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The relationship between Hirshfeld potential and cytotoxic activity: a study along a series of flavonoid and chromanone derivatives

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Supporting information: this article has supporting information at journals.iucr.org/c

The present study examines a series of six biologically-active flavonoid and chromanone derivatives by X-ray crystal structure analysis: (*E*)-3-benzylidene-2-phenylchroman-4-one, $C_{22}H_{16}O_2$, **I**, (*E*)-3-(4-methylbenzylidene)-2-phenylchroman-4-one, $C_{23}H_{18}O_2$, **II**, (*E*)-3-(3-methylbenzylidene)-2-phenylchroman-4-one, $C_{23}H_{18}O_2$, **III**, (*E*)-3-(4-methoxybenzylidene)-2-phenylchroman-4-one, $C_{23}H_{18}O_3$, **IV**, (*E*)-3-benzylidenechroman-4-one, $C_{16}H_{12}O_2$, **V**, and (*E*)-3-(4-methoxybenzylidene)chroman-4-one, $C_{17}H_{14}O_3$, **VI**. The cytotoxic activities of the presented crystal structures have been determined, together with their intermolecular interaction preferences and Hirshfeld surface characteristics. An inverse relationship was found between the contribution of C···C close contacts to the Hirshfeld surface and cytotoxic activity against the WM-115 cancer line. Dependence was also observed between the log*P* value and the percentage contribution of C···H contacts to the Hirshfeld surface.

1. Introduction

A number of our previous studies have focused on the synthesis and biological activity of chromone derivatives and flavonoids (Budzisz *et al.*, 2017; Kupcewicz *et al.*, 2016). These heterocyclic compounds include a number of plant secondary metabolites that are widely distributed in nature (Panche *et al.*, 2016). Although flavonoid compounds can be extracted from the fruits, roots and leaves of plants, they are also commonly found in beverages such as wine and tea (Landrault *et al.*, 2001). Flavonoid compounds, and their derivatives, demonstrate a wide spectrum of biological activity on plants and human health (Harborne & Williams, 2000). They protect plants against biotic stress, act as UV filters and fulfill signaling and defense functions (Panche *et al.*, 2016); in addition, aglycones, glycosides and methylated derivatives are known to possess a range of antioxidant (Soobrattee *et al.*, 2005), anti-inflammatory (Serafini *et al.*, 2010), anticancer (Ravishankar *et al.*, 2013) and antimutagenic properties, and are believed to be capable of modulating the function of cellular enzymes (Es-Safi *et al.*, 2007). The present study presents a series of new flavonoids or chromanones which exhibit promising cytotoxic activity. The compounds are (*E*)-3-benzylidene-2-phenylchroman-4-one, **I**, (*E*)-3-(4-methylbenzylidene)-2-phenylchroman-4-one, **II**, (*E*)-3-(3-methylbenzylidene)-2-phenylchroman-4-one, **III**, (*E*)-3-(4-methoxybenzylidene)-2-phenylchroman-4-one, **IV**,

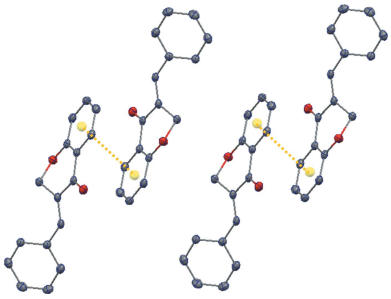


Table 1
Experimental details.

Experiments were carried out at 100 K. Absorption was corrected for by multi-scan methods, using *CrysAlis PRO* (Rigaku OD, 2015) for compounds **I**–**V** and *CrysAlis PRO* (Rigaku OD, 2019) for compound **VI**. H-atom parameters were constrained.

	I	**II**	**III**
Crystal data			
Chemical formula	$C_{22}H_{16}O_2$	$C_{23}H_{18}O_2$	$C_{23}H_{18}O_2$
M_r	312.35	326.37	326.37
Crystal system, space group	Monoclinic, *C*2/*c*	Monoclinic, $P2_1/c$	Triclinic, $P\overline{1}$
a, *b*, *c* (Å)	10.2487 (3), 16.3033 (4), 19.7539 (7)	16.6799 (5), 7.9102 (2), 13.1511 (3)	8.9094 (3), 9.6578 (3), 10.9044 (4)
α, β, γ (°)	90, 100.420 (3), 90	90, 98.558 (3), 90	104.378 (3), 109.425 (3), 91.902 (3)
V (Å^3)	3246.20 (17)	1715.85 (8)	850.24 (5)
Z	8	4	2
Radiation type	Mo *K*α	Mo *K*α	Mo *K*α
μ (mm^{-1})	0.08	0.08	0.08
Crystal size (mm)	0.28 × 0.26 × 0.08	0.28 × 0.1 × 0.1	0.28 × 0.1 × 0.08
Data collection			
Diffractometer	Rigaku OD SuperNova Dual source diffractometer with an Atlas detector	Rigaku OD SuperNova Dual source diffractometer with an Atlas detector	Rigaku OD SuperNova Dual source diffractometer with an Atlas detector
T_{min}, T_{max}	0.744, 1.000	0.833, 1.000	0.736, 1.000
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	13340, 13340, 11168	13962, 3550, 2927	7284, 3509, 2999
R_{int}	0.042	0.031	0.021
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.628	0.628	0.628
Refinement			
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, *S*	0.060, 0.153, 1.02	0.040, 0.107, 1.05	0.037, 0.098, 1.04
No. of reflections	13340	3550	3509
No. of parameters	205	227	227
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.49, −0.38	0.19, −0.23	0.24, −0.21

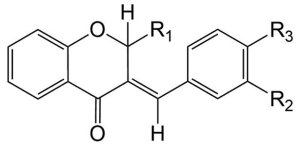
	IV	**V**	**VI**
Crystal data			
Chemical formula	$C_{23}H_{18}O_3$	$C_{16}H_{12}O_2$	$C_{17}H_{14}O_3$
M_r	342.37	236.26	266.28
Crystal system, space group	Monoclinic, $P2_1/n$	Triclinic, $P\overline{1}$	Triclinic, $P\overline{1}$
a, *b*, *c* (Å)	23.4111 (3), 8.0027 (1), 28.1094 (5)	7.7718 (3), 8.6069 (3), 9.0857 (3)	7.5570 (3), 12.3558 (4), 14.4044 (6)
α, β, γ (°)	90, 97.156 (1), 90	89.744 (3), 84.862 (3), 74.199 (3)	82.748 (3), 87.126 (3), 75.768 (3)
V (Å^3)	5225.33 (13)	582.31 (4)	1293.02 (9)
Z	12	2	4
Radiation type	Cu *K*α	Mo *K*α	Cu *K*α
μ (mm^{-1})	0.69	0.09	0.76
Crystal size (mm)	0.28 × 0.1 × 0.08	0.18 × 0.08 × 0.08	0.3 × 0.08 × 0.06
Data collection			
Diffractometer	Rigaku OD SuperNova Dual source diffractometer with an Atlas detector	Rigaku OD SuperNova Dual source diffractometer with an Atlas detector	XtaLAB Synergy Dualflex HyPix
T_{min}, T_{max}	0.997, 1.000	0.865, 1.000	0.671, 1.000
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	15971, 9323, 7742	4947, 2425, 2120	5118, 5118, 4669
R_{int}	0.022	0.023	0.020
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.599	0.628	0.595
Refinement			
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, *S*	0.038, 0.104, 1.01	0.040, 0.115, 1.06	0.070, 0.200, 1.09
No. of reflections	9323	2425	5118
No. of parameters	706	163	364
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.20, −0.21	0.29, −0.22	0.63, −0.33

Computer programs: *CrysAlis PRO* (Rigaku OD, 2015, 2019), *SHELXT* (Sheldrick, 2015*a*), *DIAMOND* (Brandenburg & Putz, 1999), *SHELXL2014* (Sheldrick, 2015*b*) and *publCIF* (Westrip, 2010).

(*E*)-3-benzylidenechroman-4-one, **V**, and (*E*)-3-(4-methoxybenzylidene)chroman-4-one, **VI** (see Scheme 1).

Quantitative structure–activity relationships (QSAR), being mathematical representations of biological activity, are widely used to predict the pharmaceutical properties of potential drug candidates, in particular their cytotoxicity (Roy *et al.*, 2015). In many cases, the pharmacological effects of drugs are determined by their interactions between the com-

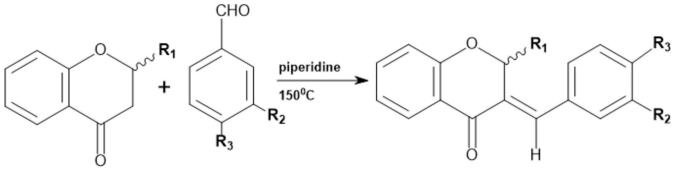
pound and the active site of other molecules; such interactions may be hydrogen bonds, electrostatic forces or van der Waals interactions (Zheng *et al.*, 2014; Deschamps, 2010). A vast number of molecular descriptors have their origin in X-ray crystallography (Cachau & Podjarny, 2005; Deschamps & George, 2003; Williams *et al.*, 2005). Most of these, such as hardness (η) and softness (σ), chemical potential (μ) and electrophilicity index (ω) are available from molecular modelling. However, the key role in pharmacology and structure-based drug design is arguably played by information about molecular structure and intermolecular interactions.



compound	R_1	R_2	R_3
I	Ph	H	H
II	Ph	H	CH_3
III	Ph	CH_3	H
IV	Ph	H	OCH_3
V	H	H	H
VI	H	H	OCH_3

Scheme 1

Flavonoid and chromanone derivatives **I**–**VI** were synthesized (Scheme 2) and their cytotoxic activities and crystal structures determined. The aim of the present study was to find the relationship between the cytotoxicity of the tested compounds and the geometric parameters of their crystal structures, as well as obtaining information by Hirshfeld surface analysis, as encouraged by previous research (Kupcewicz *et al.*, 2016).



Scheme 2

2. Experimental

2.1. Synthesis and crystallization

The general method for the synthesis of (*E*)-3-benzylidene-4-chromanone and the flavanone derivatives consists of the condensation of 4-chromanone or flavanone with the appropriate aromatic aldehyde in the presence of piperidine as a catalyst (Scheme 2). The use of excess catalyst may result in the transition of an *exo–endo* double bond between the C3 and C4 atoms in the flavanone ring, resulting in the production of 3-benzyl-2-phenylchromen-4-one derivatives (Levai, 2004). Compounds **I**–**IV** were synthesized according to Pijewska *et al.* (1993), and compounds **V** and **VI** according to Levai & Schag (1979). Both syntheses were slightly modified by Adamus-Grabicka *et al.* (2020). Analytical data for compounds **I**–**VI** are given in the supporting information. Single crystals suitable for X-ray diffraction studies were obtained from ethanol or methanol solution by slow evaporation at room temperature.

Table 2
Hydrogen-bond geometry (Å, °) for compounds **I**–**VI**.

For **I**, *Cg*2 is the centroid of the C5–C10 ring and *Cg*4 of the C18–C23 ring; for **III**, *Cg*4 is the centroid of the C21–C26 ring; for **IV**, *Cg*3 is the centroid of the C11–C16 ring, *Cg*4 of the C18–C23 ring, *Cg*8 of the C41–C46 ring, *Cg*9 of the C48–C53 ring and *Cg*13 of the C71–C76 ring.

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
Compound **I**				
C11—H11···O4^{i}	0.95	2.47	3.353 (3)	154
C17—H17···O4^{i}	0.95	2.64	3.353 (2)	132
C21—H21···O4^{ii}	0.95	2.68	3.267 (3)	120
C6—H6···*Cg*4^{iii}	0.95	2.83	3.736 (2)	160
C15—H15···*Cg*4^{iv}	0.95	2.89	3.730 (2)	148
C16—H16···*Cg*2^{iv}	0.95	2.84	3.539 (2)	131
Compound **II**				
C2—H2···O4^{v}	1.00	2.27	3.174 (2)	149
C5—H5···O1^{vi}	0.95	2.54	3.408 (2)	152
C24—H24···O4^{vii}	0.95	2.71	3.487 (2)	140
Compound **III**				
C11—H11···O4^{viii}	0.95	2.54	3.3635 (14)	145
C17—H17···O4^{viii}	0.95	2.69	3.5025 (15)	144
C24—H24···O4^{ix}	0.95	2.60	3.2731 (15)	128
C6—H6···*Cg*4^{x}	0.95	2.52	3.4654 (15)	174
Compound **IV**				
C2—H2···O34^{xi}	1.00	2.45	3.3434 (15)	148
C6—H6···O61^{xii}	0.95	2.40	3.2676 (16)	151
C14—H14···O1^{xiii}	0.95	2.46	3.3750 (15)	161
C32—H32···O64^{xiv}	1.00	2.37	3.2348 (15)	144
C36—H36···O1^{xv}	0.95	2.42	3.2733 (15)	150
C44—H44···O31^{xiii}	0.95	2.42	3.3399 (16)	162
C62—H62···O4^{xvi}	1.00	2.48	3.3629 (15)	146
C66—H66···O31^{xvii}	0.95	2.48	3.3725 (15)	157
C74—H74···O61^{xiii}	0.95	2.49	3.4018 (15)	161
C7—H7···*Cg*8^{xviii}	0.95	2.97	3.7215915)	136
C211—H21*B*···*Cg*4^{xviii}	0.98	2.71	3.6762 (15)	169
C211—H21*C*···*Cg*3^{xix}	0.98	2.91	3.6005 (17)	128
C23—H23···*Cg*9^{xx}	0.95	2.99	3.6985 (14)	133
C37—H37···*Cg*3^{xxi}	0.95	2.98	3.7434 (15)	138
C511—H51*B*···*Cg*13^{xvi}	0.98	2.75	3.6774 (15)	158
C53—H53···*Cg*4^{xxii}	0.95	2.72	3.5324 (15)	144
C811—H81*A*···*Cg*9^{xii}	0.98	2.99	3.6203 (16)	123
C811—H81*B*···*Cg*8^{xvi}	0.98	2.90	3.8631 (15)	167
Compound **V**				
C7—H7···O4^{xxiii}	0.95	2.69	3.6131 (16)	166
C11—H11···O4^{xxiv}	0.95	2.56	3.4153 (15)	150
C17—H17···O4 ^{xxiv}	0.95	2.56	3.4050 (16)	148
Compound **VI**				
C2—H2*A*···O22	0.99	2.51	3.430 (3)	154
C2—H2*B*···O1^{xxv}	0.99	2.58	3.215 (3)	122
C5—H5···O2^{xxvi}	0.95	2.55	3.319 (3)	138
C14—H14···O24^{xxvii}	0.95	2.54	3.196 (3)	127
C17—H17···O1^{xxviii}	0.95	2.45	3.349 (3)	157
C25—H25···O22^{xxvi}	0.95	2.55	3.275 (3)	133
C33—H33···O4^{xxix}	0.95	2.45	3.381 (3)	166
C37—H37···O21^{xxviii}	0.95	2.46	3.330 (3)	153
C8—H8···*Cg*3^{xxix}	0.95	2.79	3.632 (3)	149
C28—H28···*Cg*7^{xxx}	0.95	2.74	3.573(30	147

Symmetry codes: (i) $-x, y, -z+\frac{1}{2}$; (ii) $-x+\frac{1}{2}, y-\frac{1}{2}, -z+\frac{1}{2}$; (iii) $x-\frac{1}{2}, y-\frac{1}{2}, z$; (iv) $x+1, y, z$; (v) $x, -y+\frac{3}{2}, z-\frac{1}{2}$; (vi) $x, -y+\frac{3}{2}, z+\frac{1}{2}$; (vii) $x, -y+\frac{1}{2}, z-\frac{1}{2}$; (viii) $-x+1, -y, -z+1$; (ix) $-x+1, -y, -z$; (x) $x-1, y, z$; (xi) $-x+\frac{1}{2}, y+\frac{1}{2}, -z+\frac{3}{2}$; (xii) $-x+\frac{1}{2}, y+\frac{1}{2}, -z+\frac{1}{2}$; (xiii) $x, y+1, z$; (xiv) $x+\frac{1}{2}, -y+\frac{1}{2}, z+\frac{1}{2}$; (xv) $-x+\frac{1}{2}, y-\frac{1}{2}, -z+\frac{3}{2}$; (xvi) $-x+\frac{1}{2}, y-\frac{1}{2}, -z+\frac{1}{2}$; (xvii) $x-\frac{1}{2}, -y+\frac{1}{2}, z-\frac{1}{2}$; (xviii) $-x-\frac{1}{2}, -y+\frac{3}{2}, z-\frac{1}{2}$; (xix) $-x+\frac{1}{2}, y-\frac{1}{2}, -z+\frac{3}{2}$; (xx) x, y, z; (xxi) $x+\frac{1}{2}, -y+\frac{3}{2}, z+\frac{1}{2}$; (xxii) $x, y-1, z$; (xxiii) $x, y+1, z$; (xxiv) $-x+1, -y+1, -z+2$; (xxv) $-x+1, -y+3, -z+1$; (xxvi) $x, y-1, z$; (xxvii) $x, y+1, z$; (xxviii) $x+1, y, z$; (xxix) $x-1, y, z$; (xxix) $-x+1, -y+3, -z+1$; (xxx) $-x, -y+2, -z+2$.

2.2. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 1. All H atoms were fixed at calculated positions and refined in riding mode, with C—H = 0.95, 0.98, 0.99 and 1.00 Å for aromatic, methyl, methylene and methine H atoms, and with U_{iso}(H) = 1.2U_{eq}(C) for aromatic, methylene and methane H atoms, and U_{iso}(H) = 1.5U_{eq}(C) for methyl H atoms.

The structure of **IV** has three independent molecules in the asymmetric unit, and that of **VI** has two molecules in the asymmetric unit. The structure of **VI** was refined as a rotational twin rotated from the first domain by −179.9192° about the reciprocal axis [−0.04 −0.14 0.99] and the real axis 0.00 0.00 1.00. The twin law to convert hkl from the first to this domain (*SHELXL* TWIN matrix) was −1.0003 0.0011 −0.0003 −0.0024 −0.9995 −0.0002 −0.0728 −0.2831 1.0003.

3. Results and discussion

3.1. Molecular structures of compounds I–VI

The molecular structures of compounds **I**–**VI** are shown in Fig. 1. Compound **IV** crystalizes with three molecules in the asymmetric unit, while compound **VI** crystallizes with two molecules in the asymmetric unit. The main body of the presented structures consists of a chroman-4-one skeleton, *i.e.* an arene ring fused with a tetrahydropyran-4-one ring. Posi-

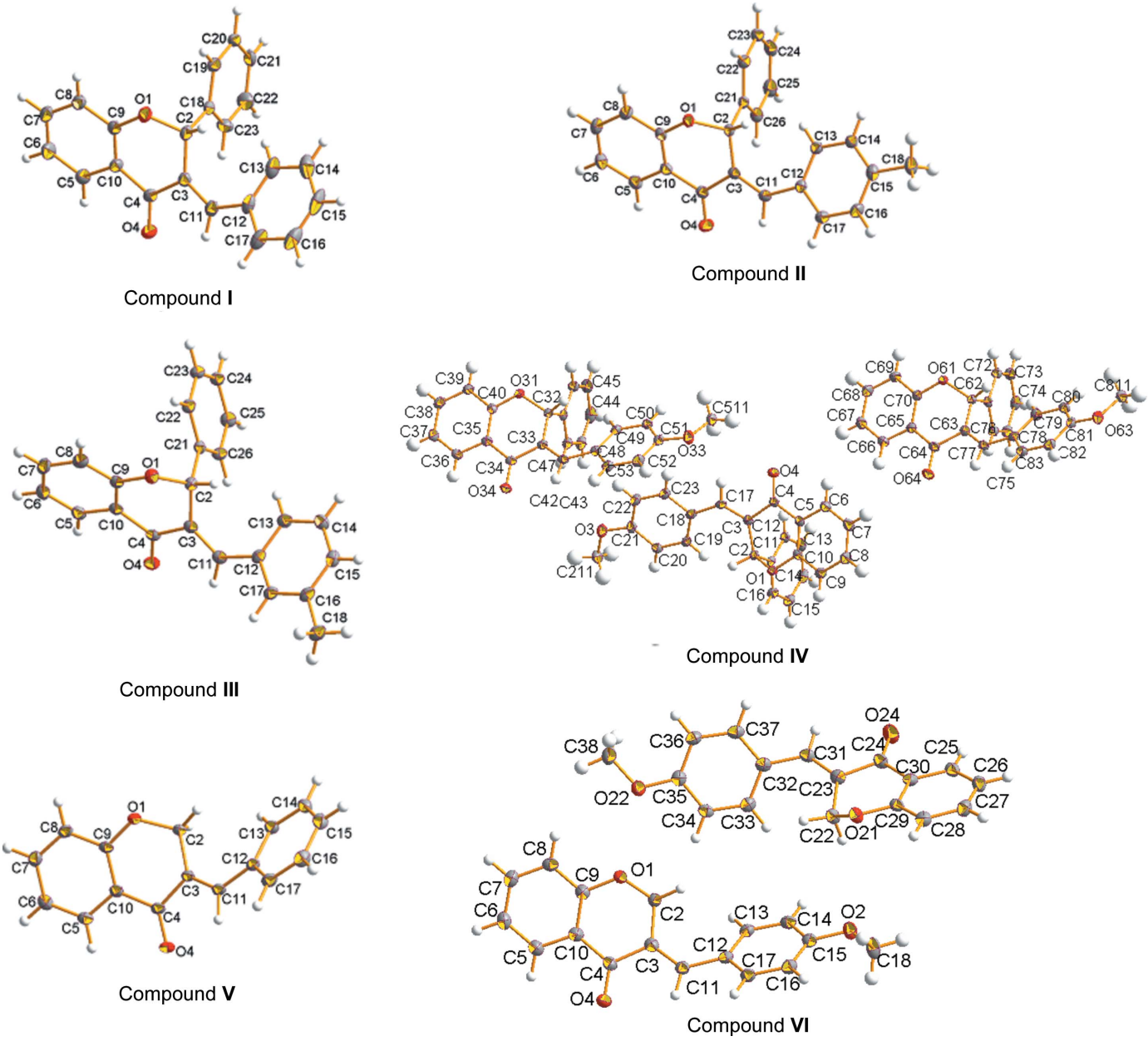


Figure 1
Views of the molecular structures of compounds **I**–**VI** with the atom-numbering schemes. Displacement ellipsoids are drawn at the 50% probability level.

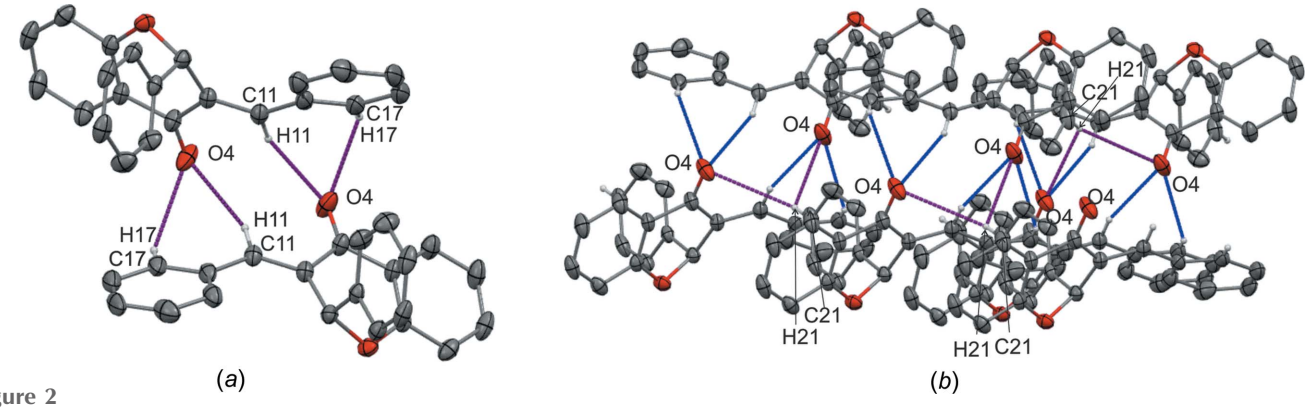


Figure 2
(*a*) Packing of compound **I** showing the C11—H11···O4^{i} and C17—H17···O4^{i} hydrogen-bonded dimers. (*b*) A fragment of the molecular structure with two chains forming a hydrogen bond *via* C21—H21···O4^{ii} (violet) hydrogen bonds, with the C11—H11···O4^{i} and C17—H17···O4^{i} hydrogen bonds drawn in blue. Generic atom labels without symmetry codes have been used.

tion 3 of the chroman moiety is connected to a benzylidene (in **I**), 4-methylbenzylidene (in **II**), 3-methylbenzylidene (in **III**), 4-methoxybenzylidene (in **IV**), benzylidene (in **V**) or 4-methoxybenzylidene (in **VI**) substituent. For all six structures, these substituents are rotated with respect to the chroman moiety, as reflected in their corresponding dihedral angles of 46.8 (2), 37.7 (2), 67.4 (2), 33.8 (2)–36.4 (2) (three molecules in the asymmetric unit), 54.9 (2) and 56.4 (2)–58.6 (2)° (two molecules in the asymmetric unit), respectively. In structures **I**–**IV**, the chroman moiety is substituted at position 2 by a phenyl ring, which is nearly perpendicular to plane of the main chroman skeleton, with the dihedral angles of 85.6 (2), 89.4 (2), 89.3 (2) and 86.9 (2)–88.2 (2)°, respectively. Compounds whose substituents at position C2 are more perpendicular with respect to the chroman moiety also have benzylidene substituents at position 3 that are more parallel (Table S1 in the supporting information). The pyran rings adopt mainly envelope (E) or screw-boat (S) conformations, with asymmetry parameters in the range ΔC_s(C2) = 1.6 (2)–10.5 (3)° (Duax & Norton, 1975) for **IV*A***, **V** and **VI*B***, and ΔC_2(O1—C2) = 7.9 (2)–13.0 (2)° (Duax & Norton, 1975) for **II**, **IV*B***, **IV*C*** and **VI*A*** (suffixes *A*–*C* denotes molecules *A*–*C*). These conformations are confirmed by the respective puckering parameters (Cremer & Pople, 1975), *i.e.* Q = 0.337 (2)–0.446 (2) Å, θ = 59.4 (2)–120.9 (2)° and φ = 43.6 (2)–233.7 (4)°. Details of the puckering parameters are given in Table S2 of the supporting information.

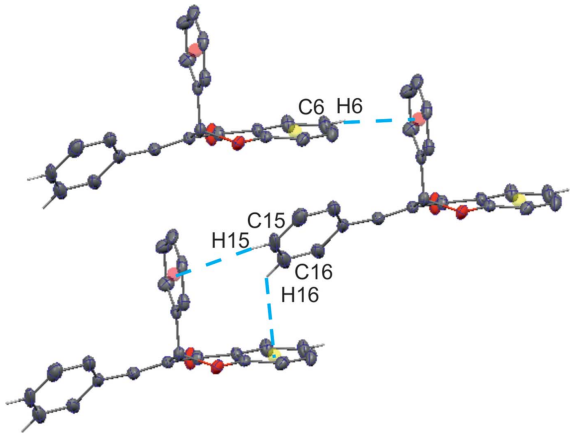


Figure 3
Fragment of the crystal packing of compound **I**, showing C6—H6···*Cg*4^{iii}, C15—H15···*Cg*4^{iv} and C16—H16···*Cg*2^{iv} interactions. *Cg*4 is a centroid of the C18–C23 ring (red dot) and *Cg*2 is the centroid of the C5–C10 ring (yellow dot). See Table 2 for symmetry codes.

3.2. Crystal structure analysis

The solid-state structures of compounds **I**–**VI** display a combination of C—H···O, C—H···π and very weak π–π interactions (see Table 2 for geometric parameters and symmetry codes). In **I**, atoms C11 and C17 act as donors in C—H···O hydrogen bonds with atom O4^{i} as acceptor, forming a dimer. The C21 atom acts as a donor to O21^{ii} as acceptor

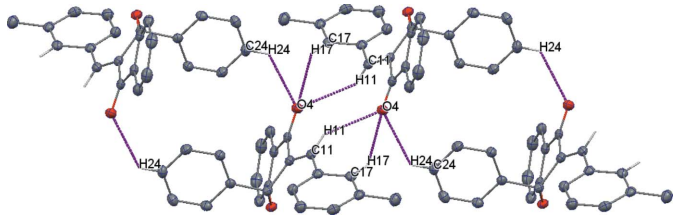


Figure 4
The crystal structure of compound **II**, showing C2—H2···O4^{v}, C5—H5···O1^{vi} and C24—H24···O4^{vii} hydrogen bonds. Generic atom labels without symmetry codes have been used.

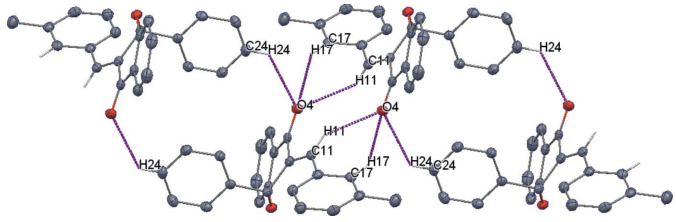


Figure 5
Fragment of the crystal structure of compound **III** showing the double chains formed by two dimers. One dimer is created by C11—H11···O4^{viii} and C17—H17···O4 ^{viii} hydrogen bonds, while the second is produced by the C24—H24···O4^{ix} hydrogen bond. Generic atom labels without symmetry codes have been used.

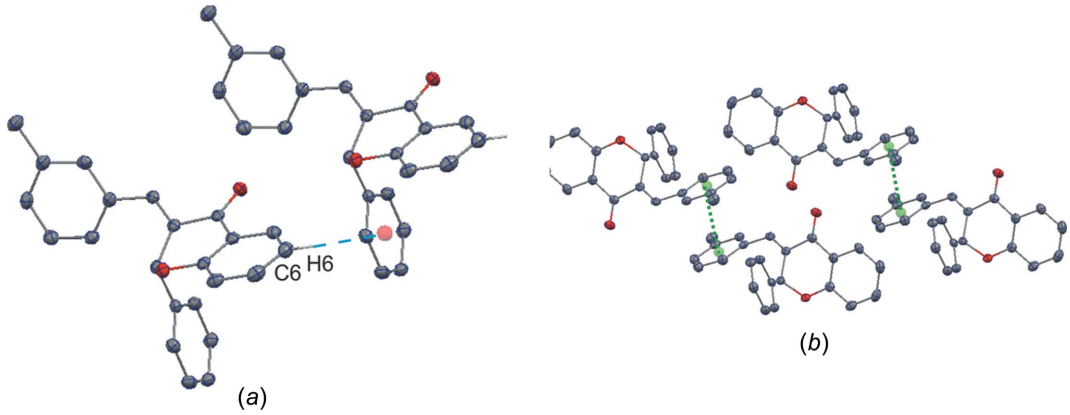


Figure 6
Fragment of the crystal packing of compound **III**, showing (*a*) C6—H6···*Cg*4^{x} and (*b*) π–π interactions between rings 3 (atoms C12–C17). *Cg*4 is the centroid of the C21–C26 ring (red dot) and the centroid of ring 3 is shown as a green dot.

(Fig. 2). This interaction generates a double chain along the [001] direction. C—H···π interactions are also observed in the crystal packing. They are formed by C6—H6···*Cg*4^{iii}, C15—H15···*Cg*4^{iv} and C16—H16···*Cg*2^{iv} (Fig. 3).

In the case of the crystal packing of compound **II**, molecules are linked *via* two hydrogen bonds, *i.e.* C2—H2···O4^{v} and C5—H5···O1^{vi}, forming chains along the [010] direction. In addition, these chains are connected *via* C24—H24···O4^{vii} hydrogen bonds (Fig. 4).

The crystal packing of **III** seems to be stabilized by a combination of intermolecular C—H···O and C—H···π hydrogen bonds and π–π stacking interactions. The C11 and C17 atoms act as donors to the O4^{viii} atom, forming dimers. The other dimer is formed by the C24—H24···O4^{ix} hydrogen bond. However, the three hydrogen bonds given above form double chains propagating along the [100] direction (Fig. 5). The crystal lattice also demonstrates a weak π–π interaction between arene rings substituted at the C3 atom of the chroman moiety, the distance between the centroids (*Cg*3···*Cg*3) being 3.854 (2) Å. Another weak interaction is C6—H6···*Cg*4^{x} (Fig. 6).

Compound **IV** crystallizes in the space group $P2_1/n$ with an asymmetric unit comprising three independent molecules; these are self-assembled into chains along the [$\bar{1}$01] direction, and are always connected by two hydrogen bonds from each molecule, *i.e.* C2—H2···O34^{xi} and C6—H6···O61^{xii} in molecule *A*, C32—H32···O64^{xiv} and C36-H36···O1^{xv} in molecule *B*, and C62—H62···O4^{xvi} and C66—H66···O31^{xvii} in molecule *C*. Each chain combines with another similarly formed chain *via* C14—H14···O1^{xiii}, C44—H44···O31^{xiii} and C74—H74···O61^{xiii} hydrogen bonds shifted by a *b* translation. C—H···π interactions are also evident in the crystal structure of **IV** (Table 2). The shortest centroid–centroid distance (*Cg*2···*Cg*7) of 3.9671 (8) Å was found between parallel arene

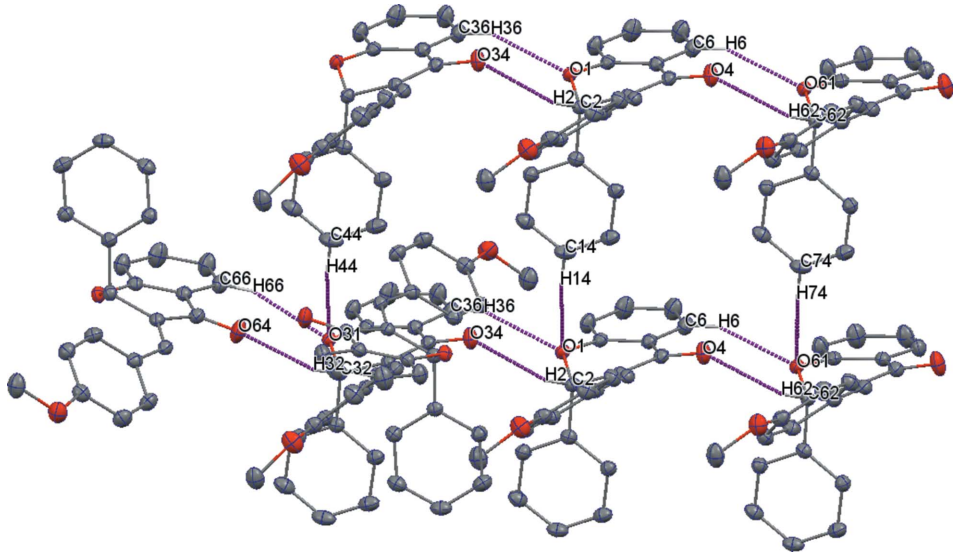


Figure 7
Fragment of the crystal structure of compound **IV**, showing two chains formed between three independent molecules *via* C2—H2···O34^{xi} and C6—H6···O61^{xii} hydrogen bonds for molecule *A*, C32—H32···O64^{xiv} and C36—H36···O1^{xv} for molecule *B*, and C62—H62···O4^{xii} and C66—H66···O31^{xvi} for molecule *C*, linked by C14—H14···O1^{xiii}, C44—H44···O31^{xiii} and C74—H74···O61^{xiii} hydrogen bonds. Generic atom labels without symmetry codes have been used.

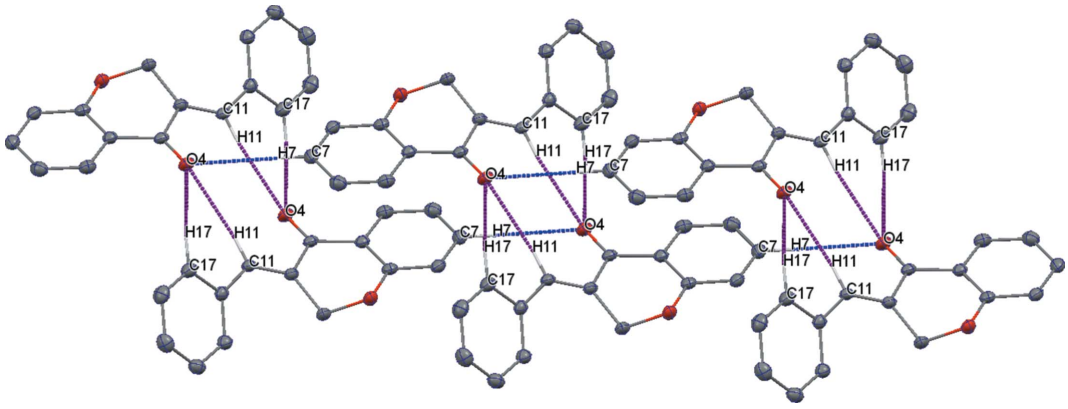


Figure 8
Fragment of the crystal packing of structure **V**, with C7—H7···O4^{xxiii} hydrogen bonds (blue line) forming chains, and C11—H11···O4^{xxiv} and C17—H17···O4x^{xiv} hydrogen bonds (violet line) forming dimers. Generic atom labels without symmetry codes have been used.

rings (fused with the pyran ring) of molecules *A* and *B*; however, it seems that π–π interactions do not play a significant role in the crystal packing (Fig. 7).

In the packing structure of **V**, the molecules are connected *via* a C7—H7···O4^{xxiii} hydrogen bond, forming chains along the [010] direction, while each molecule is also engaged in creating a dimer by a combination of C17—H17···O4^{xxiv} and C11—H11···O4^{xxiv} hydrogen bonds (Fig. 8). These interactions are also enhanced by π–π interactions between arene rings (ring 2) fused with pyran rings, with the *Cg*2···*Cg*2 distance equal to 3.770 (2) Å (Fig. 9).

Compound **VI** crystallizes in the triclinic space group $P\overline{1}$ with two independent molecules in the asymmetric unit. In the crystal structure, two independent molecules are linked by a C2—H2*A*···O22 hydrogen bond. The remaining seven hydrogen bonds (Table 2) create a three-dimensional (3D) net of hydrogen bonds (Fig. 10). In the crystal lattice of **VI**, aromatic π–π interactions are also observed between the arene ring (ring 2) fused with the pyran rings and another arene ring incorporating methoxyphenyl substituents (Fig. 11).

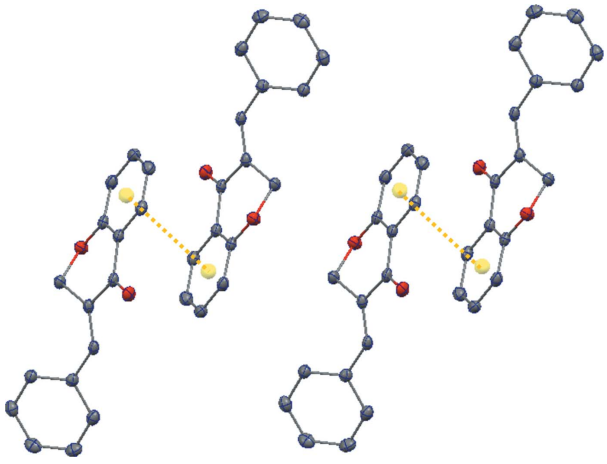


Figure 9
Fragment of the crystal packing of compound **V**, showing π–π interactions between rings 2 (atoms C5–C10).

3.3. Determination of lipophilicity

One of the key determinants influencing the bioavailability, permeability and toxicity of a potential drug is its lipophilicity (Jóźwiak *et al.*, 2001). Lipophilicity is affected by the structure of the molecule, as well as the presence of functional groups, unsaturated bonds and the molecular weight. It is usually defined as the partition coefficient between the organic and aqueous phases (*n*-octanol/water) and is designated as log*P* (Gao & Cao, 2008; Kujawski *et al.*, 2012). The log*P* factor is an important variable used in computer-based drug design, and a number of computational log*P* prediction tools have been designed, such as the *Molinspiration Cheminformatics* software package (https://www.molinspiration.com/). Unfortunately, theoretical methods do not always give results consistent with the experimental results, because they do not take into account the intermolecular interactions taking place in the reaction environment.

Therefore, in the present study, the theoretical lipophilicity values obtained using the *Molinspiration Cheminformatics* program (miLog*P*) were compared with experimental values, determined using reversed-phase thin-layer chromatography (RP-TLC) (Table 3). The experimental method for determining lipophilicity is described in the supporting information. The log*P* results indicate that the tested compounds have a lipophilic character. As the log*P* parameters for compounds **I** and **IV**–**VI** are given in a previous work (Adamus-Grabicka *et al.*, 2020), the present article only discusses the results for compounds **II** and **III**. Briefly, it was found that compound **II** is characterized by greater lipophilicity than compound **III**, probably due to the presence of a methyl group in the *para*

Table 3
The experimental (exp) and theoretical (theor) values of lipophilicity.

Compound	log*P*(exp)	milog*P*(theor)
I	4.90	5.20
II	4.45	5.65
III	3.05	5.63
IV	4.79	5.26
V	4.23	3.62
VI	4.08	3.68

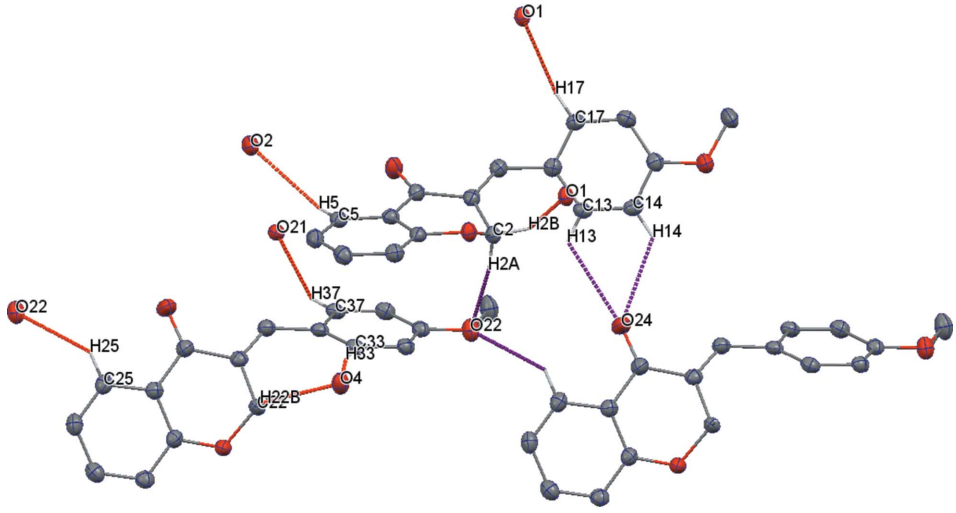


Figure 10
Fragment of the crystal packing of **VI**, demonstrating the hydrogen bond (*i.e.* C2—H2*A*···O22) present between two independent molecules and the 3D net of other hydrogen bonds. Generic atom labels without symmetry codes have been used.

position on the arene ring. Usually, the location of the methyl group in the *meta* position causes a decrease in the log*P* value (Fujita *et al.*, 1964).

All the tested compounds show high lipophilicity and high bioaccumulation potential. They also satisfy the 'rule of five' for drug-like molecules described by Lipinski *et al.* (2001). Therefore, based on the results of the lipophilicity test and Lipinski's rule, it can be assumed that the tested compounds may be good candidates for further research as potential drugs.

3.4. Biological assay

The title compounds were tested for cytotoxicity by the MTT assay against four cell lines: HL-60 (human leukemia cell line), NALM-6 (human peripheral blood leukemia cell line), WM-115 (melanoma cell line, ECACC, Salisbury, UK) and COLO-205 (human colon adenocarcinoma cells). The procedure is given in the supporting information. The results are presented as a logarithmic function in Fig. 12 and as the inhibitory concentration (IC_{50}) in μM in Table S3 in the supporting information.

The biological activity of benzylidene flavanone/chromanone derivatives and the cytotoxic activities of compounds **I**, **IV**, **V** and **VI** are discussed in detail by Adamus-Grabicka *et al.* (2020). Compound **III** exhibits similar cytotoxic activity towards all tested cell lines, although the best cytotoxic activity was observed against COLO-205 (32.64 μ*M*). It demonstrates slightly lower activity (48.81 μ*M*) against HL-60 than compound **II**, where the methyl group is in the *para* position. Compound **III** also shows better antiproliferative character towards the other three cell lines.

Comparing the tested compounds, the best cytotoxic activity was demonstrated by compound **V**, *i.e.* an unsubstituted 3-arylidenechromanone, especially towards the leukemia cell lines (HL-60 and NALM-6) (Fig. 12). All tested compounds show different levels of cytotoxic activity ranging from high (5.4 ± 0.6 μ*M*) to virtually no activity (>250 μ*M*); however, compound **V** appeared to be the best cytotoxic agent against all four cancer cell lines, as well as towards reference compounds. In addition, all compounds demonstrated a logarithmic dependence of $\frac{1}{IC_{50}}$ towards leukemia cell lines. It is worth mentioning that the compounds demonstrating greater toxicity to the HL-60 line also demonstrated greater toxicity to the NALM-6 line (Fig. S1 in the supporting information).

The effect of the synthesized and reference compounds on erythrocyte hemolysis was also investigated. Neither compound **II** nor **III** was found to have any unfavourable effect on the erythrocyte membrane over the whole tested concentration range. In addition, erythrocyte morphology was also

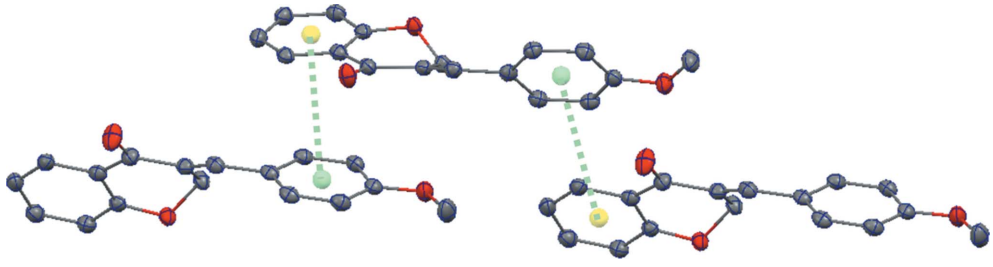


Figure 11
Fragment of the crystal packing lattice of **VI**, showing π–π interactions. The yellow dots indicate the centroids of the C5–C10 (molecule *A*) and C25–C30 (molecule *B*) rings, while the green dots indicate the centroids of the C12–C17 (molecule *A*) and C32–C37 (molecule *B*) rings.

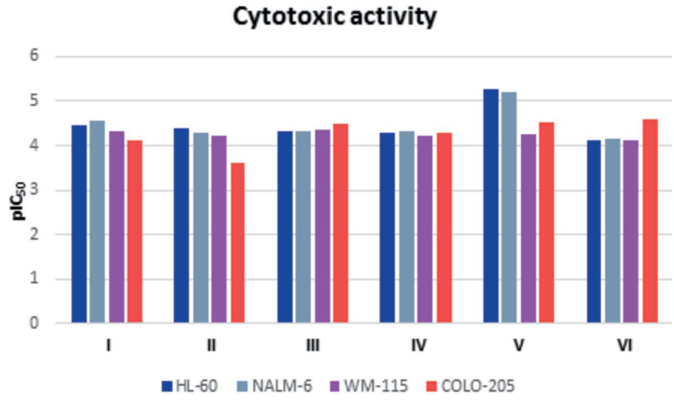


Figure 12
The cytotoxic activity (expressed as $\log\frac{1}{IC_{50}}$) against the HL-60, NALM-6, WM-115 and COLO-205 cancer cell lines.

examined as a complement to the erythrotoxicity studies; the methodology and results of the RBC lysis and morphology tests are described in Adamus-Grabicka *et al.* (2020). None of the tested compounds was found to be toxic towards red blood cells at cytotoxic concentrations, and none contributed to RBC hemolysis.

3.5. Hirshfeld surface

The Hirshfeld surfaces (Hirshfeld, 1977; Spackman & Jayatilaka, 2009) of the crystal structures of compounds **I**–**VI** were determined using the *CrystalExplorer* program (Turner *et al.*, 2017). Hirshfeld surface analysis is an efficient method for identifying intermolecular interactions in the solid state and mapping their properties in useful modes, such as normalized contact distance (d_{norm}) surface, curvedness surface, shape index and two-dimensional (2D) fingerprints (McKinnon *et al.*, 2007). The Hirshfeld surfaces mapped over d_{norm} are presented in Fig. S2 in the supporting information; this figure presents the similarities and differences in the crystal structures between compounds **I**–**VI**. The vivid red spots present contacts which correspond to the interactions described above (Fig. S2 in the supporting information, and Table 2).

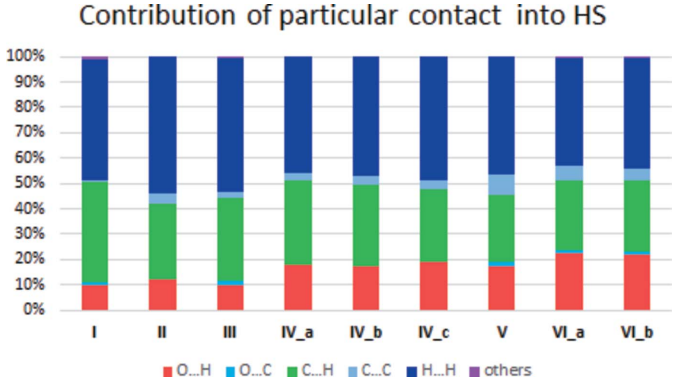


Figure 13
The percentage contributions to the Hirshfeld surface area of different close intermolecular contacts for molecules **I**–**VI**.

In addition, 2D fingerprint plots are given, and these are decomposed into fractions to provide quantitative information regarding individual interactions (Fig. S2 in the supporting information). These figures also include the percentage contribution of the particular contact in the overall Hirshfeld surface (Fig. 13). Although the 2D fingerprint plots differ between compounds **I**–**VI**, distinctive common features can be identified. The C—H$\cdots\pi$ interactions observed in the crystal structures of **I**, **III**, **IV** and **VI** occur as 'chicken-wing' shapes on two sides of the fingerprint plots. Additionally, the participation of π–π interactions in the crystal structures of **III**, **V** and **VI** shows up as a light-blue region near the centre of the plot.

The relative contribution of the different interactions to the Hirshfeld surface is presented as a histogram in Fig. 13. From this comparison, it can be clearly seen that hydrogen-bonding interactions (C—H$\cdots$O and C—H$\cdots\pi$) cover nearly 50% of the surface area, where C$\cdots$H contacts dominate. Less than 10% of the surface can be associated with C$\cdots$C contacts, often referred to as π–π interactions. The dominant interaction in all the derivatives is the H$\cdots$H interaction, constituting 43–54% of all interactions.

3.5.1. C$\cdots$C contacts contribution *versus* cytotoxic activity and lipophilicity. Simple correlations were found between the results of the Hirshfeld surface analysis and the cytotoxicity values for these compounds. The cytotoxic activity of all the compounds against WM-115 (melanoma cell line) is inversely proportional to the percentage contribution of C$\cdots$C contacts to the Hirshfeld surface (Fig. 14). This indicates that the activity of these compounds is strongly influenced by intermolecular interactions.

A comparison of the log*P* value (theoretical) with the contribution of various contacts to the Hirshfeld surface found that theoretical lipophilicity (log*P*) is also inversely proportional to the percentage contribution of the C$\cdots$C contacts to the Hirshfeld surface. This analysis has been extended by similar flavonoid compounds and benzylidenochromanone derivatives available from our previous research (Kupcewicz *et al.*, 2013; Adamus-Grabicka *et al.*, 2018; Suchojad *et al.*,

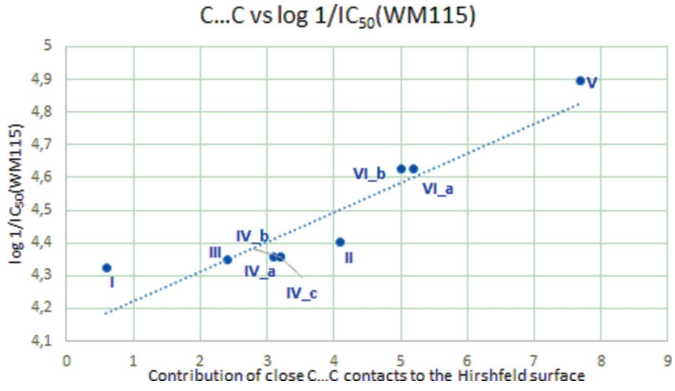


Figure 14
The relationship between cytotoxic activity (given as $\log\frac{1}{IC_{50}}$) and the percentage contribution to the Hirshfeld surface covered by C$\cdots$C contacts against cell line WM115.

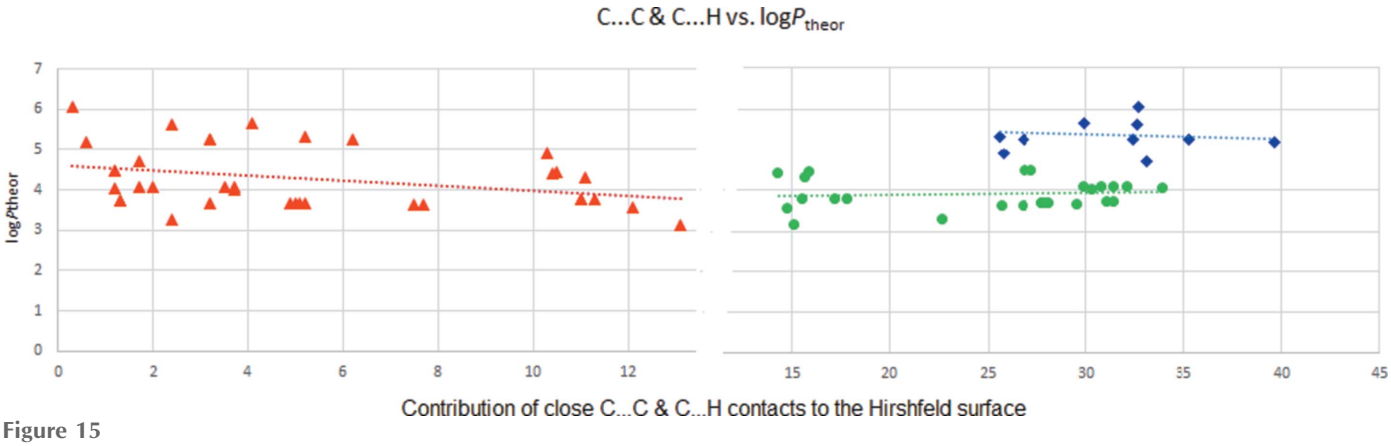


Figure 15
The relationship between the log*P* value (theoretical) and the fraction of C···C (red triangles) and C···H (green dots are flavonoids and blue squares are chromanones) interactions with respect to the Hirshfeld surface.

2019) or from the Cambridge Structural Database [Version 5.41, last update November 2019; Groom *et al.*, 2016; FAVVIH (Katrusiak *et al.*, 1987), NAMXIK, NAMXOQ, NAMXUW,-NAMYEH, NAMYOR, NAMYUX, NAMZEI, NAMZOS, NAMZUY and NANBAH (Cheng *et al.*, 2011), NANDOB (Valkonen *et al.*, 2012), SECBAF (Gopaul *et al.*, 2012*a*), YAWLEP (Gopaul *et al.*, 2012*b*) and YEJPIO (Gopaul & Koorbanally, 2012)]. The scheme used for the search is presented in Fig. S2 in the supporting information. Disordered structures were omitted. The total number of analyzed compounds is 7 flavonoids and 19 benzylidenechromanones. For all compounds, the complete Hirshfeld surface analysis were carried out with fingerprint plots and contacts contribution to the Hirshfeld surface (Fig. S3 in the supporting information). These findings provide a better correlation between the contributions of C···C contacts and log*P* than that reported previously (Małecka & Budzisz, 2014). However, it can be seen that the percentage contribution of C···H contacts in the Hirshfeld surface also shows an acceptable relationship with log*P* separately for flavonoids and for benzylidenechromanone derivatives (Fig. 15). These findings clearly indicate that intermolecular interactions have a strong influence on the activity of the tested compounds. Therefore, the cytotoxic activity of a potential drug, and some of its other descriptors, such as log*P*, can be successfully predicted based on the fraction of contacts contributing to the Hirshfeld surface. These findings support our latest QSAR model (Kupcewicz *et al.*, 2016) and could help future efforts towards structure-based drug design.

4. Conclusion

The present study examines the relationship between the structures of a group of flavonoid and chromanone derivatives and their cytotoxic activity. An inverse relationship was found between the percentage contribution of C···C contacts to the Hirshfeld surface and cytotoxic activity against the WM-115 cancer line. It was also revealed that the log*P* value is dependent upon the percentage contributions of the C···C and C···H contacts to the Hirshfeld surface. In this case, it is possible to predict the lipophilicity of a compound on the basis of X-ray crystallographic data and *vice versa*. Although these results were derived from a limited data set, they nevertheless indicate that quantitative data obtained by Hirshfeld surface analysis may be valuable in QSAR studies. QSAR models built on such descriptors become interpretable.

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supporting information

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The relationship between Hirshfeld potential and cytotoxic activity: a study along a series of flavonoid and chromanone derivatives

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Computing details

Data collection: *CrysAlis PRO* (Rigaku OD, 2015) for compound_I_AAG10, compound_II_AAG9, compound_III_AAG1, compound_V_AAG5; *CrysAlis PRO* (Agilent, 2013) for compound_IV_AAG2; *CrysAlis PRO* (Rigaku OD, 2019) for AAG4. Cell refinement: *CrysAlis PRO* (Rigaku OD, 2015) for compound_I_AAG10, compound_II_AAG9, compound_III_AAG1, compound_V_AAG5; *CrysAlis PRO* (Agilent, 2013) for compound_IV_AAG2; *CrysAlis PRO* (Rigaku OD, 2019) for AAG4. Data reduction: *CrysAlis PRO* (Rigaku OD, 2015) for compound_I_AAG10, compound_II_AAG9, compound_III_AAG1, compound_V_AAG5; *CrysAlis PRO* (Agilent, 2013) for compound_IV_AAG2; *CrysAlis PRO* (Rigaku OD, 2019) for AAG4. For all structures, program(s) used to solve structure: *SHELXS* (Sheldrick, 2015a); program(s) used to refine structure: *SHELXL2014* (Sheldrick, 2015b); molecular graphics: *DIAMOND* (Brandenburg & Putz, 1999); software used to prepare material for publication: *SHELXL2014* (Sheldrick, 2015b) and *publCIF* (Westrip, 2010).

(*E*)-3-Benzylidene-2-phenylchroman-4-one (compound_I_AAG10)

Crystal data

$C_{22}H_{16}O_2$
M_r = 312.35
Monoclinic, *C*2/*c*
a = 10.2487 (3) Å
b = 16.3033 (4) Å
c = 19.7539 (7) Å
β = 100.420 (3)°
V = 3246.20 (17) Å^3
Z = 8
F(000) = 1312
D_x = 1.278 Mg m^{-3}
Mo *Kα* radiation, *λ* = 0.71073 Å
Cell parameters from 7518 reflections
θ = 3.4–35.7°
μ = 0.08 mm^{-1}
T = 100 K
Plate, colorless
0.28 × 0.26 × 0.08 mm

Data collection

Rigaku OD SuperNova Dual source diffractometer with an Atlas detector
Radiation source: micro-focus sealed X-ray tube, SuperNova (Mo) X-ray Source
Mirror monochromator
Detector resolution: 10.4498 pixels mm^{-1}
ω scans
Absorption correction: multi-scan (CrysAlis PRO; Rigaku OD, 2015)
T_{min} = 0.744, T_{max} = 1.000
13340 measured reflections
13340 independent reflections
11168 reflections with $I > 2\sigma(I)$
R_{int} = 0.042
θ_{max} = 26.5°, θ_{min} = 3.3°
h = −12→9
k = −20→20
l = −24→24

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.060$
$wR(F^2) = 0.153$
$S = 1.02$
13340 reflections
205 parameters
0 restraints

Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.060P)^2 + 1.5246P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.49$ e Å^{-3}
$\Delta\rho_{min} = -0.38$ e Å^{-3}

Special details

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

Refinement. Refined as a 2-component perfect twin.

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

	x	*y*	*z*	U_{iso}*/U_{eq}
O1	0.32317 (14)	0.18634 (9)	0.46640 (7)	0.0255 (4)
O4	0.17916 (15)	0.30418 (10)	0.28534 (9)	0.0375 (4)
C2	0.2187 (2)	0.14416 (13)	0.41919 (11)	0.0226 (4)
H2	0.1530	0.1238	0.4471	0.027*
C3	0.14523 (19)	0.20038 (12)	0.36501 (11)	0.0220 (4)
C4	0.2246 (2)	0.26304 (13)	0.33592 (11)	0.0248 (5)
C5	0.4459 (2)	0.33109 (14)	0.34907 (13)	0.0320 (5)
H5	0.4200	0.3550	0.3048	0.038*
C6	0.5669 (2)	0.35083 (15)	0.38841 (14)	0.0377 (6)
H6	0.6254	0.3871	0.3709	0.045*
C7	0.6030 (2)	0.31731 (14)	0.45384 (14)	0.0350 (6)
H7	0.6850	0.3325	0.4816	0.042*
C8	0.5213 (2)	0.26219 (13)	0.47898 (12)	0.0288 (5)
H8	0.5471	0.2394	0.5237	0.035*
C9	0.40053 (19)	0.24013 (12)	0.43838 (11)	0.0225 (4)
C10	0.36040 (19)	0.27579 (13)	0.37392 (11)	0.0241 (5)
C11	0.01360 (19)	0.19908 (13)	0.34047 (11)	0.0249 (5)
H11	−0.0155	0.2393	0.3063	0.030*
C12	−0.09296 (11)	0.14678 (8)	0.35704 (8)	0.0276 (5)
C13	−0.07390 (11)	0.06755 (9)	0.38294 (8)	0.0342 (6)
H13	0.0124	0.0441	0.3909	0.041*
C14	−0.18105 (15)	0.02267 (8)	0.39717 (9)	0.0425 (6)
H14	−0.1680	−0.0315	0.4149	0.051*
C15	−0.30727 (12)	0.05701 (10)	0.38550 (9)	0.0448 (7)
H15	−0.3805	0.0263	0.3952	0.054*
C16	−0.32633 (10)	0.13624 (10)	0.35960 (8)	0.0412 (6)
H16	−0.4126	0.1597	0.3516	0.049*
C17	−0.21918 (13)	0.18112 (8)	0.34537 (8)	0.0334 (5)
H17	−0.2322	0.2353	0.3277	0.040*

C18	0.27922 (18)	0.06873 (12)	0.39076 (11)	0.0213 (4)
C19	0.34181 (19)	0.01096 (13)	0.43743 (12)	0.0265 (5)
H19	0.3452	0.0193	0.4853	0.032*
C20	0.3992 (2)	−0.05840 (13)	0.41512 (13)	0.0311 (5)
H20	0.4422	−0.0970	0.4477	0.037*
C21	0.3943 (2)	−0.07174 (13)	0.34587 (13)	0.0327 (5)
H21	0.4342	−0.1192	0.3305	0.039*
C22	0.3308 (2)	−0.01541 (15)	0.29893 (13)	0.0362 (6)
H22	0.3261	−0.0244	0.2510	0.043*
C23	0.2737 (2)	0.05466 (14)	0.32169 (12)	0.0302 (5)
H23	0.2304	0.0931	0.2890	0.036*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0270 (8)	0.0262 (8)	0.0219 (8)	−0.0036 (6)	0.0005 (6)	−0.0032 (6)
O4	0.0286 (8)	0.0346 (9)	0.0432 (10)	−0.0068 (7)	−0.0099 (7)	0.0148 (8)
C2	0.0199 (10)	0.0250 (11)	0.0225 (10)	−0.0053 (8)	0.0021 (8)	−0.0006 (8)
C3	0.0184 (10)	0.0196 (10)	0.0278 (11)	0.0000 (8)	0.0032 (8)	−0.0022 (8)
C4	0.0195 (10)	0.0221 (10)	0.0310 (12)	0.0009 (8)	−0.0003 (8)	0.0019 (9)
C5	0.0240 (11)	0.0291 (12)	0.0409 (14)	−0.0027 (9)	0.0006 (10)	0.0078 (10)
C6	0.0216 (11)	0.0318 (13)	0.0575 (17)	−0.0062 (10)	0.0013 (11)	0.0059 (11)
C7	0.0211 (11)	0.0277 (12)	0.0512 (15)	−0.0014 (9)	−0.0066 (10)	−0.0053 (10)
C8	0.0283 (11)	0.0240 (11)	0.0307 (12)	0.0019 (9)	−0.0038 (9)	−0.0056 (9)
C9	0.0198 (10)	0.0193 (10)	0.0279 (11)	0.0023 (8)	0.0029 (8)	−0.0040 (8)
C10	0.0161 (10)	0.0232 (10)	0.0317 (12)	0.0005 (8)	0.0008 (8)	0.0000 (8)
C11	0.0202 (10)	0.0246 (11)	0.0296 (11)	0.0000 (8)	0.0037 (9)	−0.0057 (8)
C12	0.0215 (11)	0.0351 (12)	0.0263 (11)	−0.0077 (9)	0.0044 (9)	−0.0101 (9)
C13	0.0268 (12)	0.0421 (14)	0.0351 (13)	−0.0108 (10)	0.0092 (10)	−0.0098 (10)
C14	0.0501 (16)	0.0438 (15)	0.0362 (14)	−0.0207 (12)	0.0143 (12)	−0.0061 (11)
C15	0.0267 (13)	0.075 (2)	0.0351 (14)	−0.0264 (13)	0.0111 (11)	−0.0146 (13)
C16	0.0232 (12)	0.0681 (19)	0.0325 (14)	−0.0093 (11)	0.0056 (10)	−0.0099 (12)
C17	0.0228 (11)	0.0497 (15)	0.0279 (12)	−0.0037 (10)	0.0049 (9)	−0.0109 (10)
C18	0.0162 (9)	0.0191 (10)	0.0276 (11)	−0.0041 (8)	0.0014 (8)	0.0025 (8)
C19	0.0202 (10)	0.0285 (11)	0.0286 (12)	−0.0037 (8)	−0.0012 (8)	0.0054 (9)
C20	0.0194 (10)	0.0219 (11)	0.0470 (14)	−0.0029 (9)	−0.0073 (9)	0.0093 (10)
C21	0.0231 (11)	0.0193 (11)	0.0528 (16)	−0.0017 (9)	−0.0011 (10)	−0.0071 (10)
C22	0.0438 (14)	0.0321 (13)	0.0307 (13)	0.0033 (11)	0.0016 (11)	−0.0072 (10)
C23	0.0354 (12)	0.0238 (11)	0.0291 (12)	0.0059 (9)	0.0000 (9)	0.0036 (9)

Geometric parameters (Å, º)

O1—C9	1.365 (2)	C12—C17	1.3900
O1—C2	1.459 (2)	C13—C14	1.3900
O4—C4	1.223 (3)	C13—H13	0.9500
C2—C3	1.504 (3)	C14—C15	1.3900
C2—C18	1.529 (3)	C14—H14	0.9500
C2—H2	1.0000	C15—C16	1.3900

C3—C11	1.349 (3)	C15—H15	0.9500
C3—C4	1.485 (3)	C16—C17	1.3900
C4—C10	1.472 (3)	C16—H16	0.9500
C5—C6	1.378 (3)	C17—H17	0.9500
C5—C10	1.406 (3)	C18—C23	1.375 (3)
C5—H5	0.9500	C18—C19	1.391 (3)
C6—C7	1.390 (4)	C19—C20	1.383 (3)
C6—H6	0.9500	C19—H19	0.9500
C7—C8	1.381 (3)	C20—C21	1.377 (3)
C7—H7	0.9500	C20—H20	0.9500
C8—C9	1.395 (3)	C21—C22	1.381 (3)
C8—H8	0.9500	C21—H21	0.9500
C9—C10	1.392 (3)	C22—C23	1.395 (3)
C11—C12	1.468 (2)	C22—H22	0.9500
C11—H11	0.9500	C23—H23	0.9500
C12—C13	1.3900		

C9—O1—C2	117.18 (16)	C13—C12—C11	124.12 (12)
O1—C2—C3	112.51 (16)	C17—C12—C11	115.88 (12)
O1—C2—C18	108.33 (15)	C14—C13—C12	120.0
C3—C2—C18	114.39 (17)	C14—C13—H13	120.0
O1—C2—H2	107.1	C12—C13—H13	120.0
C3—C2—H2	107.1	C13—C14—C15	120.0
C18—C2—H2	107.1	C13—C14—H14	120.0
C11—C3—C4	117.01 (19)	C15—C14—H14	120.0
C11—C3—C2	125.86 (19)	C14—C15—C16	120.0
C4—C3—C2	117.13 (17)	C14—C15—H15	120.0
O4—C4—C10	121.6 (2)	C16—C15—H15	120.0
O4—C4—C3	122.62 (19)	C17—C16—C15	120.0
C10—C4—C3	115.69 (18)	C17—C16—H16	120.0
C6—C5—C10	120.5 (2)	C15—C16—H16	120.0
C6—C5—H5	119.8	C16—C17—C12	120.0
C10—C5—H5	119.8	C16—C17—H17	120.0
C5—C6—C7	119.6 (2)	C12—C17—H17	120.0
C5—C6—H6	120.2	C23—C18—C19	118.4 (2)
C7—C6—H6	120.2	C23—C18—C2	123.52 (18)
C8—C7—C6	120.9 (2)	C19—C18—C2	118.04 (19)
C8—C7—H7	119.6	C20—C19—C18	120.9 (2)
C6—C7—H7	119.6	C20—C19—H19	119.5
C7—C8—C9	119.6 (2)	C18—C19—H19	119.5
C7—C8—H8	120.2	C21—C20—C19	120.3 (2)
C9—C8—H8	120.2	C21—C20—H20	119.8
O1—C9—C10	122.82 (18)	C19—C20—H20	119.8
O1—C9—C8	116.91 (19)	C20—C21—C22	119.3 (2)
C10—C9—C8	120.2 (2)	C20—C21—H21	120.3
C9—C10—C5	119.17 (19)	C22—C21—H21	120.3
C9—C10—C4	120.21 (19)	C21—C22—C23	120.2 (2)
C5—C10—C4	120.3 (2)	C21—C22—H22	119.9

C3—C11—C12	131.7 (2)	C23—C22—H22	119.9
C3—C11—H11	114.2	C18—C23—C22	120.8 (2)
C12—C11—H11	114.2	C18—C23—H23	119.6
C13—C12—C17	120.0	C22—C23—H23	119.6
C9—O1—C2—C3	−42.9 (2)	C3—C4—C10—C5	177.17 (19)
C9—O1—C2—C18	84.6 (2)	C4—C3—C11—C12	−179.93 (19)
O1—C2—C3—C11	−141.2 (2)	C2—C3—C11—C12	0.0 (4)
C18—C2—C3—C11	94.6 (2)	C3—C11—C12—C13	−26.0 (3)
O1—C2—C3—C4	38.7 (2)	C3—C11—C12—C17	153.9 (2)
C18—C2—C3—C4	−85.5 (2)	C17—C12—C13—C14	0.0
C11—C3—C4—O4	−10.5 (3)	C11—C12—C13—C14	179.93 (16)
C2—C3—C4—O4	169.6 (2)	C12—C13—C14—C15	0.0
C11—C3—C4—C10	166.58 (19)	C13—C14—C15—C16	0.0
C2—C3—C4—C10	−13.4 (3)	C14—C15—C16—C17	0.0
C10—C5—C6—C7	−1.7 (4)	C15—C16—C17—C12	0.0
C5—C6—C7—C8	2.4 (4)	C13—C12—C17—C16	0.0
C6—C7—C8—C9	−0.3 (3)	C11—C12—C17—C16	−179.93 (15)
C2—O1—C9—C10	21.3 (3)	O1—C2—C18—C23	−123.2 (2)
C2—O1—C9—C8	−162.10 (17)	C3—C2—C18—C23	3.2 (3)
C7—C8—C9—O1	−179.08 (19)	O1—C2—C18—C19	57.6 (2)
C7—C8—C9—C10	−2.4 (3)	C3—C2—C18—C19	−176.08 (17)
O1—C9—C10—C5	179.55 (18)	C23—C18—C19—C20	1.1 (3)
C8—C9—C10—C5	3.1 (3)	C2—C18—C19—C20	−179.64 (17)
O1—C9—C10—C4	6.5 (3)	C18—C19—C20—C21	−0.5 (3)
C8—C9—C10—C4	−170.01 (19)	C19—C20—C21—C22	−0.4 (3)
C6—C5—C10—C9	−1.0 (3)	C20—C21—C22—C23	0.7 (3)
C6—C5—C10—C4	172.0 (2)	C19—C18—C23—C22	−0.7 (3)
O4—C4—C10—C9	167.2 (2)	C2—C18—C23—C22	−180.0 (2)
C3—C4—C10—C9	−9.8 (3)	C21—C22—C23—C18	−0.2 (4)
O4—C4—C10—C5	−5.8 (3)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C11—H11···O4^{i}	0.95	2.47	3.353 (3)	154
C17—H17···O4^{i}	0.95	2.64	3.353 (2)	132
C21—H21···O4^{ii}	0.95	2.68	3.267 (3)	120

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

(*E*)-3-(4-Methylbenzylidene)-2-phenylchroman-4-one (compound_II_AAG9)

Crystal data

$C_{23}H_{18}O_2$
M_r = 326.37
Monoclinic, $P2_1/c$
a = 16.6799 (5) Å
b = 7.9102 (2) Å
c = 13.1511 (3) Å
β = 98.558 (3)°
V = 1715.85 (8) Å^3
Z = 4
F(000) = 688
D_x = 1.263 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å

Cell parameters from 4576 reflections
θ = 3.2–35.6°
μ = 0.08 mm^{-1}

T = 100 K
Needle, colorless
0.28 × 0.1 × 0.1 mm

Data collection

Rigaku OD SuperNova Dual source diffractometer with an Atlas detector
Radiation source: micro-focus sealed X-ray tube, SuperNova (Mo) X-ray Source
Mirror monochromator
Detector resolution: 10.4498 pixels mm^{-1}
ω scans
Absorption correction: multi-scan (CrysAlis PRO; Rigaku OD, 2015)

T_{min} = 0.833, T_{max} = 1.000
13962 measured reflections
3550 independent reflections
2927 reflections with $I > 2\sigma(I)$
R_{int} = 0.031
θ_{max} = 26.5°, θ_{min} = 3.1°
h = −20→20
k = −9→9
l = −11→16

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.040
$wR(F^2)$ = 0.107
S = 1.05
3550 reflections
227 parameters
0 restraints

Primary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0551P)^2 + 0.3869P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.19 e Å^{-3}
$\Delta\rho_{min}$ = −0.23 e Å^{-3}

Special details

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

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

	x	y	z	U_{iso}*/U_{eq}
O1	0.13016 (5)	0.69823 (11)	0.61728 (6)	0.0197 (2)
O4	0.24076 (6)	0.72695 (14)	0.91481 (7)	0.0330 (3)
C2	0.21464 (7)	0.64404 (15)	0.63602 (9)	0.0167 (2)
H2	0.2425	0.6974	0.5818	0.020*
C3	0.25695 (7)	0.70595 (14)	0.73857 (9)	0.0169 (2)
C4	0.20880 (8)	0.70299 (15)	0.82561 (9)	0.0199 (3)
C5	0.07131 (8)	0.65060 (17)	0.87504 (10)	0.0249 (3)
H5	0.0945	0.6518	0.9455	0.030*
C6	−0.01103 (8)	0.62868 (19)	0.84830 (11)	0.0304 (3)
H6	−0.0445	0.6141	0.9001	0.036*
C7	−0.04520 (8)	0.6279 (2)	0.74468 (11)	0.0325 (3)
H7	−0.1021	0.6131	0.7265	0.039*
C8	0.00253 (8)	0.64841 (18)	0.66811 (10)	0.0273 (3)
H8	−0.0212	0.6477	0.5978	0.033*
C9	0.08575 (8)	0.67000 (15)	0.69525 (9)	0.0195 (3)
C10	0.12126 (7)	0.67117 (15)	0.79869 (9)	0.0196 (3)

C11	0.33397 (7)	0.76156 (15)	0.75777 (9)	0.0185 (3)
H11	0.3504	0.7991	0.8263	0.022*
C12	0.39695 (7)	0.77435 (15)	0.69160 (9)	0.0192 (3)
C13	0.39774 (7)	0.68092 (16)	0.60070 (9)	0.0210 (3)
H13	0.3545	0.6054	0.5780	0.025*
C14	0.46100 (8)	0.69780 (17)	0.54379 (9)	0.0225 (3)
H14	0.4597	0.6347	0.4821	0.027*
C15	0.52641 (8)	0.80485 (17)	0.57477 (10)	0.0254 (3)
C16	0.52603 (8)	0.89659 (18)	0.66538 (11)	0.0295 (3)
H16	0.5699	0.9705	0.6884	0.035*
C17	0.46310 (8)	0.88198 (17)	0.72228 (10)	0.0253 (3)
H17	0.4646	0.9462	0.7836	0.030*
C18	0.59644 (9)	0.8178 (2)	0.51466 (12)	0.0346 (3)
H18A	0.6390	0.7379	0.5426	0.052*
H18B	0.6182	0.9331	0.5200	0.052*
H18C	0.5776	0.7908	0.4423	0.052*
C21	0.21801 (7)	0.45373 (15)	0.62104 (9)	0.0177 (3)
C22	0.18813 (7)	0.38872 (16)	0.52380 (9)	0.0220 (3)
H22	0.1658	0.4631	0.4702	0.026*
C23	0.19088 (8)	0.21637 (17)	0.50521 (10)	0.0267 (3)
H23	0.1705	0.1731	0.4390	0.032*
C24	0.22339 (8)	0.10705 (17)	0.58321 (11)	0.0284 (3)
H24	0.2254	−0.0109	0.5704	0.034*
C25	0.25287 (8)	0.17031 (17)	0.67978 (11)	0.0285 (3)
H25	0.2749	0.0955	0.7332	0.034*
C26	0.25024 (8)	0.34321 (16)	0.69859 (10)	0.0227 (3)
H26	0.2706	0.3860	0.7649	0.027*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0176 (4)	0.0237 (5)	0.0177 (4)	0.0045 (3)	0.0022 (3)	0.0018 (3)
O4	0.0259 (5)	0.0560 (7)	0.0170 (4)	−0.0066 (5)	0.0030 (4)	−0.0072 (4)
C2	0.0152 (6)	0.0182 (6)	0.0167 (5)	0.0011 (4)	0.0023 (4)	0.0012 (4)
C3	0.0197 (6)	0.0134 (6)	0.0174 (6)	0.0020 (4)	0.0022 (5)	0.0006 (4)
C4	0.0219 (6)	0.0207 (6)	0.0173 (6)	0.0009 (5)	0.0029 (5)	−0.0007 (5)
C5	0.0258 (7)	0.0278 (7)	0.0219 (6)	0.0013 (5)	0.0062 (5)	−0.0014 (5)
C6	0.0251 (7)	0.0378 (8)	0.0308 (7)	−0.0009 (6)	0.0126 (6)	−0.0028 (6)
C7	0.0180 (7)	0.0430 (9)	0.0371 (8)	0.0000 (6)	0.0055 (6)	−0.0056 (6)
C8	0.0211 (7)	0.0343 (8)	0.0254 (6)	0.0034 (6)	0.0004 (5)	−0.0019 (6)
C9	0.0202 (6)	0.0177 (6)	0.0211 (6)	0.0029 (5)	0.0047 (5)	−0.0003 (5)
C10	0.0207 (6)	0.0180 (6)	0.0203 (6)	0.0016 (5)	0.0039 (5)	−0.0017 (5)
C11	0.0220 (6)	0.0168 (6)	0.0162 (5)	0.0010 (5)	0.0011 (5)	−0.0011 (4)
C12	0.0174 (6)	0.0197 (6)	0.0199 (6)	0.0023 (5)	0.0011 (5)	0.0018 (5)
C13	0.0184 (6)	0.0213 (6)	0.0224 (6)	0.0005 (5)	0.0004 (5)	−0.0010 (5)
C14	0.0214 (6)	0.0264 (7)	0.0194 (6)	0.0046 (5)	0.0015 (5)	−0.0006 (5)
C15	0.0210 (6)	0.0269 (7)	0.0291 (7)	0.0024 (5)	0.0062 (5)	0.0031 (5)
C16	0.0214 (7)	0.0298 (7)	0.0376 (8)	−0.0074 (5)	0.0051 (6)	−0.0058 (6)

C17	0.0235 (7)	0.0270 (7)	0.0251 (6)	−0.0031 (5)	0.0024 (5)	−0.0068 (5)
C18	0.0244 (7)	0.0407 (9)	0.0413 (8)	−0.0007 (6)	0.0132 (6)	0.0009 (7)
C21	0.0146 (6)	0.0182 (6)	0.0212 (6)	−0.0013 (4)	0.0055 (5)	−0.0009 (5)
C22	0.0201 (6)	0.0230 (7)	0.0229 (6)	−0.0012 (5)	0.0030 (5)	−0.0015 (5)
C23	0.0233 (7)	0.0261 (7)	0.0320 (7)	−0.0058 (5)	0.0080 (5)	−0.0094 (5)
C24	0.0249 (7)	0.0166 (6)	0.0459 (8)	−0.0014 (5)	0.0132 (6)	−0.0038 (6)
C25	0.0272 (7)	0.0211 (7)	0.0376 (8)	0.0036 (5)	0.0066 (6)	0.0062 (6)
C26	0.0222 (6)	0.0215 (6)	0.0243 (6)	0.0012 (5)	0.0038 (5)	0.0020 (5)

Geometric parameters (Å, º)

O1—C9	1.3696 (14)	C13—C14	1.3879 (17)
O1—C2	1.4582 (14)	C13—H13	0.9500
O4—C4	1.2288 (15)	C14—C15	1.3933 (19)
C2—C3	1.5078 (16)	C14—H14	0.9500
C2—C21	1.5204 (16)	C15—C16	1.3960 (19)
C2—H2	1.0000	C15—C18	1.5082 (18)
C3—C11	1.3458 (17)	C16—C17	1.3817 (19)
C3—C4	1.4932 (16)	C16—H16	0.9500
C4—C10	1.4720 (17)	C17—H17	0.9500
C5—C6	1.3770 (19)	C18—H18A	0.9800
C5—C10	1.4064 (17)	C18—H18B	0.9800
C5—H5	0.9500	C18—H18C	0.9800
C6—C7	1.397 (2)	C21—C26	1.3893 (17)
C6—H6	0.9500	C21—C22	1.3995 (17)
C7—C8	1.3830 (19)	C22—C23	1.3870 (18)
C7—H7	0.9500	C22—H22	0.9500
C8—C9	1.3915 (18)	C23—C24	1.389 (2)
C8—H8	0.9500	C23—H23	0.9500
C9—C10	1.4006 (17)	C24—C25	1.385 (2)
C11—C12	1.4637 (17)	C24—H24	0.9500
C11—H11	0.9500	C25—C26	1.3917 (19)
C12—C17	1.4046 (17)	C25—H25	0.9500
C12—C13	1.4072 (17)	C26—H26	0.9500
C9—O1—C2	116.24 (9)	C14—C13—H13	119.6
O1—C2—C3	111.57 (9)	C12—C13—H13	119.6
O1—C2—C21	108.82 (9)	C13—C14—C15	121.73 (12)
C3—C2—C21	114.59 (10)	C13—C14—H14	119.1
O1—C2—H2	107.2	C15—C14—H14	119.1
C3—C2—H2	107.2	C14—C15—C16	117.56 (12)
C21—C2—H2	107.2	C14—C15—C18	121.46 (12)
C11—C3—C4	117.95 (10)	C16—C15—C18	120.96 (13)
C11—C3—C2	125.50 (11)	C17—C16—C15	121.27 (12)
C4—C3—C2	116.55 (10)	C17—C16—H16	119.4
O4—C4—C10	122.00 (11)	C15—C16—H16	119.4
O4—C4—C3	121.53 (11)	C16—C17—C12	121.52 (12)
C10—C4—C3	116.47 (10)	C16—C17—H17	119.2

C6—C5—C10	120.44 (12)	C12—C17—H17	119.2
C6—C5—H5	119.8	C15—C18—H18A	109.5
C10—C5—H5	119.8	C15—C18—H18B	109.5
C5—C6—C7	119.76 (12)	H18A—C18—H18B	109.5
C5—C6—H6	120.1	C15—C18—H18C	109.5
C7—C6—H6	120.1	H18A—C18—H18C	109.5
C8—C7—C6	120.96 (13)	H18B—C18—H18C	109.5
C8—C7—H7	119.5	C26—C21—C22	119.07 (12)
C6—C7—H7	119.5	C26—C21—C2	123.07 (11)
C7—C8—C9	119.21 (12)	C22—C21—C2	117.85 (11)
C7—C8—H8	120.4	C23—C22—C21	120.37 (12)
C9—C8—H8	120.4	C23—C22—H22	119.8
O1—C9—C8	117.26 (11)	C21—C22—H22	119.8
O1—C9—C10	121.89 (11)	C22—C23—C24	120.09 (12)
C8—C9—C10	120.77 (11)	C22—C23—H23	120.0
C9—C10—C5	118.87 (12)	C24—C23—H23	120.0
C9—C10—C4	119.72 (11)	C25—C24—C23	119.90 (12)
C5—C10—C4	121.33 (11)	C25—C24—H24	120.0
C3—C11—C12	131.34 (11)	C23—C24—H24	120.0
C3—C11—H11	114.3	C24—C25—C26	120.11 (13)
C12—C11—H11	114.3	C24—C25—H25	119.9
C17—C12—C13	117.13 (11)	C26—C25—H25	119.9
C17—C12—C11	118.27 (11)	C21—C26—C25	120.45 (12)
C13—C12—C11	124.55 (11)	C21—C26—H26	119.8
C14—C13—C12	120.78 (12)	C25—C26—H26	119.8

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C2—H2···O4^{i}	1.00	2.27	3.1737 (14)	149
C5—H5···O1^{ii}	0.95	2.54	3.4076 (15)	152
C24—H24···O4^{iii}	0.95	2.71	3.4871 (17)	140

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

(*E*)-3-(3-Methylbenzylidene)-2-phenylchroman-4-one (compound_III_AAG1)

Crystal data

$C_{23}H_{18}O_2$
M_r = 326.37
Triclinic, $P\overline{1}$
a = 8.9094 (3) Å
b = 9.6578 (3) Å
c = 10.9044 (4) Å
α = 104.378 (3)°
β = 109.425 (3)°
γ = 91.902 (3)°
V = 850.24 (5) Å^3

Z = 2
$F(000)$ = 344
D_x = 1.275 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 3948 reflections
θ = 3.5–36.1°
μ = 0.08 mm^{-1}
T = 100 K
Needle, colorless
0.28 × 0.1 × 0.08 mm

Data collection

Rigaku OD SuperNova Dual source diffractometer with an Atlas detector
Radiation source: micro-focus sealed X-ray tube, SuperNova (Mo) X-ray Source
Detector resolution: 10.4498 pixels mm^{-1}
ω scans
Absorption correction: multi-scan (CrysAlis PRO; Rigaku OD, 2015)
T_{min} = 0.736, T_{max} = 1.000

7284 measured reflections
3509 independent reflections
2999 reflections with $I > 2\sigma(I)$
R_{int} = 0.021
θ_{max} = 26.5°, θ_{min} = 3.1°
h = −11→11
k = −12→12
l = −13→10

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.037
$wR(F^2)$ = 0.098
S = 1.04
3509 reflections
227 parameters
0 restraints

Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0439P)^2 + 0.2079P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.24 e Å^{-3}
$\Delta\rho_{min}$ = −0.21 e Å^{-3}

Special details

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

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

	x	y	z	U_{iso}*/U_{eq}
O1	0.57118 (10)	0.44640 (9)	0.37188 (8)	0.0200 (2)
O4	0.38766 (10)	0.03505 (9)	0.32719 (8)	0.0229 (2)
C21	0.66313 (13)	0.29543 (13)	0.20350 (11)	0.0170 (2)
C12	0.89045 (14)	0.16807 (12)	0.54388 (12)	0.0192 (3)
C2	0.66990 (14)	0.33222 (12)	0.35024 (11)	0.0172 (2)
H2	0.7834	0.3701	0.4101	0.021*
C3	0.61964 (14)	0.20436 (12)	0.39022 (11)	0.0176 (2)
C11	0.71562 (14)	0.13781 (13)	0.47467 (11)	0.0188 (3)
H11	0.6638	0.0611	0.4920	0.023*
C9	0.40917 (14)	0.40489 (13)	0.31451 (11)	0.0188 (3)
C4	0.44417 (14)	0.15501 (13)	0.33244 (11)	0.0179 (2)
C26	0.66779 (14)	0.15547 (13)	0.13257 (12)	0.0204 (3)
H26	0.6718	0.0796	0.1744	0.024*
C10	0.34138 (14)	0.26263 (13)	0.28726 (11)	0.0188 (3)
C13	0.99590 (15)	0.20421 (13)	0.48267 (12)	0.0221 (3)
H13	0.9549	0.2119	0.3926	0.026*
C17	0.95460 (15)	0.15826 (13)	0.67717 (12)	0.0203 (3)
H17	0.8844	0.1306	0.7185	0.024*
C16	1.11883 (15)	0.18806 (13)	0.75045 (12)	0.0215 (3)
C5	0.17385 (15)	0.22886 (14)	0.22882 (12)	0.0228 (3)
H5	0.1266	0.1325	0.2086	0.027*

C22	0.65751 (14)	0.40519 (13)	0.14018 (12)	0.0208 (3)
H22	0.6544	0.5013	0.1876	0.025*
C25	0.66659 (15)	0.12572 (14)	0.00015 (12)	0.0230 (3)
H25	0.6695	0.0297	−0.0476	0.028*
C24	0.66114 (14)	0.23528 (14)	−0.06185 (12)	0.0221 (3)
H24	0.6607	0.2149	−0.1518	0.027*
C14	1.16003 (15)	0.22879 (14)	0.55301 (13)	0.0249 (3)
H14	1.2311	0.2514	0.5103	0.030*
C15	1.22082 (15)	0.22054 (13)	0.68555 (12)	0.0234 (3)
H15	1.3335	0.2373	0.7327	0.028*
C23	0.65634 (15)	0.37523 (14)	0.00852 (12)	0.0232 (3)
H23	0.6522	0.4508	−0.0336	0.028*
C8	0.31165 (16)	0.51168 (14)	0.28802 (12)	0.0243 (3)
H8	0.3579	0.6084	0.3085	0.029*
C6	0.07720 (15)	0.33358 (16)	0.20038 (13)	0.0274 (3)
H6	−0.0359	0.3095	0.1599	0.033*
C7	0.14682 (16)	0.47552 (15)	0.23157 (13)	0.0279 (3)
H7	0.0801	0.5482	0.2138	0.033*
C18	1.18325 (16)	0.18957 (15)	0.89766 (12)	0.0283 (3)
H18A	1.1300	0.1062	0.9099	0.042*
H18B	1.1624	0.2785	0.9527	0.042*
H18C	1.2991	0.1850	0.9259	0.042*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0226 (4)	0.0158 (4)	0.0191 (4)	0.0016 (3)	0.0072 (3)	0.0009 (3)
O4	0.0240 (4)	0.0210 (5)	0.0226 (4)	−0.0023 (3)	0.0066 (4)	0.0069 (4)
C21	0.0138 (5)	0.0185 (6)	0.0176 (5)	0.0001 (4)	0.0045 (4)	0.0046 (5)
C12	0.0228 (6)	0.0134 (6)	0.0191 (6)	0.0031 (4)	0.0056 (5)	0.0028 (4)
C2	0.0176 (5)	0.0157 (6)	0.0168 (5)	0.0013 (4)	0.0051 (4)	0.0031 (4)
C3	0.0212 (6)	0.0166 (6)	0.0141 (5)	0.0012 (4)	0.0071 (4)	0.0014 (4)
C11	0.0230 (6)	0.0162 (6)	0.0167 (5)	0.0001 (5)	0.0083 (5)	0.0022 (4)
C9	0.0222 (6)	0.0217 (6)	0.0121 (5)	0.0032 (5)	0.0072 (4)	0.0025 (5)
C4	0.0225 (6)	0.0191 (6)	0.0123 (5)	0.0004 (5)	0.0078 (4)	0.0027 (4)
C26	0.0240 (6)	0.0176 (6)	0.0191 (6)	0.0009 (5)	0.0068 (5)	0.0056 (5)
C10	0.0214 (6)	0.0221 (6)	0.0138 (5)	0.0032 (5)	0.0079 (4)	0.0043 (5)
C13	0.0244 (6)	0.0227 (7)	0.0180 (6)	0.0027 (5)	0.0068 (5)	0.0048 (5)
C17	0.0252 (6)	0.0155 (6)	0.0211 (6)	0.0038 (5)	0.0088 (5)	0.0055 (5)
C16	0.0286 (7)	0.0141 (6)	0.0181 (6)	0.0043 (5)	0.0050 (5)	0.0026 (5)
C5	0.0224 (6)	0.0290 (7)	0.0182 (6)	0.0013 (5)	0.0086 (5)	0.0067 (5)
C22	0.0219 (6)	0.0169 (6)	0.0245 (6)	0.0032 (5)	0.0091 (5)	0.0057 (5)
C25	0.0268 (6)	0.0195 (6)	0.0200 (6)	0.0015 (5)	0.0076 (5)	0.0019 (5)
C24	0.0212 (6)	0.0289 (7)	0.0167 (5)	0.0035 (5)	0.0062 (5)	0.0075 (5)
C14	0.0251 (6)	0.0256 (7)	0.0241 (6)	0.0016 (5)	0.0105 (5)	0.0044 (5)
C15	0.0204 (6)	0.0200 (6)	0.0242 (6)	0.0017 (5)	0.0037 (5)	0.0019 (5)
C23	0.0243 (6)	0.0247 (7)	0.0250 (6)	0.0052 (5)	0.0099 (5)	0.0127 (5)
C8	0.0328 (7)	0.0217 (7)	0.0194 (6)	0.0075 (5)	0.0111 (5)	0.0043 (5)

C6	0.0206 (6)	0.0400 (8)	0.0231 (6)	0.0072 (5)	0.0085 (5)	0.0098 (6)
C7	0.0301 (7)	0.0331 (8)	0.0238 (6)	0.0156 (6)	0.0115 (5)	0.0096 (6)
C18	0.0315 (7)	0.0295 (7)	0.0205 (6)	0.0051 (5)	0.0045 (5)	0.0073 (5)

Geometric parameters (Å, º)

O1—C9	1.3682 (14)	C17—H17	0.9500
O1—C2	1.4555 (14)	C16—C15	1.3942 (18)
O4—C4	1.2281 (14)	C16—C18	1.5105 (16)
C21—C26	1.3903 (17)	C5—C6	1.3771 (19)
C21—C22	1.3953 (17)	C5—H5	0.9500
C21—C2	1.5305 (15)	C22—C23	1.3887 (17)
C12—C13	1.4004 (17)	C22—H22	0.9500
C12—C17	1.4027 (16)	C25—C24	1.3841 (18)
C12—C11	1.4695 (16)	C25—H25	0.9500
C2—C3	1.5086 (16)	C24—C23	1.3885 (18)
C2—H2	1.0000	C24—H24	0.9500
C3—C11	1.3413 (17)	C14—C15	1.3885 (18)
C3—C4	1.4881 (15)	C14—H14	0.9500
C11—H11	0.9500	C15—H15	0.9500
C9—C8	1.3938 (17)	C23—H23	0.9500
C9—C10	1.4012 (17)	C8—C7	1.3839 (18)
C4—C10	1.4762 (17)	C8—H8	0.9500
C26—C25	1.3969 (16)	C6—C7	1.397 (2)
C26—H26	0.9500	C6—H6	0.9500
C10—C5	1.4043 (16)	C7—H7	0.9500
C13—C14	1.3868 (17)	C18—H18A	0.9800
C13—H13	0.9500	C18—H18B	0.9800
C17—C16	1.3937 (17)	C18—H18C	0.9800
C9—O1—C2	115.14 (9)	C17—C16—C18	120.52 (11)
C26—C21—C22	118.86 (11)	C15—C16—C18	121.13 (11)
C26—C21—C2	121.58 (10)	C6—C5—C10	120.87 (12)
C22—C21—C2	119.53 (11)	C6—C5—H5	119.6
C13—C12—C17	118.55 (11)	C10—C5—H5	119.6
C13—C12—C11	123.22 (10)	C23—C22—C21	120.62 (12)
C17—C12—C11	118.22 (11)	C23—C22—H22	119.7
O1—C2—C3	110.50 (9)	C21—C22—H22	119.7
O1—C2—C21	109.10 (9)	C24—C25—C26	120.39 (12)
C3—C2—C21	113.52 (9)	C24—C25—H25	119.8
O1—C2—H2	107.8	C26—C25—H25	119.8
C3—C2—H2	107.8	C25—C24—C23	119.46 (11)
C21—C2—H2	107.8	C25—C24—H24	120.3
C11—C3—C4	118.88 (10)	C23—C24—H24	120.3
C11—C3—C2	126.48 (11)	C13—C14—C15	120.26 (12)
C4—C3—C2	114.58 (10)	C13—C14—H14	119.9
C3—C11—C12	128.46 (11)	C15—C14—H14	119.9
C3—C11—H11	115.8	C14—C15—C16	120.92 (11)

C12—C11—H11	115.8	C14—C15—H15	119.5
O1—C9—C8	117.18 (11)	C16—C15—H15	119.5
O1—C9—C10	122.27 (11)	C24—C23—C22	120.31 (11)
C8—C9—C10	120.51 (11)	C24—C23—H23	119.8
O4—C4—C10	121.96 (11)	C22—C23—H23	119.8
O4—C4—C3	122.58 (11)	C7—C8—C9	119.42 (12)
C10—C4—C3	115.40 (10)	C7—C8—H8	120.3
C21—C26—C25	120.37 (11)	C9—C8—H8	120.3
C21—C26—H26	119.8	C5—C6—C7	119.48 (12)
C25—C26—H26	119.8	C5—C6—H6	120.3
C9—C10—C5	118.78 (11)	C7—C6—H6	120.3
C9—C10—C4	119.87 (11)	C8—C7—C6	120.90 (12)
C5—C10—C4	121.05 (11)	C8—C7—H7	119.5
C14—C13—C12	120.24 (11)	C6—C7—H7	119.5
C14—C13—H13	119.9	C16—C18—H18A	109.5
C12—C13—H13	119.9	C16—C18—H18B	109.5
C16—C17—C12	121.63 (11)	H18A—C18—H18B	109.5
C16—C17—H17	119.2	C16—C18—H18C	109.5
C12—C17—H17	119.2	H18A—C18—H18C	109.5
C17—C16—C15	118.32 (11)	H18B—C18—H18C	109.5

C9—O1—C2—C3	−51.97 (12)	C3—C4—C10—C9	−7.36 (15)
C9—O1—C2—C21	73.51 (11)	O4—C4—C10—C5	−3.72 (17)
C26—C21—C2—O1	−145.49 (10)	C3—C4—C10—C5	178.94 (10)
C22—C21—C2—O1	36.67 (14)	C17—C12—C13—C14	0.34 (18)
C26—C21—C2—C3	−21.79 (15)	C11—C12—C13—C14	−178.77 (12)
C22—C21—C2—C3	160.38 (10)	C13—C12—C17—C16	2.12 (18)
O1—C2—C3—C11	−127.71 (12)	C11—C12—C17—C16	−178.73 (11)
C21—C2—C3—C11	109.35 (13)	C12—C17—C16—C15	−3.55 (18)
O1—C2—C3—C4	49.49 (12)	C12—C17—C16—C18	174.54 (12)
C21—C2—C3—C4	−73.45 (13)	C9—C10—C5—C6	−0.92 (17)
C4—C3—C11—C12	−179.62 (11)	C4—C10—C5—C6	172.84 (11)
C2—C3—C11—C12	−2.5 (2)	C26—C21—C22—C23	0.09 (17)
C13—C12—C11—C3	−39.46 (19)	C2—C21—C22—C23	177.99 (11)
C17—C12—C11—C3	141.43 (13)	C21—C26—C25—C24	0.16 (18)
C2—O1—C9—C8	−157.01 (10)	C26—C25—C24—C23	−0.21 (18)
C2—O1—C9—C10	25.25 (14)	C12—C13—C14—C15	−1.3 (2)
C11—C3—C4—O4	−20.52 (17)	C13—C14—C15—C16	−0.2 (2)
C2—C3—C4—O4	162.05 (11)	C17—C16—C15—C14	2.57 (19)
C11—C3—C4—C10	156.80 (11)	C18—C16—C15—C14	−175.51 (12)
C2—C3—C4—C10	−20.63 (14)	C25—C24—C23—C22	0.20 (18)
C22—C21—C26—C25	−0.10 (17)	C21—C22—C23—C24	−0.15 (18)
C2—C21—C26—C25	−177.95 (11)	O1—C9—C8—C7	−179.21 (10)
O1—C9—C10—C5	179.68 (10)	C10—C9—C8—C7	−1.43 (17)
C8—C9—C10—C5	2.01 (17)	C10—C5—C6—C7	−0.74 (18)
O1—C9—C10—C4	5.84 (16)	C9—C8—C7—C6	−0.26 (18)
C8—C9—C10—C4	−171.83 (10)	C5—C6—C7—C8	1.35 (19)
O4—C4—C10—C9	169.98 (10)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C11—H11···O4^{i}	0.95	2.54	3.3635 (14)	145
C17—H17···O4^{i}	0.95	2.69	3.5025 (15)	144
C24—H24···O4^{ii}	0.95	2.60	3.2731 (15)	128

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

(*E*)-3-(4-Methoxybenzylidene)-2-phenylchroman-4-one (compound_IV_AAG2)

Crystal data

$C_{23}H_{18}O_3$
M_r = 342.37
Monoclinic, $P2_1/n$
a = 23.4111 (3) Å
b = 8.0027 (1) Å
c = 28.1094 (5) Å
β = 97.156 (1)°
V = 5225.33 (13) Å^3
Z = 12
$F(000)$ = 2160
D_x = 1.306 Mg m^{-3}
Cu $K\alpha$ radiation, λ = 1.54184 Å
Cell parameters from 7810 reflections
θ = 2.6–74.1°
μ = 0.69 mm^{-1}
T = 100 K
Needle, colorless
0.28 × 0.1 × 0.08 mm

Data collection

Rigaku OD SuperNova Dual source diffractometer with an Atlas detector
ω scans
Absorption correction: multi-scan (CrysAlis PRO; Agilent, 2013)
T_{min} = 0.997, T_{max} = 1.000
15971 measured reflections
9323 independent reflections
7742 reflections with $I > 2\sigma(I)$
R_{int} = 0.022
θ_{max} = 67.5°, θ_{min} = 2.3°
h = −26→28
k = −9→7
l = −33→30

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.038
$wR(F^2)$ = 0.104
S = 1.01
9323 reflections
706 parameters
0 restraints
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0515P)^2 + 0.9818P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.20 e Å^{-3}
$\Delta\rho_{min}$ = −0.21 e Å^{-3}

Special details

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

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

	x	*y*	*z*	U_{iso}*/U_{eq}
O1	0.10459 (3)	0.83638 (11)	0.53474 (3)	0.01769 (18)
O4	0.26145 (4)	0.77397 (13)	0.48650 (3)	0.0270 (2)
O3	0.31526 (4)	0.78657 (13)	0.78489 (3)	0.0293 (2)

C5	0.16094 (5)	0.81050 (15)	0.46836 (4)	0.0195 (3)
C4	0.21516 (5)	0.80412 (15)	0.50102 (4)	0.0190 (3)
C6	0.16063 (6)	0.79075 (18)	0.41871 (5)	0.0248 (3)
H6	0.1960	0.7805	0.4057	0.030*
C3	0.21023 (5)	0.83055 (14)	0.55297 (4)	0.0171 (2)
C17	0.25594 (5)	0.78916 (15)	0.58462 (5)	0.0186 (2)
H17	0.2877	0.7476	0.5702	0.022*
C21	0.29676 (6)	0.79508 (16)	0.73726 (5)	0.0221 (3)
C9	0.05653 (6)	0.82182 (18)	0.45664 (5)	0.0253 (3)
H9	0.0210	0.8326	0.4694	0.030*
C19	0.23508 (5)	0.88924 (15)	0.66704 (4)	0.0187 (2)
H19	0.2033	0.9545	0.6534	0.022*
C23	0.31335 (5)	0.70289 (17)	0.65885 (5)	0.0229 (3)
H23	0.3355	0.6391	0.6393	0.028*
C18	0.26619 (5)	0.79578 (15)	0.63689 (5)	0.0192 (3)
C16	0.10377 (5)	1.16182 (16)	0.58418 (5)	0.0214 (3)
H16	0.0767	1.0927	0.5973	0.026*
C2	0.15441 (5)	0.89981 (15)	0.56591 (4)	0.0162 (2)
H2	0.1505	0.8617	0.5992	0.019*
C14	0.13611 (6)	1.43522 (16)	0.56434 (5)	0.0252 (3)
H14	0.1310	1.5530	0.5636	0.030*
C15	0.09655 (6)	1.33437 (17)	0.58351 (5)	0.0251 (3)
H15	0.0646	1.3830	0.5961	0.030*
C10	0.10837 (5)	0.82584 (15)	0.48687 (4)	0.0184 (2)
C11	0.15050 (5)	1.08964 (15)	0.56562 (4)	0.0173 (2)
C12	0.19031 (5)	1.19147 (16)	0.54688 (4)	0.0194 (3)
H12	0.2224	1.1431	0.5345	0.023*
C20	0.24966 (5)	0.88868 (16)	0.71658 (5)	0.0202 (3)
H20	0.2276	0.9519	0.7363	0.024*
C13	0.18315 (6)	1.36457 (17)	0.54629 (5)	0.0236 (3)
H13	0.2104	1.4341	0.5336	0.028*
C8	0.05741 (6)	0.8019 (2)	0.40788 (5)	0.0317 (3)
H8	0.0222	0.7989	0.3871	0.038*
C22	0.32840 (6)	0.70158 (17)	0.70782 (5)	0.0251 (3)
H22	0.3603	0.6370	0.7215	0.030*
C7	0.10937 (6)	0.7862 (2)	0.38870 (5)	0.0307 (3)
H7	0.1094	0.7724	0.3551	0.037*
C211	0.28785 (6)	0.8935 (2)	0.81596 (5)	0.0321 (3)
H21A	0.2464	0.8712	0.8119	0.048*
H21B	0.2946	1.0105	0.8080	0.048*
H21C	0.3038	0.8721	0.8493	0.048*
O31	0.56062 (3)	0.27594 (11)	0.79683 (3)	0.01768 (18)
O34	0.40526 (4)	0.20680 (13)	0.84703 (3)	0.0264 (2)
C40	0.55790 (5)	0.26249 (15)	0.84483 (4)	0.0185 (2)
O33	0.34425 (4)	0.32326 (14)	0.55019 (3)	0.0311 (2)
C41	0.51536 (5)	0.53223 (15)	0.76805 (4)	0.0174 (2)
C42	0.47572 (6)	0.63647 (16)	0.78610 (4)	0.0209 (3)
H42	0.4431	0.5901	0.7983	0.025*

C33	0.45460 (5)	0.27542 (15)	0.78040 (4)	0.0175 (2)
C46	0.56266 (6)	0.60212 (17)	0.74971 (5)	0.0228 (3)
H46	0.5895	0.5315	0.7369	0.027*
C48	0.39692 (5)	0.26975 (16)	0.69692 (5)	0.0194 (3)
C36	0.50724 (6)	0.22330 (17)	0.91374 (5)	0.0244 (3)
H36	0.4722	0.2119	0.9272	0.029*
C47	0.40779 (5)	0.24452 (15)	0.74877 (5)	0.0192 (3)
H47	0.3763	0.1982	0.7625	0.023*
C52	0.33472 (6)	0.19928 (18)	0.62375 (5)	0.0274 (3)
H52	0.3035	0.1363	0.6081	0.033*
C35	0.50597 (5)	0.24518 (15)	0.86402 (5)	0.0196 (3)
C43	0.48379 (6)	0.80906 (17)	0.78637 (5)	0.0267 (3)
H43	0.4567	0.8801	0.7987	0.032*
C34	0.45110 (5)	0.24053 (15)	0.83209 (5)	0.0191 (3)
C45	0.57070 (6)	0.77351 (18)	0.75011 (5)	0.0279 (3)
H45	0.6031	0.8203	0.7377	0.034*
C37	0.55895 (6)	0.2183 (2)	0.94308 (5)	0.0305 (3)
H37	0.5596	0.2035	0.9767	0.037*
C51	0.36454 (6)	0.31041 (17)	0.59743 (5)	0.0223 (3)
C50	0.41129 (5)	0.39894 (16)	0.62069 (5)	0.0200 (3)
H50	0.4324	0.4729	0.6031	0.024*
C44	0.53120 (7)	0.87706 (17)	0.76868 (5)	0.0292 (3)
H44	0.5368	0.9947	0.7692	0.035*
C49	0.42686 (5)	0.37853 (16)	0.66989 (4)	0.0195 (3)
H49	0.4586	0.4400	0.6854	0.023*
C39	0.61035 (6)	0.25690 (18)	0.87438 (5)	0.0252 (3)
H39	0.6456	0.2681	0.8611	0.030*
C32	0.51044 (5)	0.34316 (15)	0.76700 (4)	0.0166 (2)
H32	0.5133	0.3069	0.7333	0.020*
C53	0.35057 (6)	0.18097 (17)	0.67222 (5)	0.0248 (3)
H53	0.3295	0.1059	0.6896	0.030*
C38	0.61048 (6)	0.2350 (2)	0.92305 (5)	0.0310 (3)
H38	0.6461	0.2311	0.9433	0.037*
C511	0.36840 (6)	0.4486 (2)	0.52261 (5)	0.0298 (3)
H51A	0.4095	0.4262	0.5222	0.045*
H51B	0.3636	0.5585	0.5370	0.045*
H51C	0.3487	0.4473	0.4897	0.045*
O61	0.22635 (4)	0.40047 (11)	0.13133 (3)	0.01906 (19)
O64	0.07332 (4)	0.32928 (14)	0.18334 (3)	0.0316 (2)
O63	0.01585 (4)	0.29424 (13)	−0.11462 (3)	0.0294 (2)
C63	0.12093 (5)	0.39015 (15)	0.11531 (4)	0.0186 (3)
C70	0.22422 (5)	0.40593 (15)	0.17964 (5)	0.0193 (3)
C62	0.17574 (5)	0.46126 (15)	0.10066 (4)	0.0175 (2)
H62	0.1788	0.4219	0.0673	0.021*
C76	0.13695 (5)	0.75279 (16)	0.11604 (5)	0.0208 (3)
H76	0.1035	0.7045	0.1263	0.025*
C77	0.07469 (5)	0.34220 (15)	0.08504 (5)	0.0199 (3)
H77	0.0438	0.3011	0.1006	0.024*

C64	0.11821 (5)	0.36726 (16)	0.16759 (5)	0.0207 (3)
C66	0.17394 (6)	0.38554 (17)	0.24932 (5)	0.0242 (3)
H66	0.1391	0.3727	0.2631	0.029*
C75	0.14402 (6)	0.92594 (17)	0.11667 (5)	0.0242 (3)
H75	0.1153	0.9953	0.1275	0.029*
C65	0.17270 (5)	0.38931 (15)	0.19937 (5)	0.0196 (3)
C74	0.19253 (6)	0.99728 (16)	0.10159 (5)	0.0245 (3)
H74	0.1972	1.1152	0.1023	0.029*
C73	0.23435 (6)	0.89628 (17)	0.08536 (5)	0.0237 (3)
H73	0.2676	0.9450	0.0748	0.028*
C78	0.06315 (5)	0.34154 (16)	0.03287 (5)	0.0201 (3)
C71	0.17897 (5)	0.65101 (15)	0.10037 (4)	0.0179 (2)
C69	0.27641 (6)	0.41947 (19)	0.20914 (5)	0.0278 (3)
H69	0.3115	0.4303	0.1956	0.033*
C83	0.01889 (6)	0.23595 (17)	0.01259 (5)	0.0243 (3)
H83	−0.0016	0.1715	0.0333	0.029*
C79	0.09117 (6)	0.43776 (16)	0.00123 (5)	0.0226 (3)
H79	0.1207	0.5129	0.0137	0.027*
C72	0.22748 (5)	0.72430 (16)	0.08461 (5)	0.0208 (3)
H72	0.2560	0.6555	0.0733	0.025*
C82	0.00403 (6)	0.22232 (18)	−0.03621 (5)	0.0261 (3)
H82	−0.0259	0.1487	−0.0488	0.031*
C80	0.07665 (6)	0.42563 (17)	−0.04811 (5)	0.0239 (3)
H80	0.0964	0.4915	−0.0689	0.029*
C68	0.27653 (6)	0.4169 (2)	0.25813 (5)	0.0343 (3)
H68	0.3120	0.4266	0.2784	0.041*
C811	0.04246 (6)	0.3967 (2)	−0.14750 (5)	0.0337 (3)
H81A	0.0339	0.5144	−0.1420	0.051*
H81B	0.0275	0.3661	−0.1805	0.051*
H81C	0.0842	0.3794	−0.1425	0.051*
C67	0.22546 (6)	0.4003 (2)	0.27861 (5)	0.0314 (3)
H67	0.2262	0.3993	0.3125	0.038*
C81	0.03315 (6)	0.31704 (17)	−0.06707 (5)	0.0228 (3)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0148 (4)	0.0201 (4)	0.0181 (4)	−0.0028 (3)	0.0019 (3)	−0.0018 (3)
O4	0.0196 (5)	0.0398 (6)	0.0227 (5)	0.0045 (4)	0.0063 (4)	0.0012 (4)
O3	0.0321 (5)	0.0350 (5)	0.0194 (5)	0.0056 (4)	−0.0023 (4)	0.0010 (4)
C5	0.0205 (6)	0.0170 (6)	0.0211 (6)	−0.0014 (5)	0.0033 (5)	0.0018 (5)
C4	0.0195 (6)	0.0168 (6)	0.0212 (6)	−0.0001 (5)	0.0048 (5)	0.0024 (5)
C6	0.0235 (7)	0.0295 (7)	0.0221 (7)	−0.0030 (5)	0.0051 (5)	0.0015 (5)
C3	0.0180 (6)	0.0133 (5)	0.0202 (6)	−0.0011 (5)	0.0034 (5)	0.0006 (5)
C17	0.0172 (6)	0.0157 (6)	0.0233 (6)	0.0006 (5)	0.0044 (5)	−0.0010 (5)
C21	0.0230 (6)	0.0228 (6)	0.0194 (6)	−0.0039 (5)	−0.0017 (5)	0.0007 (5)
C9	0.0177 (6)	0.0333 (7)	0.0248 (7)	−0.0030 (5)	0.0024 (5)	0.0004 (6)
C19	0.0160 (6)	0.0176 (6)	0.0220 (6)	−0.0002 (5)	0.0005 (5)	0.0008 (5)

C23	0.0195 (6)	0.0241 (6)	0.0246 (7)	0.0034 (5)	0.0005 (5)	−0.0023 (5)
C18	0.0165 (6)	0.0175 (6)	0.0232 (6)	−0.0020 (5)	0.0008 (5)	0.0004 (5)
C16	0.0197 (6)	0.0207 (6)	0.0238 (6)	−0.0002 (5)	0.0029 (5)	−0.0011 (5)
C2	0.0148 (6)	0.0172 (6)	0.0160 (6)	−0.0009 (5)	−0.0002 (4)	−0.0001 (4)
C14	0.0316 (7)	0.0158 (6)	0.0265 (7)	0.0016 (5)	−0.0029 (5)	−0.0006 (5)
C15	0.0235 (7)	0.0224 (7)	0.0290 (7)	0.0047 (5)	0.0012 (5)	−0.0035 (5)
C10	0.0217 (6)	0.0161 (6)	0.0178 (6)	−0.0021 (5)	0.0035 (5)	0.0015 (5)
C11	0.0176 (6)	0.0180 (6)	0.0153 (6)	0.0010 (5)	−0.0016 (4)	0.0001 (5)
C12	0.0201 (6)	0.0197 (6)	0.0180 (6)	−0.0013 (5)	0.0009 (5)	−0.0004 (5)
C20	0.0193 (6)	0.0198 (6)	0.0217 (6)	−0.0024 (5)	0.0032 (5)	−0.0008 (5)
C13	0.0285 (7)	0.0195 (6)	0.0221 (6)	−0.0040 (5)	0.0005 (5)	0.0023 (5)
C8	0.0233 (7)	0.0474 (9)	0.0232 (7)	−0.0053 (6)	−0.0024 (5)	0.0018 (6)
C22	0.0219 (6)	0.0256 (7)	0.0266 (7)	0.0052 (5)	−0.0023 (5)	0.0013 (5)
C7	0.0299 (7)	0.0441 (8)	0.0177 (6)	−0.0063 (6)	0.0013 (5)	0.0012 (6)
C211	0.0318 (7)	0.0444 (9)	0.0197 (7)	0.0020 (7)	0.0012 (5)	−0.0018 (6)
O31	0.0153 (4)	0.0193 (4)	0.0186 (4)	0.0028 (3)	0.0028 (3)	0.0012 (3)
O34	0.0194 (5)	0.0377 (5)	0.0231 (5)	−0.0037 (4)	0.0069 (4)	0.0002 (4)
C40	0.0219 (6)	0.0154 (6)	0.0185 (6)	0.0019 (5)	0.0035 (5)	−0.0017 (5)
O33	0.0304 (5)	0.0389 (6)	0.0217 (5)	−0.0076 (4)	−0.0052 (4)	0.0028 (4)
C41	0.0184 (6)	0.0182 (6)	0.0147 (5)	−0.0008 (5)	−0.0018 (4)	−0.0005 (5)
C42	0.0234 (6)	0.0201 (6)	0.0184 (6)	0.0018 (5)	−0.0002 (5)	−0.0004 (5)
C33	0.0181 (6)	0.0141 (5)	0.0206 (6)	0.0010 (5)	0.0043 (5)	−0.0014 (5)
C46	0.0210 (6)	0.0219 (6)	0.0251 (6)	−0.0016 (5)	0.0013 (5)	0.0010 (5)
C48	0.0162 (6)	0.0197 (6)	0.0222 (6)	0.0006 (5)	0.0020 (5)	−0.0019 (5)
C36	0.0236 (7)	0.0304 (7)	0.0201 (6)	0.0027 (5)	0.0059 (5)	−0.0010 (5)
C47	0.0180 (6)	0.0168 (6)	0.0235 (6)	−0.0008 (5)	0.0050 (5)	−0.0006 (5)
C52	0.0226 (6)	0.0297 (7)	0.0283 (7)	−0.0068 (6)	−0.0029 (5)	−0.0007 (6)
C35	0.0201 (6)	0.0176 (6)	0.0214 (6)	0.0006 (5)	0.0038 (5)	−0.0014 (5)
C43	0.0361 (8)	0.0201 (6)	0.0227 (7)	0.0062 (6)	−0.0013 (5)	−0.0021 (5)
C34	0.0195 (6)	0.0165 (6)	0.0219 (6)	0.0004 (5)	0.0049 (5)	−0.0029 (5)
C45	0.0283 (7)	0.0238 (7)	0.0303 (7)	−0.0072 (6)	−0.0014 (5)	0.0051 (6)
C37	0.0293 (7)	0.0443 (9)	0.0177 (6)	0.0058 (6)	0.0022 (5)	−0.0009 (6)
C51	0.0211 (6)	0.0243 (6)	0.0206 (6)	0.0031 (5)	−0.0010 (5)	−0.0014 (5)
C50	0.0183 (6)	0.0192 (6)	0.0227 (6)	0.0018 (5)	0.0034 (5)	0.0006 (5)
C44	0.0411 (8)	0.0171 (6)	0.0266 (7)	−0.0035 (6)	−0.0065 (6)	0.0005 (5)
C49	0.0163 (6)	0.0193 (6)	0.0222 (6)	0.0001 (5)	0.0003 (5)	−0.0021 (5)
C39	0.0175 (6)	0.0325 (7)	0.0258 (7)	0.0033 (5)	0.0033 (5)	−0.0013 (6)
C32	0.0152 (6)	0.0178 (6)	0.0167 (6)	0.0011 (5)	0.0015 (4)	−0.0003 (5)
C53	0.0209 (6)	0.0265 (7)	0.0264 (7)	−0.0045 (5)	0.0001 (5)	0.0027 (5)
C38	0.0218 (7)	0.0453 (9)	0.0245 (7)	0.0047 (6)	−0.0023 (5)	−0.0015 (6)
C511	0.0240 (7)	0.0426 (8)	0.0220 (7)	−0.0002 (6)	0.0003 (5)	0.0053 (6)
O61	0.0178 (4)	0.0199 (4)	0.0201 (4)	0.0030 (3)	0.0051 (3)	0.0015 (3)
O64	0.0210 (5)	0.0501 (6)	0.0242 (5)	−0.0092 (4)	0.0054 (4)	0.0043 (5)
O63	0.0277 (5)	0.0389 (6)	0.0208 (5)	−0.0022 (4)	−0.0005 (4)	−0.0014 (4)
C63	0.0205 (6)	0.0139 (5)	0.0219 (6)	0.0001 (5)	0.0044 (5)	0.0010 (5)
C70	0.0220 (6)	0.0153 (6)	0.0210 (6)	0.0019 (5)	0.0041 (5)	0.0010 (5)
C62	0.0179 (6)	0.0175 (6)	0.0173 (6)	0.0008 (5)	0.0028 (4)	0.0006 (5)
C76	0.0209 (6)	0.0213 (6)	0.0202 (6)	−0.0002 (5)	0.0027 (5)	0.0004 (5)

C77	0.0193 (6)	0.0175 (6)	0.0235 (6)	−0.0004 (5)	0.0051 (5)	0.0012 (5)
C64	0.0211 (6)	0.0191 (6)	0.0224 (6)	−0.0015 (5)	0.0049 (5)	0.0011 (5)
C66	0.0224 (6)	0.0279 (7)	0.0233 (7)	−0.0007 (5)	0.0061 (5)	0.0029 (5)
C75	0.0281 (7)	0.0203 (6)	0.0237 (6)	0.0050 (5)	0.0015 (5)	−0.0017 (5)
C65	0.0203 (6)	0.0160 (6)	0.0228 (6)	0.0004 (5)	0.0040 (5)	0.0013 (5)
C74	0.0316 (7)	0.0157 (6)	0.0243 (6)	−0.0016 (5)	−0.0041 (5)	0.0006 (5)
C73	0.0230 (6)	0.0218 (6)	0.0255 (7)	−0.0058 (5)	−0.0009 (5)	0.0038 (5)
C78	0.0186 (6)	0.0176 (6)	0.0239 (6)	0.0016 (5)	0.0025 (5)	−0.0003 (5)
C71	0.0200 (6)	0.0179 (6)	0.0151 (6)	−0.0019 (5)	0.0001 (4)	0.0003 (5)
C69	0.0188 (6)	0.0368 (8)	0.0280 (7)	0.0006 (6)	0.0038 (5)	0.0043 (6)
C83	0.0206 (6)	0.0253 (7)	0.0267 (7)	−0.0034 (5)	0.0020 (5)	0.0033 (5)
C79	0.0235 (6)	0.0193 (6)	0.0243 (6)	−0.0030 (5)	0.0008 (5)	0.0006 (5)
C72	0.0202 (6)	0.0207 (6)	0.0215 (6)	−0.0003 (5)	0.0022 (5)	0.0007 (5)
C82	0.0215 (6)	0.0286 (7)	0.0269 (7)	−0.0043 (5)	−0.0021 (5)	−0.0007 (6)
C80	0.0247 (7)	0.0233 (6)	0.0237 (7)	−0.0010 (5)	0.0033 (5)	0.0027 (5)
C68	0.0210 (7)	0.0527 (10)	0.0277 (7)	−0.0027 (6)	−0.0026 (5)	0.0051 (7)
C811	0.0291 (7)	0.0505 (9)	0.0214 (7)	−0.0002 (7)	0.0030 (5)	0.0030 (6)
C67	0.0279 (7)	0.0454 (8)	0.0208 (7)	−0.0024 (6)	0.0020 (5)	0.0048 (6)
C81	0.0220 (6)	0.0243 (6)	0.0215 (6)	0.0043 (5)	0.0000 (5)	−0.0004 (5)

Geometric parameters (Å, º)

O1—C10	1.3623 (15)	C52—C53	1.374 (2)
O1—C2	1.4600 (14)	C52—C51	1.398 (2)
O4—C4	1.2287 (16)	C52—H52	0.9500
O3—C21	1.3565 (16)	C35—C34	1.4730 (17)
O3—C211	1.4308 (18)	C43—C44	1.383 (2)
C5—C10	1.4000 (18)	C43—H43	0.9500
C5—C6	1.4038 (19)	C45—C44	1.391 (2)
C5—C4	1.4717 (17)	C45—H45	0.9500
C4—C3	1.4941 (17)	C37—C38	1.400 (2)
C6—C7	1.3786 (19)	C37—H37	0.9500
C6—H6	0.9500	C51—C50	1.3959 (18)
C3—C17	1.3451 (18)	C50—C49	1.3952 (18)
C3—C2	1.5052 (16)	C50—H50	0.9500
C17—C18	1.4597 (18)	C44—H44	0.9500
C17—H17	0.9500	C49—H49	0.9500
C21—C22	1.395 (2)	C39—C38	1.379 (2)
C21—C20	1.3983 (18)	C39—H39	0.9500
C9—C8	1.383 (2)	C32—H32	1.0000
C9—C10	1.3923 (18)	C53—H53	0.9500
C9—H9	0.9500	C38—H38	0.9500
C19—C20	1.3919 (18)	C511—H51A	0.9800
C19—C18	1.4007 (18)	C511—H51B	0.9800
C19—H19	0.9500	C511—H51C	0.9800
C23—C22	1.3778 (19)	O61—C70	1.3656 (16)
C23—C18	1.4082 (18)	O61—C62	1.4592 (14)
C23—H23	0.9500	O64—C64	1.2284 (16)

C16—C15	1.3910 (19)	O63—C81	1.3597 (16)
C16—C11	1.3946 (18)	O63—C811	1.4345 (18)
C16—H16	0.9500	C63—C77	1.3466 (18)
C2—C11	1.5218 (17)	C63—C64	1.4902 (18)
C2—H2	1.0000	C63—C62	1.5072 (17)
C14—C15	1.387 (2)	C70—C69	1.3927 (19)
C14—C13	1.389 (2)	C70—C65	1.3953 (18)
C14—H14	0.9500	C62—C71	1.5205 (17)
C15—H15	0.9500	C62—H62	1.0000
C11—C12	1.3902 (18)	C76—C71	1.3904 (18)
C12—C13	1.3953 (19)	C76—C75	1.3954 (19)
C12—H12	0.9500	C76—H76	0.9500
C20—H20	0.9500	C77—C78	1.4576 (18)
C13—H13	0.9500	C77—H77	0.9500
C8—C7	1.396 (2)	C64—C65	1.4738 (18)
C8—H8	0.9500	C66—C67	1.378 (2)
C22—H22	0.9500	C66—C65	1.4013 (19)
C7—H7	0.9500	C66—H66	0.9500
C211—H21A	0.9800	C75—C74	1.384 (2)
C211—H21B	0.9800	C75—H75	0.9500
C211—H21C	0.9800	C74—C73	1.390 (2)
O31—C40	1.3631 (15)	C74—H74	0.9500
O31—C32	1.4584 (14)	C73—C72	1.3855 (19)
O34—C34	1.2299 (16)	C73—H73	0.9500
C40—C39	1.3948 (18)	C78—C79	1.3998 (18)
C40—C35	1.3970 (18)	C78—C83	1.4020 (19)
O33—C51	1.3572 (16)	C71—C72	1.3982 (18)
O33—C511	1.4271 (18)	C69—C68	1.377 (2)
C41—C42	1.3900 (18)	C69—H69	0.9500
C41—C46	1.3953 (18)	C83—C82	1.377 (2)
C41—C32	1.5174 (17)	C83—H83	0.9500
C42—C43	1.3939 (19)	C79—C80	1.3896 (19)
C42—H42	0.9500	C79—H79	0.9500
C33—C47	1.3453 (18)	C72—H72	0.9500
C33—C34	1.4918 (17)	C82—C81	1.393 (2)
C33—C32	1.5057 (16)	C82—H82	0.9500
C46—C45	1.384 (2)	C80—C81	1.3932 (19)
C46—H46	0.9500	C80—H80	0.9500
C48—C49	1.3998 (18)	C68—C67	1.397 (2)
C48—C53	1.4061 (18)	C68—H68	0.9500
C48—C47	1.4619 (18)	C811—H81A	0.9800
C36—C37	1.3779 (19)	C811—H81B	0.9800
C36—C35	1.4051 (18)	C811—H81C	0.9800
C36—H36	0.9500	C67—H67	0.9500
C47—H47	0.9500		

C10—O1—C2	117.97 (9)	C46—C45—H45	120.1
C21—O3—C211	117.40 (11)	C44—C45—H45	120.1

C10—C5—C6	118.91 (12)	C36—C37—C38	119.56 (13)
C10—C5—C4	120.07 (11)	C36—C37—H37	120.2
C6—C5—C4	120.90 (12)	C38—C37—H37	120.2
O4—C4—C5	121.84 (12)	O33—C51—C50	125.32 (12)
O4—C4—C3	122.08 (11)	O33—C51—C52	115.40 (12)
C5—C4—C3	116.05 (11)	C50—C51—C52	119.28 (12)
C7—C6—C5	120.50 (13)	C49—C50—C51	119.85 (12)
C7—C6—H6	119.8	C49—C50—H50	120.1
C5—C6—H6	119.8	C51—C50—H50	120.1
C17—C3—C4	117.37 (11)	C43—C44—C45	120.09 (13)
C17—C3—C2	125.02 (11)	C43—C44—H44	120.0
C4—C3—C2	117.61 (10)	C45—C44—H44	120.0
C3—C17—C18	132.39 (11)	C50—C49—C48	121.62 (12)
C3—C17—H17	113.8	C50—C49—H49	119.2
C18—C17—H17	113.8	C48—C49—H49	119.2
O3—C21—C22	115.51 (12)	C38—C39—C40	119.20 (12)
O3—C21—C20	125.13 (12)	C38—C39—H39	120.4
C22—C21—C20	119.35 (12)	C40—C39—H39	120.4
C8—C9—C10	119.21 (12)	O31—C32—C33	112.68 (10)
C8—C9—H9	120.4	O31—C32—C41	107.63 (9)
C10—C9—H9	120.4	C33—C32—C41	114.85 (10)
C20—C19—C18	121.50 (11)	O31—C32—H32	107.1
C20—C19—H19	119.2	C33—C32—H32	107.1
C18—C19—H19	119.2	C41—C32—H32	107.1
C22—C23—C18	122.06 (12)	C52—C53—C48	122.15 (13)
C22—C23—H23	119.0	C52—C53—H53	118.9
C18—C23—H23	119.0	C48—C53—H53	118.9
C19—C18—C23	117.07 (12)	C39—C38—C37	121.05 (12)
C19—C18—C17	126.57 (11)	C39—C38—H38	119.5
C23—C18—C17	116.33 (11)	C37—C38—H38	119.5
C15—C16—C11	120.38 (12)	O33—C511—H51A	109.5
C15—C16—H16	119.8	O33—C511—H51B	109.5
C11—C16—H16	119.8	H51A—C511—H51B	109.5
O1—C2—C3	112.32 (9)	O33—C511—H51C	109.5
O1—C2—C11	107.50 (9)	H51A—C511—H51C	109.5
C3—C2—C11	114.82 (10)	H51B—C511—H51C	109.5
O1—C2—H2	107.3	C70—O61—C62	116.70 (9)
C3—C2—H2	107.3	C81—O63—C811	117.16 (11)
C11—C2—H2	107.3	C77—C63—C64	117.28 (11)
C15—C14—C13	120.21 (12)	C77—C63—C62	125.43 (11)
C15—C14—H14	119.9	C64—C63—C62	117.27 (11)
C13—C14—H14	119.9	O61—C70—C69	117.16 (11)
C14—C15—C16	119.80 (13)	O61—C70—C65	122.19 (11)
C14—C15—H15	120.1	C69—C70—C65	120.56 (12)
C16—C15—H15	120.1	O61—C62—C63	111.65 (10)
O1—C10—C9	116.44 (11)	O61—C62—C71	107.38 (10)
O1—C10—C5	122.73 (11)	C63—C62—C71	115.04 (10)
C9—C10—C5	120.74 (12)	O61—C62—H62	107.5

C12—C11—C16	119.55 (12)	C63—C62—H62	107.5
C12—C11—C2	122.99 (11)	C71—C62—H62	107.5
C16—C11—C2	117.45 (11)	C71—C76—C75	119.88 (12)
C11—C12—C13	120.08 (12)	C71—C76—H76	120.1
C11—C12—H12	120.0	C75—C76—H76	120.1
C13—C12—H12	120.0	C63—C77—C78	132.10 (12)
C19—C20—C21	120.00 (12)	C63—C77—H77	114.0
C19—C20—H20	120.0	C78—C77—H77	114.0
C21—C20—H20	120.0	O64—C64—C65	121.73 (12)
C14—C13—C12	119.96 (13)	O64—C64—C63	121.96 (12)
C14—C13—H13	120.0	C65—C64—C63	116.29 (11)
C12—C13—H13	120.0	C67—C66—C65	120.28 (12)
C9—C8—C7	120.96 (13)	C67—C66—H66	119.9
C9—C8—H8	119.5	C65—C66—H66	119.9
C7—C8—H8	119.5	C74—C75—C76	120.42 (13)
C23—C22—C21	120.01 (12)	C74—C75—H75	119.8
C23—C22—H22	120.0	C76—C75—H75	119.8
C21—C22—H22	120.0	C70—C65—C66	119.30 (12)
C6—C7—C8	119.69 (13)	C70—C65—C64	119.75 (11)
C6—C7—H7	120.2	C66—C65—C64	120.87 (11)
C8—C7—H7	120.2	C75—C74—C73	119.94 (12)
O3—C211—H21A	109.5	C75—C74—H74	120.0
O3—C211—H21B	109.5	C73—C74—H74	120.0
H21A—C211—H21B	109.5	C72—C73—C74	119.84 (12)
O3—C211—H21C	109.5	C72—C73—H73	120.1
H21A—C211—H21C	109.5	C74—C73—H73	120.1
H21B—C211—H21C	109.5	C79—C78—C83	117.02 (12)
C40—O31—C32	117.69 (9)	C79—C78—C77	126.46 (12)
O31—C40—C39	116.48 (11)	C83—C78—C77	116.50 (11)
O31—C40—C35	122.69 (11)	C76—C71—C72	119.30 (12)
C39—C40—C35	120.73 (12)	C76—C71—C62	123.07 (11)
C51—O33—C511	118.08 (11)	C72—C71—C62	117.61 (11)
C42—C41—C46	119.40 (12)	C68—C69—C70	119.13 (13)
C42—C41—C32	123.58 (12)	C68—C69—H69	120.4
C46—C41—C32	117.02 (11)	C70—C69—H69	120.4
C41—C42—C43	120.03 (13)	C82—C83—C78	122.35 (12)
C41—C42—H42	120.0	C82—C83—H83	118.8
C43—C42—H42	120.0	C78—C83—H83	118.8
C47—C33—C34	118.04 (11)	C80—C79—C78	121.33 (12)
C47—C33—C32	124.20 (11)	C80—C79—H79	119.3
C34—C33—C32	117.76 (10)	C78—C79—H79	119.3
C45—C46—C41	120.51 (13)	C73—C72—C71	120.60 (12)
C45—C46—H46	119.7	C73—C72—H72	119.7
C41—C46—H46	119.7	C71—C72—H72	119.7
C49—C48—C53	117.00 (12)	C83—C82—C81	119.67 (12)
C49—C48—C47	126.19 (11)	C83—C82—H82	120.2
C53—C48—C47	116.77 (11)	C81—C82—H82	120.2
C37—C36—C35	120.48 (12)	C79—C80—C81	120.11 (12)

C37—C36—H36	119.8	C79—C80—H80	119.9
C35—C36—H36	119.8	C81—C80—H80	119.9
C33—C47—C48	131.28 (12)	C69—C68—C67	121.23 (13)
C33—C47—H47	114.4	C69—C68—H68	119.4
C48—C47—H47	114.4	C67—C68—H68	119.4
C53—C52—C51	120.09 (12)	O63—C811—H81A	109.5
C53—C52—H52	120.0	O63—C811—H81B	109.5
C51—C52—H52	120.0	H81A—C811—H81B	109.5
C40—C35—C36	118.98 (12)	O63—C811—H81C	109.5
C40—C35—C34	120.14 (11)	H81A—C811—H81C	109.5
C36—C35—C34	120.78 (11)	H81B—C811—H81C	109.5
C44—C43—C42	120.14 (13)	C66—C67—C68	119.50 (13)
C44—C43—H43	119.9	C66—C67—H67	120.3
C42—C43—H43	119.9	C68—C67—H67	120.3
O34—C34—C35	121.95 (12)	O63—C81—C82	115.48 (12)
O34—C34—C33	121.99 (11)	O63—C81—C80	125.02 (12)
C35—C34—C33	116.04 (11)	C82—C81—C80	119.50 (12)
C46—C45—C44	119.82 (13)		

C10—C5—C4—O4	172.55 (12)	C511—O33—C51—C52	172.73 (12)
C6—C5—C4—O4	−3.4 (2)	C53—C52—C51—O33	−177.89 (13)
C10—C5—C4—C3	−5.40 (17)	C53—C52—C51—C50	1.6 (2)
C6—C5—C4—C3	178.69 (11)	O33—C51—C50—C49	177.97 (12)
C10—C5—C6—C7	−0.1 (2)	C52—C51—C50—C49	−1.43 (19)
C4—C5—C6—C7	175.82 (13)	C42—C43—C44—C45	0.6 (2)
O4—C4—C3—C17	−12.43 (18)	C46—C45—C44—C43	−0.5 (2)
C5—C4—C3—C17	165.51 (11)	C51—C50—C49—C48	0.45 (19)
O4—C4—C3—C2	168.13 (12)	C53—C48—C49—C50	0.38 (18)
C5—C4—C3—C2	−13.93 (16)	C47—C48—C49—C50	−177.01 (12)
C4—C3—C17—C18	−179.20 (12)	O31—C40—C39—C38	−176.80 (12)
C2—C3—C17—C18	0.2 (2)	C35—C40—C39—C38	−0.4 (2)
C211—O3—C21—C22	173.76 (12)	C40—O31—C32—C33	−42.30 (14)
C211—O3—C21—C20	−6.13 (19)	C40—O31—C32—C41	85.35 (12)
C20—C19—C18—C23	−0.82 (18)	C47—C33—C32—O31	−145.24 (12)
C20—C19—C18—C17	−178.53 (12)	C34—C33—C32—O31	34.79 (14)
C22—C23—C18—C19	0.60 (19)	C47—C33—C32—C41	91.03 (15)
C22—C23—C18—C17	178.55 (12)	C34—C33—C32—C41	−88.94 (13)
C3—C17—C18—C19	−18.2 (2)	C42—C41—C32—O31	−117.82 (12)
C3—C17—C18—C23	164.12 (13)	C46—C41—C32—O31	62.14 (14)
C10—O1—C2—C3	−42.24 (14)	C42—C41—C32—C33	8.55 (17)
C10—O1—C2—C11	85.01 (12)	C46—C41—C32—C33	−171.49 (11)
C17—C3—C2—O1	−142.84 (12)	C51—C52—C53—C48	−0.7 (2)
C4—C3—C2—O1	36.55 (14)	C49—C48—C53—C52	−0.2 (2)
C17—C3—C2—C11	93.93 (14)	C47—C48—C53—C52	177.40 (13)
C4—C3—C2—C11	−86.68 (13)	C40—C39—C38—C37	0.0 (2)
C13—C14—C15—C16	−0.7 (2)	C36—C37—C38—C39	0.2 (2)
C11—C16—C15—C14	−0.1 (2)	C62—O61—C70—C69	−153.01 (11)
C2—O1—C10—C9	−158.52 (11)	C62—O61—C70—C65	30.56 (16)

C2—O1—C10—C5	24.89 (16)	C70—O61—C62—C63	−47.30 (13)
C8—C9—C10—O1	−176.47 (12)	C70—O61—C62—C71	79.67 (12)
C8—C9—C10—C5	0.2 (2)	C77—C63—C62—O61	−141.66 (12)
C6—C5—C10—O1	176.38 (11)	C64—C63—C62—O61	36.81 (14)
C4—C5—C10—O1	0.38 (18)	C77—C63—C62—C71	95.64 (15)
C6—C5—C10—C9	−0.07 (19)	C64—C63—C62—C71	−85.89 (13)
C4—C5—C10—C9	−176.07 (12)	C64—C63—C77—C78	−177.96 (12)
C15—C16—C11—C12	0.72 (19)	C62—C63—C77—C78	0.5 (2)
C15—C16—C11—C2	−177.83 (12)	C77—C63—C64—O64	−10.26 (19)
O1—C2—C11—C12	−114.83 (12)	C62—C63—C64—O64	171.15 (12)
C3—C2—C11—C12	10.94 (17)	C77—C63—C64—C65	168.52 (11)
O1—C2—C11—C16	63.66 (14)	C62—C63—C64—C65	−10.07 (16)
C3—C2—C11—C16	−170.57 (10)	C71—C76—C75—C74	0.3 (2)
C16—C11—C12—C13	−0.59 (18)	O61—C70—C65—C66	176.02 (11)
C2—C11—C12—C13	177.87 (11)	C69—C70—C65—C66	−0.30 (19)
C18—C19—C20—C21	0.85 (19)	O61—C70—C65—C64	−0.83 (18)
O3—C21—C20—C19	179.27 (12)	C69—C70—C65—C64	−177.15 (12)
C22—C21—C20—C19	−0.62 (19)	C67—C66—C65—C70	0.9 (2)
C15—C14—C13—C12	0.8 (2)	C67—C66—C65—C64	177.67 (13)
C11—C12—C13—C14	−0.16 (19)	O64—C64—C65—C70	169.77 (13)
C10—C9—C8—C7	−0.1 (2)	C63—C64—C65—C70	−9.01 (17)
C18—C23—C22—C21	−0.4 (2)	O64—C64—C65—C66	−7.0 (2)
O3—C21—C22—C23	−179.49 (12)	C63—C64—C65—C66	174.19 (11)
C20—C21—C22—C23	0.4 (2)	C76—C75—C74—C73	0.5 (2)
C5—C6—C7—C8	0.2 (2)	C75—C74—C73—C72	−0.4 (2)
C9—C8—C7—C6	−0.1 (2)	C63—C77—C78—C79	−21.0 (2)
C32—O31—C40—C39	−157.69 (11)	C63—C77—C78—C83	160.18 (14)
C32—O31—C40—C35	25.97 (16)	C75—C76—C71—C72	−1.11 (19)
C46—C41—C42—C43	−0.85 (18)	C75—C76—C71—C62	177.25 (12)
C32—C41—C42—C43	179.11 (12)	O61—C62—C71—C76	−119.98 (12)
C42—C41—C46—C45	1.01 (19)	C63—C62—C71—C76	4.98 (17)
C32—C41—C46—C45	−178.95 (12)	O61—C62—C71—C72	58.40 (14)
C34—C33—C47—C48	177.83 (12)	C63—C62—C71—C72	−176.64 (11)
C32—C33—C47—C48	−2.1 (2)	O61—C70—C69—C68	−176.75 (13)
C49—C48—C47—C33	−20.9 (2)	C65—C70—C69—C68	−0.3 (2)
C53—C48—C47—C33	161.75 (14)	C79—C78—C83—C82	1.7 (2)
O31—C40—C35—C36	176.70 (11)	C77—C78—C83—C82	−179.34 (12)
C39—C40—C35—C36	0.51 (19)	C83—C78—C79—C80	−1.62 (19)
O31—C40—C35—C34	0.11 (19)	C77—C78—C79—C80	179.56 (12)
C39—C40—C35—C34	−176.08 (12)	C74—C73—C72—C71	−0.5 (2)
C37—C36—C35—C40	−0.3 (2)	C76—C71—C72—C73	1.22 (19)
C37—C36—C35—C34	176.28 (13)	C62—C71—C72—C73	−177.23 (12)
C41—C42—C43—C44	0.0 (2)	C78—C83—C82—C81	−0.6 (2)
C40—C35—C34—O34	171.68 (12)	C78—C79—C80—C81	0.5 (2)
C36—C35—C34—O34	−4.8 (2)	C70—C69—C68—C67	0.3 (2)
C40—C35—C34—C33	−7.01 (17)	C65—C66—C67—C68	−0.8 (2)
C36—C35—C34—C33	176.46 (11)	C69—C68—C67—C66	0.3 (3)
C47—C33—C34—O34	−9.97 (18)	C811—O63—C81—C82	176.45 (12)

C32—C33—C34—O34	170.01 (12)	C811—O63—C81—C80	−3.50 (19)
C47—C33—C34—O34	168.73 (11)	C83—C82—C81—O63	179.47 (12)
C32—C33—C34—C35	−11.30 (16)	C83—C82—C81—C80	−0.6 (2)
C41—C46—C45—C44	−0.4 (2)	C79—C80—C81—O63	−179.39 (12)
C35—C36—C37—C38	0.0 (2)	C79—C80—C81—C82	0.7 (2)
C511—O33—C51—C50	−6.7 (2)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C2—H2···O34^{i}	1.00	2.45	3.3434 (15)	148
C6—H6···O61^{ii}	0.95	2.40	3.2676 (16)	151
C14—H14···O1^{iii}	0.95	2.46	3.3750 (15)	161
C32—H32···O64^{iv}	1.00	2.37	3.2348 (15)	144
C36—H36···O1^{v}	0.95	2.42	3.2733 (15)	150
C44—H44···O31^{iii}	0.95	2.42	3.3399 (16)	162
C62—H62···O4^{vi}	1.00	2.48	3.3629 (15)	146
C66—H66···O31^{vii}	0.95	2.48	3.3725 (15)	157
C74—H74···O61^{iii}	0.95	2.49	3.4018 (15)	161

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

(*E*)-3-Benzylidenechroman-4-one (compound_V_AAG5)

Crystal data

$C_{16}H_{12}O_2$
M_r = 236.26
Triclinic, $P\overline{1}$
a = 7.7718 (3) Å
b = 8.6069 (3) Å
c = 9.0857 (3) Å
α = 89.744 (3)°
β = 84.862 (3)°
γ = 74.199 (3)°
V = 582.31 (4) Å^3
Z = 2
F(000) = 248
D_x = 1.347 Mg m^{-3}
Mo *Kα* radiation, *λ* = 0.71073 Å
Cell parameters from 3741 reflections
θ = 3.7–35.8°
μ = 0.09 mm^{-1}
T = 100 K
Needle, colorless
0.18 × 0.08 × 0.08 mm

Data collection

Rigaku OD SuperNova Dual source diffractometer with an Atlas detector
Radiation source: micro-focus sealed X-ray tube, SuperNova (Mo) X-ray Source
Mirror monochromator
Detector resolution: 10.4498 pixels mm^{-1}
ω scans
Absorption correction: multi-scan (CrysAlis PRO; Rigaku OD, 2015)
T_{min} = 0.865, T_{max} = 1.000
4947 measured reflections
2425 independent reflections
2120 reflections with $I > 2\sigma(I)$
R_{int} = 0.023
θ_{max} = 26.5°, θ_{min} = 3.3°
h = −9→9
k = −9→10
l = −11→11

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.040
$wR(F^2)$ = 0.115
S = 1.06
2425 reflections
163 parameters
0 restraints

Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0597P)^2 + 0.1583P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.29$ e Å^{-3}
$\Delta\rho_{min} = -0.22$ e Å^{-3}

Special details

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

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

	x	*y*	*z*	U_{iso}*/U_{eq}
O1	0.56491 (12)	0.96012 (10)	0.67017 (9)	0.0203 (2)
O4	0.31184 (12)	0.69758 (10)	0.94932 (9)	0.0221 (2)
C9	0.43926 (16)	1.03656 (15)	0.78021 (13)	0.0174 (3)
C4	0.38354 (17)	0.77597 (15)	0.86245 (13)	0.0175 (3)
C12	0.74274 (17)	0.43694 (14)	0.64267 (13)	0.0182 (3)
C3	0.52016 (17)	0.69819 (15)	0.73982 (13)	0.0172 (3)
C10	0.34234 (16)	0.95369 (14)	0.87368 (13)	0.0177 (3)
C8	0.41493 (18)	1.20162 (15)	0.79971 (14)	0.0208 (3)
H8	0.4843	1.2560	0.7383	0.025*
C11	0.60958 (17)	0.54181 (15)	0.74961 (13)	0.0181 (3)
H11	0.5826	0.4918	0.8389	0.022*
C5	0.21682 (17)	1.04169 (16)	0.98441 (14)	0.0207 (3)
H5	0.1504	0.9873	1.0488	0.025*
C13	0.74448 (17)	0.45644 (15)	0.48910 (14)	0.0201 (3)
H13	0.6581	0.5435	0.4503	0.024*
C6	0.18851 (18)	1.20637 (16)	1.00108 (14)	0.0236 (3)
H6	0.1009	1.2652	1.0748	0.028*
C17	0.87029 (18)	0.30509 (15)	0.69590 (14)	0.0225 (3)
H17	0.8701	0.2881	0.7993	0.027*
C7	0.28933 (18)	1.28633 (15)	0.90890 (14)	0.0225 (3)
H7	0.2714	1.3993	0.9214	0.027*
C2	0.54324 (18)	0.80948 (14)	0.61596 (13)	0.0194 (3)
H2A	0.6499	0.7553	0.5486	0.023*
H2B	0.4369	0.8322	0.5587	0.023*
C16	0.99632 (18)	0.19957 (16)	0.59981 (16)	0.0262 (3)
H16	1.0826	0.1117	0.6377	0.031*
C15	0.99711 (18)	0.22168 (16)	0.44774 (16)	0.0257 (3)
H15	1.0836	0.1492	0.3819	0.031*
C14	0.87098 (18)	0.35004 (16)	0.39325 (15)	0.0234 (3)
H14	0.8711	0.3653	0.2896	0.028*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0259 (5)	0.0181 (4)	0.0172 (4)	−0.0084 (4)	0.0039 (3)	0.0016 (3)
O4	0.0288 (5)	0.0228 (5)	0.0160 (4)	−0.0104 (4)	0.0013 (4)	0.0049 (3)
C9	0.0191 (6)	0.0210 (6)	0.0126 (5)	−0.0055 (5)	−0.0035 (4)	0.0035 (4)
C4	0.0207 (6)	0.0212 (6)	0.0126 (6)	−0.0080 (5)	−0.0045 (4)	0.0047 (4)
C12	0.0199 (6)	0.0184 (6)	0.0185 (6)	−0.0085 (5)	−0.0026 (5)	0.0024 (5)
C3	0.0213 (6)	0.0201 (6)	0.0125 (6)	−0.0094 (5)	−0.0017 (4)	0.0024 (4)
C10	0.0201 (6)	0.0200 (6)	0.0142 (6)	−0.0063 (5)	−0.0046 (5)	0.0031 (5)
C8	0.0261 (7)	0.0211 (6)	0.0177 (6)	−0.0098 (5)	−0.0057 (5)	0.0057 (5)
C11	0.0232 (6)	0.0210 (6)	0.0128 (6)	−0.0098 (5)	−0.0040 (5)	0.0039 (5)
C5	0.0209 (6)	0.0250 (6)	0.0166 (6)	−0.0071 (5)	−0.0017 (5)	0.0035 (5)
C13	0.0219 (6)	0.0208 (6)	0.0185 (6)	−0.0064 (5)	−0.0041 (5)	0.0022 (5)
C6	0.0233 (7)	0.0256 (7)	0.0197 (6)	−0.0029 (5)	−0.0014 (5)	−0.0013 (5)
C17	0.0248 (7)	0.0236 (6)	0.0206 (6)	−0.0086 (5)	−0.0043 (5)	0.0055 (5)
C7	0.0282 (7)	0.0181 (6)	0.0215 (6)	−0.0053 (5)	−0.0077 (5)	0.0019 (5)
C2	0.0267 (7)	0.0184 (6)	0.0138 (6)	−0.0076 (5)	0.0000 (5)	0.0022 (5)
C16	0.0227 (7)	0.0230 (7)	0.0312 (7)	−0.0026 (5)	−0.0050 (5)	0.0040 (5)
C15	0.0204 (7)	0.0268 (7)	0.0290 (7)	−0.0066 (5)	0.0029 (5)	−0.0045 (5)
C14	0.0256 (7)	0.0279 (7)	0.0183 (6)	−0.0105 (5)	0.0001 (5)	−0.0013 (5)

Geometric parameters (Å, º)

O1—C9	1.3664 (14)	C5—C6	1.3808 (18)
O1—C2	1.4462 (14)	C5—H5	0.9500
O4—C4	1.2288 (15)	C13—C14	1.3882 (18)
C9—C8	1.3912 (17)	C13—H13	0.9500
C9—C10	1.4035 (17)	C6—C7	1.4000 (19)
C4—C10	1.4770 (17)	C6—H6	0.9500
C4—C3	1.4915 (17)	C17—C16	1.3842 (18)
C12—C13	1.4040 (17)	C17—H17	0.9500
C12—C17	1.4045 (17)	C7—H7	0.9500
C12—C11	1.4659 (17)	C2—H2A	0.9900
C3—C11	1.3428 (17)	C2—H2B	0.9900
C3—C2	1.5046 (16)	C16—C15	1.394 (2)
C10—C5	1.4022 (17)	C16—H16	0.9500
C8—C7	1.3834 (18)	C15—C14	1.3852 (19)
C8—H8	0.9500	C15—H15	0.9500
C11—H11	0.9500	C14—H14	0.9500
C9—O1—C2	115.34 (9)	C14—C13—H13	119.6
O1—C9—C8	116.88 (11)	C12—C13—H13	119.6
O1—C9—C10	122.40 (11)	C5—C6—C7	119.76 (12)
C8—C9—C10	120.69 (12)	C5—C6—H6	120.1
O4—C4—C10	122.27 (11)	C7—C6—H6	120.1
O4—C4—C3	122.37 (11)	C16—C17—C12	120.92 (12)
C10—C4—C3	115.35 (10)	C16—C17—H17	119.5

C13—C12—C17	118.03 (11)	C12—C17—H17	119.5
C13—C12—C11	123.52 (11)	C8—C7—C6	120.34 (12)
C17—C12—C11	118.37 (11)	C8—C7—H7	119.8
C11—C3—C4	118.95 (11)	C6—C7—H7	119.8
C11—C3—C2	126.81 (11)	O1—C2—C3	111.90 (10)
C4—C3—C2	114.23 (10)	O1—C2—H2A	109.2
C5—C10—C9	118.55 (11)	C3—C2—H2A	109.2
C5—C10—C4	121.13 (11)	O1—C2—H2B	109.2
C9—C10—C4	120.13 (11)	C3—C2—H2B	109.2
C7—C8—C9	119.77 (11)	H2A—C2—H2B	107.9
C7—C8—H8	120.1	C17—C16—C15	120.25 (12)
C9—C8—H8	120.1	C17—C16—H16	119.9
C3—C11—C12	129.45 (11)	C15—C16—H16	119.9
C3—C11—H11	115.3	C14—C15—C16	119.59 (12)
C12—C11—H11	115.3	C14—C15—H15	120.2
C6—C5—C10	120.84 (12)	C16—C15—H15	120.2
C6—C5—H5	119.6	C15—C14—C13	120.42 (12)
C10—C5—H5	119.6	C15—C14—H14	119.8
C14—C13—C12	120.78 (12)	C13—C14—H14	119.8

C2—O1—C9—C8	−158.11 (11)	C13—C12—C11—C3	−29.7 (2)
C2—O1—C9—C10	24.05 (16)	C17—C12—C11—C3	153.47 (13)
O4—C4—C3—C11	−21.46 (17)	C9—C10—C5—C6	0.20 (18)
C10—C4—C3—C11	157.66 (11)	C4—C10—C5—C6	175.21 (11)
O4—C4—C3—C2	159.17 (12)	C17—C12—C13—C14	−1.21 (18)
C10—C4—C3—C2	−21.72 (14)	C11—C12—C13—C14	−178.02 (11)
O1—C9—C10—C5	179.55 (10)	C10—C5—C6—C7	−1.62 (19)
C8—C9—C10—C5	1.79 (18)	C13—C12—C17—C16	1.34 (18)
O1—C9—C10—C4	4.49 (18)	C11—C12—C17—C16	178.32 (11)
C8—C9—C10—C4	−173.27 (11)	C9—C8—C7—C6	0.90 (19)
O4—C4—C10—C5	−0.65 (18)	C5—C6—C7—C8	1.08 (19)
C3—C4—C10—C5	−179.76 (10)	C9—O1—C2—C3	−50.13 (14)
O4—C4—C10—C9	174.29 (11)	C11—C3—C2—O1	−130.51 (13)
C3—C4—C10—C9	−4.83 (16)	C4—C3—C2—O1	48.81 (14)
O1—C9—C8—C7	179.78 (11)	C12—C17—C16—C15	−0.8 (2)
C10—C9—C8—C7	−2.35 (18)	C17—C16—C15—C14	0.0 (2)
C4—C3—C11—C12	177.04 (11)	C16—C15—C14—C13	0.1 (2)
C2—C3—C11—C12	−3.7 (2)	C12—C13—C14—C15	0.52 (19)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C11—H11···O4^{i}	0.95	2.56	3.4153 (15)	150
C17—H17···O4^{i}	0.95	2.56	3.4050 (16)	148
C7—H7···O4^{ii}	0.95	2.69	3.6131 (16)	166

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

(*E*)-3-(4-Methoxybenzylidene)chroman-4-one (AAG4)

Crystal data

$C_{17}H_{14}O_3$
$M_r = 266.28$
Triclinic, $P\overline{1}$
$a = 7.5570$ (3) Å
$b = 12.3558$ (4) Å
$c = 14.4044$ (6) Å
$\alpha = 82.748$ (3)°
$\beta = 87.126$ (3)°
$\gamma = 75.768$ (3)°
$V = 1293.02$ (9) Å^3
$Z = 4$
$F(000) = 560$
$D_x = 1.368$ Mg m^{-3}
Cu $K\alpha$ radiation, $\lambda = 1.54184$ Å
Cell parameters from 8925 reflections
$\theta = 3.7$–$71.9°$
$\mu = 0.76$ mm^{-1}
$T = 100$ K
Needle, colorless
0.3 × 0.08 × 0.06 mm

Data collection

XtaLAB Synergy Dualflex HyPix diffractometer
Radiation source: micro-focus sealed X-ray tube, PhotonJet (Cu) X-ray Source
Mirror monochromator
Detector resolution: 10.0000 pixels mm^{-1}
ω scans
Absorption correction: multi-scan (CrysAlis PRO; Rigaku OD, 2019)
$T_{min} = 0.671$, $T_{max} = 1.000$
5118 measured reflections
5118 independent reflections
4669 reflections with $I > 2\sigma(I)$
$R_{int} = 0.020$
$\theta_{max} = 66.6°$, $\theta_{min} = 3.1°$
$h = -8 \rightarrow 8$
$k = -14 \rightarrow 14$
$l = -17 \rightarrow 17$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.070$
$wR(F^2) = 0.200$
$S = 1.09$
5118 reflections
364 parameters
0 restraints
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.1415P)^2 + 0.4525P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.63$ e Å^{-3}
$\Delta\rho_{min} = -0.33$ e Å^{-3}

Special details

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

Refinement. Refined as a 2-component twin.

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

	x	*y*	*z*	U_{iso}*/U_{eq}
O22	0.4818 (3)	0.40499 (15)	0.86434 (13)	0.0291 (4)
O21	−0.0050 (2)	−0.08126 (14)	0.93266 (12)	0.0242 (4)
O24	0.4539 (3)	−0.26141 (17)	0.81175 (16)	0.0385 (5)
C32	0.4383 (3)	0.0781 (2)	0.85181 (17)	0.0219 (5)
C34	0.3282 (3)	0.2796 (2)	0.81810 (17)	0.0229 (5)
H34	0.2396	0.3429	0.7911	0.028*
C27	−0.1267 (4)	−0.3485 (2)	0.94295 (18)	0.0282 (6)
H27	−0.2211	−0.3822	0.9690	0.034*

C29	0.0045 (3)	−0.1894 (2)	0.91708 (16)	0.0217 (5)
C37	0.5883 (3)	0.0959 (2)	0.89517 (17)	0.0245 (5)
H37	0.6791	0.0328	0.9205	0.029*
C31	0.4290 (3)	−0.0379 (2)	0.84604 (17)	0.0241 (5)
H31	0.5435	−0.0899	0.8398	0.029*
C30	0.1541 (3)	−0.2517 (2)	0.86905 (17)	0.0223 (5)
C33	0.3082 (3)	0.1718 (2)	0.81231 (17)	0.0232 (5)
H33	0.2062	0.1616	0.7815	0.028*
C28	−0.1342 (4)	−0.2376 (2)	0.95432 (17)	0.0260 (5)
H28	−0.2338	−0.1951	0.9874	0.031*
C24	0.3103 (4)	−0.2035 (2)	0.83950 (18)	0.0253 (6)
C36	0.6085 (3)	0.2030 (2)	0.90225 (17)	0.0251 (5)
H36	0.7102	0.2134	0.9332	0.030*
C23	0.2839 (3)	−0.0813 (2)	0.84823 (17)	0.0229 (5)
C22	0.0881 (3)	−0.0182 (2)	0.86382 (18)	0.0242 (5)
H22A	0.0863	0.0552	0.8852	0.029*
H22B	0.0228	−0.0034	0.8039	0.029*
C35	0.4763 (3)	0.2955 (2)	0.86302 (17)	0.0233 (5)
C25	0.1572 (4)	−0.3633 (2)	0.85732 (17)	0.0249 (5)
H25	0.2560	−0.4061	0.8240	0.030*
C26	0.0184 (4)	−0.4111 (2)	0.89352 (18)	0.0276 (6)
H26	0.0213	−0.4866	0.8849	0.033*
C38	0.6384 (4)	0.4274 (2)	0.9020 (2)	0.0372 (7)
H38A	0.6447	0.4006	0.9691	0.056*
H38B	0.6293	0.5085	0.8929	0.056*
H38C	0.7487	0.3884	0.8700	0.056*
O1	0.5023 (2)	0.60541 (14)	0.43853 (12)	0.0229 (4)
O4	0.0517 (2)	0.82132 (15)	0.32039 (14)	0.0303 (5)
O2	0.0227 (3)	0.14104 (15)	0.35386 (13)	0.0298 (4)
C12	0.0649 (3)	0.4712 (2)	0.35122 (17)	0.0226 (5)
C14	0.1829 (3)	0.2797 (2)	0.31505 (17)	0.0239 (5)
H14	0.2757	0.2250	0.2883	0.029*
C3	0.2212 (3)	0.6308 (2)	0.35305 (16)	0.0217 (5)
C4	0.1961 (3)	0.7548 (2)	0.34556 (17)	0.0230 (5)
C11	0.0749 (3)	0.5888 (2)	0.34865 (17)	0.0225 (5)
H11	−0.0389	0.6428	0.3431	0.027*
C8	0.6381 (3)	0.7532 (2)	0.45601 (17)	0.0252 (5)
H8	0.7344	0.7008	0.4892	0.030*
C7	0.6363 (4)	0.8663 (2)	0.44207 (18)	0.0276 (6)
H7	0.7320	0.8916	0.4661	0.033*
C2	0.4134 (3)	0.5615 (2)	0.37033 (17)	0.0214 (5)
H2A	0.4103	0.4829	0.3930	0.026*
H2B	0.4846	0.5612	0.3107	0.026*
C15	0.0292 (3)	0.2501 (2)	0.35630 (17)	0.0241 (5)
C13	0.2016 (3)	0.3887 (2)	0.31268 (17)	0.0224 (5)
H13	0.3080	0.4080	0.2847	0.027*
C10	0.3523 (3)	0.7936 (2)	0.37333 (16)	0.0219 (5)
C5	0.3548 (3)	0.9078 (2)	0.35931 (17)	0.0235 (5)

H5	0.2587	0.9606	0.3263	0.028*
C9	0.4980 (3)	0.7173 (2)	0.42096 (16)	0.0215 (5)
C17	−0.0894 (3)	0.4396 (2)	0.39111 (17)	0.0235 (5)
H17	−0.1845	0.4945	0.4161	0.028*
C16	−0.1079 (3)	0.3297 (2)	0.39524 (18)	0.0259 (5)
H16	−0.2127	0.3093	0.4242	0.031*
C6	0.4947 (4)	0.9441 (2)	0.39283 (18)	0.0277 (6)
H6	0.4954	1.0215	0.3827	0.033*
C18	−0.1317 (4)	0.1054 (2)	0.3944 (2)	0.0360 (7)
H18A	−0.2428	0.1530	0.3650	0.054*
H18B	−0.1379	0.1117	0.4617	0.054*
H18C	−0.1208	0.0270	0.3844	0.054*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O22	0.0341 (10)	0.0234 (9)	0.0310 (10)	−0.0096 (8)	−0.0063 (8)	−0.0002 (7)
O21	0.0247 (9)	0.0204 (9)	0.0266 (9)	−0.0054 (7)	0.0066 (7)	−0.0024 (7)
O24	0.0279 (10)	0.0297 (10)	0.0585 (14)	−0.0065 (8)	0.0143 (9)	−0.0143 (9)
C32	0.0189 (11)	0.0262 (13)	0.0194 (11)	−0.0050 (9)	0.0049 (9)	−0.0010 (9)
C34	0.0221 (12)	0.0237 (12)	0.0207 (12)	−0.0038 (9)	0.0008 (9)	0.0022 (9)
C27	0.0312 (14)	0.0313 (14)	0.0245 (13)	−0.0133 (11)	−0.0008 (10)	−0.0003 (11)
C29	0.0227 (12)	0.0211 (12)	0.0195 (11)	−0.0024 (9)	−0.0027 (9)	−0.0008 (9)
C37	0.0188 (12)	0.0294 (13)	0.0233 (12)	−0.0048 (9)	0.0023 (9)	0.0015 (10)
C31	0.0219 (12)	0.0249 (13)	0.0232 (12)	−0.0031 (9)	0.0015 (10)	−0.0004 (10)
C30	0.0217 (12)	0.0240 (12)	0.0198 (12)	−0.0037 (9)	−0.0019 (9)	−0.0003 (9)
C33	0.0224 (12)	0.0285 (13)	0.0194 (11)	−0.0086 (10)	0.0015 (9)	−0.0008 (10)
C28	0.0270 (13)	0.0281 (13)	0.0224 (12)	−0.0068 (10)	0.0011 (10)	−0.0018 (10)
C24	0.0256 (13)	0.0233 (13)	0.0260 (13)	−0.0038 (10)	0.0027 (10)	−0.0040 (10)
C36	0.0221 (12)	0.0308 (14)	0.0224 (12)	−0.0076 (10)	−0.0022 (10)	0.0003 (10)
C23	0.0240 (12)	0.0246 (13)	0.0191 (11)	−0.0046 (10)	0.0021 (9)	−0.0026 (9)
C22	0.0224 (12)	0.0213 (12)	0.0280 (13)	−0.0064 (9)	0.0057 (10)	0.0001 (10)
C35	0.0257 (13)	0.0251 (13)	0.0193 (12)	−0.0082 (10)	0.0033 (9)	−0.0016 (9)
C25	0.0298 (13)	0.0220 (12)	0.0213 (12)	−0.0024 (10)	−0.0038 (10)	−0.0028 (10)
C26	0.0358 (14)	0.0217 (12)	0.0262 (13)	−0.0087 (10)	−0.0052 (11)	−0.0003 (10)
C38	0.0448 (17)	0.0300 (14)	0.0411 (16)	−0.0170 (12)	−0.0111 (13)	−0.0009 (12)
O1	0.0236 (8)	0.0196 (8)	0.0249 (9)	−0.0051 (7)	−0.0048 (7)	0.0009 (7)
O4	0.0236 (9)	0.0231 (9)	0.0421 (11)	−0.0036 (7)	−0.0078 (8)	0.0030 (8)
O2	0.0324 (10)	0.0237 (9)	0.0347 (10)	−0.0108 (8)	0.0047 (8)	−0.0035 (8)
C12	0.0218 (12)	0.0240 (13)	0.0217 (12)	−0.0047 (10)	−0.0051 (9)	−0.0018 (10)
C14	0.0211 (12)	0.0277 (13)	0.0213 (12)	−0.0025 (9)	−0.0006 (9)	−0.0037 (10)
C3	0.0259 (13)	0.0214 (12)	0.0168 (11)	−0.0045 (10)	−0.0002 (9)	−0.0012 (9)
C4	0.0252 (13)	0.0213 (13)	0.0209 (12)	−0.0042 (10)	0.0006 (10)	0.0009 (9)
C11	0.0230 (12)	0.0233 (13)	0.0205 (12)	−0.0045 (10)	−0.0023 (9)	−0.0018 (9)
C8	0.0247 (12)	0.0275 (13)	0.0221 (12)	−0.0045 (10)	−0.0021 (10)	−0.0012 (10)
C7	0.0307 (13)	0.0303 (14)	0.0252 (13)	−0.0131 (11)	0.0000 (10)	−0.0048 (10)
C2	0.0216 (12)	0.0209 (12)	0.0226 (12)	−0.0067 (9)	−0.0016 (9)	−0.0025 (9)
C15	0.0265 (13)	0.0240 (13)	0.0218 (12)	−0.0069 (10)	−0.0024 (10)	−0.0001 (10)

C13	0.0193 (11)	0.0275 (13)	0.0208 (12)	−0.0069 (10)	−0.0008 (9)	−0.0014 (10)
C10	0.0216 (12)	0.0237 (12)	0.0192 (12)	−0.0045 (9)	0.0025 (9)	−0.0007 (9)
C5	0.0253 (13)	0.0234 (13)	0.0203 (12)	−0.0045 (10)	0.0037 (9)	−0.0011 (10)
C9	0.0238 (12)	0.0219 (12)	0.0179 (11)	−0.0049 (10)	0.0030 (9)	−0.0018 (9)
C17	0.0207 (12)	0.0257 (13)	0.0237 (12)	−0.0042 (9)	0.0003 (9)	−0.0043 (10)
C16	0.0227 (12)	0.0295 (13)	0.0262 (13)	−0.0084 (10)	0.0013 (10)	−0.0019 (10)
C6	0.0340 (14)	0.0241 (13)	0.0268 (13)	−0.0100 (10)	0.0030 (10)	−0.0050 (10)
C18	0.0433 (16)	0.0287 (14)	0.0398 (16)	−0.0180 (12)	0.0112 (13)	−0.0049 (12)

Geometric parameters (Å, º)

O22—C35	1.366 (3)	O1—C9	1.366 (3)
O22—C38	1.427 (3)	O1—C2	1.446 (3)
O21—C29	1.366 (3)	O4—C4	1.233 (3)
O21—C22	1.446 (3)	O2—C15	1.365 (3)
O24—C24	1.223 (3)	O2—C18	1.419 (3)
C32—C37	1.397 (4)	C12—C17	1.394 (4)
C32—C33	1.400 (4)	C12—C13	1.408 (3)
C32—C31	1.465 (3)	C12—C11	1.469 (3)
C34—C35	1.387 (4)	C14—C13	1.384 (4)
C34—C33	1.391 (3)	C14—C15	1.386 (4)
C34—H34	0.9500	C14—H14	0.9500
C27—C28	1.388 (4)	C3—C11	1.339 (4)
C27—C26	1.397 (4)	C3—C4	1.488 (3)
C27—H27	0.9500	C3—C2	1.509 (3)
C29—C28	1.382 (4)	C4—C10	1.469 (4)
C29—C30	1.407 (3)	C11—H11	0.9500
C37—C36	1.385 (4)	C8—C9	1.383 (4)
C37—H37	0.9500	C8—C7	1.384 (4)
C31—C23	1.331 (4)	C8—H8	0.9500
C31—H31	0.9500	C7—C6	1.404 (4)
C30—C25	1.404 (3)	C7—H7	0.9500
C30—C24	1.470 (4)	C2—H2A	0.9900
C33—H33	0.9500	C2—H2B	0.9900
C28—H28	0.9500	C15—C16	1.391 (4)
C24—C23	1.494 (3)	C13—H13	0.9500
C36—C35	1.398 (4)	C10—C5	1.404 (3)
C36—H36	0.9500	C10—C9	1.405 (3)
C23—C22	1.514 (3)	C5—C6	1.375 (4)
C22—H22A	0.9900	C5—H5	0.9500
C22—H22B	0.9900	C17—C16	1.393 (4)
C25—C26	1.376 (4)	C17—H17	0.9500
C25—H25	0.9500	C16—H16	0.9500
C26—H26	0.9500	C6—H6	0.9500
C38—H38A	0.9800	C18—H18A	0.9800
C38—H38B	0.9800	C18—H18B	0.9800
C38—H38C	0.9800	C18—H18C	0.9800

C35—O22—C38	118.4 (2)	C9—O1—C2	115.39 (18)
C29—O21—C22	115.55 (18)	C15—O2—C18	118.5 (2)
C37—C32—C33	118.5 (2)	C17—C12—C13	117.9 (2)
C37—C32—C31	118.4 (2)	C17—C12—C11	118.9 (2)
C33—C32—C31	123.1 (2)	C13—C12—C11	123.3 (2)
C35—C34—C33	120.5 (2)	C13—C14—C15	120.2 (2)
C35—C34—H34	119.8	C13—C14—H14	119.9
C33—C34—H34	119.8	C15—C14—H14	119.9
C28—C27—C26	120.6 (2)	C11—C3—C4	119.2 (2)
C28—C27—H27	119.7	C11—C3—C2	125.0 (2)
C26—C27—H27	119.7	C4—C3—C2	115.8 (2)
O21—C29—C28	117.6 (2)	O4—C4—C10	121.8 (2)
O21—C29—C30	121.5 (2)	O4—C4—C3	122.4 (2)
C28—C29—C30	120.9 (2)	C10—C4—C3	115.8 (2)
C36—C37—C32	121.9 (2)	C3—C11—C12	129.4 (2)
C36—C37—H37	119.0	C3—C11—H11	115.3
C32—C37—H37	119.0	C12—C11—H11	115.3
C23—C31—C32	129.6 (2)	C9—C8—C7	119.2 (2)
C23—C31—H31	115.2	C9—C8—H8	120.4
C32—C31—H31	115.2	C7—C8—H8	120.4
C25—C30—C29	118.5 (2)	C8—C7—C6	120.7 (2)
C25—C30—C24	121.2 (2)	C8—C7—H7	119.7
C29—C30—C24	120.1 (2)	C6—C7—H7	119.7
C34—C33—C32	120.1 (2)	O1—C2—C3	111.52 (19)
C34—C33—H33	120.0	O1—C2—H2A	109.3
C32—C33—H33	120.0	C3—C2—H2A	109.3
C29—C28—C27	119.5 (2)	O1—C2—H2B	109.3
C29—C28—H28	120.3	C3—C2—H2B	109.3
C27—C28—H28	120.3	H2A—C2—H2B	108.0
O24—C24—C30	121.4 (2)	O2—C15—C14	115.7 (2)
O24—C24—C23	122.7 (2)	O2—C15—C16	124.0 (2)
C30—C24—C23	115.9 (2)	C14—C15—C16	120.3 (2)
C37—C36—C35	118.7 (2)	C14—C13—C12	120.8 (2)
C37—C36—H36	120.6	C14—C13—H13	119.6
C35—C36—H36	120.6	C12—C13—H13	119.6
C31—C23—C24	119.4 (2)	C5—C10—C9	118.3 (2)
C31—C23—C22	125.5 (2)	C5—C10—C4	121.4 (2)
C24—C23—C22	115.0 (2)	C9—C10—C4	120.1 (2)
O21—C22—C23	111.40 (19)	C6—C5—C10	120.8 (2)
O21—C22—H22A	109.3	C6—C5—H5	119.6
C23—C22—H22A	109.3	C10—C5—H5	119.6
O21—C22—H22B	109.3	O1—C9—C8	117.3 (2)
C23—C22—H22B	109.3	O1—C9—C10	121.3 (2)
H22A—C22—H22B	108.0	C8—C9—C10	121.4 (2)
O22—C35—C34	115.4 (2)	C16—C17—C12	121.7 (2)
O22—C35—C36	124.3 (2)	C16—C17—H17	119.1
C34—C35—C36	120.3 (2)	C12—C17—H17	119.1
C26—C25—C30	120.7 (2)	C15—C16—C17	119.1 (2)

C26—C25—H25	119.6	C15—C16—H16	120.5
C30—C25—H25	119.6	C17—C16—H16	120.5
C25—C26—C27	119.8 (2)	C5—C6—C7	119.7 (2)
C25—C26—H26	120.1	C5—C6—H6	120.2
C27—C26—H26	120.1	C7—C6—H6	120.2
O22—C38—H38A	109.5	O2—C18—H18A	109.5
O22—C38—H38B	109.5	O2—C18—H18B	109.5
H38A—C38—H38B	109.5	H18A—C18—H18B	109.5
O22—C38—H38C	109.5	O2—C18—H18C	109.5
H38A—C38—H38C	109.5	H18A—C18—H18C	109.5
H38B—C38—H38C	109.5	H18B—C18—H18C	109.5

C22—O21—C29—C28	−154.3 (2)	C11—C3—C4—O4	−12.1 (4)
C22—O21—C29—C30	28.9 (3)	C2—C3—C4—O4	171.0 (2)
C33—C32—C37—C36	1.8 (4)	C11—C3—C4—C10	165.8 (2)
C31—C32—C37—C36	179.1 (2)	C2—C3—C4—C10	−11.1 (3)
C37—C32—C31—C23	147.3 (3)	C4—C3—C11—C12	177.8 (2)
C33—C32—C31—C23	−35.7 (4)	C2—C3—C11—C12	−5.6 (4)
O21—C29—C30—C25	178.6 (2)	C17—C12—C11—C3	148.4 (3)
C28—C29—C30—C25	1.8 (3)	C13—C12—C11—C3	−33.6 (4)
O21—C29—C30—C24	3.8 (3)	C9—C8—C7—C6	−0.2 (4)
C28—C29—C30—C24	−172.9 (2)	C9—O1—C2—C3	−52.0 (3)
C35—C34—C33—C32	−0.1 (4)	C11—C3—C2—O1	−134.7 (2)
C37—C32—C33—C34	−1.1 (4)	C4—C3—C2—O1	42.0 (3)
C31—C32—C33—C34	−178.2 (2)	C18—O2—C15—C14	−179.6 (2)
O21—C29—C28—C27	−177.8 (2)	C18—O2—C15—C16	−0.9 (4)
C30—C29—C28—C27	−0.9 (4)	C13—C14—C15—O2	179.1 (2)
C26—C27—C28—C29	−0.7 (4)	C13—C14—C15—C16	0.3 (4)
C25—C30—C24—O24	−6.8 (4)	C15—C14—C13—C12	−0.5 (4)
C29—C30—C24—O24	167.8 (2)	C17—C12—C13—C14	−0.4 (3)
C25—C30—C24—C23	174.9 (2)	C11—C12—C13—C14	−178.4 (2)
C29—C30—C24—C23	−10.5 (3)	O4—C4—C10—C5	−9.7 (4)
C32—C37—C36—C35	−1.3 (4)	C3—C4—C10—C5	172.4 (2)
C32—C31—C23—C24	179.0 (2)	O4—C4—C10—C9	165.8 (2)
C32—C31—C23—C22	−4.1 (4)	C3—C4—C10—C9	−12.1 (3)
O24—C24—C23—C31	−15.2 (4)	C9—C10—C5—C6	−1.1 (3)
C30—C24—C23—C31	163.1 (2)	C4—C10—C5—C6	174.4 (2)
O24—C24—C23—C22	167.7 (2)	C2—O1—C9—C8	−152.8 (2)
C30—C24—C23—C22	−14.0 (3)	C2—O1—C9—C10	30.3 (3)
C29—O21—C22—C23	−52.1 (3)	C7—C8—C9—O1	−178.2 (2)
C31—C23—C22—O21	−132.7 (3)	C7—C8—C9—C10	−1.3 (4)
C24—C23—C22—O21	44.2 (3)	C5—C10—C9—O1	178.7 (2)
C38—O22—C35—C34	−174.6 (2)	C4—C10—C9—O1	3.2 (3)
C38—O22—C35—C36	4.7 (4)	C5—C10—C9—C8	2.0 (3)
C33—C34—C35—O22	180.0 (2)	C4—C10—C9—C8	−173.6 (2)
C33—C34—C35—C36	0.6 (4)	C13—C12—C17—C16	1.5 (4)
C37—C36—C35—O22	−179.2 (2)	C11—C12—C17—C16	179.6 (2)
C37—C36—C35—C34	0.1 (4)	O2—C15—C16—C17	−177.9 (2)

C29—C30—C25—C26	−1.2 (4)	C14—C15—C16—C17	0.8 (4)
C24—C30—C25—C26	173.5 (2)	C12—C17—C16—C15	−1.7 (4)
C30—C25—C26—C27	−0.3 (4)	C10—C5—C6—C7	−0.3 (4)
C28—C27—C26—C25	1.3 (4)	C8—C7—C6—C5	1.0 (4)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C2—H2*B*···O22^{i}	0.99	2.51	3.430 (3)	154
C2—H2*A*···O1^{i}	0.99	2.58	3.215 (3)	122
C5—H5···O2^{ii}	0.95	2.55	3.319 (3)	138
C14—H14···O24^{iii}	0.95	2.54	3.196 (3)	127
C17—H17···O1^{iv}	0.95	2.45	3.349 (3)	157
C25—H25···O22^{v}	0.95	2.55	3.275 (3)	133
C33—H33···O4^{vi}	0.95	2.45	3.381 (3)	166
C37—H37···O21^{vii}	0.95	2.46	3.330 (3)	153

Symmetry codes: (i) −*x*+1, −*y*+1, −*z*+1; (ii) *x*, *y*+1, *z*; (iii) −*x*+1, −*y*, −*z*+1; (iv) *x*−1, *y*, *z*; (v) *x*, *y*−1, *z*; (vi) −*x*, −*y*+1, −*z*+1; (vii) *x*+1, *y*, *z*.