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Crystal structure of (4-chlorophenyl)[2-(10-hydroxyphenanthren-9-yl)phenanthro[9,10-*b*]-furan-3-yl]methanone

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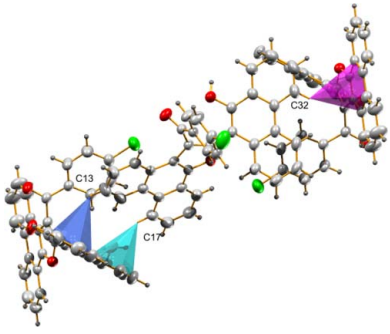
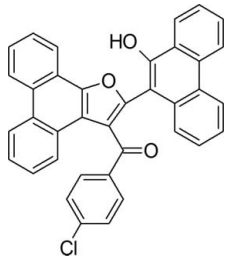
In the title compound, $C_{37}H_{21}ClO_3$, the dihedral angle between the two phenanthrene moieties is 57.79 (5)°. The furan and one of the phenanthrene groups are fused in an almost coplanar arrangement [dihedral angle = 5.14 (8)°] and the furan unit makes dihedral angles of 70.27 (11) and 57.58 (8)° with the planes of the phenyl and the second phenanthrene group, respectively. In the crystal, neighbouring molecules are connected *via* two intermolecular hydrogen-bonding interactions (O—H···O and C—H···O) towards the carbonyl O atom with donor–acceptor distances of 2.824 (2) and 3.277 (3) Å, creating an inversion dimer. A non-classical C—H···Cl interaction [3.564 (2) Å] and three C—H···π interactions, with C···π distances of 3.709 (3), 3.745 (2) and 3.628 (3) Å, connect the molecules, forming a three-dimensional supramolecular architecture in the solid state.

1. Chemical context

Furan and its derivatives have in recent years again attracted the attention of researchers from various areas of chemistry (Uchuskin *et al.*, 2014; Liu *et al.*, 2013). The dihydrofuran core framework was identified in many natural products and in drugs with remarkable biological activities (Michael, 2000; Lipshutz, 1986), inspiring the development of new synthetic methods for the construction of functionalized furans (Singh & Batra, 2008; Snider, 1996; Ranu *et al.*, 2008; Redon *et al.*, 2008; Adamo *et al.*, 2009). As for most organic syntheses, furans are often synthesized in stepwise sequences. However, it is much more efficient if one can form several bonds in one sequence without isolating the intermediates, changing the reaction conditions, or adding reagents (Tietze & Beifuss, 1993). This type of reaction, commonly termed a domino reaction (Muthusaravanan *et al.*, 2013; Kadzimirsz *et al.*, 2008; Criado *et al.*, 2013) would allow a substantial reduction of waste compared to stepwise reactions. The amount of solvents, reagents, adsorbents, and energy would also be dramatically decreased.





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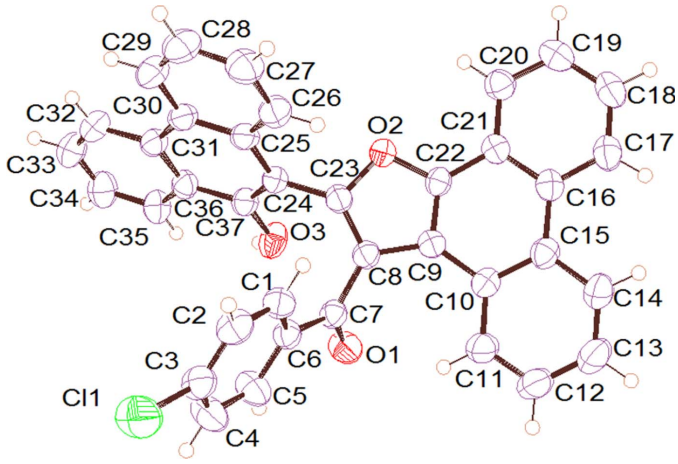

Figure 1
View of the title compound (3) with atom labelling. Displacement ellipsoids are drawn at the 50% probability level.

The title compound of this report has been obtained using such a domino reaction. Using a tandem Michael–aldol reaction of phenanthrenequinone (1) with 4-chloroacetophenone (2) we were able to obtain the highly substituted furan (3) and the 3(2*H*)-furanone (4) (Jacob *et al.*, 2005) in one simple multicomponent reaction.

2. Structural commentary

In the title compound, (3), the two phenanthrene moieties make a dihedral angle of 57.79 (5)°, while one of the phenanthrene moieties is fused together with the furan ring in an almost coplanar arrangement [5.14 (8)°] (Fig. 1). The central furan ring makes dihedral angles of 70.27 (11) and 57.58 (8)° with the phenyl ring and the other phenanthrene moieties, respectively. These two attached rings are twisted so that the C═O oxygen atom points away from the phenanthrene ring. This conformation is stabilized by intramolecular hydrogen bonds between the H atoms attached to atoms C11 and C26 towards O1 and O2, respectively (see Table 1 for numerical values).

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

*Cg*1 is the centroid of the C31–C36 ring, *Cg*2 is the centroid of the C25–C30 ring and Cg3 is the centroid of the C9/C10/C15/C16/C21/C22 ring.

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O3—H3···O1^{i}	0.86 (1)	2.01 (2)	2.824 (2)	156 (3)
C20—H20···Cl1^{ii}	0.93	2.68	3.564 (2)	158
C35—H35···O1^{i}	0.93	2.52	3.277 (3)	139
C11—H11···O1	0.93	2.53	3.275 (3)	137
C26—H26···O2	0.93	2.52	3.057 (3)	117
C13—H13···*Cg*1^{iii}	0.93	3.00	3.709 (3)	134
C17—H17···*Cg*2^{iii}	0.93	2.94	3.745 (2)	146
C32—H32···*Cg*3^{ii}	0.93	2.92	3.628 (3)	134

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

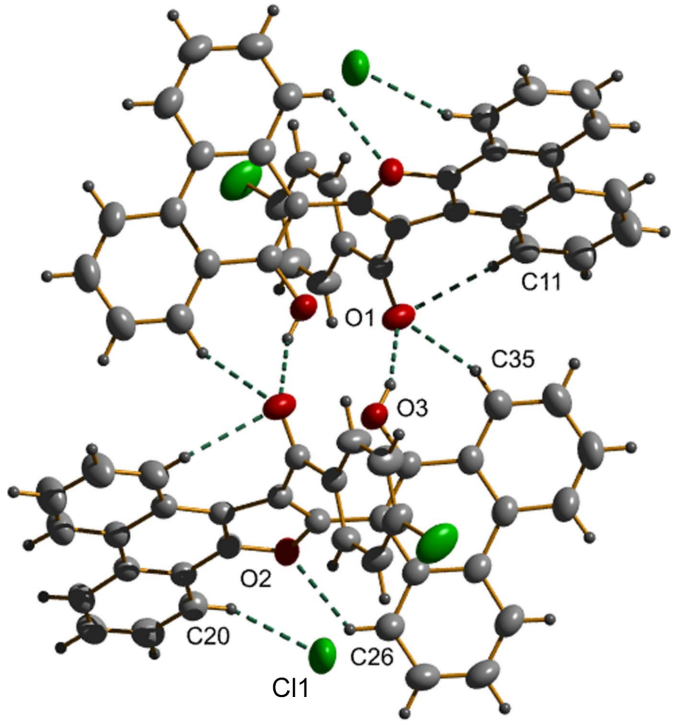

Figure 2
Hydrogen-bonding interactions found in the title compound (see Table 1 for details).

3. Supramolecular features

There are several intermolecular hydrogen-bonding interactions present in the molecular crystal. Carbonyl atom O1 acts as an acceptor for three hydrogen bonds; the intramolecular C—H···O hydrogen bond with the H atom attached to C11, see above, and two intermolecular hydrogen bonds involving atoms O3 and C35 of a neighbouring mol-

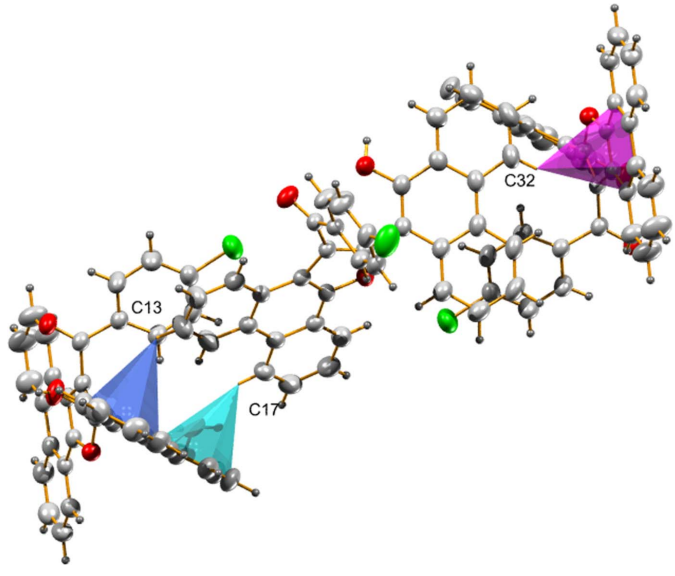

Figure 3
C—H···π interactions found in the title compound.

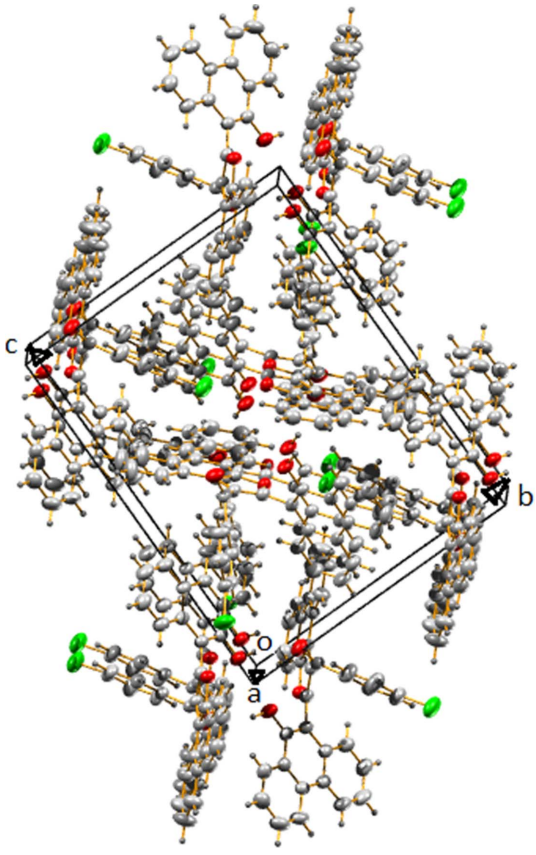

Figure 4
Packing diagram of the title compound along the *a* axis.

ecule. The latter two intermolecular hydrogen-bonding interactions lead to formation of an inversion dimer. Another non-classical hydrogen-bonding interaction with the Cl atom of a neighbouring molecule as the acceptor connects these dimers, forming zigzag chains propagating in the *b*-axis direction (Fig. 2). Three C—H···π interactions (Fig. 3) are found in the crystal. The first two C—H···π interactions are between the H atoms attached to C13 and C17 and the outer two aromatic rings of one of the phenanthrene moieties of an adjacent molecule with C···π distances of 3.709 (3) and 3.745 (2) Å. The third C—H···π interaction occurs between atom C32 and the central aromatic ring of the other phenanthrene moiety (see Table 1 for numerical values and symmetry operators of O—H···O, C—H···O and C—H···π interactions). Fig. 4 shows the packing diagram of the title compound along *a* axis.

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{37}H_{21}ClO_3$
M_r	548.99
Crystal system, space group	Monoclinic, $P2_1/n$
Temperature (K)	296
a, b, c (Å)	11.6682 (12), 13.4448 (15), 17.071 (2)
β (°)	93.091 (5)
V (Å^3)	2674.1 (5)
Z	4
Radiation type	Mo $K\alpha$
μ (mm^{-1})	0.18
Crystal size (mm)	0.40 × 0.35 × 0.30
Data collection	
Diffractometer	Bruker Kappa APEXII CCD
Absorption correction	Multi-scan (*SADABS*; Bruker, 2007)
T_{min}, T_{max}	0.918, 0.920
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	19672, 5790, 3869
R_{int}	0.027
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.639
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.046, 0.147, 1.01
No. of reflections	5790
No. of parameters	374
No. of restraints	1
H-atom treatment	H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.28, −0.36

Computer programs: *APEX2*, *SAINT* and *XPREP* (Bruker, 2007), *SHELXS2012* and *SHELXL97*(Sheldrick, 2008), *ORTEP-3 for Windows* (Farrugia, 2012), *DIAMOND* (Brandenburg, 2010), and *publCIF* (Westrip, 2010).

4. Synthesis and crystallization

A mixture of phenanthrenequinone (1) (5.2 g, 25 mmol), 4-chloroacetophenone (2) (4.2 g, 27 mmol) and powdered potassium hydroxide (1 g) in methanol (30 ml) was stirred at 333 K for 4 h and then kept in a refrigerator for 48 h. The main product obtained was a 3(2*H*)-furanone [2-(4-chlorophenyl)-2-hydroxy-1-oxacyclopenta[*l*]phenanthren-3-one] (4) (65%), which was purified by recrystallization from a mixture of methanol and dichloromethane (2:1 *v*/*v*). The title compound (3) was the minor product formed along with (4) during the reaction (Fig. 5). The reaction mixture was filtered and the filtrate was concentrated and subjected to column chromatography over silica gel. The title compound (14%) was sepa-

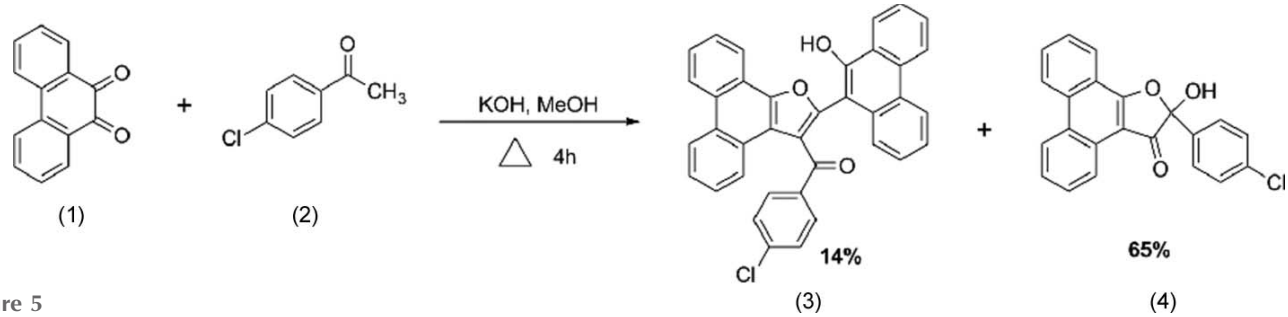

Figure 5
Reaction scheme showing the synthesis of the title compound (3).

rated on elution with a mixture of hexane and ethyl acetate (2:3 *v*/*v*). Diffraction-quality single crystals were generated by slow evaporation from methanol. Yield 1.90 g (14%); m.p. 459 K; IR (KBr, ν_{max}): 3374 (OH), 1591 (C=O) cm^{-1}; ^1H NMR ($CDCl_3$): δ 8.79–7.26 (*m*, 20H), 8.69 (*s*, 1H); MS: *m*/*z* 548 (M^+). Analysis calculated for $C_{37}H_{21}ClO_3$: C 80.94, H 3.86%; found: C 80.82, H 3.66%.

5. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2. The C-bound H atoms were placed in calculated positions and treated as riding with C—H = 0.93 Å and with $U_{iso}(H) = 1.2U_{eq}(C)$. The phenanthroline atom H3 was located from a difference Fourier map and refined with a distance restraint of O—H = 0.86 (1) Å. The reflection 101 was omitted owing to bad agreement.

Acknowledgements

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References

Adamo, M., Suresh, S. & Piras, L. (2009). *Tetrahedron*, **65**, 5402–5408.
Brandenburg, K. (2010). *DIAMOND*. Crystal Impact GbR, Bonn, Germany.
Bruker (2007). *APEX2*, *SAINT* and *SADABS*. Bruker AXS Inc., Madison, Wisconsin, USA.
Criado, A., Vilas-Varela, M., Cobas, A., Perez, D., Pena, D. & Guitian, E. (2013). *J. Org. Chem.* **78**, 12637–12649.
Farrugia, L. J. (2012). *J. Appl. Cryst.* **45**, 849–854.
Jacob, A. M., Thumpakkara, R. K., Prathapan, S. & Jose, B. (2005). *Tetrahedron*, **61**, 4601–4607.
Kadzimirsz, D., Kramer, D., Sripanom, L., Oppel, I. M., Rodziewicz, P., Doltsinis, N. L. & Dyker, G. (2008). *J. Org. Chem.* **73**, 4644–4649.
Lipshutz, B. H. (1986). *Chem. Rev.* **86**, 795–819.
Liu, Z., Fang, J. & Yan, C. (2013). *Chin. J. Chem.* **31**, 1054–1058.
Michael, J. P. (2000). *Nat. Prod. Rep.* **17**, 603–620.
Muthusaravanan, S., Bala, B. D. & Perumal, S. (2013). *Tetrahedron Lett.* **54**, 5302–5306.
Ranu, B. C., Adak, L. & Banerjee, S. (2008). *Tetrahedron Lett.* **49**, 4613–4617.
Redon, S., Leleu, S., Pannecoucke, X., Franck, X. & Outurquin, F. (2008). *Tetrahedron*, **64**, 9293–9304.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Singh, V. & Batra, S. (2008). *Tetrahedron*, **64**, 4511–4574.
Snider, B. B. (1996). *Chem. Rev.* **96**, 339–364.
Tietze, L. F. & Beifuss, U. (1993). *Angew. Chem.* **105**, 137–170.
Uchuskin, G. M., Molodtsova, V. N., Lysenko, A. S., Strelnikov, N. V., Trushkov, V. I. & Butin, V. A. (2014). *Eur. J. Org. Chem.* pp. 2508–2515.
Westrip, S. P. (2010). *J. Appl. Cryst.* **43**, 920–925.

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Crystal structure of (4-chlorophenyl)[2-(10-hydroxyphenanthren-9-yl)phenanthro[9,10-*b*]furan-3-yl]methanone

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Computing details

Data collection: *APEX2* (Bruker, 2007); cell refinement: *APEX2* and *SAINT* (Bruker, 2007); data reduction: *SAINT* and *XPREP* (Bruker, 2007); program(s) used to solve structure: *SHELXS2012* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL2012* (Sheldrick, 2008); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 2012) and *DIAMOND* (Brandenburg, 2010); software used to prepare material for publication: *SHELXL97* (Sheldrick, 2008) and *publCIF* (Westrip, 2010).

(4-Chlorophenyl)[2-(10-hydroxyphenanthren-9-yl)phenanthro[9,10-*b*]furan-3-yl]methanone

Crystal data

$C_{37}H_{21}ClO_3$
M_r = 548.99
Monoclinic, $P2_1/n$
a = 11.6682 (12) Å
b = 13.4448 (15) Å
c = 17.071 (2) Å
β = 93.091 (5)°
V = 2674.1 (5) Å^3
Z = 4
$F(000)$ = 1136
D_x = 1.364 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 6603 reflections
θ = 2.4–27.5°
μ = 0.18 mm^{-1}
T = 296 K
Block, yellow
0.40 × 0.35 × 0.30 mm

Data collection

Bruker Kappa APEXII CCD diffractometer
Detector resolution: 8.33 pixels mm^{-1}
ω and φ scan
Absorption correction: multi-scan (*SADABS*; Bruker, 2007)
T_{min} = 0.918, T_{max} = 0.920
19672 measured reflections
5790 independent reflections
3869 reflections with $I > 2\sigma(I)$
R_{int} = 0.027
θ_{max} = 27.0°, θ_{min} = 2.3°
h = −10→14
k = −16→17
l = −21→21

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.046
$wR(F^2)$ = 0.147
S = 1.01
5790 reflections
374 parameters
1 restraint
Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0706P)^2 + 0.9152P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.28 e Å^{-3}
$\Delta\rho_{min}$ = −0.36 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}
C1	0.40391 (18)	0.20554 (15)	0.31866 (12)	0.0457 (5)
H1	0.3254	0.2178	0.3152	0.055*
C2	0.4531 (2)	0.15068 (17)	0.26111 (13)	0.0518 (5)
H2	0.4082	0.1250	0.2192	0.062*
C3	0.5692 (2)	0.13462 (17)	0.26653 (13)	0.0522 (5)
C4	0.6369 (2)	0.1693 (2)	0.32875 (15)	0.0642 (7)
H4	0.7153	0.1566	0.3320	0.077*
C5	0.58738 (18)	0.22282 (19)	0.38599 (13)	0.0562 (6)
H5	0.6328	0.2463	0.4285	0.067*
C6	0.47072 (16)	0.24266 (14)	0.38182 (11)	0.0390 (4)
C7	0.42073 (16)	0.29570 (15)	0.44766 (11)	0.0391 (4)
C8	0.29803 (16)	0.32528 (14)	0.44139 (11)	0.0383 (4)
C9	0.20746 (16)	0.29570 (14)	0.49057 (11)	0.0367 (4)
C10	0.19633 (17)	0.22173 (14)	0.55080 (11)	0.0397 (4)
C11	0.28787 (19)	0.16154 (17)	0.57835 (14)	0.0531 (6)
H11	0.3595	0.1690	0.5576	0.064*
C12	0.2732 (2)	0.09222 (19)	0.63515 (17)	0.0680 (7)
H12	0.3348	0.0527	0.6528	0.082*
C13	0.1672 (2)	0.0801 (2)	0.66681 (18)	0.0751 (8)
H13	0.1581	0.0334	0.7062	0.090*
C14	0.0758 (2)	0.13712 (19)	0.64004 (15)	0.0634 (7)
H14	0.0049	0.1281	0.6616	0.076*
C15	0.08630 (17)	0.20866 (15)	0.58108 (12)	0.0439 (5)
C16	−0.01135 (17)	0.26868 (15)	0.55198 (11)	0.0410 (4)
C17	−0.12259 (18)	0.25754 (17)	0.57918 (13)	0.0516 (5)
H17	−0.1354	0.2092	0.6167	0.062*
C18	−0.21172 (19)	0.31559 (18)	0.55206 (14)	0.0558 (6)
H18	−0.2839	0.3067	0.5716	0.067*
C19	−0.19613 (19)	0.38775 (18)	0.49571 (14)	0.0535 (6)
H19	−0.2572	0.4277	0.4780	0.064*
C20	−0.09032 (18)	0.39981 (16)	0.46640 (12)	0.0466 (5)
H20	−0.0798	0.4476	0.4281	0.056*
C21	0.00207 (16)	0.34092 (14)	0.49349 (11)	0.0379 (4)
C22	0.11395 (16)	0.34856 (14)	0.46446 (11)	0.0367 (4)
C23	0.25215 (16)	0.39259 (15)	0.38951 (11)	0.0393 (4)
C24	0.30128 (16)	0.45162 (14)	0.32712 (11)	0.0385 (4)
C25	0.25286 (17)	0.44683 (15)	0.24773 (11)	0.0406 (4)
C26	0.16075 (18)	0.38356 (17)	0.22663 (13)	0.0506 (5)
H26	0.1304	0.3432	0.2646	0.061*

C27	0.1146 (2)	0.38008 (19)	0.15127 (14)	0.0590 (6)
H27	0.0536	0.3375	0.1386	0.071*
C28	0.1583 (2)	0.4396 (2)	0.09386 (14)	0.0630 (6)
H28	0.1259	0.4379	0.0429	0.076*
C29	0.2489 (2)	0.50093 (18)	0.11228 (13)	0.0567 (6)
H29	0.2783	0.5399	0.0731	0.068*
C30	0.29902 (17)	0.50670 (15)	0.18884 (12)	0.0433 (5)
C31	0.39590 (17)	0.57061 (15)	0.20959 (12)	0.0430 (5)
C32	0.4470 (2)	0.63220 (18)	0.15423 (14)	0.0582 (6)
H32	0.4176	0.6322	0.1025	0.070*
C33	0.5380 (2)	0.69143 (19)	0.17483 (16)	0.0657 (7)
H33	0.5692	0.7318	0.1372	0.079*
C34	0.5849 (2)	0.69255 (18)	0.25086 (15)	0.0611 (6)
H34	0.6471	0.7335	0.2644	0.073*
C35	0.53931 (19)	0.63313 (17)	0.30604 (13)	0.0516 (5)
H35	0.5719	0.6330	0.3570	0.062*
C36	0.44394 (17)	0.57218 (15)	0.28718 (11)	0.0416 (5)
C37	0.39348 (17)	0.51105 (15)	0.34560 (11)	0.0408 (4)
O1	0.47866 (12)	0.31117 (12)	0.50815 (9)	0.0543 (4)
O2	0.13803 (11)	0.40874 (10)	0.40306 (7)	0.0401 (3)
O3	0.43790 (14)	0.51167 (13)	0.42054 (8)	0.0555 (4)
Cl1	0.63277 (7)	0.06692 (6)	0.19461 (4)	0.0826 (3)
H3	0.479 (2)	0.5630 (16)	0.4332 (18)	0.099 (11)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0406 (11)	0.0519 (12)	0.0441 (11)	−0.0030 (9)	−0.0032 (9)	0.0002 (9)
C2	0.0611 (14)	0.0545 (13)	0.0386 (11)	−0.0019 (11)	−0.0075 (10)	−0.0051 (9)
C3	0.0636 (15)	0.0546 (13)	0.0385 (11)	0.0101 (11)	0.0025 (10)	−0.0030 (9)
C4	0.0445 (13)	0.0922 (19)	0.0558 (14)	0.0109 (13)	0.0016 (11)	−0.0152 (13)
C5	0.0399 (12)	0.0809 (16)	0.0470 (13)	0.0004 (11)	−0.0050 (9)	−0.0157 (11)
C6	0.0388 (11)	0.0428 (11)	0.0352 (10)	−0.0024 (8)	0.0004 (8)	0.0008 (8)
C7	0.0388 (10)	0.0444 (11)	0.0339 (10)	−0.0028 (8)	0.0005 (8)	0.0020 (8)
C8	0.0378 (10)	0.0441 (11)	0.0329 (10)	−0.0011 (8)	0.0007 (8)	−0.0003 (8)
C9	0.0366 (10)	0.0405 (10)	0.0327 (9)	−0.0026 (8)	−0.0008 (8)	−0.0040 (7)
C10	0.0439 (11)	0.0379 (10)	0.0369 (10)	−0.0058 (8)	−0.0023 (8)	0.0002 (8)
C11	0.0457 (12)	0.0529 (13)	0.0601 (14)	−0.0026 (10)	−0.0046 (10)	0.0108 (10)
C12	0.0579 (15)	0.0615 (15)	0.0833 (19)	−0.0006 (12)	−0.0085 (13)	0.0300 (14)
C13	0.0690 (17)	0.0701 (17)	0.086 (2)	−0.0087 (14)	−0.0023 (15)	0.0397 (15)
C14	0.0563 (14)	0.0634 (15)	0.0707 (17)	−0.0102 (12)	0.0057 (12)	0.0228 (12)
C15	0.0464 (12)	0.0421 (11)	0.0428 (11)	−0.0081 (9)	−0.0003 (9)	0.0024 (8)
C16	0.0420 (11)	0.0433 (11)	0.0379 (10)	−0.0074 (9)	0.0026 (8)	−0.0044 (8)
C17	0.0476 (13)	0.0578 (13)	0.0501 (13)	−0.0111 (10)	0.0086 (10)	0.0020 (10)
C18	0.0404 (12)	0.0696 (15)	0.0581 (14)	−0.0078 (11)	0.0099 (10)	−0.0057 (12)
C19	0.0417 (12)	0.0598 (14)	0.0592 (14)	0.0051 (10)	0.0025 (10)	−0.0077 (11)
C20	0.0468 (12)	0.0471 (12)	0.0461 (12)	0.0031 (9)	0.0039 (9)	−0.0017 (9)
C21	0.0382 (10)	0.0403 (10)	0.0352 (10)	−0.0026 (8)	0.0026 (8)	−0.0065 (8)

C22	0.0415 (11)	0.0373 (10)	0.0313 (9)	−0.0025 (8)	0.0028 (8)	−0.0013 (7)
C23	0.0358 (10)	0.0459 (11)	0.0364 (10)	−0.0012 (8)	0.0025 (8)	−0.0010 (8)
C24	0.0383 (10)	0.0433 (11)	0.0342 (10)	0.0037 (8)	0.0040 (8)	0.0028 (8)
C25	0.0396 (10)	0.0449 (11)	0.0372 (10)	0.0083 (9)	0.0012 (8)	0.0009 (8)
C26	0.0470 (12)	0.0622 (14)	0.0423 (12)	−0.0011 (10)	−0.0015 (9)	0.0021 (10)
C27	0.0507 (13)	0.0736 (16)	0.0516 (14)	−0.0023 (12)	−0.0082 (10)	−0.0070 (12)
C28	0.0632 (15)	0.0834 (18)	0.0406 (13)	0.0033 (13)	−0.0123 (11)	0.0005 (12)
C29	0.0623 (14)	0.0685 (15)	0.0389 (12)	0.0073 (12)	−0.0002 (10)	0.0078 (10)
C30	0.0465 (11)	0.0464 (11)	0.0369 (11)	0.0115 (9)	0.0013 (8)	0.0032 (8)
C31	0.0473 (12)	0.0425 (11)	0.0394 (11)	0.0086 (9)	0.0060 (9)	0.0055 (8)
C32	0.0616 (15)	0.0648 (15)	0.0482 (13)	0.0038 (12)	0.0042 (11)	0.0201 (11)
C33	0.0688 (16)	0.0640 (15)	0.0654 (17)	−0.0054 (13)	0.0128 (13)	0.0249 (12)
C34	0.0632 (15)	0.0562 (14)	0.0649 (16)	−0.0125 (12)	0.0140 (12)	0.0021 (11)
C35	0.0547 (13)	0.0554 (13)	0.0454 (12)	−0.0070 (10)	0.0097 (10)	−0.0038 (10)
C36	0.0453 (11)	0.0417 (11)	0.0385 (11)	0.0030 (9)	0.0086 (9)	−0.0008 (8)
C37	0.0438 (11)	0.0456 (11)	0.0333 (10)	0.0032 (9)	0.0037 (8)	−0.0012 (8)
O1	0.0476 (9)	0.0749 (11)	0.0398 (8)	0.0036 (7)	−0.0046 (7)	−0.0107 (7)
O2	0.0392 (7)	0.0454 (7)	0.0361 (7)	0.0021 (6)	0.0048 (6)	0.0047 (6)
O3	0.0629 (10)	0.0691 (11)	0.0340 (8)	−0.0196 (8)	−0.0022 (7)	0.0013 (7)
Cl1	0.1005 (6)	0.0945 (5)	0.0529 (4)	0.0339 (4)	0.0043 (3)	−0.0198 (3)

Geometric parameters (Å, º)

C1—C2	1.378 (3)	C19—C20	1.367 (3)
C1—C6	1.389 (3)	C19—H19	0.9300
C1—H1	0.9300	C20—C21	1.396 (3)
C2—C3	1.370 (3)	C20—H20	0.9300
C2—H2	0.9300	C21—C22	1.425 (3)
C3—C4	1.370 (3)	C22—O2	1.365 (2)
C3—Cl1	1.728 (2)	C23—O2	1.381 (2)
C4—C5	1.367 (3)	C23—C24	1.469 (3)
C4—H4	0.9300	C24—C37	1.363 (3)
C5—C6	1.385 (3)	C24—C25	1.441 (3)
C5—H5	0.9300	C25—C26	1.402 (3)
C6—C7	1.477 (3)	C25—C30	1.417 (3)
C7—O1	1.221 (2)	C26—C27	1.369 (3)
C7—C8	1.484 (3)	C26—H26	0.9300
C8—C23	1.356 (3)	C27—C28	1.384 (3)
C8—C9	1.441 (3)	C27—H27	0.9300
C9—C22	1.357 (3)	C28—C29	1.364 (3)
C9—C10	1.441 (3)	C28—H28	0.9300
C10—C11	1.401 (3)	C29—C30	1.405 (3)
C10—C15	1.420 (3)	C29—H29	0.9300
C11—C12	1.362 (3)	C30—C31	1.449 (3)
C11—H11	0.9300	C31—C36	1.410 (3)
C12—C13	1.386 (4)	C31—C32	1.412 (3)
C12—H12	0.9300	C32—C33	1.359 (4)
C13—C14	1.372 (4)	C32—H32	0.9300

C13—H13	0.9300	C33—C34	1.381 (4)
C14—C15	1.402 (3)	C33—H33	0.9300
C14—H14	0.9300	C34—C35	1.365 (3)
C15—C16	1.461 (3)	C34—H34	0.9300
C16—C21	1.408 (3)	C35—C36	1.405 (3)
C16—C17	1.410 (3)	C35—H35	0.9300
C17—C18	1.361 (3)	C36—C37	1.442 (3)
C17—H17	0.9300	C37—O3	1.354 (2)
C18—C19	1.385 (3)	O3—H3	0.864 (10)
C18—H18	0.9300		

C2—C1—C6	120.52 (19)	C19—C20—C21	120.5 (2)
C2—C1—H1	119.7	C19—C20—H20	119.7
C6—C1—H1	119.7	C21—C20—H20	119.7
C3—C2—C1	119.0 (2)	C20—C21—C16	120.84 (18)
C3—C2—H2	120.5	C20—C21—C22	123.34 (18)
C1—C2—H2	120.5	C16—C21—C22	115.82 (18)
C2—C3—C4	121.7 (2)	C9—C22—O2	111.56 (16)
C2—C3—Cl1	119.73 (17)	C9—C22—C21	125.70 (18)
C4—C3—Cl1	118.58 (18)	O2—C22—C21	122.69 (17)
C5—C4—C3	119.0 (2)	C8—C23—O2	110.23 (16)
C5—C4—H4	120.5	C8—C23—C24	132.70 (18)
C3—C4—H4	120.5	O2—C23—C24	116.99 (16)
C4—C5—C6	121.1 (2)	C37—C24—C25	120.54 (18)
C4—C5—H5	119.4	C37—C24—C23	118.86 (17)
C6—C5—H5	119.4	C25—C24—C23	120.60 (17)
C5—C6—C1	118.66 (19)	C26—C25—C30	118.55 (19)
C5—C6—C7	118.75 (18)	C26—C25—C24	121.64 (18)
C1—C6—C7	122.41 (17)	C30—C25—C24	119.81 (18)
O1—C7—C6	120.15 (17)	C27—C26—C25	121.3 (2)
O1—C7—C8	120.22 (18)	C27—C26—H26	119.4
C6—C7—C8	119.56 (16)	C25—C26—H26	119.4
C23—C8—C9	106.78 (17)	C26—C27—C28	120.3 (2)
C23—C8—C7	124.84 (17)	C26—C27—H27	119.8
C9—C8—C7	128.25 (17)	C28—C27—H27	119.8
C22—C9—C8	105.43 (17)	C29—C28—C27	119.8 (2)
C22—C9—C10	119.59 (17)	C29—C28—H28	120.1
C8—C9—C10	134.70 (18)	C27—C28—H28	120.1
C11—C10—C15	119.61 (18)	C28—C29—C30	121.8 (2)
C11—C10—C9	122.79 (18)	C28—C29—H29	119.1
C15—C10—C9	117.58 (17)	C30—C29—H29	119.1
C12—C11—C10	120.8 (2)	C29—C30—C25	118.3 (2)
C12—C11—H11	119.6	C29—C30—C31	122.72 (19)
C10—C11—H11	119.6	C25—C30—C31	119.02 (18)
C11—C12—C13	120.4 (2)	C36—C31—C32	117.3 (2)
C11—C12—H12	119.8	C36—C31—C30	120.26 (18)
C13—C12—H12	119.8	C32—C31—C30	122.4 (2)
C14—C13—C12	120.0 (2)	C33—C32—C31	121.5 (2)

C14—C13—H13	120.0	C33—C32—H32	119.2
C12—C13—H13	120.0	C31—C32—H32	119.2
C13—C14—C15	121.7 (2)	C32—C33—C34	120.9 (2)
C13—C14—H14	119.2	C32—C33—H33	119.5
C15—C14—H14	119.2	C34—C33—H33	119.5
C14—C15—C10	117.5 (2)	C35—C34—C33	119.5 (2)
C14—C15—C16	121.72 (19)	C35—C34—H34	120.2
C10—C15—C16	120.77 (18)	C33—C34—H34	120.2
C21—C16—C17	116.52 (19)	C34—C35—C36	121.1 (2)
C21—C16—C15	120.50 (17)	C34—C35—H35	119.5
C17—C16—C15	122.98 (19)	C36—C35—H35	119.5
C18—C17—C16	121.8 (2)	C35—C36—C31	119.58 (19)
C18—C17—H17	119.1	C35—C36—C37	121.44 (19)
C16—C17—H17	119.1	C31—C36—C37	118.98 (18)
C17—C18—C19	120.7 (2)	O3—C37—C24	118.63 (17)
C17—C18—H18	119.6	O3—C37—C36	120.00 (18)
C19—C18—H18	119.6	C24—C37—C36	121.37 (18)
C20—C19—C18	119.5 (2)	C22—O2—C23	105.99 (14)
C20—C19—H19	120.2	C37—O3—H3	115 (2)
C18—C19—H19	120.2		

C6—C1—C2—C3	−1.0 (3)	C10—C9—C22—C21	2.5 (3)
C1—C2—C3—C4	1.9 (4)	C20—C21—C22—C9	177.58 (19)
C1—C2—C3—Cl1	−179.48 (17)	C16—C21—C22—C9	−2.9 (3)
C2—C3—C4—C5	−1.3 (4)	C20—C21—C22—O2	−5.4 (3)
Cl1—C3—C4—C5	−179.9 (2)	C16—C21—C22—O2	174.09 (16)
C3—C4—C5—C6	−0.2 (4)	C9—C8—C23—O2	0.7 (2)
C4—C5—C6—C1	1.1 (4)	C7—C8—C23—O2	−175.36 (17)
C4—C5—C6—C7	176.4 (2)	C9—C8—C23—C24	177.5 (2)
C2—C1—C6—C5	−0.4 (3)	C7—C8—C23—C24	1.5 (3)
C2—C1—C6—C7	−175.59 (19)	C8—C23—C24—C37	−55.3 (3)
C5—C6—C7—O1	−8.4 (3)	O2—C23—C24—C37	121.35 (19)
C1—C6—C7—O1	166.7 (2)	C8—C23—C24—C25	124.7 (2)
C5—C6—C7—C8	174.80 (19)	O2—C23—C24—C25	−58.6 (2)
C1—C6—C7—C8	−10.1 (3)	C37—C24—C25—C26	177.61 (19)
O1—C7—C8—C23	117.9 (2)	C23—C24—C25—C26	−2.4 (3)
C6—C7—C8—C23	−65.3 (3)	C37—C24—C25—C30	−2.0 (3)
O1—C7—C8—C9	−57.2 (3)	C23—C24—C25—C30	177.96 (17)
C6—C7—C8—C9	119.5 (2)	C30—C25—C26—C27	−1.0 (3)
C23—C8—C9—C22	−0.4 (2)	C24—C25—C26—C27	179.4 (2)
C7—C8—C9—C22	175.41 (19)	C25—C26—C27—C28	−0.1 (4)
C23—C8—C9—C10	173.2 (2)	C26—C27—C28—C29	1.0 (4)
C7—C8—C9—C10	−11.0 (3)	C27—C28—C29—C30	−0.9 (4)
C22—C9—C10—C11	177.33 (19)	C28—C29—C30—C25	−0.1 (3)
C8—C9—C10—C11	4.4 (3)	C28—C29—C30—C31	179.5 (2)
C22—C9—C10—C15	−0.9 (3)	C26—C25—C30—C29	1.0 (3)
C8—C9—C10—C15	−173.8 (2)	C24—C25—C30—C29	−179.31 (18)
C15—C10—C11—C12	−1.5 (3)	C26—C25—C30—C31	−178.58 (18)

C9—C10—C11—C12	−179.7 (2)	C24—C25—C30—C31	1.1 (3)
C10—C11—C12—C13	−0.1 (4)	C29—C30—C31—C36	−178.97 (19)
C11—C12—C13—C14	1.1 (5)	C25—C30—C31—C36	0.6 (3)
C12—C13—C14—C15	−0.3 (4)	C29—C30—C31—C32	0.2 (3)
C13—C14—C15—C10	−1.3 (4)	C25—C30—C31—C32	179.77 (19)
C13—C14—C15—C16	179.4 (2)	C36—C31—C32—C33	−0.7 (3)
C11—C10—C15—C14	2.2 (3)	C30—C31—C32—C33	−179.8 (2)
C9—C10—C15—C14	−179.59 (19)	C31—C32—C33—C34	0.8 (4)
C11—C10—C15—C16	−178.45 (19)	C32—C33—C34—C35	0.2 (4)
C9—C10—C15—C16	−0.2 (3)	C33—C34—C35—C36	−1.3 (4)
C14—C15—C16—C21	179.1 (2)	C34—C35—C36—C31	1.4 (3)
C10—C15—C16—C21	−0.2 (3)	C34—C35—C36—C37	−178.4 (2)
C14—C15—C16—C17	−1.7 (3)	C32—C31—C36—C35	−0.4 (3)
C10—C15—C16—C17	178.98 (19)	C30—C31—C36—C35	178.76 (19)
C21—C16—C17—C18	−1.9 (3)	C32—C31—C36—C37	179.40 (18)
C15—C16—C17—C18	178.9 (2)	C30—C31—C36—C37	−1.4 (3)
C16—C17—C18—C19	0.6 (3)	C25—C24—C37—O3	−178.54 (18)
C17—C18—C19—C20	0.8 (3)	C23—C24—C37—O3	1.5 (3)
C18—C19—C20—C21	−0.8 (3)	C25—C24—C37—C36	1.2 (3)
C19—C20—C21—C16	−0.6 (3)	C23—C24—C37—C36	−178.75 (17)
C19—C20—C21—C22	178.89 (19)	C35—C36—C37—O3	0.1 (3)
C17—C16—C21—C20	1.9 (3)	C31—C36—C37—O3	−179.74 (18)
C15—C16—C21—C20	−178.83 (18)	C35—C36—C37—C24	−179.69 (19)
C17—C16—C21—C22	−177.64 (18)	C31—C36—C37—C24	0.5 (3)
C15—C16—C21—C22	1.6 (3)	C9—C22—O2—C23	0.4 (2)
C8—C9—C22—O2	0.0 (2)	C21—C22—O2—C23	−177.00 (17)
C10—C9—C22—O2	−174.73 (16)	C8—C23—O2—C22	−0.6 (2)
C8—C9—C22—C21	177.31 (17)	C24—C23—O2—C22	−178.04 (16)

Hydrogen-bond geometry (Å, º)

Cg1 is the centroid of the C31–C36 ring, Cg2 is the centroid of the C25–C30 ring and Cg3 is the centroid of the C9/C10/C15/C16/C21/C22 ring.

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O3—H3···O1^{i}	0.86 (1)	2.01 (2)	2.824 (2)	156 (3)
C20—H20···Cl1^{ii}	0.93	2.68	3.564 (2)	158
C35—H35···O1^{i}	0.93	2.52	3.277 (3)	139
C11—H11···O1	0.93	2.53	3.275 (3)	137
C26—H26···O2	0.93	2.52	3.057 (3)	117
C13—H13···*Cg*1^{iii}	0.93	3.00	3.709 (3)	134
C17—H17···*Cg*2^{iii}	0.93	2.94	3.745 (2)	146
C32—H32···*Cg*3^{ii}	0.93	2.92	3.628 (3)	134

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