemistry at the lowest stereogenic locant, the reference molecules of (II) and (III) appear to have the opposite configuration at the point of connection to the 4-isopropylphenyl substituent. Compound (III) also exhibits enantiomeric disorder (Fig. 3), with a fraction of 0.923 (3) of (1*R*,6*S*) molecules at the reference site and a fraction of 0.077 (3) of (1*S*,6*R*) molecules, *i.e.* with very much less extensive disorder than that found in compound (II). For the dominant form, the ring-puckering parameters, calculated for the atom sequence C11–C12–C13–C14–C15–C16, are Q = 0.497 (3) Å, θ = 129.1 (3)° and φ = 133.3 (5)°, again indicative of an envelope form. Here the fold of the envelope is across the C11···C15 line, so that in each of compounds (II) and (III), the point of the envelope corresponds to the C atom which carries the 4-isopropylphenyl substituent.

Compound (IV) crystallizes with $Z' = 2$ in the space group $P\overline{1}$ (Fig. 4), although there is no disorder in this structure; a detailed comparison of the atomic coordinates for corresponding pairs of atoms in the two independent molecules shows that no additional symmetry is present. It will be convenient to refer to the molecules containing atoms O125 and O225 as molecules of types 1 and 2, respectively. In each of the two independent molecules, there is an O—H···O hydrogen bond (Table 3) forming an *S*(6) (Bernstein *et al.*, 1995) motif, and these may account for the fairly small dihedral angles between the carboxylate groups and the adjacent aryl rings, *i.e.* 7.90 (16)° in molecule 1 and 15.20 (14)° in molecule 2. By contrast, in compound (III), where there is no intramolecular O—H···O hydrogen bond to influence the orientation of the ester group, the dihedral angle between the carboxylate group and the mean plane through atoms C11–C15 is 83.60 (12)° (*cf.* Figs. 3 and 4). In molecule 1 of compound (IV), the central aryl ring (C121–C126) makes dihedral angles with the two outer rings (C111–C116 and C131–C136) of 28.64 (14) and 71.83 (14)°, respectively, with a dihedral angle of 63.13 (14)° between the planes of the two outer rings; the corresponding values for molecule 2 are 27.30 (13)°, 61.53 (12)° and 72.82 (14) Å, respectively, indicating an absence of internal symmetry relating both molecules.

The only direction-specific intermolecular interaction involving the major orientation of compound (I) is a C—

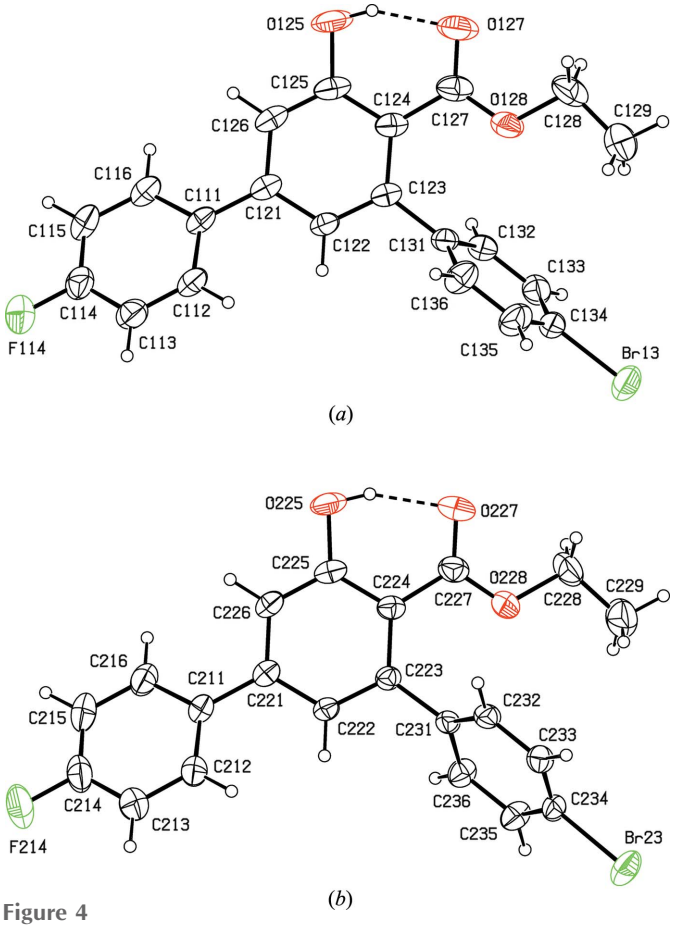


Figure 4
The structures of the two independent molecules of compound (IV), showing the atom-labelling scheme and the intramolecular O—H···O hydrogen bonds for (*a*) a type 1 molecule and (*b*) a type 2 molecule. Displacement ellipsoids are drawn at the 30% probability level.

Table 3
Hydrogen bonds and short intermolecular contacts (Å, °) for compounds (I)–(IV).

*Cg*1 represents the centroid of the C131–C136 ring, *Cg*2 represents the centroid of the C231–C236 ring, *Cg*3 represents the centroid of the C141–C146 ring and *Cg*4 represents the centroid of the C221–C226 ring C221–C226.

Compound	D—H···A	D—H	H···A	D···A	D—H···A
(I)	C12—H12···O111^{i}	0.93	2.49	3.15 (2)	128
	C232—H232···*Cg*1^{ii}	0.93	2.74	3.67 (4)	174
	C232—H232···*Cg*2^{ii}	0.93	2.76	3.67 (4)	166
(II)	C132—H132···O11^{iii}	0.93	2.56	3.28 (4))	134
	C232—H232···O21^{iii}	0.93	2.54	3.18 (7)	126
(III)	C162—H162···O111^{iv}	0.93	2.58	3.499 (4)	169
	C25—H25*A*···O221^{iv}	0.96	2.36	3.11 (6)	134
	C11—H11···*Cg*3^{v}	0.98	2.78	3.651 (3)	149
(IV)	O125—H125···O127	0.85 (5)	1.76 (5)	2.542 (4)	151 (5)
	O225—H225···O227	0.85 (4)	1.75 (4)	2.556 (3)	157 (4)
	C128—H18*A*···O127^{vi}	0.97	2.57	3.463 (5)	153
	C236—H236···*Cg*4^{vii}	0.93	2.78	3.541 (3)	140

Symmetry codes: (i) $-x+\frac{3}{2}, y-\frac{1}{2}, z$; (ii) $-x+\frac{3}{2}, y+\frac{1}{2}, z$; (iii) $-x, -y+1, -z+1$; (iv) $x, -y+\frac{3}{2}, z-\frac{1}{2}$; (v) $-x+1, -y+1, -z+1$; (vi) $-x+1, -y+2, -z$; (vii) $-x+2, -y+1, -z+1$.

H···O contact having a rather small D—H···A angle (Table 3); such a small value of this angle makes it very unlikely that this contact could be regarded as structurally significant (Wood *et al.*, 2009). There are two C—H···π(arene) interactions, both involving the same C—H bond in the minor component and the nonhalogenated rings of both major and minor components, but there is no corresponding interaction involving a C—H bond from the major component. If this C—H bond had been part of a molecule with unit occupancy, the C—H···π(arene) interaction would have given rise to a hydrogen-bonded chain running parallel to the [010] direction. However, because these interactions involve only *ca* one third of the molecules, any such chain will be badly fragmented into two- and three-molecule sections. The only direction-specific intermolecular interactions in the structure of compound (II) are two C—H···O contacts both having D—H···A angles below 135° (Table 3), so that neither of these is likely to be structurally significant (Wood *et al.*, 2009).

In the structure of compound (III), inversion-related pairs of the major disorder component are linked by paired C—H···π(arene) hydrogen bonds (Table 3) to form cyclic dimers, with the reference dimer lying across $(\frac{1}{2}, \frac{1}{2}, \frac{1}{2})$ (Fig. 5). In addition, the major disorder components of molecules related by the *c*-glide plane at $y = \frac{3}{4}$ are linked by an almost linear C—H···O hydrogen bond to form a *C*(7) (Bernstein *et al.*, 1995) chain running parallel to the [001] direction (Fig. 6). The C—H···O hydrogen bond also links the reference dimer centred at $(\frac{1}{2}, \frac{1}{2}, \frac{1}{2})$ directly to the four symmetry-related dimers centred at $(\frac{1}{2}, 0, 0)$, $(\frac{1}{2}, 1, 0)$, $(\frac{1}{2}, 0, 1)$ and $(\frac{1}{2}, 1, 1)$, thereby forming a sheet lying parallel to (100) (Fig. 7). By contrast to these interactions involving the major disorder component, there is only a single C—H···O contact involving the minor component (Table 3),

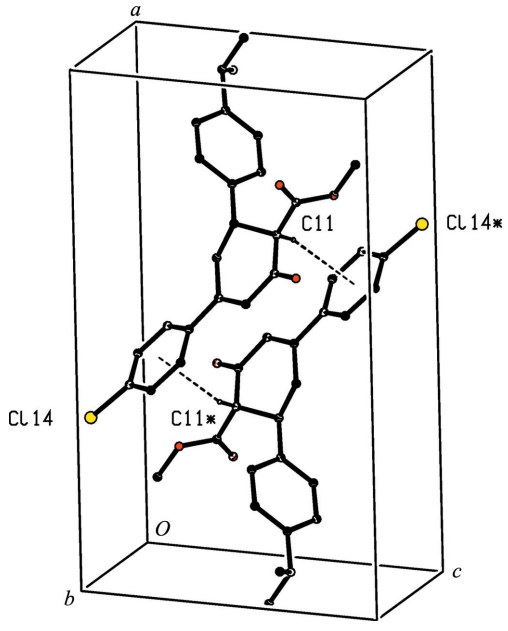


Figure 5
Part of the crystal structure of compound (III), showing the formation of a centrosymmetric dimer built from C—H···π(arene) hydrogen bonds. For the sake of clarity, the minor disorder component and H atoms not involved in the motif shown have been omitted. Atoms marked with an asterisk (*) are at the symmetry position $(-x+1, -y+1, -z+1)$.

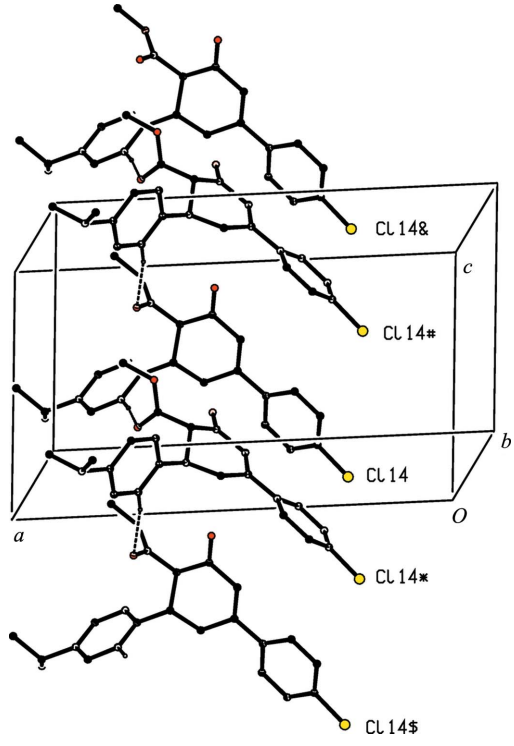


Figure 6
Part of the crystal structure of compound (III), showing the formation of a *C*(7) hydrogen-bonded chain running parallel to [001]. For the sake of clarity, the minor disorder component and H atoms not involved in the motif shown have been omitted. Atoms marked with an asterisk (*), a hash (#), a dollar sign ($) or an ampersand (&) are at the symmetry positions $(x, -y+\frac{3}{2}, z-\frac{1}{2})$, $(x, -y+\frac{3}{2}, z+\frac{1}{2})$, $(x, y, z-1)$ and $(x, y, z+1)$, respectively.

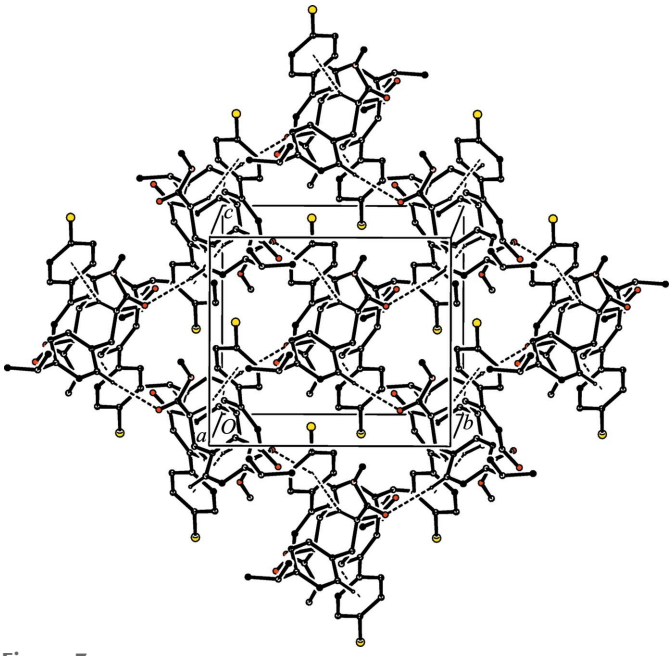


Figure 7
Part of the crystal structure of compound (III), showing the formation of a hydrogen-bonded sheet lying parallel to (100). For the sake of clarity, the minor disorder component and H atoms not involved in the motifs shown have been omitted.

which has a small D—H···A angle such that it cannot be regarded as structurally significant.

As noted above, each of the two independent molecules of compound (IV) contains an intramolecular O—H···O hydrogen bond. In addition, each type of molecule is linked into centrosymmetric dimers, but by different means. The type 1 molecules are linked by inversion-related C—H···O hydrogen bonds, while the type 2 molecules are linked by inversion-related C—H···π(arene) hydrogen bonds, so forming dimers lying across ($\frac{1}{2}$, 1, 0) and (1, $\frac{1}{2}$, $\frac{1}{2}$), respectively (Fig. 8).

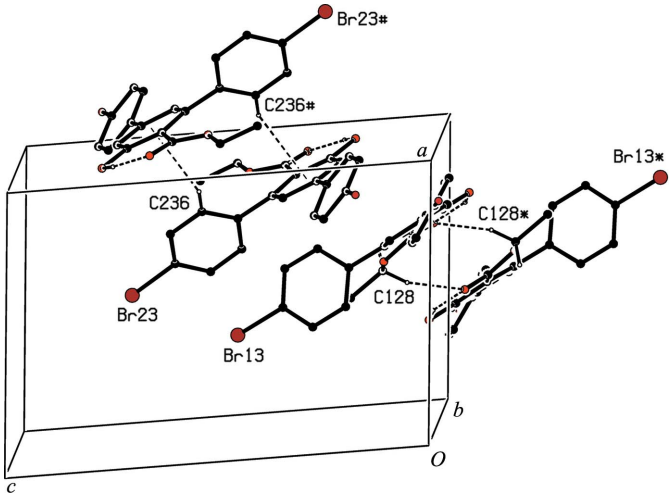
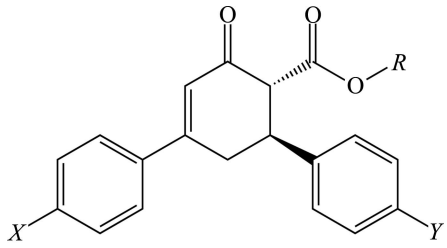
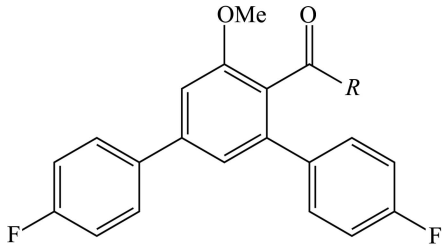


Figure 8
Part of the crystal structure of compound (IV), showing the intramolecular O—H···O hydrogen bonds and the two different hydrogen-bonded dimers formed by the type 1 and type 2 molecules. For the sake of clarity, H atoms bonded to C atoms which are not involved in the motifs shown have been omitted. Atoms marked with an asterisk (*) or a hash (#) are at the symmetry positions ($-x+1, -y+2, -z$) and ($-x+2, -y+1, -z+1$), respectively.



(V) R = Me, X = Y = F
(VI) R = Et, X = Y = F
(VII) R = Et, X = F, Y = Cl
(VIII) R = Et, X = F, Y = Br



(IX) R = Me
(X) R = OMe
Scheme 3

It is of interest briefly to note the structures of some related cyclohexenone and terphenyl derivatives (see Scheme 3). Compound (V), prepared using the cycloaddition reaction between (2*E*)-1,3-bis(4-fluorophenyl)prop-2-en-1-one and methyl 3-oxobutanoate, also crystallizes with Z' = 2 as a racemic mixture of the (1*R*,6*S*) and (1*S*,6*R*) forms, although the stereochemistry of the compound was not mentioned anywhere in the original report (Fun *et al.*, 2010). Compound (VI), the precursor for compound (IV) reported here, was described (Dutkiewicz *et al.*, 2011) as having a three-dimensional supramolecular structure, built from C—H···O, C—H···F and C—H···π(arene) hydrogen bonds; however, the C—H···O contact has a D—H···A angle of only 130° and so cannot be regarded as structurally significant (Wood *et al.*, 2009), and it is well established (Howard *et al.*, 1996) that F atoms bonded to C atoms are not effective acceptors of hydrogen bonds. Hence, the supramolecular aggregation in compound (VI) depends solely on the C—H···π(arene) interaction which generates isolated centrosymmetric dimers. Whereas compound (VI) crystallizes in the space group $P2_1/n$ with Z' = 1, the two closely related analogues (VII) (Sapnakumari *et al.*, 2013) and (VIII) (Kant *et al.*, 2012) both crystallize in the space group $P\overline{1}$ with Z' = 2. The unit cells of these two compounds have very similar repeat vectors, but in compound (VII), the inter-cell angles lie in the order $\beta > \alpha > \gamma$, while in compound (VIII), the order is $\gamma > \alpha > \beta$, equivalent, in effect, to an interchange of the b and c axes. The close similarity between the two structures is not readily recognized because of the different atomic numbering schemes employed,

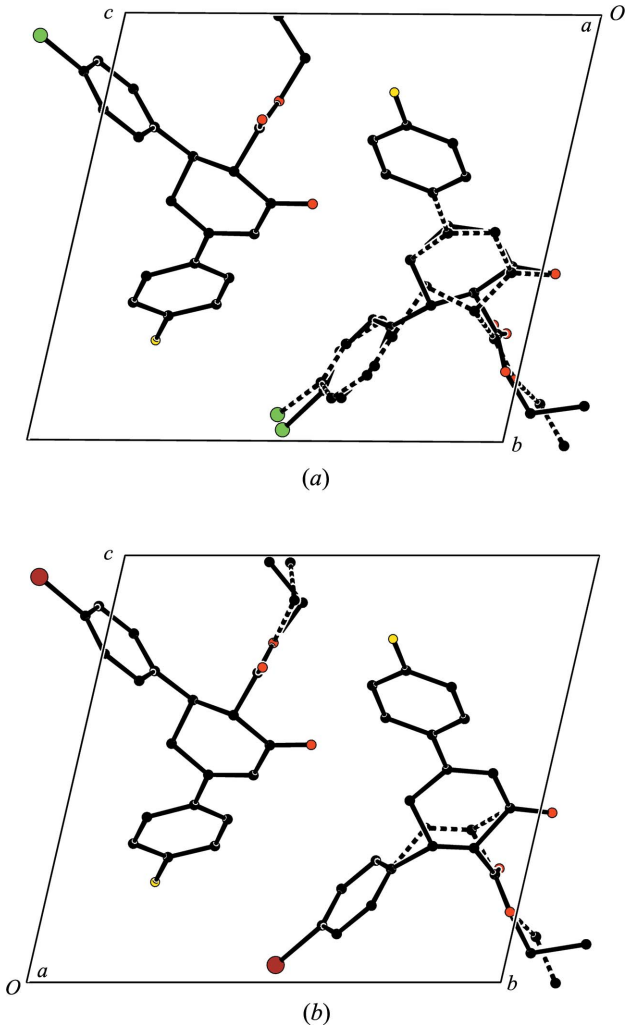


Figure 9
Projections of parts of the crystal structures of compounds (VII) and (VIII), showing the two independent molecules within the unit cells for (*a*) compound (VII) and (*b*) compound (VIII). For compound (VII), the original atomic coordinates (Sapnakumari *et al.*, 2013) were employed, but for compound (VIII) (Kant *et al.*, 2012), both molecules have been subjected to simple translations within the unit cell. The projections are viewed along [100] and, for the sake of clarity, H atoms have been omitted. Note the different locations of the origin and the different orientations of the unit-cell repeat vectors.

the different choice of origin and the different placing of the two independent molecules within the unit cells; however, when these factors are taken into consideration, the similarities become immediately apparent (Fig. 9). When a diarylcyclohexenone of type (*B*) (Scheme 1) is oxidized using iodine in methanol, rather than a quinone as used here for the formation of compound (IV), the resulting terphenyls carry a 5-methoxy substituent instead of the 5-hydroxy substituent observed in (IV), as exemplified by compounds (IX) (Fun *et al.*, 2012) and (X) (Fun *et al.* 2011) (see Scheme 3). Neither of compounds (IX) and (X) contains an intramolecular O—H···O hydrogen bond and, in each, the carbonyl substituent is approximately orthogonal to the adjacent ring, as found here in (III) but not in (IV). There are no significant direction-specific intermolecular interactions in the structures of (IX) and (X).

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supporting information

Acta Cryst. (2015). C**71**, 610-617 [doi:10.1107/S2053229615011961]

A chalcone showing positional disorder, two related diarylcyclohexenones showing enantiomeric disorder and a related hydroxyterphenyl, all derived from simple carbonyl precursors

Vinutha V. Salian, Badiadka Narayana, Hemmige S. Yathirajan, Mehmet Akkurt, Ömer Çelik, Cem Cüneyt Ersanlı and Christopher Glidewell

Computing details

For all compounds, data collection: *APEX2* (Bruker, 2007); cell refinement: *SAINT* (Bruker, 2007); data reduction: *SAINT* (Bruker, 2007); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL2014* (Sheldrick, 2015); molecular graphics: *PLATON* (Spek, 2009); software used to prepare material for publication: *SHELXL2014* (Sheldrick, 2015) and *PLATON* (Spek, 2009).

(I) (2*E*)-1-(2,4-Dichlorophenyl)-3-[4-(prop-1-en-2-yl)phenyl]prop-2-en-1-one

Crystal data

$C_{18}H_{14}Cl_2O$
M_r = 317.19
Orthorhombic, *Pbca*
a = 13.4314 (4) Å
b = 8.2405 (4) Å
c = 29.9252 (9) Å
V = 3312.2 (2) Å^3
Z = 8
F(000) = 1312
D_x = 1.272 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 6379 reflections
θ = 2.0–33.4°
μ = 0.39 mm^{-1}
T = 296 K
Block, colourless
0.40 × 0.30 × 0.15 mm

Data collection

Bruker APEXII CCD diffractometer
Radiation source: sealed tube
Graphite monochromator
φ and ω scans
17929 measured reflections
3450 independent reflections
2028 reflections with $I > 2\sigma(I)$
R_{int} = 0.033
θ_{max} = 26.5°, θ_{min} = 2.0°
h = −16→16
k = −9→10
l = −37→37

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.058
$wR(F^2)$ = 0.206
S = 1.03
3450 reflections
254 parameters
52 restraints
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.1057P)^2 + 0.9703P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.46 e Å^{-3}
$\Delta\rho_{min}$ = −0.31 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.

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

	x	*y*	*z*	U_{iso}*/U_{eq}	Occ. (<1)
C11	0.7567 (7)	0.739 (2)	0.57053 (18)	0.058 (2)	0.664 (6)
C12	0.7031 (13)	0.672 (3)	0.6098 (2)	0.061 (2)	0.664 (6)
H12	0.6526	0.5967	0.6050	0.073*	0.664 (6)
C13	0.7246 (15)	0.715 (2)	0.6513 (2)	0.059 (2)	0.664 (6)
H13	0.7798	0.7812	0.6553	0.071*	0.664 (6)
O111	0.8328 (7)	0.8177 (10)	0.5735 (3)	0.0665 (19)	0.664 (6)
C111	0.7210 (6)	0.687 (2)	0.5250 (2)	0.0568 (14)	0.664 (6)
C112	0.6231 (12)	0.693 (5)	0.5100 (7)	0.0673 (15)	0.664 (6)
Cl12	0.5308 (6)	0.7807 (12)	0.5433 (3)	0.0976 (16)	0.664 (6)
C113	0.5956 (11)	0.642 (5)	0.4680 (6)	0.0796 (13)	0.664 (6)
H113	0.5296	0.6473	0.4587	0.095*	0.664 (6)
C114	0.6684 (5)	0.5834 (15)	0.4401 (2)	0.076 (2)	0.664 (6)
Cl14	0.6356 (5)	0.5116 (14)	0.38744 (18)	0.1276 (12)	0.664 (6)
C115	0.7653 (5)	0.5726 (11)	0.4536 (2)	0.073 (2)	0.664 (6)
H115	0.8138	0.5328	0.4343	0.088*	0.664 (6)
C116	0.7905 (7)	0.622 (5)	0.4964 (6)	0.071 (3)	0.664 (6)
H116	0.8560	0.6105	0.5060	0.085*	0.664 (6)
C131	0.6701 (12)	0.668 (3)	0.69147 (17)	0.0554 (16)	0.664 (6)
C132	0.7062 (14)	0.714 (2)	0.7332 (2)	0.0750 (17)	0.664 (6)
H132	0.7627	0.7792	0.7349	0.090*	0.664 (6)
C133	0.6598 (6)	0.6653 (14)	0.77220 (18)	0.0766 (15)	0.664 (6)
H133	0.6812	0.7075	0.7994	0.092*	0.664 (6)
C134	0.5829 (4)	0.5561 (9)	0.77170 (15)	0.0591 (13)	0.664 (6)
C135	0.5471 (5)	0.5098 (11)	0.73047 (17)	0.0652 (12)	0.664 (6)
H135	0.4924	0.4408	0.7291	0.078*	0.664 (6)
C136	0.5903 (6)	0.5631 (14)	0.69100 (15)	0.0589 (13)	0.664 (6)
H136	0.5649	0.5275	0.6638	0.071*	0.664 (6)
C137	0.5321 (4)	0.4974 (8)	0.81457 (18)	0.0795 (17)	0.664 (6)
C138	0.4602 (6)	0.5849 (10)	0.8300 (3)	0.128 (3)	0.664 (6)
H18A	0.4379	0.5697	0.8591	0.153*	0.664 (6)
H18B	0.4306	0.6632	0.8120	0.153*	0.664 (6)
C139	0.5860 (10)	0.3732 (16)	0.8368 (4)	0.136 (4)	0.664 (6)
H19A	0.6353	0.3293	0.8170	0.204*	0.664 (6)
H19B	0.6181	0.4176	0.8627	0.204*	0.664 (6)
H19C	0.5410	0.2887	0.8458	0.204*	0.664 (6)
C21	0.7590 (13)	0.715 (5)	0.5734 (4)	0.058 (2)	0.336 (6)
C22	0.694 (3)	0.687 (6)	0.6128 (4)	0.061 (2)	0.336 (6)
H22	0.6343	0.6309	0.6090	0.073*	0.336 (6)
C23	0.719 (3)	0.739 (5)	0.6530 (4)	0.059 (2)	0.336 (6)

H23	0.7623	0.8265	0.6543	0.071*	0.336 (6)
O211	0.8417 (14)	0.773 (2)	0.5760 (6)	0.0665 (19)	0.336 (6)
C211	0.7176 (12)	0.674 (5)	0.5281 (4)	0.0568 (14)	0.336 (6)
C212	0.622 (2)	0.706 (11)	0.5133 (14)	0.0673 (15)	0.336 (6)
Cl22	0.5407 (12)	0.820 (3)	0.5466 (6)	0.0976 (16)	0.336 (6)
C213	0.587 (2)	0.656 (10)	0.4722 (13)	0.0796 (13)	0.336 (6)
H213	0.5239	0.6839	0.4623	0.095*	0.336 (6)
C214	0.6501 (12)	0.564 (4)	0.4463 (5)	0.076 (2)	0.336 (6)
Cl24	0.6118 (12)	0.505 (3)	0.3931 (4)	0.1276 (12)	0.336 (6)
C215	0.7469 (11)	0.538 (3)	0.4584 (5)	0.073 (2)	0.336 (6)
H215	0.7885	0.4755	0.4404	0.088*	0.336 (6)
C216	0.7821 (15)	0.606 (10)	0.4976 (12)	0.071 (3)	0.336 (6)
H216	0.8501	0.6051	0.5035	0.085*	0.336 (6)
C231	0.685 (3)	0.673 (5)	0.6957 (3)	0.0554 (16)	0.336 (6)
C232	0.718 (3)	0.747 (5)	0.7350 (4)	0.0750 (17)	0.336 (6)
H232	0.7619	0.8344	0.7335	0.090*	0.336 (6)
C233	0.6871 (15)	0.691 (3)	0.7763 (3)	0.0766 (15)	0.336 (6)
H233	0.7226	0.7190	0.8018	0.092*	0.336 (6)
C234	0.6047 (10)	0.593 (2)	0.7801 (3)	0.0591 (13)	0.336 (6)
C235	0.5687 (11)	0.525 (2)	0.7412 (3)	0.0652 (12)	0.336 (6)
H235	0.5167	0.4511	0.7429	0.078*	0.336 (6)
C236	0.6080 (14)	0.564 (3)	0.6997 (3)	0.0589 (13)	0.336 (6)
H236	0.5818	0.5163	0.6741	0.071*	0.336 (6)
C237	0.5705 (9)	0.5291 (15)	0.8256 (3)	0.0795 (17)	0.336 (6)
C238	0.5270 (11)	0.6295 (19)	0.8524 (5)	0.128 (3)	0.336 (6)
H28A	0.5234	0.6064	0.8828	0.153*	0.336 (6)
H28B	0.4993	0.7248	0.8414	0.153*	0.336 (6)
C239	0.616 (3)	0.379 (3)	0.8369 (9)	0.136 (4)	0.336 (6)
H29A	0.6427	0.3295	0.8104	0.204*	0.336 (6)
H29B	0.6693	0.3981	0.8578	0.204*	0.336 (6)
H29C	0.5679	0.3082	0.8502	0.204*	0.336 (6)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C11	0.0644 (16)	0.051 (5)	0.0593 (15)	0.003 (2)	−0.0007 (13)	0.007 (2)
C12	0.063 (3)	0.060 (5)	0.0603 (17)	−0.002 (4)	−0.0005 (17)	−0.001 (2)
C13	0.064 (3)	0.053 (5)	0.0599 (15)	−0.001 (4)	−0.0027 (13)	−0.0011 (16)
O111	0.067 (2)	0.061 (5)	0.0719 (16)	−0.006 (3)	−0.0051 (13)	0.004 (3)
C111	0.0633 (16)	0.054 (3)	0.0534 (15)	−0.0073 (17)	0.0025 (12)	0.008 (2)
C112	0.0640 (16)	0.079 (7)	0.059 (3)	−0.0073 (16)	0.0020 (18)	0.006 (2)
Cl12	0.0659 (16)	0.148 (5)	0.0791 (13)	0.016 (2)	−0.0005 (11)	−0.0095 (17)
C113	0.072 (3)	0.100 (8)	0.067 (4)	−0.020 (3)	−0.006 (3)	0.004 (3)
C114	0.095 (4)	0.082 (4)	0.050 (3)	−0.034 (4)	0.009 (2)	0.003 (3)
Cl14	0.142 (4)	0.1750 (17)	0.0659 (15)	−0.057 (3)	0.0023 (14)	−0.0272 (17)
C115	0.086 (3)	0.072 (5)	0.061 (2)	−0.018 (3)	0.022 (2)	0.002 (3)
C116	0.070 (2)	0.075 (8)	0.0676 (19)	−0.001 (4)	0.005 (2)	0.003 (3)
C131	0.056 (5)	0.057 (2)	0.0534 (16)	−0.001 (3)	−0.007 (2)	0.002 (2)

C132	0.082 (5)	0.078 (8)	0.0647 (18)	−0.034 (3)	−0.0052 (18)	−0.007 (2)
C133	0.070 (6)	0.104 (5)	0.0558 (19)	−0.029 (3)	−0.011 (2)	−0.008 (2)
C134	0.065 (3)	0.060 (4)	0.052 (2)	−0.005 (2)	−0.006 (2)	−0.004 (2)
C135	0.052 (3)	0.069 (3)	0.074 (3)	−0.013 (2)	−0.013 (2)	0.002 (3)
C136	0.057 (3)	0.070 (2)	0.050 (2)	−0.008 (3)	−0.019 (2)	0.000 (3)
C137	0.075 (4)	0.099 (4)	0.065 (3)	0.008 (3)	0.007 (2)	0.018 (3)
C138	0.112 (5)	0.144 (6)	0.127 (6)	0.045 (5)	0.064 (4)	0.051 (5)
C139	0.108 (10)	0.190 (6)	0.111 (3)	0.042 (6)	0.045 (5)	0.077 (4)
C21	0.0644 (16)	0.051 (5)	0.0593 (15)	0.003 (2)	−0.0007 (13)	0.007 (2)
C22	0.063 (3)	0.060 (5)	0.0603 (17)	−0.002 (4)	−0.0005 (17)	−0.001 (2)
C23	0.064 (3)	0.053 (5)	0.0599 (15)	−0.001 (4)	−0.0027 (13)	−0.0011 (16)
O211	0.067 (2)	0.061 (5)	0.0719 (16)	−0.006 (3)	−0.0051 (13)	0.004 (3)
C211	0.0633 (16)	0.054 (3)	0.0534 (15)	−0.0073 (17)	0.0025 (12)	0.008 (2)
C212	0.0640 (16)	0.079 (7)	0.059 (3)	−0.0073 (16)	0.0020 (18)	0.006 (2)
Cl22	0.0659 (16)	0.148 (5)	0.0791 (13)	0.016 (2)	−0.0005 (11)	−0.0095 (17)
C213	0.072 (3)	0.100 (8)	0.067 (4)	−0.020 (3)	−0.006 (3)	0.004 (3)
C214	0.095 (4)	0.082 (4)	0.050 (3)	−0.034 (4)	0.009 (2)	0.003 (3)
Cl24	0.142 (4)	0.1750 (17)	0.0659 (15)	−0.057 (3)	0.0023 (14)	−0.0272 (17)
C215	0.086 (3)	0.072 (5)	0.061 (2)	−0.018 (3)	0.022 (2)	0.002 (3)
C216	0.070 (2)	0.075 (8)	0.0676 (19)	−0.001 (4)	0.005 (2)	0.003 (3)
C231	0.056 (5)	0.057 (2)	0.0534 (16)	−0.001 (3)	−0.007 (2)	0.002 (2)
C232	0.082 (5)	0.078 (8)	0.0647 (18)	−0.034 (3)	−0.0052 (18)	−0.007 (2)
C233	0.070 (6)	0.104 (5)	0.0558 (19)	−0.029 (3)	−0.011 (2)	−0.008 (2)
C234	0.065 (3)	0.060 (4)	0.052 (2)	−0.005 (2)	−0.006 (2)	−0.004 (2)
C235	0.052 (3)	0.069 (3)	0.074 (3)	−0.013 (2)	−0.013 (2)	0.002 (3)
C236	0.057 (3)	0.070 (2)	0.050 (2)	−0.008 (3)	−0.019 (2)	0.000 (3)
C237	0.075 (4)	0.099 (4)	0.065 (3)	0.008 (3)	0.007 (2)	0.018 (3)
C238	0.112 (5)	0.144 (6)	0.127 (6)	0.045 (5)	0.064 (4)	0.051 (5)
C239	0.108 (10)	0.190 (6)	0.111 (3)	0.042 (6)	0.045 (5)	0.077 (4)

Geometric parameters (Å, º)

C11—O111	1.215 (4)	C21—O211	1.214 (5)
C11—C12	1.485 (8)	C21—C22	1.485 (8)
C11—C111	1.507 (4)	C21—C211	1.505 (5)
C12—C13	1.323 (4)	C22—C23	1.323 (5)
C12—H12	0.9300	C22—H22	0.9300
C13—C131	1.460 (6)	C23—C231	1.459 (8)
C13—H13	0.9300	C23—H23	0.9300
C111—C116	1.376 (4)	C211—C216	1.376 (5)
C111—C112	1.390 (6)	C211—C212	1.390 (7)
C112—C113	1.376 (4)	C212—C213	1.376 (5)
C112—Cl12	1.747 (5)	C212—Cl22	1.746 (7)
C113—C114	1.373 (9)	C213—C214	1.374 (10)
C113—H113	0.9300	C213—H213	0.9300
C114—C115	1.366 (5)	C214—C215	1.366 (6)
C114—Cl14	1.741 (4)	C214—Cl24	1.742 (5)
C115—C116	1.385 (6)	C215—C216	1.384 (8)

C115—H115	0.9300	C215—H215	0.9300
C116—H116	0.9300	C216—H216	0.9300
C131—C136	1.376 (4)	C231—C236	1.376 (5)
C131—C132	1.394 (5)	C231—C232	1.396 (7)
C132—C133	1.382 (5)	C232—C233	1.383 (7)
C132—H132	0.9300	C232—H232	0.9300
C133—C134	1.369 (5)	C233—C234	1.371 (6)
C133—H133	0.9300	C233—H233	0.9300
C134—C135	1.378 (5)	C234—C235	1.379 (6)
C134—C137	1.531 (5)	C234—C237	1.532 (6)
C135—C136	1.387 (5)	C235—C236	1.389 (6)
C135—H135	0.9300	C235—H235	0.9300
C136—H136	0.9300	C236—H236	0.9300
C137—C138	1.291 (6)	C237—C238	1.292 (7)
C137—C139	1.419 (7)	C237—C239	1.422 (7)
C138—H18A	0.9300	C238—H28A	0.9300
C138—H18B	0.9300	C238—H28B	0.9300
C139—H19A	0.9600	C239—H29A	0.9600
C139—H19B	0.9600	C239—H29B	0.9600
C139—H19C	0.9600	C239—H29C	0.9600

O111—C11—C12	123.3 (4)	O211—C21—C22	123.4 (6)
O111—C11—C111	119.0 (3)	O211—C21—C211	119.1 (6)
C12—C11—C111	117.2 (5)	C22—C21—C211	117.5 (6)
C13—C12—C11	122.6 (5)	C23—C22—C21	121.5 (8)
C13—C12—H12	118.7	C23—C22—H22	119.2
C11—C12—H12	118.7	C21—C22—H22	119.2
C12—C13—C131	126.4 (5)	C22—C23—C231	126.8 (10)
C12—C13—H13	116.8	C22—C23—H23	116.6
C131—C13—H13	116.8	C231—C23—H23	116.6
C116—C111—C112	117.1 (3)	C216—C211—C212	116.9 (6)
C116—C111—C11	117.3 (3)	C216—C211—C21	117.2 (5)
C112—C111—C11	125.6 (4)	C212—C211—C21	125.8 (6)
C113—C112—C111	122.6 (6)	C213—C212—C211	122.4 (6)
C113—C112—Cl12	117.1 (4)	C213—C212—Cl22	117.5 (7)
C111—C112—Cl12	120.2 (3)	C211—C212—Cl22	120.0 (5)
C114—C113—C112	118.0 (6)	C214—C213—C212	117.7 (8)
C114—C113—H113	121.0	C214—C213—H213	121.2
C112—C113—H113	121.0	C212—C213—H213	121.2
C115—C114—C113	121.6 (3)	C215—C214—C213	121.5 (6)
C115—C114—Cl14	119.1 (3)	C215—C214—Cl24	118.6 (6)
C113—C114—Cl14	119.3 (4)	C213—C214—Cl24	119.2 (7)
C114—C115—C116	119.1 (4)	C214—C215—C216	119.1 (7)
C114—C115—H115	120.4	C214—C215—H215	120.5
C116—C115—H115	120.4	C216—C215—H215	120.5
C111—C116—C115	121.6 (4)	C211—C216—C215	120.7 (9)
C111—C116—H116	119.2	C211—C216—H216	119.6
C115—C116—H116	119.2	C215—C216—H216	119.6

C136—C131—C132	116.9 (3)	C236—C231—C232	116.9 (6)
C136—C131—C13	123.3 (4)	C236—C231—C23	123.3 (8)
C132—C131—C13	119.4 (5)	C232—C231—C23	118.6 (9)
C133—C132—C131	121.3 (5)	C233—C232—C231	120.6 (9)
C133—C132—H132	119.4	C233—C232—H232	119.7
C131—C132—H132	119.4	C231—C232—H232	119.7
C134—C133—C132	121.5 (4)	C234—C233—C232	121.1 (7)
C134—C133—H133	119.3	C234—C233—H233	119.5
C132—C133—H133	119.3	C232—C233—H233	119.5
C133—C134—C135	117.1 (3)	C233—C234—C235	116.7 (5)
C133—C134—C137	122.3 (4)	C233—C234—C237	121.2 (7)
C135—C134—C137	120.4 (4)	C235—C234—C237	120.3 (7)
C134—C135—C136	121.9 (4)	C234—C235—C236	121.9 (6)
C134—C135—H135	119.1	C234—C235—H235	119.1
C136—C135—H135	119.1	C236—C235—H235	119.1
C131—C136—C135	121.0 (3)	C231—C236—C235	120.9 (6)
C131—C136—H136	119.5	C231—C236—H236	119.6
C135—C136—H136	119.5	C235—C236—H236	119.6
C138—C137—C139	128.1 (5)	C238—C237—C239	127.2 (8)
C138—C137—C134	117.1 (5)	C238—C237—C234	117.8 (7)
C139—C137—C134	113.2 (4)	C239—C237—C234	112.4 (6)
C137—C138—H18A	120.0	C237—C238—H28A	120.0
C137—C138—H18B	120.0	C237—C238—H28B	120.0
H18A—C138—H18B	120.0	H28A—C238—H28B	120.0
C137—C139—H19A	109.5	C237—C239—H29A	109.5
C137—C139—H19B	109.5	C237—C239—H29B	109.5
H19A—C139—H19B	109.5	H29A—C239—H29B	109.5
C137—C139—H19C	109.5	C237—C239—H29C	109.5
H19A—C139—H19C	109.5	H29A—C239—H29C	109.5
H19B—C139—H19C	109.5	H29B—C239—H29C	109.5

O111—C11—C12—C13	12 (4)	O211—C21—C22—C23	7 (8)
C111—C11—C12—C13	−177 (2)	C211—C21—C22—C23	−171 (4)
C11—C12—C13—C131	174 (2)	C21—C22—C23—C231	−156 (5)
O111—C11—C111—C116	46 (2)	O211—C21—C211—C216	40 (5)
C12—C11—C111—C116	−126 (3)	C22—C21—C211—C216	−142 (6)
O111—C11—C111—C112	−137 (3)	O211—C21—C211—C212	−136 (6)
C12—C11—C111—C112	51 (3)	C22—C21—C211—C212	42 (7)
C116—C111—C112—C113	−2 (5)	C216—C211—C212—C213	8 (10)
C11—C111—C112—C113	−179 (3)	C21—C211—C212—C213	−176 (6)
C116—C111—C112—Cl12	−178 (3)	C216—C211—C212—Cl22	−170 (7)
C11—C111—C112—Cl12	5 (5)	C21—C211—C212—Cl22	6 (10)
C111—C112—C113—C114	0 (6)	C211—C212—C213—C214	3 (13)
Cl12—C112—C113—C114	175 (3)	Cl22—C212—C213—C214	−179 (6)
C112—C113—C114—C115	1 (5)	C212—C213—C214—C215	−7 (10)
C112—C113—C114—Cl14	178 (3)	C212—C213—C214—Cl24	−177 (6)
C113—C114—C115—C116	0 (3)	C213—C214—C215—C216	0 (7)
Cl14—C114—C115—C116	−177 (2)	Cl24—C214—C215—C216	170 (4)

C112—C111—C116—C115	4 (4)	C212—C211—C216—C215	−15 (8)
C11—C111—C116—C115	−179 (2)	C21—C211—C216—C215	168 (5)
C114—C115—C116—C111	−3 (4)	C214—C215—C216—C211	11 (8)
C12—C13—C131—C136	4 (4)	C22—C23—C231—C236	−12 (8)
C12—C13—C131—C132	176 (3)	C22—C23—C231—C232	−179 (5)
C136—C131—C132—C133	−4 (3)	C236—C231—C232—C233	11 (7)
C13—C131—C132—C133	−177 (2)	C23—C231—C232—C233	179 (5)
C131—C132—C133—C134	7 (3)	C231—C232—C233—C234	−18 (6)
C132—C133—C134—C135	−6.5 (19)	C232—C233—C234—C235	14 (4)
C132—C133—C134—C137	178.5 (14)	C232—C233—C234—C237	179 (3)
C133—C134—C135—C136	3.8 (14)	C233—C234—C235—C236	−6 (3)
C137—C134—C135—C136	178.9 (8)	C237—C234—C235—C236	−170.8 (18)
C132—C131—C136—C135	1 (3)	C232—C231—C236—C235	−3 (6)
C13—C131—C136—C135	173.6 (18)	C23—C231—C236—C235	−170 (4)
C134—C135—C136—C131	−1.3 (19)	C234—C235—C236—C231	0 (4)
C133—C134—C137—C138	86.2 (10)	C233—C234—C237—C238	75 (2)
C135—C134—C137—C138	−88.7 (9)	C235—C234—C237—C238	−120.8 (19)
C133—C134—C137—C139	−80.6 (12)	C233—C234—C237—C239	−89 (3)
C135—C134—C137—C139	104.5 (12)	C235—C234—C237—C239	76 (3)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C12—H12···O111^{i}	0.93	2.49	3.15 (2)	128
C232—H232···*Cg*1^{ii}	0.93	2.76	3.67 (4)	166

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

(II) (5*RS*)-3-(4-Chlorophenyl)-5-[4-(propan-2-yl)phenyl]cyclohex-2-en-1-one

Crystal data

$C_{21}H_{21}ClO$
M_r = 324.83
Monoclinic, $P2_1/n$
a = 6.6259 (8) Å
b = 8.6868 (11) Å
c = 30.472 (4) Å
β = 91.765 (4)°
V = 1753.1 (4) Å^3
Z = 4
$F(000)$ = 688
D_x = 1.231 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 4357 reflections
θ = 3.1–28.4°
μ = 0.22 mm^{-1}
T = 293 K
Block, colourless
0.19 × 0.15 × 0.11 mm

Data collection

Bruker APEXII CCD diffractometer
Radiation source: sealed tube
Graphite monochromator
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2003)
T_{min} = 0.663, T_{max} = 0.976
21442 measured reflections
3272 independent reflections
1931 reflections with $I > 2\sigma(I)$
R_{int} = 0.082
θ_{max} = 25.6°, θ_{min} = 3.1°
h = −7→8
k = −10→10
l = −36→36

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.059$
$wR(F^2) = 0.138$
$S = 1.03$
3272 reflections
279 parameters
60 restraints
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0413P)^2 + 0.9497P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.31$ e Å^{-3}
$\Delta\rho_{min} = -0.17$ e Å^{-3}
Extinction correction: *SHELXL2014* (Sheldrick, 2015), $Fc^* = kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.0100 (15)

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.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}	Occ. (<1)
C11	0.091 (3)	0.455 (4)	0.4102 (4)	0.0585 (17)	0.662 (4)
O11	−0.076 (5)	0.399 (6)	0.4156 (6)	0.0840 (8)	0.662 (4)
C12	0.199 (4)	0.541 (5)	0.4441 (4)	0.0547 (12)	0.662 (4)
H12	0.1387	0.5518	0.4711	0.066*	0.662 (4)
C13	0.3797 (16)	0.6067 (15)	0.4392 (2)	0.040 (2)	0.662 (4)
C14	0.4731 (13)	0.6029 (14)	0.3949 (2)	0.0505 (18)	0.662 (4)
H14A	0.5770	0.5242	0.3952	0.061*	0.662 (4)
H14B	0.5381	0.7011	0.3899	0.061*	0.662 (4)
C15	0.3230 (6)	0.5705 (5)	0.35659 (12)	0.0503 (10)	0.662 (4)
H15	0.2306	0.6583	0.3540	0.060*	0.662 (4)
C16	0.1990 (7)	0.4287 (6)	0.36797 (17)	0.0557 (16)	0.662 (4)
H16A	0.1012	0.4076	0.3444	0.067*	0.662 (4)
H16B	0.2873	0.3401	0.3712	0.067*	0.662 (4)
C131	0.4866 (14)	0.6938 (18)	0.4747 (2)	0.042 (2)	0.662 (4)
C132	0.389 (2)	0.737 (4)	0.5126 (5)	0.060 (2)	0.662 (4)
H132	0.2599	0.7003	0.5172	0.072*	0.662 (4)
C133	0.4794 (15)	0.8342 (15)	0.5436 (2)	0.060 (3)	0.662 (4)
H133	0.4071	0.8686	0.5673	0.072*	0.662 (4)
C134	0.6760 (16)	0.8788 (19)	0.5389 (3)	0.057 (3)	0.662 (4)
Cl14	0.7993 (13)	0.9868 (6)	0.5794 (2)	0.0839 (10)	0.662 (4)
C135	0.784 (2)	0.827 (3)	0.5034 (4)	0.062 (3)	0.662 (4)
H135	0.9198	0.8508	0.5012	0.075*	0.662 (4)
C136	0.6858 (14)	0.7401 (16)	0.4713 (2)	0.051 (2)	0.662 (4)
H136	0.7559	0.7115	0.4466	0.061*	0.662 (4)
C151	0.4284 (8)	0.5538 (5)	0.31305 (13)	0.0474 (13)	0.662 (4)
C156	0.3465 (9)	0.6243 (6)	0.2764 (2)	0.0593 (16)	0.662 (4)
H156	0.2277	0.6803	0.2788	0.071*	0.662 (4)
C155	0.4332 (11)	0.6151 (7)	0.2366 (2)	0.0573 (18)	0.662 (4)

H155	0.3723	0.6660	0.2128	0.069*	0.662 (4)
C154	0.6030 (14)	0.5355 (10)	0.23038 (19)	0.0448 (16)	0.662 (4)
C153	0.6942 (19)	0.4645 (16)	0.2673 (3)	0.067 (2)	0.662 (4)
H153	0.8163	0.4130	0.2646	0.080*	0.662 (4)
C152	0.6029 (16)	0.4705 (19)	0.3082 (3)	0.070 (2)	0.662 (4)
H152	0.6605	0.4180	0.3320	0.084*	0.662 (4)
C157	0.6890 (15)	0.5244 (12)	0.1850 (2)	0.0602 (17)	0.662 (4)
H157	0.6346	0.6118	0.1681	0.072*	0.662 (4)
C158	0.613 (3)	0.382 (2)	0.1624 (6)	0.091 (4)	0.662 (4)
H18A	0.4684	0.3784	0.1633	0.136*	0.662 (4)
H18B	0.6531	0.3825	0.1324	0.136*	0.662 (4)
H18C	0.6690	0.2928	0.1770	0.136*	0.662 (4)
C159	0.9162 (14)	0.539 (2)	0.1850 (5)	0.088 (4)	0.662 (4)
H19A	0.9567	0.6315	0.1998	0.132*	0.662 (4)
H19B	0.9760	0.4517	0.1999	0.132*	0.662 (4)
H19C	0.9601	0.5413	0.1553	0.132*	0.662 (4)
C21	0.071 (6)	0.459 (7)	0.4108 (7)	0.0585 (17)	0.338 (4)
O21	−0.089 (9)	0.395 (13)	0.4187 (11)	0.0840 (8)	0.338 (4)
C22	0.188 (8)	0.543 (10)	0.4439 (8)	0.0547 (12)	0.338 (4)
H22	0.1469	0.5371	0.4728	0.066*	0.338 (4)
C23	0.353 (4)	0.627 (3)	0.4359 (4)	0.040 (2)	0.338 (4)
C24	0.443 (3)	0.617 (3)	0.3914 (4)	0.0505 (18)	0.338 (4)
H24A	0.5883	0.6112	0.3951	0.061*	0.338 (4)
H24B	0.4113	0.7117	0.3755	0.061*	0.338 (4)
C25	0.3695 (12)	0.4802 (9)	0.3632 (2)	0.0503 (10)	0.338 (4)
H25	0.4228	0.3862	0.3771	0.060*	0.338 (4)
C26	0.1399 (12)	0.4725 (16)	0.3641 (4)	0.0557 (16)	0.338 (4)
H26A	0.0826	0.5645	0.3506	0.067*	0.338 (4)
H26B	0.0923	0.3842	0.3473	0.067*	0.338 (4)
C231	0.464 (3)	0.715 (4)	0.4705 (5)	0.042 (2)	0.338 (4)
C232	0.381 (4)	0.745 (8)	0.5109 (11)	0.060 (2)	0.338 (4)
H232	0.2438	0.7271	0.5144	0.072*	0.338 (4)
C233	0.496 (3)	0.802 (4)	0.5461 (5)	0.060 (3)	0.338 (4)
H233	0.4412	0.8079	0.5738	0.072*	0.338 (4)
C234	0.690 (3)	0.850 (4)	0.5398 (6)	0.057 (3)	0.338 (4)
Cl24	0.821 (3)	0.9531 (14)	0.5803 (5)	0.0839 (10)	0.338 (4)
C235	0.771 (4)	0.838 (7)	0.4984 (9)	0.062 (3)	0.338 (4)
H235	0.8963	0.8805	0.4929	0.075*	0.338 (4)
C236	0.662 (3)	0.763 (4)	0.4654 (5)	0.051 (2)	0.338 (4)
H236	0.7230	0.7448	0.4389	0.061*	0.338 (4)
C251	0.4490 (15)	0.4875 (11)	0.3168 (2)	0.0474 (13)	0.338 (4)
C252	0.640 (3)	0.433 (4)	0.3101 (5)	0.070 (2)	0.338 (4)
H252	0.7184	0.4001	0.3342	0.084*	0.338 (4)
C253	0.718 (4)	0.425 (4)	0.2694 (5)	0.067 (2)	0.338 (4)
H253	0.8370	0.3712	0.2655	0.080*	0.338 (4)
C254	0.628 (3)	0.494 (3)	0.2347 (4)	0.0448 (16)	0.338 (4)
C255	0.434 (2)	0.5552 (17)	0.2403 (4)	0.0573 (18)	0.338 (4)
H255	0.3622	0.5963	0.2163	0.069*	0.338 (4)

C256	0.3471 (18)	0.5551 (15)	0.2818 (3)	0.0593 (16)	0.338 (4)
H256	0.2216	0.6003	0.2856	0.071*	0.338 (4)
C257	0.719 (3)	0.489 (3)	0.1897 (4)	0.0602 (17)	0.338 (4)
H257	0.6675	0.5796	0.1740	0.072*	0.338 (4)
C258	0.640 (7)	0.352 (5)	0.1645 (11)	0.091 (4)	0.338 (4)
H28A	0.4957	0.3506	0.1652	0.136*	0.338 (4)
H28B	0.6813	0.3594	0.1346	0.136*	0.338 (4)
H28C	0.6940	0.2598	0.1775	0.136*	0.338 (4)
C259	0.946 (3)	0.502 (5)	0.1906 (11)	0.088 (4)	0.338 (4)
H29A	0.9862	0.5912	0.2072	0.132*	0.338 (4)
H29B	1.0040	0.4117	0.2040	0.132*	0.338 (4)
H29C	0.9913	0.5112	0.1611	0.132*	0.338 (4)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C11	0.050 (4)	0.064 (3)	0.0622 (18)	0.000 (4)	0.0137 (16)	0.0109 (15)
O11	0.055 (4)	0.111 (3)	0.087 (3)	−0.022 (3)	0.0196 (17)	0.007 (4)
C12	0.054 (3)	0.0629 (19)	0.0480 (15)	0.002 (3)	0.0191 (14)	0.0050 (14)
C13	0.040 (3)	0.040 (4)	0.0403 (16)	0.017 (3)	0.0084 (18)	0.013 (2)
C14	0.047 (3)	0.062 (3)	0.0432 (18)	−0.003 (2)	0.0121 (19)	0.0041 (18)
C15	0.054 (2)	0.052 (3)	0.0451 (19)	0.008 (2)	0.0101 (17)	0.004 (2)
C16	0.048 (3)	0.064 (4)	0.056 (2)	0.000 (3)	0.004 (2)	−0.001 (2)
C131	0.050 (3)	0.040 (5)	0.035 (2)	0.013 (3)	0.0084 (18)	0.015 (3)
C132	0.058 (2)	0.078 (4)	0.0448 (19)	0.006 (3)	0.0131 (18)	0.004 (3)
C133	0.082 (3)	0.057 (7)	0.0414 (17)	0.025 (3)	0.0137 (19)	0.004 (3)
C134	0.087 (3)	0.036 (7)	0.0466 (16)	0.014 (3)	0.0017 (17)	0.004 (2)
Cl14	0.123 (2)	0.062 (3)	0.0658 (6)	0.005 (2)	−0.0093 (8)	−0.0123 (17)
C135	0.067 (2)	0.065 (5)	0.055 (4)	−0.004 (3)	0.006 (3)	0.005 (4)
C136	0.057 (3)	0.057 (5)	0.040 (2)	0.007 (3)	0.011 (2)	0.006 (3)
C151	0.053 (2)	0.043 (4)	0.0460 (18)	−0.009 (3)	0.0099 (16)	−0.004 (2)
C156	0.0504 (19)	0.071 (5)	0.057 (2)	0.002 (3)	0.0087 (17)	0.011 (3)
C155	0.0540 (19)	0.067 (6)	0.051 (2)	−0.004 (3)	0.0052 (16)	0.015 (3)
C154	0.055 (3)	0.040 (6)	0.0398 (18)	−0.002 (2)	0.0040 (19)	0.0021 (19)
C153	0.076 (3)	0.066 (8)	0.059 (2)	0.027 (3)	0.017 (2)	−0.002 (2)
C152	0.076 (5)	0.089 (9)	0.0456 (18)	0.021 (3)	0.005 (2)	0.005 (2)
C157	0.069 (3)	0.064 (5)	0.048 (2)	0.012 (3)	0.010 (2)	0.008 (2)
C158	0.110 (6)	0.101 (8)	0.062 (3)	0.016 (5)	0.005 (3)	−0.029 (4)
C159	0.066 (4)	0.126 (10)	0.073 (4)	0.012 (4)	0.020 (4)	0.014 (5)
C21	0.050 (4)	0.064 (3)	0.0622 (18)	0.000 (4)	0.0137 (16)	0.0109 (15)
O21	0.055 (4)	0.111 (3)	0.087 (3)	−0.022 (3)	0.0196 (17)	0.007 (4)
C22	0.054 (3)	0.0629 (19)	0.0480 (15)	0.002 (3)	0.0191 (14)	0.0050 (14)
C23	0.040 (3)	0.040 (4)	0.0403 (16)	0.017 (3)	0.0084 (18)	0.013 (2)
C24	0.047 (3)	0.062 (3)	0.0432 (18)	−0.003 (2)	0.0121 (19)	0.0041 (18)
C25	0.054 (2)	0.052 (3)	0.0451 (19)	0.008 (2)	0.0101 (17)	0.004 (2)
C26	0.048 (3)	0.064 (4)	0.056 (2)	0.000 (3)	0.004 (2)	−0.001 (2)
C231	0.050 (3)	0.040 (5)	0.035 (2)	0.013 (3)	0.0084 (18)	0.015 (3)
C232	0.058 (2)	0.078 (4)	0.0448 (19)	0.006 (3)	0.0131 (18)	0.004 (3)

C233	0.082 (3)	0.057 (7)	0.0414 (17)	0.025 (3)	0.0137 (19)	0.004 (3)
C234	0.087 (3)	0.036 (7)	0.0466 (16)	0.014 (3)	0.0017 (17)	0.004 (2)
Cl24	0.123 (2)	0.062 (3)	0.0658 (6)	0.005 (2)	−0.0093 (8)	−0.0123 (17)
C235	0.067 (2)	0.065 (5)	0.055 (4)	−0.004 (3)	0.006 (3)	0.005 (4)
C236	0.057 (3)	0.057 (5)	0.040 (2)	0.007 (3)	0.011 (2)	0.006 (3)
C251	0.053 (2)	0.043 (4)	0.0460 (18)	−0.009 (3)	0.0099 (16)	−0.004 (2)
C252	0.076 (5)	0.089 (9)	0.0456 (18)	0.021 (3)	0.005 (2)	0.005 (2)
C253	0.076 (3)	0.066 (8)	0.059 (2)	0.027 (3)	0.017 (2)	−0.002 (2)
C254	0.055 (3)	0.040 (6)	0.0398 (18)	−0.002 (2)	0.0040 (19)	0.0021 (19)
C255	0.0540 (19)	0.067 (6)	0.051 (2)	−0.004 (3)	0.0052 (16)	0.015 (3)
C256	0.0504 (19)	0.071 (5)	0.057 (2)	0.002 (3)	0.0087 (17)	0.011 (3)
C257	0.069 (3)	0.064 (5)	0.048 (2)	0.012 (3)	0.010 (2)	0.008 (2)
C258	0.110 (6)	0.101 (8)	0.062 (3)	0.016 (5)	0.005 (3)	−0.029 (4)
C259	0.066 (4)	0.126 (10)	0.073 (4)	0.012 (4)	0.020 (4)	0.014 (5)

Geometric parameters (Å, º)

C11—O11	1.227 (5)	C21—O21	1.227 (6)
C11—C12	1.448 (4)	C21—C22	1.448 (5)
C11—C16	1.511 (8)	C21—C26	1.511 (10)
C12—C13	1.339 (6)	C22—C23	1.339 (8)
C12—H12	0.9300	C22—H22	0.9300
C13—C131	1.482 (4)	C23—C231	1.482 (5)
C13—C14	1.501 (3)	C23—C24	1.502 (4)
C14—C15	1.537 (7)	C24—C25	1.537 (8)
C14—H14A	0.9700	C24—H24A	0.9700
C14—H14B	0.9700	C24—H24B	0.9700
C15—C151	1.525 (4)	C25—C26	1.523 (6)
C15—C16	1.527 (5)	C25—C251	1.526 (5)
C15—H15	0.9800	C25—H25	0.9800
C16—H16A	0.9700	C26—H26A	0.9700
C16—H16B	0.9700	C26—H26B	0.9700
C131—C136	1.386 (4)	C231—C236	1.386 (5)
C131—C132	1.392 (5)	C231—C232	1.393 (5)
C132—C133	1.386 (9)	C232—C233	1.387 (10)
C132—H132	0.9300	C232—H232	0.9300
C133—C134	1.371 (4)	C233—C234	1.372 (5)
C133—H133	0.9300	C233—H233	0.9300
C134—C135	1.389 (9)	C234—C235	1.390 (10)
C134—Cl14	1.734 (3)	C234—Cl24	1.736 (4)
C135—C136	1.381 (5)	C235—C236	1.381 (5)
C135—H135	0.9300	C235—H235	0.9300
C136—H136	0.9300	C236—H236	0.9300
C151—C156	1.372 (7)	C251—C252	1.372 (9)
C151—C152	1.376 (7)	C251—C256	1.376 (9)
C156—C155	1.360 (8)	C252—C253	1.361 (9)
C156—H156	0.9300	C252—H252	0.9300
C155—C154	1.339 (8)	C253—C254	1.338 (9)

C155—H155	0.9300	C253—H253	0.9300
C154—C153	1.402 (8)	C254—C255	1.405 (10)
C154—C157	1.516 (4)	C254—C257	1.516 (5)
C153—C152	1.404 (8)	C255—C256	1.405 (9)
C153—H153	0.9300	C255—H255	0.9300
C152—H152	0.9300	C256—H256	0.9300
C157—C158	1.497 (5)	C257—C258	1.495 (6)
C157—C159	1.510 (5)	C257—C259	1.507 (6)
C157—H157	0.9800	C257—H257	0.9800
C158—H18A	0.9600	C258—H28A	0.9600
C158—H18B	0.9600	C258—H28B	0.9600
C158—H18C	0.9600	C258—H28C	0.9600
C159—H19A	0.9600	C259—H29A	0.9600
C159—H19B	0.9600	C259—H29B	0.9600
C159—H19C	0.9600	C259—H29C	0.9600

O11—C11—C12	122.7 (6)	O21—C21—C22	122.5 (6)
O11—C11—C16	120.5 (7)	O21—C21—C26	120.4 (11)
C12—C11—C16	116.7 (6)	C22—C21—C26	116.7 (9)
C13—C12—C11	124.4 (5)	C23—C22—C21	124.4 (9)
C13—C12—H12	117.8	C23—C22—H22	117.8
C11—C12—H12	117.8	C21—C22—H22	117.8
C12—C13—C131	123.0 (3)	C22—C23—C231	122.9 (6)
C12—C13—C14	119.1 (3)	C22—C23—C24	118.9 (6)
C131—C13—C14	117.8 (3)	C231—C23—C24	117.8 (5)
C13—C14—C15	114.4 (5)	C23—C24—C25	114.8 (7)
C13—C14—H14A	108.7	C23—C24—H24A	108.6
C15—C14—H14A	108.7	C25—C24—H24A	108.6
C13—C14—H14B	108.7	C23—C24—H24B	108.6
C15—C14—H14B	108.7	C25—C24—H24B	108.6
H14A—C14—H14B	107.6	H24A—C24—H24B	107.6
C151—C15—C16	112.6 (3)	C26—C25—C251	113.0 (5)
C151—C15—C14	112.1 (3)	C26—C25—C24	108.8 (7)
C16—C15—C14	108.3 (5)	C251—C25—C24	111.9 (6)
C151—C15—H15	107.9	C26—C25—H25	107.6
C16—C15—H15	107.9	C251—C25—H25	107.6
C14—C15—H15	107.9	C24—C25—H25	107.6
C11—C16—C15	110.2 (9)	C21—C26—C25	110.4 (14)
C11—C16—H16A	109.6	C21—C26—H26A	109.6
C15—C16—H16A	109.6	C25—C26—H26A	109.6
C11—C16—H16B	109.6	C21—C26—H26B	109.6
C15—C16—H16B	109.6	C25—C26—H26B	109.6
H16A—C16—H16B	108.1	H26A—C26—H26B	108.1
C136—C131—C132	116.7 (3)	C236—C231—C232	116.3 (5)
C136—C131—C13	121.9 (3)	C236—C231—C23	122.1 (5)
C132—C131—C13	121.3 (3)	C232—C231—C23	121.5 (5)
C133—C132—C131	121.9 (5)	C233—C232—C231	121.9 (8)
C133—C132—H132	119.1	C233—C232—H232	119.1

C131—C132—H132	119.1	C231—C232—H232	119.1
C134—C133—C132	119.5 (4)	C234—C233—C232	119.6 (7)
C134—C133—H133	120.3	C234—C233—H233	120.2
C132—C133—H133	120.3	C232—C233—H233	120.2
C133—C134—C135	120.1 (4)	C233—C234—C235	119.6 (6)
C133—C134—Cl14	120.5 (3)	C233—C234—Cl24	120.4 (5)
C135—C134—Cl14	119.2 (4)	C235—C234—Cl24	119.1 (7)
C136—C135—C134	119.1 (5)	C236—C235—C234	119.2 (7)
C136—C135—H135	120.4	C236—C235—H235	120.4
C134—C135—H135	120.4	C234—C235—H235	120.4
C135—C136—C131	122.3 (4)	C235—C236—C231	122.4 (5)
C135—C136—H136	118.9	C235—C236—H236	118.8
C131—C136—H136	118.9	C231—C236—H236	118.8
C156—C151—C152	117.4 (3)	C252—C251—C256	117.7 (5)
C156—C151—C15	119.1 (4)	C252—C251—C25	118.0 (7)
C152—C151—C15	123.6 (4)	C256—C251—C25	124.1 (6)
C155—C156—C151	122.2 (5)	C253—C252—C251	122.0 (9)
C155—C156—H156	118.9	C253—C252—H252	119.0
C151—C156—H156	118.9	C251—C252—H252	119.0
C154—C155—C156	122.4 (5)	C254—C253—C252	121.8 (9)
C154—C155—H155	118.8	C254—C253—H253	119.1
C156—C155—H155	118.8	C252—C253—H253	119.1
C155—C154—C153	117.2 (3)	C253—C254—C255	117.4 (5)
C155—C154—C157	120.2 (5)	C253—C254—C257	121.3 (8)
C153—C154—C157	122.6 (5)	C255—C254—C257	120.9 (9)
C154—C153—C152	120.6 (5)	C256—C255—C254	120.7 (8)
C154—C153—H153	119.7	C256—C255—H255	119.7
C152—C153—H153	119.7	C254—C255—H255	119.7
C151—C152—C153	120.1 (5)	C251—C256—C255	119.6 (8)
C151—C152—H152	119.9	C251—C256—H256	120.2
C153—C152—H152	119.9	C255—C256—H256	120.2
C158—C157—C159	112.8 (4)	C258—C257—C259	113.6 (6)
C158—C157—C154	110.0 (4)	C258—C257—C254	110.2 (6)
C159—C157—C154	113.4 (4)	C259—C257—C254	114.1 (6)
C158—C157—H157	106.7	C258—C257—H257	106.1
C159—C157—H157	106.7	C259—C257—H257	106.1
C154—C157—H157	106.7	C254—C257—H257	106.1
C157—C158—H18A	109.5	C257—C258—H28A	109.5
C157—C158—H18B	109.5	C257—C258—H28B	109.5
H18A—C158—H18B	109.5	H28A—C258—H28B	109.5
C157—C158—H18C	109.5	C257—C258—H28C	109.5
H18A—C158—H18C	109.5	H28A—C258—H28C	109.5
H18B—C158—H18C	109.5	H28B—C258—H28C	109.5
C157—C159—H19A	109.5	C257—C259—H29A	109.5
C157—C159—H19B	109.5	C257—C259—H29B	109.5
H19A—C159—H19B	109.5	H29A—C259—H29B	109.5
C157—C159—H19C	109.5	C257—C259—H29C	109.5
H19A—C159—H19C	109.5	H29A—C259—H29C	109.5

H19B—C159—H19C	109.5	H29B—C259—H29C	109.5
O11—C11—C12—C13	−179 (5)	O21—C21—C22—C23	−173 (10)
C16—C11—C12—C13	5 (6)	C26—C21—C22—C23	−1 (11)
C11—C12—C13—C131	−180 (3)	C21—C22—C23—C231	178 (6)
C11—C12—C13—C14	5 (5)	C21—C22—C23—C24	−10 (11)
C12—C13—C14—C15	19 (3)	C22—C23—C24—C25	−15 (6)
C131—C13—C14—C15	−156.6 (11)	C231—C23—C24—C25	158 (2)
C13—C14—C15—C151	−174.9 (8)	C23—C24—C25—C26	48 (2)
C13—C14—C15—C16	−50.0 (11)	C23—C24—C25—C251	173.8 (19)
O11—C11—C16—C15	146 (4)	O21—C21—C26—C25	−152 (8)
C12—C11—C16—C15	−37 (3)	C22—C21—C26—C25	36 (7)
C151—C15—C16—C11	−177.3 (13)	C251—C25—C26—C21	178 (3)
C14—C15—C16—C11	58.1 (14)	C24—C25—C26—C21	−57 (3)
C12—C13—C131—C136	170 (3)	C22—C23—C231—C236	161 (6)
C14—C13—C131—C136	−15 (2)	C24—C23—C231—C236	−12 (5)
C12—C13—C131—C132	−12 (4)	C22—C23—C231—C232	−15 (8)
C14—C13—C131—C132	163 (2)	C24—C23—C231—C232	172 (5)
C136—C131—C132—C133	6 (4)	C236—C231—C232—C233	−8 (9)
C13—C131—C132—C133	−172 (2)	C23—C231—C232—C233	168 (5)
C131—C132—C133—C134	−5 (4)	C231—C232—C233—C234	9 (9)
C132—C133—C134—C135	0 (3)	C232—C233—C234—C235	−1 (7)
C132—C133—C134—Cl14	−175 (2)	C232—C233—C234—Cl24	168 (5)
C133—C134—C135—C136	5 (3)	C233—C234—C235—C236	−7 (8)
Cl14—C134—C135—C136	179.5 (19)	Cl24—C234—C235—C236	−176 (4)
C134—C135—C136—C131	−4 (3)	C234—C235—C236—C231	8 (8)
C132—C131—C136—C135	−1 (3)	C232—C231—C236—C235	0 (7)
C13—C131—C136—C135	177 (2)	C23—C231—C236—C235	−176 (4)
C16—C15—C151—C156	101.2 (5)	C26—C25—C251—C252	−154 (2)
C14—C15—C151—C156	−136.3 (7)	C24—C25—C251—C252	83 (3)
C16—C15—C151—C152	−78.6 (10)	C26—C25—C251—C256	30.9 (13)
C14—C15—C151—C152	43.9 (12)	C24—C25—C251—C256	−92.3 (16)
C152—C151—C156—C155	−0.7 (11)	C256—C251—C252—C253	−9 (5)
C15—C151—C156—C155	179.5 (5)	C25—C251—C252—C253	176 (3)
C151—C156—C155—C154	0.7 (11)	C251—C252—C253—C254	11 (6)
C156—C155—C154—C153	−2.1 (15)	C252—C253—C254—C255	−9 (5)
C156—C155—C154—C157	177.8 (8)	C252—C253—C254—C257	178 (3)
C155—C154—C153—C152	4 (2)	C253—C254—C255—C256	5 (4)
C157—C154—C153—C152	−176.3 (14)	C257—C254—C255—C256	177.9 (19)
C156—C151—C152—C153	2.2 (19)	C252—C251—C256—C255	5 (3)
C15—C151—C152—C153	−178.0 (12)	C25—C251—C256—C255	−179.9 (12)
C154—C153—C152—C151	−4 (2)	C254—C255—C256—C251	−3 (2)
C155—C154—C157—C158	−93.5 (11)	C253—C254—C257—C258	91 (3)
C153—C154—C157—C158	86.4 (15)	C255—C254—C257—C258	−82 (3)
C155—C154—C157—C159	139.1 (11)	C253—C254—C257—C259	−38 (3)
C153—C154—C157—C159	−41.0 (15)	C255—C254—C257—C259	149 (2)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C132—H132···O11^{i}	0.93	2.56	3.28 (4)	134
C232—H232···O21^{i}	0.93	2.54	3.18 (7)	126

Symmetry code: (i) −*x*, −*y*+1, −*z*+1.

(III) Methyl (1*RS*, 6*SR*)-4-(4-chlorophenyl)-6-[4-(propan-2-yl)phenyl]-2-oxocyclohex-3-ene-1-carboxylate

Crystal data

$C_{23}H_{23}ClO_3$
M_r = 382.86
Monoclinic, $P2_1/c$
a = 17.4871 (11) Å
b = 11.5900 (8) Å
c = 10.0167 (7) Å
β = 95.300 (2)°
V = 2021.5 (2) Å^3
Z = 4
$F(000)$ = 808
D_x = 1.258 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 4991 reflections
θ = 3.0–28.3°
μ = 0.21 mm^{-1}
T = 296 K
Block, colourless
0.26 × 0.19 × 0.15 mm

Data collection

Bruker APEXII CCD diffractometer
Radiation source: sealed tube
Graphite monochromator
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2003)
T_{min} = 0.834, T_{max} = 0.969
20398 measured reflections
4991 independent reflections
3514 reflections with $I > 2\sigma(I)$
R_{int} = 0.041
θ_{max} = 28.3°, θ_{min} = 3.0°
h = −22→23
k = −15→15
l = −13→12

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.059
$wR(F^2)$ = 0.161
S = 1.04
4991 reflections
326 parameters
70 restraints
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0564P)^2 + 1.5457P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.40 e Å^{-3}
$\Delta\rho_{min}$ = −0.34 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.

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

	x	*y*	*z*	U_{iso}*/U_{eq}	Occ. (<1)
C11	0.67921 (11)	0.59732 (19)	0.5968 (2)	0.0337 (5)	0.923 (3)
H11	0.6653	0.5238	0.6364	0.040*	0.923 (3)
C12	0.61362 (12)	0.6821 (2)	0.6066 (2)	0.0382 (6)	0.923 (3)

C13	0.54809 (13)	0.6725 (5)	0.5041 (4)	0.0362 (4)	0.923 (3)
H13	0.5047	0.7170	0.5142	0.043*	0.923 (3)
C14	0.54750 (14)	0.6035 (4)	0.3975 (3)	0.0290 (6)	0.923 (3)
C15	0.61648 (13)	0.5296 (3)	0.3766 (3)	0.0325 (5)	0.923 (3)
H15A	0.6222	0.5246	0.2813	0.039*	0.923 (3)
H15B	0.6078	0.4522	0.4089	0.039*	0.923 (3)
C16	0.69109 (11)	0.57701 (18)	0.4490 (2)	0.0309 (5)	0.923 (3)
H16	0.7009	0.6522	0.4093	0.037*	0.923 (3)
C111	0.75056 (13)	0.6434 (2)	0.6760 (2)	0.0407 (6)	0.923 (3)
O111	0.78881 (15)	0.7201 (2)	0.6392 (3)	0.0617 (6)	0.923 (3)
O112	0.76537 (13)	0.5883 (2)	0.7915 (2)	0.0607 (6)	0.923 (3)
C112	0.8325 (2)	0.6272 (4)	0.8761 (4)	0.0795 (10)	0.923 (3)
H12A	0.8380	0.5820	0.9567	0.119*	0.923 (3)
H12B	0.8775	0.6185	0.8288	0.119*	0.923 (3)
H12C	0.8263	0.7070	0.8986	0.119*	0.923 (3)
O121	0.61591 (15)	0.7541 (3)	0.6946 (3)	0.0678 (12)	0.923 (3)
C141	0.48122 (15)	0.5978 (3)	0.2927 (3)	0.0290 (7)	0.923 (3)
C142	0.4247 (2)	0.6836 (4)	0.2815 (5)	0.0377 (10)	0.923 (3)
H142	0.4265	0.7428	0.3444	0.045*	0.923 (3)
C143	0.36581 (13)	0.6821 (2)	0.1783 (3)	0.0408 (8)	0.923 (3)
H143	0.3289	0.7400	0.1714	0.049*	0.923 (3)
C144	0.36301 (13)	0.5935 (2)	0.0861 (2)	0.0384 (7)	0.923 (3)
Cl14	0.29254 (6)	0.59274 (11)	−0.0478 (1)	0.0614 (3)	0.923 (3)
C145	0.41591 (15)	0.5058 (2)	0.0971 (3)	0.0440 (9)	0.923 (3)
H145	0.4125	0.4451	0.0363	0.053*	0.923 (3)
C146	0.4746 (3)	0.5090 (4)	0.2005 (6)	0.0398 (11)	0.923 (3)
H146	0.5105	0.4495	0.2077	0.048*	0.923 (3)
C161	0.75879 (11)	0.4996 (2)	0.4274 (2)	0.0323 (5)	0.923 (3)
C162	0.80382 (14)	0.5237 (2)	0.3245 (3)	0.0435 (6)	0.923 (3)
H162	0.7929	0.5881	0.2709	0.052*	0.923 (3)
C163	0.86508 (19)	0.4531 (3)	0.3000 (3)	0.0549 (8)	0.923 (3)
H163	0.8940	0.4705	0.2292	0.066*	0.923 (3)
C164	0.88409 (17)	0.3575 (3)	0.3784 (4)	0.0528 (8)	0.923 (3)
C165	0.8390 (2)	0.3333 (4)	0.4809 (5)	0.0586 (10)	0.923 (3)
H165	0.8502	0.2691	0.5349	0.070*	0.923 (3)
C166	0.7770 (2)	0.4033 (4)	0.5051 (4)	0.0511 (7)	0.923 (3)
H166	0.7475	0.3849	0.5746	0.061*	0.923 (3)
C167	0.95445 (19)	0.2861 (3)	0.3538 (4)	0.0814 (12)	0.923 (3)
H167	0.9644	0.3016	0.2607	0.098*	0.923 (3)
C168	1.0243 (2)	0.3329 (7)	0.4403 (5)	0.125 (2)	0.923 (3)
H18A	1.0275	0.4149	0.4285	0.188*	0.923 (3)
H18B	1.0192	0.3160	0.5329	0.188*	0.923 (3)
H18C	1.0701	0.2972	0.4139	0.188*	0.923 (3)
C169	0.9422 (3)	0.1599 (4)	0.3619 (6)	0.136 (2)	0.923 (3)
H19A	0.9886	0.1203	0.3455	0.204*	0.923 (3)
H19B	0.9283	0.1404	0.4496	0.204*	0.923 (3)
H19C	0.9017	0.1373	0.2959	0.204*	0.923 (3)
C21	0.6907 (8)	0.6585 (14)	0.5361 (17)	0.0337 (5)	0.077 (3)

H21	0.7033	0.6996	0.4555	0.040*	0.077 (3)
C22	0.6177 (10)	0.711 (2)	0.580 (3)	0.0382 (6)	0.077 (3)
C23	0.5453 (10)	0.673 (5)	0.507 (4)	0.0362 (4)	0.077 (3)
H23	0.4993	0.7021	0.5326	0.043*	0.077 (3)
C24	0.5427 (15)	0.597 (4)	0.406 (4)	0.0290 (6)	0.077 (3)
C25	0.6120 (13)	0.525 (3)	0.383 (3)	0.0325 (5)	0.077 (3)
H25A	0.6319	0.5492	0.3008	0.039*	0.077 (3)
H25B	0.5960	0.4450	0.3724	0.039*	0.077 (3)
C26	0.6767 (7)	0.5320 (14)	0.4976 (18)	0.0309 (5)	0.077 (3)
H26	0.6596	0.4917	0.5756	0.037*	0.077 (3)
C211	0.7564 (11)	0.6763 (18)	0.643 (2)	0.0407 (6)	0.077 (3)
O211	0.8025 (18)	0.752 (3)	0.640 (3)	0.0617 (6)	0.077 (3)
O212	0.7534 (17)	0.604 (3)	0.745 (3)	0.0607 (6)	0.077 (3)
C212	0.814 (3)	0.616 (6)	0.854 (4)	0.0795 (10)	0.077 (3)
H22A	0.8068	0.5602	0.9222	0.119*	0.077 (3)
H22B	0.8631	0.6028	0.8196	0.119*	0.077 (3)
H22C	0.8132	0.6922	0.8907	0.119*	0.077 (3)
O221	0.6194 (16)	0.782 (4)	0.669 (5)	0.0678 (12)	0.077 (3)
C241	0.475 (2)	0.588 (4)	0.304 (5)	0.0290 (7)	0.077 (3)
C242	0.419 (3)	0.674 (5)	0.290 (7)	0.0377 (10)	0.077 (3)
H242	0.4240	0.7394	0.3447	0.045*	0.077 (3)
C243	0.3550 (16)	0.664 (3)	0.197 (3)	0.0408 (8)	0.077 (3)
H243	0.3139	0.7148	0.1985	0.049*	0.077 (3)
C244	0.3537 (17)	0.577 (3)	0.103 (3)	0.0384 (7)	0.077 (3)
Cl24	0.2789 (8)	0.5690 (16)	−0.0233 (14)	0.0614 (3)	0.077 (3)
C245	0.407 (2)	0.490 (3)	0.115 (4)	0.0440 (9)	0.077 (3)
H245	0.4017	0.4255	0.0599	0.053*	0.077 (3)
C246	0.470 (4)	0.500 (6)	0.211 (8)	0.0398 (11)	0.077 (3)
H246	0.5089	0.4458	0.2122	0.048*	0.077 (3)
C261	0.7482 (10)	0.4714 (16)	0.458 (2)	0.0323 (5)	0.077 (3)
C262	0.7817 (14)	0.510 (3)	0.346 (3)	0.0435 (6)	0.077 (3)
H262	0.7552	0.5610	0.2873	0.052*	0.077 (3)
C263	0.8543 (18)	0.472 (4)	0.321 (4)	0.0549 (8)	0.077 (3)
H263	0.8814	0.5119	0.2597	0.066*	0.077 (3)
C264	0.8868 (16)	0.376 (3)	0.385 (4)	0.0528 (8)	0.077 (3)
C265	0.852 (3)	0.334 (5)	0.493 (7)	0.0586 (10)	0.077 (3)
H265	0.8735	0.2721	0.5417	0.070*	0.077 (3)
C266	0.785 (3)	0.384 (4)	0.532 (5)	0.0511 (7)	0.077 (3)
H266	0.7639	0.3570	0.6083	0.061*	0.077 (3)
C267	0.9673 (13)	0.338 (3)	0.357 (4)	0.0814 (12)	0.077 (3)
H267	0.9891	0.4018	0.3092	0.098*	0.077 (3)
C268	1.017 (2)	0.327 (8)	0.491 (5)	0.125 (2)	0.077 (3)
H28A	1.0128	0.3964	0.5421	0.188*	0.077 (3)
H28B	0.9992	0.2627	0.5404	0.188*	0.077 (3)
H28C	1.0694	0.3148	0.4744	0.188*	0.077 (3)
C269	0.968 (3)	0.236 (4)	0.268 (6)	0.136 (2)	0.077 (3)
H29A	1.0200	0.2167	0.2542	0.204*	0.077 (3)
H29B	0.9439	0.1721	0.3077	0.204*	0.077 (3)

H29C	0.9403	0.2541	0.1828	0.204*	0.077 (3)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C11	0.0297 (9)	0.0379 (12)	0.0332 (10)	−0.0004 (8)	0.0011 (8)	−0.0046 (9)
C12	0.0333 (10)	0.0449 (14)	0.0370 (12)	0.0009 (9)	0.0068 (8)	−0.0095 (10)
C13	0.0270 (9)	0.0414 (11)	0.0407 (10)	0.0049 (8)	0.0060 (8)	−0.0077 (9)
C14	0.0247 (8)	0.0299 (11)	0.0332 (10)	−0.0015 (8)	0.0056 (8)	0.0011 (9)
C15	0.0283 (9)	0.0353 (11)	0.0335 (10)	0.0020 (8)	0.0003 (8)	−0.0063 (8)
C16	0.0259 (9)	0.0338 (11)	0.0330 (10)	0.0002 (8)	0.0028 (7)	−0.0023 (8)
C111	0.0338 (10)	0.0491 (15)	0.0390 (14)	0.0011 (10)	0.0029 (9)	−0.0109 (11)
O111	0.0524 (13)	0.0661 (18)	0.0651 (12)	−0.0226 (11)	−0.0019 (9)	−0.0048 (12)
O112	0.0551 (12)	0.0767 (14)	0.0469 (13)	−0.0079 (10)	−0.0145 (9)	0.0018 (11)
C112	0.065 (3)	0.101 (3)	0.067 (2)	−0.0024 (19)	−0.0294 (15)	−0.014 (2)
O121	0.0518 (10)	0.084 (2)	0.0655 (18)	0.0163 (11)	−0.0052 (10)	−0.0443 (18)
C141	0.0224 (9)	0.0314 (11)	0.0337 (11)	−0.0017 (8)	0.0051 (9)	0.0018 (10)
C142	0.0332 (12)	0.0330 (13)	0.0462 (14)	0.0002 (11)	0.0007 (11)	−0.0040 (12)
C143	0.0324 (11)	0.0392 (14)	0.0501 (14)	0.0040 (10)	−0.0001 (10)	0.0063 (11)
C144	0.0290 (11)	0.0492 (14)	0.0361 (12)	−0.0057 (10)	−0.0013 (9)	0.0036 (11)
Cl14	0.0478 (5)	0.0821 (7)	0.0505 (5)	−0.0013 (4)	−0.0147 (4)	0.0015 (4)
C145	0.0378 (13)	0.0495 (15)	0.0443 (15)	−0.0026 (11)	0.0010 (11)	−0.0136 (13)
C146	0.0330 (12)	0.0412 (15)	0.0451 (16)	0.0038 (12)	0.0035 (12)	−0.0077 (13)
C161	0.0255 (9)	0.0357 (12)	0.0351 (12)	0.0004 (8)	−0.0012 (8)	−0.0036 (9)
C162	0.0380 (13)	0.0497 (14)	0.0431 (13)	0.0059 (11)	0.0062 (10)	0.0008 (10)
C163	0.0432 (14)	0.069 (2)	0.0542 (17)	0.0111 (12)	0.0149 (11)	−0.0047 (15)
C164	0.0348 (11)	0.0578 (18)	0.0652 (16)	0.0117 (11)	0.0022 (10)	−0.0096 (14)
C165	0.0479 (19)	0.0510 (15)	0.076 (2)	0.0165 (15)	0.0014 (17)	0.0085 (14)
C166	0.0417 (14)	0.053 (2)	0.060 (2)	0.0107 (11)	0.0114 (14)	0.0103 (13)
C167	0.0595 (18)	0.087 (3)	0.099 (2)	0.0361 (19)	0.0181 (17)	−0.004 (2)
C168	0.0409 (18)	0.197 (5)	0.139 (5)	0.043 (3)	0.010 (2)	0.003 (5)
C169	0.144 (4)	0.092 (3)	0.178 (5)	0.069 (3)	0.043 (4)	−0.009 (3)
C21	0.0297 (9)	0.0379 (12)	0.0332 (10)	−0.0004 (8)	0.0011 (8)	−0.0046 (9)
C22	0.0333 (10)	0.0449 (14)	0.0370 (12)	0.0009 (9)	0.0068 (8)	−0.0095 (10)
C23	0.0270 (9)	0.0414 (11)	0.0407 (10)	0.0049 (8)	0.0060 (8)	−0.0077 (9)
C24	0.0247 (8)	0.0299 (11)	0.0332 (10)	−0.0015 (8)	0.0056 (8)	0.0011 (9)
C25	0.0283 (9)	0.0353 (11)	0.0335 (10)	0.0020 (8)	0.0003 (8)	−0.0063 (8)
C26	0.0259 (9)	0.0338 (11)	0.0330 (10)	0.0002 (8)	0.0028 (7)	−0.0023 (8)
C211	0.0338 (10)	0.0491 (15)	0.0390 (14)	0.0011 (10)	0.0029 (9)	−0.0109 (11)
O211	0.0524 (13)	0.0661 (18)	0.0651 (12)	−0.0226 (11)	−0.0019 (9)	−0.0048 (12)
O212	0.0551 (12)	0.0767 (14)	0.0469 (13)	−0.0079 (10)	−0.0145 (9)	0.0018 (11)
C212	0.065 (3)	0.101 (3)	0.067 (2)	−0.0024 (19)	−0.0294 (15)	−0.014 (2)
O221	0.0518 (10)	0.084 (2)	0.0655 (18)	0.0163 (11)	−0.0052 (10)	−0.0443 (18)
C241	0.0224 (9)	0.0314 (11)	0.0337 (11)	−0.0017 (8)	0.0051 (9)	0.0018 (10)
C242	0.0332 (12)	0.0330 (13)	0.0462 (14)	0.0002 (11)	0.0007 (11)	−0.0040 (12)
C243	0.0324 (11)	0.0392 (14)	0.0501 (14)	0.0040 (10)	−0.0001 (10)	0.0063 (11)
C244	0.0290 (11)	0.0492 (14)	0.0361 (12)	−0.0057 (10)	−0.0013 (9)	0.0036 (11)
Cl24	0.0478 (5)	0.0821 (7)	0.0505 (5)	−0.0013 (4)	−0.0147 (4)	0.0015 (4)

C245	0.0378 (13)	0.0495 (15)	0.0443 (15)	−0.0026 (11)	0.0010 (11)	−0.0136 (13)
C246	0.0330 (12)	0.0412 (15)	0.0451 (16)	0.0038 (12)	0.0035 (12)	−0.0077 (13)
C261	0.0255 (9)	0.0357 (12)	0.0351 (12)	0.0004 (8)	−0.0012 (8)	−0.0036 (9)
C262	0.0380 (13)	0.0497 (14)	0.0431 (13)	0.0059 (11)	0.0062 (10)	0.0008 (10)
C263	0.0432 (14)	0.069 (2)	0.0542 (17)	0.0111 (12)	0.0149 (11)	−0.0047 (15)
C264	0.0348 (11)	0.0578 (18)	0.0652 (16)	0.0117 (11)	0.0022 (10)	−0.0096 (14)
C265	0.0479 (19)	0.0510 (15)	0.076 (2)	0.0165 (15)	0.0014 (17)	0.0085 (14)
C266	0.0417 (14)	0.053 (2)	0.060 (2)	0.0107 (11)	0.0114 (14)	0.0103 (13)
C267	0.0595 (18)	0.087 (3)	0.099 (2)	0.0361 (19)	0.0181 (17)	−0.004 (2)
C268	0.0409 (18)	0.197 (5)	0.139 (5)	0.043 (3)	0.010 (2)	0.003 (5)
C269	0.144 (4)	0.092 (3)	0.178 (5)	0.069 (3)	0.043 (4)	−0.009 (3)

Geometric parameters (Å, º)

C11—C111	1.513 (3)	C21—C211	1.510 (5)
C11—C12	1.521 (3)	C21—C22	1.518 (5)
C11—C16	1.532 (3)	C21—C26	1.529 (6)
C11—H11	0.9800	C21—H21	0.9800
C12—O121	1.212 (3)	C22—O221	1.211 (5)
C12—C13	1.470 (3)	C22—C23	1.472 (6)
C13—C14	1.335 (3)	C23—C24	1.336 (5)
C13—H13	0.9300	C23—H23	0.9300
C14—C141	1.491 (2)	C24—C241	1.493 (5)
C14—C15	1.509 (2)	C24—C25	1.508 (5)
C15—C16	1.535 (3)	C25—C26	1.536 (6)
C15—H15A	0.9700	C25—H25A	0.9700
C15—H15B	0.9700	C25—H25B	0.9700
C16—C161	1.516 (3)	C26—C261	1.520 (5)
C16—H16	0.9800	C26—H26	0.9800
C111—O111	1.191 (3)	C211—O211	1.193 (6)
C111—O112	1.326 (3)	C211—O212	1.326 (6)
O112—C112	1.455 (3)	O212—C212	1.455 (6)
C112—H12A	0.9600	C212—H22A	0.9600
C112—H12B	0.9600	C212—H22B	0.9600
C112—H12C	0.9600	C212—H22C	0.9600
C141—C146	1.381 (3)	C241—C246	1.381 (5)
C141—C142	1.399 (3)	C241—C242	1.400 (5)
C142—C143	1.391 (3)	C242—C243	1.392 (6)
C142—H142	0.9300	C242—H242	0.9300
C143—C144	1.379 (3)	C243—C244	1.380 (5)
C143—H143	0.9300	C243—H243	0.9300
C144—C145	1.371 (3)	C244—C245	1.372 (6)
C144—Cl14	1.736 (2)	C244—Cl24	1.736 (5)
C145—C146	1.389 (3)	C245—C246	1.390 (6)
C145—H145	0.9300	C245—H245	0.9300
C146—H146	0.9300	C246—H246	0.9300
C161—C166	1.381 (3)	C261—C266	1.379 (6)
C161—C162	1.382 (3)	C261—C262	1.381 (6)

C162—C163	1.388 (3)	C262—C263	1.389 (6)
C162—H162	0.9300	C262—H262	0.9300
C163—C164	1.380 (4)	C263—C264	1.382 (6)
C163—H163	0.9300	C263—H263	0.9300
C164—C165	1.380 (4)	C264—C265	1.382 (6)
C164—C167	1.521 (3)	C264—C267	1.524 (6)
C165—C166	1.393 (4)	C265—C266	1.393 (6)
C165—H165	0.9300	C265—H265	0.9300
C166—H166	0.9300	C266—H266	0.9300
C167—C169	1.482 (6)	C267—C269	1.482 (8)
C167—C168	1.531 (6)	C267—C268	1.532 (8)
C167—H167	0.9800	C267—H267	0.9800
C168—H18A	0.9600	C268—H28A	0.9600
C168—H18B	0.9600	C268—H28B	0.9600
C168—H18C	0.9600	C268—H28C	0.9600
C169—H19A	0.9600	C269—H29A	0.9600
C169—H19B	0.9600	C269—H29B	0.9600
C169—H19C	0.9600	C269—H29C	0.9600

C111—C11—C12	109.16 (18)	C211—C21—C22	110.1 (7)
C111—C11—C16	112.47 (17)	C211—C21—C26	113.8 (7)
C12—C11—C16	109.41 (17)	C22—C21—C26	109.9 (7)
C111—C11—H11	108.6	C211—C21—H21	107.6
C12—C11—H11	108.6	C22—C21—H21	107.6
C16—C11—H11	108.6	C26—C21—H21	107.6
O121—C12—C13	122.1 (2)	O221—C22—C23	122.2 (8)
O121—C12—C11	121.2 (2)	O221—C22—C21	121.7 (7)
C13—C12—C11	116.74 (18)	C23—C22—C21	116.1 (6)
C14—C13—C12	123.52 (19)	C24—C23—C22	122.9 (9)
C14—C13—H13	118.2	C24—C23—H23	118.6
C12—C13—H13	118.2	C22—C23—H23	118.6
C13—C14—C141	122.54 (17)	C23—C24—C241	122.3 (7)
C13—C14—C15	120.27 (17)	C23—C24—C25	120.4 (6)
C141—C14—C15	117.16 (16)	C241—C24—C25	117.0 (7)
C14—C15—C16	112.76 (18)	C24—C25—C26	113.9 (7)
C14—C15—H15A	109.0	C24—C25—H25A	108.8
C16—C15—H15A	109.0	C26—C25—H25A	108.8
C14—C15—H15B	109.0	C24—C25—H25B	108.8
C16—C15—H15B	109.0	C26—C25—H25B	108.8
H15A—C15—H15B	107.8	H25A—C25—H25B	107.7
C161—C16—C11	113.92 (16)	C261—C26—C21	113.2 (7)
C161—C16—C15	111.22 (17)	C261—C26—C25	110.2 (7)
C11—C16—C15	109.0 (2)	C21—C26—C25	109.2 (8)
C161—C16—H16	107.5	C261—C26—H26	108.0
C11—C16—H16	107.5	C21—C26—H26	108.0
C15—C16—H16	107.5	C25—C26—H26	108.0
O111—C111—O112	124.0 (2)	O211—C211—O212	123.6 (8)
O111—C111—C11	124.1 (3)	O211—C211—C21	123.8 (9)

O112—C111—C11	111.9 (2)	O212—C211—C21	112.3 (7)
C111—O112—C112	116.1 (2)	C211—O212—C212	116.2 (9)
O112—C112—H12A	109.5	O212—C212—H22A	109.5
O112—C112—H12B	109.5	O212—C212—H22B	109.5
H12A—C112—H12B	109.5	H22A—C212—H22B	109.5
O112—C112—H12C	109.5	O212—C212—H22C	109.5
H12A—C112—H12C	109.5	H22A—C212—H22C	109.5
H12B—C112—H12C	109.5	H22B—C212—H22C	109.5
C146—C141—C142	117.49 (18)	C246—C241—C242	117.2 (7)
C146—C141—C14	121.01 (17)	C246—C241—C24	121.0 (6)
C142—C141—C14	121.48 (18)	C242—C241—C24	121.5 (7)
C143—C142—C141	121.3 (2)	C243—C242—C241	121.2 (8)
C143—C142—H142	119.3	C243—C242—H242	119.4
C141—C142—H142	119.3	C241—C242—H242	119.4
C144—C143—C142	119.04 (19)	C244—C243—C242	118.9 (8)
C144—C143—H143	120.5	C244—C243—H243	120.6
C142—C143—H143	120.5	C242—C243—H243	120.6
C145—C144—C143	121.04 (19)	C245—C244—C243	120.6 (7)
C145—C144—Cl14	118.97 (17)	C245—C244—Cl24	119.1 (6)
C143—C144—Cl14	119.98 (17)	C243—C244—Cl24	120.0 (6)
C144—C145—C146	119.2 (2)	C244—C245—C246	119.0 (7)
C144—C145—H145	120.4	C244—C245—H245	120.5
C146—C145—H145	120.4	C246—C245—H245	120.5
C141—C146—C145	121.9 (2)	C241—C246—C245	121.9 (8)
C141—C146—H146	119.1	C241—C246—H246	119.0
C145—C146—H146	119.1	C245—C246—H246	119.0
C166—C161—C162	117.7 (2)	C266—C261—C262	117.6 (6)
C166—C161—C16	122.7 (2)	C266—C261—C26	123.2 (7)
C162—C161—C16	119.5 (2)	C262—C261—C26	119.1 (7)
C161—C162—C163	121.0 (2)	C261—C262—C263	120.7 (8)
C161—C162—H162	119.5	C261—C262—H262	119.6
C163—C162—H162	119.5	C263—C262—H262	119.6
C164—C163—C162	121.4 (2)	C264—C263—C262	120.7 (8)
C164—C163—H163	119.3	C264—C263—H263	119.6
C162—C163—H163	119.3	C262—C263—H263	119.6
C163—C164—C165	117.5 (2)	C263—C264—C265	117.0 (7)
C163—C164—C167	120.0 (3)	C263—C264—C267	119.7 (9)
C165—C164—C167	122.4 (3)	C265—C264—C267	121.7 (9)
C164—C165—C166	121.2 (3)	C264—C265—C266	121.3 (7)
C164—C165—H165	119.4	C264—C265—H265	119.3
C166—C165—H165	119.4	C266—C265—H265	119.3
C161—C166—C165	121.1 (2)	C261—C266—C265	120.8 (7)
C161—C166—H166	119.5	C261—C266—H266	119.6
C165—C166—H166	119.5	C265—C266—H266	119.6
C169—C167—C164	113.9 (3)	C269—C267—C264	113.5 (9)
C169—C167—C168	115.5 (4)	C269—C267—C268	115.4 (9)
C164—C167—C168	109.3 (3)	C264—C267—C268	108.8 (8)
C169—C167—H167	105.8	C269—C267—H267	106.1

C164—C167—H167	105.8	C264—C267—H267	106.1
C168—C167—H167	105.8	C268—C267—H267	106.1
C167—C168—H18A	109.5	C267—C268—H28A	109.5
C167—C168—H18B	109.5	C267—C268—H28B	109.5
H18A—C168—H18B	109.5	H28A—C268—H28B	109.5
C167—C168—H18C	109.5	C267—C268—H28C	109.5
H18A—C168—H18C	109.5	H28A—C268—H28C	109.5
H18B—C168—H18C	109.5	H28B—C268—H28C	109.5
C167—C169—H19A	109.5	C267—C269—H29A	109.5
C167—C169—H19B	109.5	C267—C269—H29B	109.5
H19A—C169—H19B	109.5	H29A—C269—H29B	109.5
C167—C169—H19C	109.5	C267—C269—H29C	109.5
H19A—C169—H19C	109.5	H29A—C269—H29C	109.5
H19B—C169—H19C	109.5	H29B—C269—H29C	109.5
C111—C11—C12—O121	−18.8 (3)	C211—C21—C22—O221	18 (5)
C16—C11—C12—O121	−142.3 (3)	C26—C21—C22—O221	144 (5)
C111—C11—C12—C13	160.9 (3)	C211—C21—C22—C23	−163 (3)
C16—C11—C12—C13	37.4 (4)	C26—C21—C22—C23	−37 (4)
O121—C12—C13—C14	172.0 (4)	O221—C22—C23—C24	178 (6)
C11—C12—C13—C14	−7.7 (6)	C21—C22—C23—C24	−1 (7)
C12—C13—C14—C141	−178.1 (4)	C22—C23—C24—C241	−158 (5)
C12—C13—C14—C15	0.0 (7)	C22—C23—C24—C25	16 (8)
C13—C14—C15—C16	−23.3 (5)	C23—C24—C25—C26	10 (6)
C141—C14—C15—C16	154.9 (3)	C241—C24—C25—C26	−176 (4)
C111—C11—C16—C161	54.7 (2)	C211—C21—C26—C261	−53.7 (13)
C12—C11—C16—C161	176.15 (17)	C22—C21—C26—C261	−177.7 (12)
C111—C11—C16—C15	179.6 (2)	C211—C21—C26—C25	−176.9 (15)
C12—C11—C16—C15	−59.0 (2)	C22—C21—C26—C25	59.1 (15)
C14—C15—C16—C161	179.1 (3)	C24—C25—C26—C261	−171 (3)
C14—C15—C16—C11	52.7 (4)	C24—C25—C26—C21	−47 (3)
C12—C11—C111—O111	−75.3 (3)	C22—C21—C211—O211	−98 (4)
C16—C11—C111—O111	46.3 (3)	C26—C21—C211—O211	138 (4)
C12—C11—C111—O112	104.6 (2)	C22—C21—C211—O212	77 (3)
C16—C11—C111—O112	−133.8 (2)	C26—C21—C211—O212	−47 (3)
O111—C111—O112—C112	0.7 (5)	O211—C211—O212—C212	−5 (7)
C11—C111—O112—C112	−179.3 (3)	C21—C211—O212—C212	−180 (4)
C13—C14—C141—C146	−165.6 (6)	C23—C24—C241—C246	−171 (8)
C15—C14—C141—C146	16.2 (6)	C25—C24—C241—C246	14 (8)
C13—C14—C141—C142	16.3 (7)	C23—C24—C241—C242	14 (9)
C15—C14—C141—C142	−161.9 (5)	C25—C24—C241—C242	−160 (7)
C146—C141—C142—C143	−2.6 (8)	C246—C241—C242—C243	7 (12)
C14—C141—C142—C143	175.6 (4)	C24—C241—C242—C243	−178 (6)
C141—C142—C143—C144	0.6 (7)	C241—C242—C243—C244	−10 (10)
C142—C143—C144—C145	1.8 (5)	C242—C243—C244—C245	12 (8)
C142—C143—C144—Cl14	−177.3 (4)	C242—C243—C244—Cl24	−174 (6)
C143—C144—C145—C146	−2.2 (5)	C243—C244—C245—C246	−10 (9)
Cl14—C144—C145—C146	176.9 (4)	Cl24—C244—C245—C246	176 (6)

C142—C141—C146—C145	2.2 (9)	C242—C241—C246—C245	−6 (13)
C14—C141—C146—C145	−176.0 (5)	C24—C241—C246—C245	180 (7)
C144—C145—C146—C141	0.1 (9)	C244—C245—C246—C241	7 (12)
C11—C16—C161—C166	39.4 (4)	C21—C26—C261—C266	113 (4)
C15—C16—C161—C166	−84.4 (4)	C25—C26—C261—C266	−124 (4)
C11—C16—C161—C162	−142.1 (2)	C21—C26—C261—C262	−63 (3)
C15—C16—C161—C162	94.2 (3)	C25—C26—C261—C262	60 (3)
C166—C161—C162—C163	0.2 (5)	C266—C261—C262—C263	−9 (6)
C16—C161—C162—C163	−178.4 (3)	C26—C261—C262—C263	168 (3)
C161—C162—C163—C164	−1.0 (5)	C261—C262—C263—C264	16 (6)
C162—C163—C164—C165	1.2 (6)	C262—C263—C264—C265	−13 (7)
C162—C163—C164—C167	−176.4 (4)	C262—C263—C264—C267	−179 (4)
C163—C164—C165—C166	−0.5 (7)	C263—C264—C265—C266	3 (9)
C167—C164—C165—C166	176.9 (5)	C267—C264—C265—C266	169 (7)
C162—C161—C166—C165	0.4 (6)	C262—C261—C266—C265	−1 (9)
C16—C161—C166—C165	179.0 (4)	C26—C261—C266—C265	−177 (6)
C164—C165—C166—C161	−0.2 (8)	C264—C265—C266—C261	4 (12)
C163—C164—C167—C169	−138.4 (5)	C263—C264—C267—C269	−103 (5)
C165—C164—C167—C169	44.2 (6)	C265—C264—C267—C269	91 (6)
C163—C164—C167—C168	90.7 (5)	C263—C264—C267—C268	127 (5)
C165—C164—C167—C168	−86.7 (5)	C265—C264—C267—C268	−39 (6)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C162—H162···O111^{i}	0.93	2.58	3.499 (4)	169
C25—H25*A*···O221^{i}	0.96	2.36	3.11 (6)	134
C11—H11···*Cg*2^{ii}	0.98	2.78	3.651 (3)	149

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

(IV) Ethyl 4″-bromo-4-fluoro-5′-hydroxy-1,1′:3′,1″-terphenyl-4′-carboxylate

Crystal data

$C_{21}H_{16}BrFO_3$
M_r = 415.24
Triclinic, $P\overline{1}$
a = 10.0725 (3) Å
b = 12.5793 (3) Å
c = 14.9438 (4) Å
α = 104.332 (1)°
β = 92.467 (1)°
γ = 94.565 (1)°
V = 1824.84 (9) Å^3
Z = 4
F(000) = 840
D_x = 1.511 Mg m^{-3}
Mo *Kα* radiation, *λ* = 0.71073 Å
Cell parameters from 13987 reflections
θ = 1.7–33.4°
μ = 2.28 mm^{-1}
T = 296 K
Block, colourless
0.45 × 0.45 × 0.45 mm

Data collection

Bruker APEXII CCD diffractometer
Graphite monochromator
φ and *ω* scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2003)
T_{min} = 0.215, T_{max} = 0.358
34603 measured reflections
8396 independent reflections

5320 reflections with $I > 2\sigma(I)$
$R_{int} = 0.034$
$\theta_{max} = 27.6°$, $\theta_{min} = 1.7°$

$h = -13 \rightarrow 12$
$k = -16 \rightarrow 16$
$l = -19 \rightarrow 18$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.043$
$wR(F^2) = 0.118$
$S = 1.03$
8396 reflections
478 parameters
0 restraints
Hydrogen site location: mixed

H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0477P)^2 + 0.8208P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.62$ e Å^{-3}
$\Delta\rho_{min} = -0.71$ e Å^{-3}
Extinction correction: *SHELXL2014* (Sheldrick, 2015), $Fc^* = kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.0048 (6)

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.

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

	x	*y*	*z*	U_{iso}*/U_{eq}
C111	0.7438 (3)	0.4110 (2)	0.02870 (16)	0.0537 (6)
C112	0.6640 (3)	0.3319 (2)	0.05730 (18)	0.0627 (7)
H112	0.5865	0.3515	0.0862	0.075*
C113	0.6970 (3)	0.2254 (3)	0.0438 (2)	0.0730 (9)
H113	0.6437	0.1735	0.0643	0.088*
C114	0.8098 (3)	0.1976 (3)	−0.0004 (2)	0.0712 (8)
F114	0.8412 (2)	0.09202 (16)	−0.01424 (15)	0.1008 (7)
C115	0.8904 (3)	0.2710 (3)	−0.0308 (2)	0.0728 (8)
H115	0.9665	0.2496	−0.0609	0.087*
C116	0.8573 (3)	0.3777 (3)	−0.01614 (18)	0.0634 (7)
H116	0.9120	0.4287	−0.0367	0.076*
C121	0.7093 (3)	0.5255 (2)	0.04528 (17)	0.0534 (6)
C122	0.6388 (3)	0.5722 (2)	0.12179 (17)	0.0517 (6)
H122	0.6144	0.5298	0.1624	0.062*
C123	0.6043 (2)	0.6786 (2)	0.13892 (17)	0.0500 (6)
C124	0.6402 (3)	0.7445 (2)	0.07832 (19)	0.0573 (7)
C125	0.7146 (3)	0.6980 (3)	0.0033 (2)	0.0667 (8)
C126	0.7472 (3)	0.5915 (3)	−0.01258 (18)	0.0626 (7)
H126	0.7958	0.5630	−0.0632	0.075*
O125	0.7573 (3)	0.7555 (2)	−0.05715 (18)	0.0963 (9)
H125	0.715 (5)	0.813 (4)	−0.045 (4)	0.145*
C131	0.5319 (3)	0.7191 (2)	0.22405 (17)	0.0485 (6)
C132	0.5948 (3)	0.7925 (2)	0.30136 (18)	0.0541 (6)
H132	0.6839	0.8179	0.3003	0.065*
C133	0.5281 (3)	0.8288 (2)	0.38011 (19)	0.0632 (7)

H133	0.5715	0.8786	0.4316	0.076*
C134	0.3983 (3)	0.7912 (2)	0.3821 (2)	0.0625 (8)
Br13	0.30565 (5)	0.84128 (4)	0.49028 (3)	0.10301 (18)
C135	0.3335 (3)	0.7169 (3)	0.3080 (2)	0.0788 (9)
H135	0.2450	0.6909	0.3104	0.095*
C136	0.4015 (3)	0.6805 (3)	0.2290 (2)	0.0720 (8)
H136	0.3581	0.6290	0.1785	0.086*
C127	0.5985 (3)	0.8562 (3)	0.0855 (2)	0.0678 (8)
O127	0.6350 (3)	0.9124 (2)	0.03382 (19)	0.0978 (8)
O128	0.5159 (2)	0.88957 (17)	0.14927 (15)	0.0712 (6)
C128	0.4673 (4)	0.9966 (3)	0.1563 (3)	0.0845 (10)
H18A	0.4197	0.9979	0.0988	0.101*
H18B	0.5412	1.0537	0.1691	0.101*
C129	0.3772 (5)	1.0151 (4)	0.2325 (3)	0.1152 (15)
H19A	0.4265	1.0176	0.2895	0.173*
H19B	0.3071	0.9560	0.2205	0.173*
H19C	0.3391	1.0837	0.2371	0.173*
C211	0.9069 (3)	0.2370 (2)	0.25683 (17)	0.0515 (6)
C212	0.8119 (3)	0.1662 (2)	0.2821 (2)	0.0629 (7)
H212	0.7443	0.1953	0.3184	0.076*
C213	0.8150 (4)	0.0540 (3)	0.2549 (2)	0.0757 (9)
H213	0.7511	0.0073	0.2729	0.091*
C214	0.9137 (4)	0.0125 (3)	0.2008 (2)	0.0767 (9)
F214	0.9156 (3)	−0.09838 (16)	0.17351 (16)	0.1110 (7)
C215	1.0078 (4)	0.0784 (3)	0.1735 (2)	0.0808 (10)
H215	1.0734	0.0478	0.1359	0.097*
C216	1.0058 (3)	0.1906 (3)	0.2016 (2)	0.0684 (8)
H216	1.0711	0.2361	0.1835	0.082*
C221	0.9022 (2)	0.3578 (2)	0.28631 (16)	0.0475 (6)
C222	0.8445 (2)	0.40629 (19)	0.36756 (16)	0.0451 (5)
H222	0.8097	0.3613	0.4034	0.054*
C223	0.8368 (2)	0.51852 (19)	0.39710 (16)	0.0423 (5)
C224	0.8946 (2)	0.5873 (2)	0.34509 (17)	0.0469 (6)
C225	0.9489 (3)	0.5377 (2)	0.26158 (17)	0.0547 (6)
C226	0.9521 (3)	0.4251 (2)	0.23327 (17)	0.0531 (6)
H226	0.9884	0.3941	0.1776	0.064*
O225	1.0025 (2)	0.59677 (19)	0.20535 (15)	0.0769 (6)
H225	0.990 (4)	0.662 (3)	0.234 (3)	0.115*
C231	0.7598 (2)	0.55789 (18)	0.47978 (16)	0.0416 (5)
C232	0.6439 (2)	0.60762 (19)	0.47208 (18)	0.0490 (6)
H232	0.6170	0.6203	0.4156	0.059*
C233	0.5676 (3)	0.6387 (2)	0.54666 (19)	0.0535 (6)
H233	0.4894	0.6716	0.5407	0.064*
C234	0.6081 (2)	0.62052 (19)	0.62961 (18)	0.0494 (6)
Br23	0.50253 (4)	0.66094 (3)	0.73227 (2)	0.08328 (15)
C235	0.7215 (3)	0.5699 (2)	0.63936 (18)	0.0586 (7)
H235	0.7474	0.5571	0.6960	0.070*
C236	0.7967 (3)	0.5382 (2)	0.56382 (18)	0.0535 (6)

H236	0.8732	0.5031	0.5696	0.064*
C227	0.9102 (3)	0.7084 (2)	0.3756 (2)	0.0574 (7)
O227	0.9440 (3)	0.76698 (18)	0.32523 (17)	0.0903 (7)
O228	0.8893 (2)	0.74953 (15)	0.46187 (15)	0.0705 (6)
C228	0.9047 (4)	0.8684 (2)	0.4957 (3)	0.0862 (11)
H28A	0.9984	0.8952	0.5017	0.103*
H28B	0.8589	0.9019	0.4527	0.103*
C229	0.8482 (5)	0.8968 (3)	0.5846 (3)	0.1020 (13)
H29A	0.8934	0.8627	0.6264	0.153*
H29B	0.7551	0.8714	0.5778	0.153*
H29C	0.8587	0.9754	0.6088	0.153*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C111	0.0517 (15)	0.0693 (17)	0.0351 (12)	−0.0027 (12)	0.0084 (11)	0.0054 (12)
C112	0.0617 (17)	0.0742 (19)	0.0483 (15)	−0.0008 (14)	0.0178 (13)	0.0079 (13)
C113	0.086 (2)	0.069 (2)	0.0596 (18)	−0.0056 (16)	0.0182 (16)	0.0083 (15)
C114	0.085 (2)	0.0658 (19)	0.0544 (17)	0.0069 (16)	0.0114 (16)	−0.0013 (14)
F114	0.1267 (18)	0.0747 (13)	0.0961 (15)	0.0228 (12)	0.0301 (13)	0.0041 (11)
C115	0.0664 (19)	0.083 (2)	0.0572 (17)	0.0068 (16)	0.0180 (15)	−0.0058 (15)
C116	0.0589 (17)	0.0757 (19)	0.0485 (15)	−0.0044 (14)	0.0148 (13)	0.0036 (13)
C121	0.0456 (14)	0.0735 (18)	0.0412 (13)	−0.0025 (12)	0.0055 (11)	0.0169 (12)
C122	0.0563 (15)	0.0598 (16)	0.0416 (13)	0.0000 (12)	0.0119 (11)	0.0177 (11)
C123	0.0446 (14)	0.0647 (16)	0.0441 (13)	−0.0019 (11)	0.0037 (11)	0.0218 (12)
C124	0.0512 (15)	0.0737 (18)	0.0557 (16)	0.0027 (13)	0.0047 (12)	0.0331 (14)
C125	0.0587 (17)	0.097 (2)	0.0564 (17)	0.0012 (16)	0.0095 (13)	0.0435 (16)
C126	0.0564 (16)	0.091 (2)	0.0439 (14)	0.0041 (15)	0.0154 (12)	0.0228 (14)
O125	0.0996 (19)	0.131 (2)	0.0887 (17)	0.0189 (15)	0.0418 (14)	0.0760 (17)
C131	0.0517 (15)	0.0508 (14)	0.0489 (14)	0.0057 (11)	0.0074 (11)	0.0226 (12)
C132	0.0569 (16)	0.0552 (15)	0.0533 (15)	0.0033 (12)	−0.0006 (12)	0.0210 (13)
C133	0.081 (2)	0.0573 (16)	0.0514 (16)	0.0156 (15)	−0.0026 (14)	0.0124 (13)
C134	0.080 (2)	0.0639 (17)	0.0543 (16)	0.0300 (15)	0.0203 (15)	0.0257 (14)
Br13	0.1318 (4)	0.1190 (3)	0.0773 (3)	0.0688 (3)	0.0504 (2)	0.0354 (2)
C135	0.0612 (19)	0.091 (2)	0.085 (2)	0.0021 (17)	0.0275 (17)	0.021 (2)
C136	0.0597 (18)	0.083 (2)	0.0641 (18)	−0.0120 (15)	0.0101 (14)	0.0052 (16)
C127	0.0573 (18)	0.086 (2)	0.073 (2)	0.0034 (15)	−0.0009 (15)	0.0463 (18)
O127	0.0971 (18)	0.1112 (19)	0.119 (2)	0.0211 (14)	0.0313 (15)	0.0844 (17)
O128	0.0809 (14)	0.0709 (13)	0.0776 (14)	0.0185 (11)	0.0125 (11)	0.0435 (11)
C128	0.087 (2)	0.074 (2)	0.109 (3)	0.0179 (18)	0.000 (2)	0.051 (2)
C129	0.134 (4)	0.099 (3)	0.134 (4)	0.058 (3)	0.041 (3)	0.050 (3)
C211	0.0549 (15)	0.0559 (15)	0.0410 (13)	0.0077 (12)	0.0082 (11)	0.0056 (11)
C212	0.0696 (18)	0.0532 (16)	0.0631 (17)	0.0081 (13)	0.0174 (14)	0.0063 (13)
C213	0.089 (2)	0.0580 (18)	0.075 (2)	0.0023 (16)	0.0127 (18)	0.0077 (15)
C214	0.100 (3)	0.0544 (18)	0.068 (2)	0.0187 (17)	0.0026 (18)	−0.0041 (15)
F214	0.148 (2)	0.0594 (11)	0.1143 (17)	0.0280 (12)	0.0165 (15)	−0.0069 (11)
C215	0.088 (2)	0.075 (2)	0.071 (2)	0.0286 (18)	0.0179 (18)	−0.0044 (17)
C216	0.073 (2)	0.0720 (19)	0.0576 (17)	0.0136 (15)	0.0193 (14)	0.0061 (14)

C221	0.0426 (13)	0.0579 (15)	0.0418 (13)	0.0043 (11)	0.0049 (10)	0.0119 (11)
C222	0.0449 (13)	0.0481 (13)	0.0452 (13)	0.0044 (10)	0.0126 (10)	0.0156 (11)
C223	0.0388 (12)	0.0455 (13)	0.0455 (13)	0.0064 (10)	0.0072 (10)	0.0153 (10)
C224	0.0423 (13)	0.0512 (14)	0.0512 (14)	0.0028 (10)	0.0063 (10)	0.0206 (11)
C225	0.0506 (15)	0.0712 (18)	0.0468 (14)	−0.0037 (12)	0.0068 (11)	0.0261 (13)
C226	0.0537 (15)	0.0663 (17)	0.0378 (13)	0.0015 (12)	0.0103 (11)	0.0105 (12)
O225	0.0946 (16)	0.0837 (15)	0.0603 (13)	−0.0087 (13)	0.0263 (11)	0.0351 (11)
C231	0.0397 (12)	0.0374 (12)	0.0499 (13)	0.0035 (9)	0.0111 (10)	0.0138 (10)
C232	0.0523 (15)	0.0471 (13)	0.0546 (14)	0.0129 (11)	0.0106 (11)	0.0224 (11)
C233	0.0475 (14)	0.0513 (14)	0.0679 (17)	0.0177 (11)	0.0158 (12)	0.0206 (13)
C234	0.0479 (14)	0.0447 (13)	0.0543 (15)	0.0053 (11)	0.0186 (11)	0.0071 (11)
Br23	0.0795 (2)	0.1001 (3)	0.0700 (2)	0.02292 (18)	0.03781 (17)	0.01091 (18)
C235	0.0568 (16)	0.0752 (18)	0.0465 (14)	0.0126 (14)	0.0081 (12)	0.0177 (13)
C236	0.0439 (14)	0.0662 (16)	0.0549 (15)	0.0171 (12)	0.0088 (11)	0.0188 (13)
C227	0.0509 (15)	0.0558 (15)	0.0710 (19)	0.0029 (12)	0.0118 (13)	0.0255 (14)
O227	0.119 (2)	0.0675 (13)	0.0990 (17)	0.0002 (13)	0.0289 (15)	0.0482 (13)
O228	0.0878 (15)	0.0432 (10)	0.0787 (14)	−0.0063 (9)	0.0260 (11)	0.0122 (9)
C228	0.097 (3)	0.0420 (16)	0.114 (3)	−0.0059 (16)	0.025 (2)	0.0098 (17)
C229	0.150 (4)	0.070 (2)	0.078 (2)	0.011 (2)	0.004 (2)	0.0029 (19)

Geometric parameters (Å, º)

C111—C112	1.389 (4)	C211—C212	1.384 (4)
C111—C116	1.391 (4)	C211—C216	1.392 (4)
C111—C121	1.473 (4)	C211—C221	1.478 (4)
C112—C113	1.375 (4)	C212—C213	1.372 (4)
C112—H112	0.9300	C212—H212	0.9300
C113—C114	1.365 (4)	C213—C214	1.363 (5)
C113—H113	0.9300	C213—H213	0.9300
C114—C115	1.356 (4)	C214—C215	1.352 (5)
C114—F114	1.357 (4)	C214—F214	1.355 (3)
C115—C116	1.376 (4)	C215—C216	1.371 (4)
C115—H115	0.9300	C215—H215	0.9300
C116—H116	0.9300	C216—H216	0.9300
C121—C126	1.384 (4)	C221—C226	1.377 (3)
C121—C122	1.400 (3)	C221—C222	1.391 (3)
C122—C123	1.375 (4)	C222—C223	1.380 (3)
C122—H122	0.9300	C222—H222	0.9300
C123—C124	1.410 (3)	C223—C224	1.409 (3)
C123—C131	1.492 (3)	C223—C231	1.488 (3)
C124—C125	1.406 (4)	C224—C225	1.405 (4)
C124—C127	1.478 (4)	C224—C227	1.471 (4)
C125—O125	1.353 (3)	C225—O225	1.354 (3)
C125—C126	1.371 (4)	C225—C226	1.378 (4)
C126—H126	0.9300	C226—H226	0.9300
O125—H125	0.85 (5)	O225—H225	0.85 (4)
C131—C136	1.375 (4)	C231—C236	1.380 (3)
C131—C132	1.380 (4)	C231—C232	1.381 (3)

C132—C133	1.377 (4)	C232—C233	1.375 (3)
C132—H132	0.9300	C232—H232	0.9300
C133—C134	1.358 (4)	C233—C234	1.366 (4)
C133—H133	0.9300	C233—H233	0.9300
C134—C135	1.362 (5)	C234—C235	1.371 (4)
C134—Br13	1.897 (3)	C234—Br23	1.893 (2)
C135—C136	1.388 (4)	C235—C236	1.380 (3)
C135—H135	0.9300	C235—H235	0.9300
C136—H136	0.9300	C236—H236	0.9300
C127—O127	1.219 (3)	C227—O227	1.217 (3)
C127—O128	1.304 (4)	C227—O228	1.298 (3)
O128—C128	1.450 (4)	O228—C228	1.449 (3)
C128—C129	1.472 (5)	C228—C229	1.442 (5)
C128—H18A	0.9700	C228—H28A	0.9700
C128—H18B	0.9700	C228—H28B	0.9700
C129—H19A	0.9600	C229—H29A	0.9600
C129—H19B	0.9600	C229—H29B	0.9600
C129—H19C	0.9600	C229—H29C	0.9600
C112—C111—C116	117.4 (3)	C212—C211—C216	117.7 (3)
C112—C111—C121	121.2 (2)	C212—C211—C221	121.0 (2)
C116—C111—C121	121.4 (2)	C216—C211—C221	121.2 (2)
C113—C112—C111	121.6 (3)	C213—C212—C211	121.6 (3)
C113—C112—H112	119.2	C213—C212—H212	119.2
C111—C112—H112	119.2	C211—C212—H212	119.2
C114—C113—C112	118.4 (3)	C214—C213—C212	118.5 (3)
C114—C113—H113	120.8	C214—C213—H213	120.7
C112—C113—H113	120.8	C212—C213—H213	120.7
C115—C114—F114	119.3 (3)	C215—C214—F214	119.7 (3)
C115—C114—C113	122.6 (3)	C215—C214—C213	122.1 (3)
F114—C114—C113	118.1 (3)	F214—C214—C213	118.3 (3)
C114—C115—C116	118.6 (3)	C214—C215—C216	119.5 (3)
C114—C115—H115	120.7	C214—C215—H215	120.3
C116—C115—H115	120.7	C216—C215—H215	120.3
C115—C116—C111	121.4 (3)	C215—C216—C211	120.7 (3)
C115—C116—H116	119.3	C215—C216—H216	119.7
C111—C116—H116	119.3	C211—C216—H216	119.7
C126—C121—C122	117.6 (3)	C226—C221—C222	118.1 (2)
C126—C121—C111	121.6 (2)	C226—C221—C211	121.0 (2)
C122—C121—C111	120.8 (2)	C222—C221—C211	120.8 (2)
C123—C122—C121	122.2 (2)	C223—C222—C221	122.7 (2)
C123—C122—H122	118.9	C223—C222—H222	118.7
C121—C122—H122	118.9	C221—C222—H222	118.7
C122—C123—C124	119.9 (2)	C222—C223—C224	118.8 (2)
C122—C123—C131	116.8 (2)	C222—C223—C231	116.86 (19)
C124—C123—C131	123.3 (2)	C224—C223—C231	124.2 (2)
C125—C124—C123	117.5 (3)	C225—C224—C223	118.3 (2)
C125—C124—C127	118.1 (2)	C225—C224—C227	117.5 (2)

C123—C124—C127	124.3 (3)	C223—C224—C227	124.1 (2)
O125—C125—C126	116.5 (3)	O225—C225—C226	116.3 (2)
O125—C125—C124	122.0 (3)	O225—C225—C224	122.6 (3)
C126—C125—C124	121.5 (2)	C226—C225—C224	121.1 (2)
C125—C126—C121	121.3 (3)	C221—C226—C225	120.9 (2)
C125—C126—H126	119.4	C221—C226—H226	119.6
C121—C126—H126	119.4	C225—C226—H226	119.6
C125—O125—H125	104 (3)	C225—O225—H225	102 (3)
C136—C131—C132	117.9 (2)	C236—C231—C232	118.5 (2)
C136—C131—C123	120.7 (2)	C236—C231—C223	120.9 (2)
C132—C131—C123	121.3 (2)	C232—C231—C223	120.3 (2)
C133—C132—C131	121.2 (3)	C233—C232—C231	121.0 (2)
C133—C132—H132	119.4	C233—C232—H232	119.5
C131—C132—H132	119.4	C231—C232—H232	119.5
C134—C133—C132	119.5 (3)	C234—C233—C232	119.2 (2)
C134—C133—H133	120.2	C234—C233—H233	120.4
C132—C133—H133	120.2	C232—C233—H233	120.4
C133—C134—C135	121.1 (3)	C233—C234—C235	121.3 (2)
C133—C134—Br13	119.7 (2)	C233—C234—Br23	119.57 (18)
C135—C134—Br13	119.1 (2)	C235—C234—Br23	119.1 (2)
C134—C135—C136	119.0 (3)	C234—C235—C236	118.9 (2)
C134—C135—H135	120.5	C234—C235—H235	120.5
C136—C135—H135	120.5	C236—C235—H235	120.5
C131—C136—C135	121.2 (3)	C231—C236—C235	120.9 (2)
C131—C136—H136	119.4	C231—C236—H236	119.5
C135—C136—H136	119.4	C235—C236—H236	119.5
O127—C127—O128	121.9 (3)	O227—C227—O228	121.6 (3)
O127—C127—C124	122.6 (3)	O227—C227—C224	123.2 (3)
O128—C127—C124	115.4 (2)	O228—C227—C224	115.2 (2)
C127—O128—C128	117.5 (2)	C227—O228—C228	117.4 (2)
O128—C128—C129	107.1 (3)	C229—C228—O228	108.1 (3)
O128—C128—H18A	110.3	C229—C228—H28A	110.1
C129—C128—H18A	110.3	O228—C228—H28A	110.1
O128—C128—H18B	110.3	C229—C228—H28B	110.1
C129—C128—H18B	110.3	O228—C228—H28B	110.1
H18A—C128—H18B	108.6	H28A—C228—H28B	108.4
C128—C129—H19A	109.5	C228—C229—H29A	109.5
C128—C129—H19B	109.5	C228—C229—H29B	109.5
H19A—C129—H19B	109.5	H29A—C229—H29B	109.5
C128—C129—H19C	109.5	C228—C229—H29C	109.5
H19A—C129—H19C	109.5	H29A—C229—H29C	109.5
H19B—C129—H19C	109.5	H29B—C229—H29C	109.5
C116—C111—C112—C113	1.4 (4)	C216—C211—C212—C213	0.7 (4)
C121—C111—C112—C113	−178.6 (3)	C221—C211—C212—C213	179.7 (3)
C111—C112—C113—C114	−1.2 (5)	C211—C212—C213—C214	−0.7 (5)
C112—C113—C114—C115	0.4 (5)	C212—C213—C214—C215	−0.1 (5)
C112—C113—C114—F114	−179.4 (3)	C212—C213—C214—F214	−179.5 (3)

F114—C114—C115—C116	−179.9 (3)	F214—C214—C215—C216	−179.7 (3)
C113—C114—C115—C116	0.3 (5)	C213—C214—C215—C216	0.8 (6)
C114—C115—C116—C111	−0.1 (5)	C214—C215—C216—C211	−0.8 (5)
C112—C111—C116—C115	−0.7 (4)	C212—C211—C216—C215	0.1 (4)
C121—C111—C116—C115	179.3 (3)	C221—C211—C216—C215	−178.9 (3)
C112—C111—C121—C126	−151.8 (3)	C212—C211—C221—C226	−150.9 (3)
C116—C111—C121—C126	28.2 (4)	C216—C211—C221—C226	28.0 (4)
C112—C111—C121—C122	29.5 (4)	C212—C211—C221—C222	27.7 (4)
C116—C111—C121—C122	−150.5 (3)	C216—C211—C221—C222	−153.3 (3)
C126—C121—C122—C123	1.7 (4)	C226—C221—C222—C223	−0.7 (4)
C111—C121—C122—C123	−179.6 (2)	C211—C221—C222—C223	−179.3 (2)
C121—C122—C123—C124	−0.1 (4)	C221—C222—C223—C224	−2.8 (4)
C121—C122—C123—C131	−178.6 (2)	C221—C222—C223—C231	173.7 (2)
C122—C123—C124—C125	−1.9 (4)	C222—C223—C224—C225	4.6 (3)
C131—C123—C124—C125	176.6 (2)	C231—C223—C224—C225	−171.5 (2)
C122—C123—C124—C127	174.5 (3)	C222—C223—C224—C227	−171.0 (2)
C131—C123—C124—C127	−7.0 (4)	C231—C223—C224—C227	12.9 (4)
C123—C124—C125—O125	−178.0 (3)	C223—C224—C225—O225	177.7 (2)
C127—C124—C125—O125	5.4 (4)	C227—C224—C225—O225	−6.4 (4)
C123—C124—C125—C126	2.2 (4)	C223—C224—C225—C226	−3.3 (4)
C127—C124—C125—C126	−174.4 (3)	C227—C224—C225—C226	172.6 (2)
O125—C125—C126—C121	179.6 (3)	C222—C221—C226—C225	2.2 (4)
C124—C125—C126—C121	−0.6 (5)	C211—C221—C226—C225	−179.2 (2)
C122—C121—C126—C125	−1.3 (4)	O225—C225—C226—C221	178.9 (2)
C111—C121—C126—C125	179.9 (3)	C224—C225—C226—C221	−0.2 (4)
C122—C123—C131—C136	−71.5 (3)	C222—C223—C231—C236	60.2 (3)
C124—C123—C131—C136	110.0 (3)	C224—C223—C231—C236	−123.6 (3)
C122—C123—C131—C132	105.8 (3)	C222—C223—C231—C232	−114.8 (3)
C124—C123—C131—C132	−72.7 (3)	C224—C223—C231—C232	61.4 (3)
C136—C131—C132—C133	−2.0 (4)	C236—C231—C232—C233	1.1 (4)
C123—C131—C132—C133	−179.4 (2)	C223—C231—C232—C233	176.2 (2)
C131—C132—C133—C134	0.4 (4)	C231—C232—C233—C234	0.5 (4)
C132—C133—C134—C135	1.0 (4)	C232—C233—C234—C235	−1.5 (4)
C132—C133—C134—Br13	−179.81 (19)	C232—C233—C234—Br23	−179.07 (19)
C133—C134—C135—C136	−0.9 (5)	C233—C234—C235—C236	0.9 (4)
Br13—C134—C135—C136	180.0 (3)	Br23—C234—C235—C236	178.5 (2)
C132—C131—C136—C135	2.2 (5)	C232—C231—C236—C235	−1.6 (4)
C123—C131—C136—C135	179.6 (3)	C223—C231—C236—C235	−176.7 (2)
C134—C135—C136—C131	−0.8 (5)	C234—C235—C236—C231	0.7 (4)
C125—C124—C127—O127	−6.1 (5)	C225—C224—C227—O227	14.3 (4)
C123—C124—C127—O127	177.5 (3)	C223—C224—C227—O227	−170.1 (3)
C125—C124—C127—O128	171.7 (3)	C225—C224—C227—O228	−163.6 (2)
C123—C124—C127—O128	−4.7 (4)	C223—C224—C227—O228	12.0 (4)
O127—C127—O128—C128	0.6 (5)	O227—C227—O228—C228	1.5 (4)
C124—C127—O128—C128	−177.3 (3)	C224—C227—O228—C228	179.4 (3)
C127—O128—C128—C129	−179.9 (3)	C227—O228—C228—C229	167.1 (3)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O125—H125···O127	0.85 (5)	1.76 (5)	2.542 (4)	151 (5)
O225—H225···O227	0.85 (4)	1.75 (4)	2.556 (3)	157 (4)
C128—H18*A*···O127^{i}	0.97	2.57	3.463 (5)	153
C236—H236···*Cg*3^{ii}	0.93	2.78	3.541 (3)	140

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