

Crystal structures of (*E*)-3-(4-hydroxybenzylidene)-chroman-4-one and (*E*)-3-(3-hydroxybenzylidene)-2-phenylchroman-4-one

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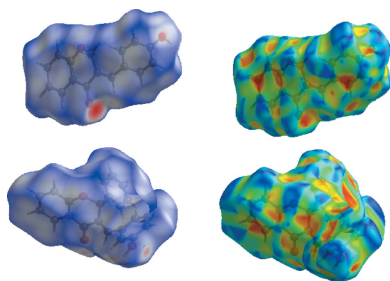
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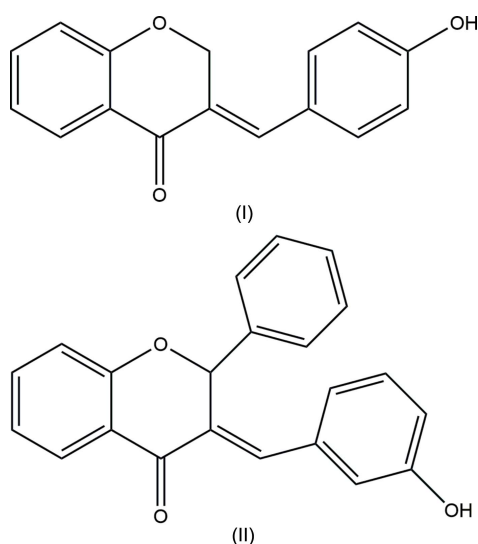
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The synthesis and crystal structures of (*E*)-3-(4-hydroxybenzylidene)chroman-4-one, C₁₆H₁₂O₃, **I**, and (*E*)-3-(3-hydroxybenzylidene)-2-phenylchroman-4-one, C₂₂H₁₆O₃, **II**, are reported. These compounds are of interest with respect to biological activity. Both structures display intermolecular C—H···O and O—H···O hydrogen bonding, forming layers in the crystal lattice. The crystal structure of compound **I** is consolidated by π – π interactions. The lipophilicity ($\log P$) was determined as it is one of the parameters qualifying compounds as potential drugs. The $\log P$ value for compound **I** is associated with a larger contribution of C···H interaction in the Hirshfeld surface.

1. Chemical context

Chromanone (chroman-4-one) and flavanone (2-phenylchroman-4-one) belong to the class of heterocyclic compounds and are composed of a benzene ring fused to a 2,3-dihydro- γ -pyranone ring (Emami & Ghanbarimasir, 2015). 3-Arylidenechromanones/flavanones and their derivatives are naturally occurring homoisoflavones, and can be obtained by condensing the corresponding aryl aldehydes with chromanone/flavanone. These compounds were synthesized for the first time by Robinson in the early 1920s by the condensation reaction of chromanone or flavanone with the appropriate aryl aldehyde using a catalyst (alcohol potassium hydroxide) (Perkin *et al.*, 1926). In 1979, Levai and Schag synthesized *E*-3-arylidenechroman-4-one using piperidine as a catalyst (Levai & Schag, 1979). Several years later, in 1993, Pijewska and coworkers (Pijewska *et al.*, 1993) obtained the series of 3-arylidene flavanones derivatives substituted by various groups using flavanones with aromatic aldehydes in the presence of piperidine. Flavonoid compounds belong to one of the largest and most interesting groups of chemical compounds. They are of interest to many scientists because they show biological properties (Nijveldt *et al.*, 2001; Williams *et al.*, 2004). Natural and synthetic flavonoids have a wide range of antioxidant, anti-allergic, anti-inflammatory, antimicrobial, anti-coagulant, anti-cholesterol or anti-cancer activities (Czaplińska *et al.*, 2012).





2. Structural commentary

The molecular structures of **I** and **II** are shown in Fig. 1. The main chroman skeleton of each molecule consists of a benzene ring fused with a pyran ring. In position 3 of the chroman moiety, a *para*-hydroxybenzylidene (**I**) or a *meta*-hydroxybenzylidene (**II**) substituent is connected to give the *E*-isomer, similar to the previously mentioned structure (Kupcewicz, *et al.*, 2013). Moreover in compound **II**, the chroman moiety is substituted at position 2 by a phenyl ring. The pyran rings adopt an envelope conformation with puckering parameters $Q_T = 0.371(2) \text{ \AA}$, $\varphi_2 = 233.8(4)^\circ$, $\theta_2 = 120.0(3)^\circ$ for **I**, and $Q_T = 0.423(3) \text{ \AA}$, $\varphi_2 = 65.9(5)^\circ$, $\theta_2 = 58.5(4)^\circ$ for **II**. The dihedral angles between the hydroxybenzylidene rings and the main chroman skeleton are $47.54(8)$ and $69.46(12)^\circ$, respectively, for **I** and **II** (Fig. 2).

3. Supramolecular features

In the crystal packing of **I**, molecules are connected into layers parallel to the *bc* plane *via* C—H...O and O—H...O hydrogen bonds (Table 1, Fig. 3). The stability of the layers is further enhanced by π – π stacking interactions occurring between the benzene rings fused with the pyran rings and the

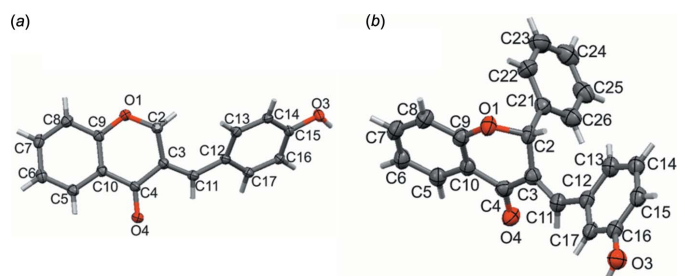


Figure 1
The molecular structures of compounds **I** and **II** with displacement ellipsoids drawn at the 50% probability level.

Table 1
Hydrogen-bond geometry (\AA , $^\circ$) for **I**.

| <i>D</i> —H... <i>A</i> | <i>D</i> —H | H... <i>A</i> | <i>D</i> ... <i>A</i> | <i>D</i> —H... <i>A</i> |
|----------------------------|-------------|---------------|-----------------------|-------------------------|
| C11—H11...O3 ⁱ | 0.95 | 2.55 | 3.264 (2) | 132 |
| C11—H11...O3 ⁱⁱ | 0.95 | 2.52 | 3.194 (2) | 129 |
| O3—H3...O4 ⁱⁱⁱ | 0.84 | 1.85 | 2.6852 (19) | 172 |
| C2—H2A...O1 ^{iv} | 0.99 | 2.53 | 3.397 (3) | 147 |
| C11—H11...O4 | 0.95 | 2.45 | 2.818 (2) | 103 |

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

aromatic rings of adjacent hydroxybenzylidene groups (Table 2). In the crystal packing of **II**, molecules are also linked by O—H...O and C—H...O hydrogen bonds (Table 3, Fig. 4) into layers parallel to the *ab* plane.

4. Database survey

A search of the Cambridge Structural Database (CSD version 5.40, last update November 2018; Groom *et al.*, 2016) using the scheme presented in Fig. 5 found 41 chromanone (Ishikawa *et al.*, 2013a,b; Zimmerman *et al.*, 2015; Marx, Suresh *et al.*, 2007; Katrusiak *et al.*, 1987; Brien *et al.*, 2012; Suresh *et al.*, 2007; Boonsri *et al.*, 2005; Biruntha *et al.*, 2018; Talhi *et al.*, 2016; Wu, Liu *et al.*, 2011; Marx *et al.*, 2008; Cheng *et al.*, 2011; Valkonen *et al.*, 2012; Lepitre *et al.*, 2017; Gopaul, Shaikh, Koorbanally *et al.*, 2012; Gopaul, Koorbanally *et al.*, 2012; Marx, Manivannan *et al.*, 2007; Suresh *et al.*, 2007; Marx *et al.*, 2008; Hassaine *et al.*, 2016; Chantrapromma *et al.*, 2006; Zhang *et al.*, 2012; Augustine *et al.*, 2008; Gopaul, Shaikh, Ramjugernath *et al.*, 2012; Gopaul & Koorbanally, 2012; Zhang *et al.*, 2013) and four flavanone structures (Zhong *et al.*, 2013; Kupcewicz *et al.*, 2013; Wu, Zeng *et al.*, 2011; Monserrat *et al.*, 2013). In the flavanone structures, the phenyl substituent at the C2 position is always nearly perpendicular to the chroman moiety, with the

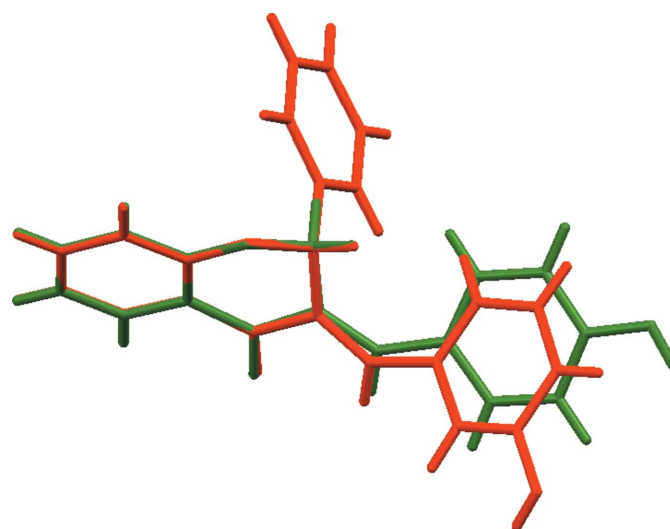


Figure 2
Overlay of compound **I** (green) and compound **II** (red).

Table 2
Geometrical parameters (Å, °) for the π - π stacking interactions for compound **I**.

$Cg(1)$ and $Cg(2)$ are the centroids of the C5–C10 and C12–C17 rings, respectively; α refers to the dihedral angle between planes (I) and (J); β refers to the angle between the $Cg(I)$ – $Cg(J)$ vector and normal to plane (I); γ refers to the angle between the $Cg(I)$ – $Cg(J)$ vector and normal to plane (J).

| | $Cg(I)\cdots Cg(J)$ | $Cg(I)_{\text{Perp}}$ | $Cg(J)_{\text{Perp}}$ | α | β | γ |
|--------------------------|---------------------|-----------------------|-----------------------|-----------|---------|----------|
| $Cg(1)\cdots Cg(1)^i$ | 3.8508 (13) | 3.5260 (9) | –3.5259 (9) | 0.03 (10) | 23.7 | 23.7 |
| $Cg(1)\cdots Cg(1)^{ii}$ | 3.8512 (13) | 3.5260 (9) | –3.5262 (9) | 0.03 (10) | 23.7 | 23.7 |
| $Cg(2)\cdots Cg(2)^i$ | 3.8510 (13) | 3.3739 (8) | –3.3738 (8) | 0.03 (10) | 28.8 | 28.8 |
| $Cg(2)\cdots Cg(2)^{ii}$ | 3.8510 (13) | 3.3740 (8) | –3.3738 (8) | 0.03 (10) | 28.8 | 28.8 |

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

Table 3
Hydrogen-bond geometry (Å, °) for **II**.

| $D-H\cdots A$ | $D-H$ | $H\cdots A$ | $D\cdots A$ | $D-H\cdots A$ |
|-----------------------|-------|-------------|-------------|---------------|
| $O3-H3\cdots O4^i$ | 0.84 | 1.89 | 2.728 (3) | 172 |
| $C17-H17\cdots O4^i$ | 0.95 | 2.49 | 3.184 (4) | 130 |
| $C6-H6\cdots O3^{ii}$ | 0.95 | 2.45 | 3.265 (4) | 143 |
| $C11-H11\cdots O4$ | 0.95 | 2.43 | 2.807 (3) | 103 |

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

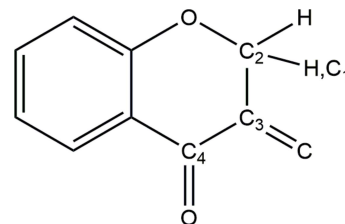


Figure 5
Reference moiety for database survey.

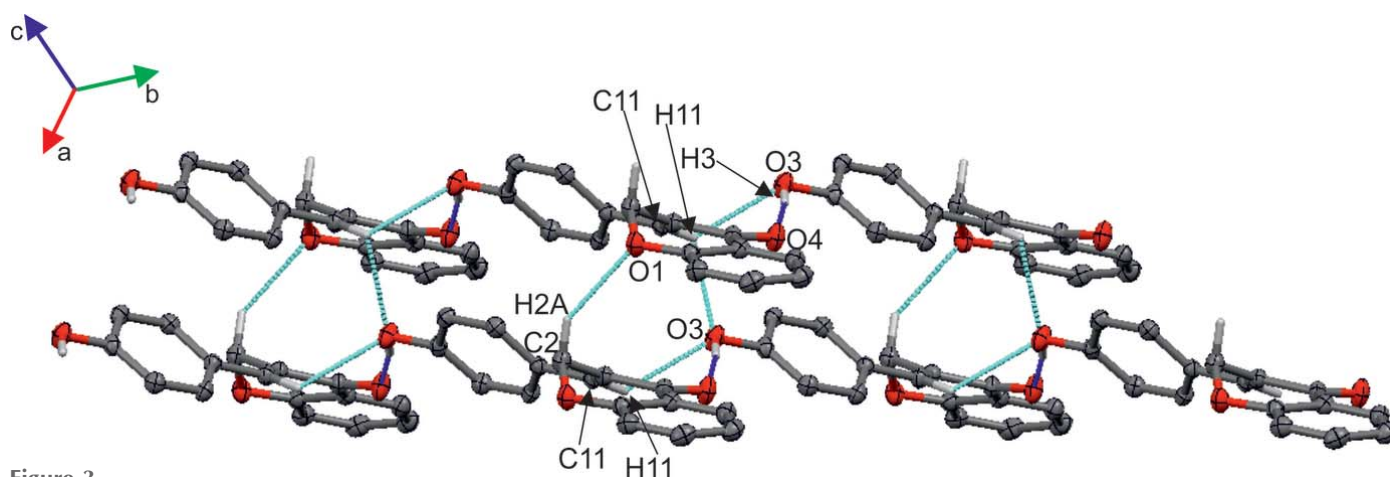


Figure 3
Partial packing of compound **I** showing the O–H \cdots O (blue dotted lines) and C–H \cdots O (cyan dotted lines) hydrogen-bonding network.

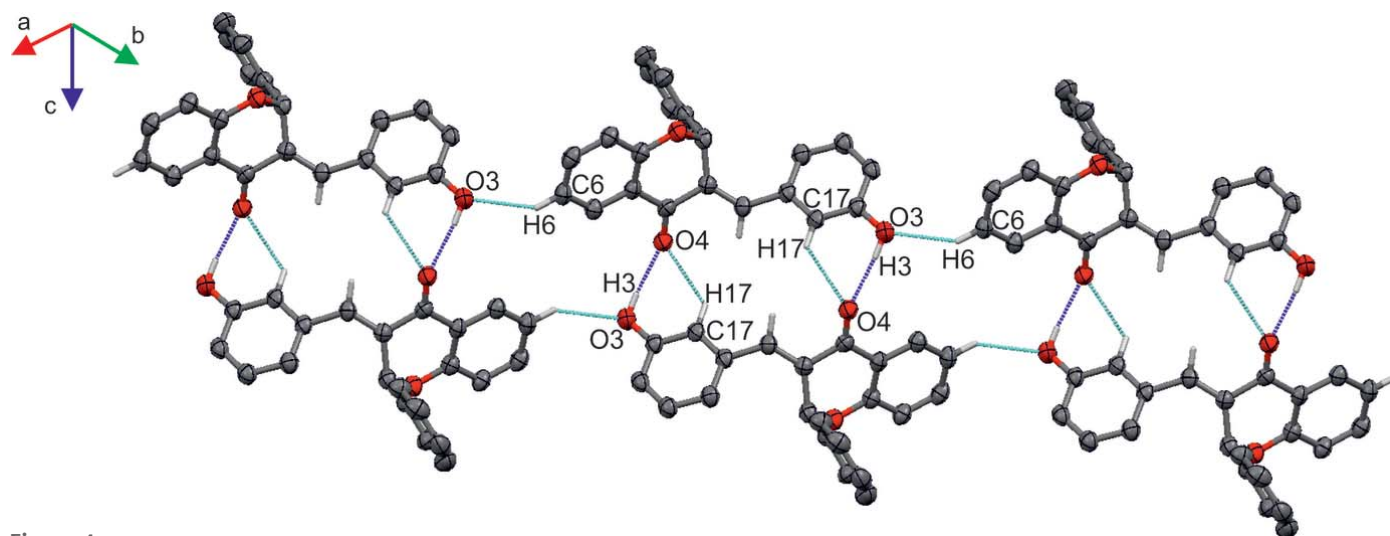


Figure 4
Partial packing of compound **II** showing the O–H \cdots O (blue dotted lines) and C–H \cdots O (cyan dotted lines) hydrogen-bonding network.

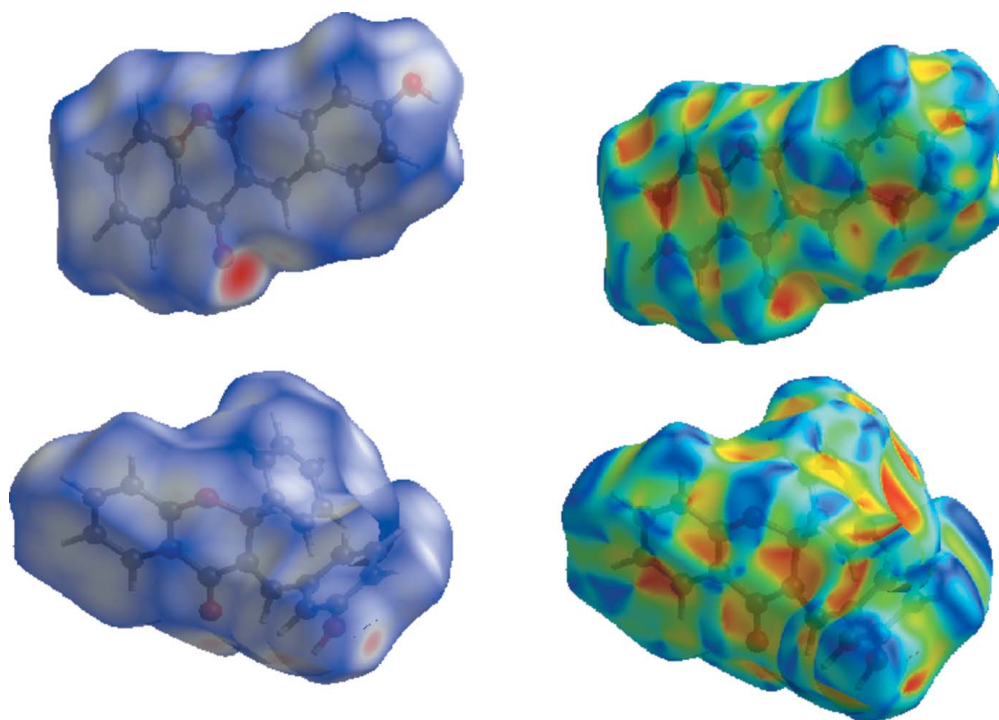


Figure 6
View of the three-dimensional Hirshfeld surfaces of the title compounds plotted over d_{norm} (left) and shape-index (right); first row: compound **I**, second row: compound **II**.

C(phen)–C2–C3–C4 torsion angle in the range 82.44–107.90°. In both chromanone and flavanone structures, the pyran ring adopts a slightly distorted envelope conformation. In the 41 chromanone derivatives, the bond distances and angles within the chroman moiety are in good agreement with those found in compound **I**.

5. Experimental and theoretical lipophilicity of compounds **I** and **II**

Lipophilicity is one of the descriptors that is currently used in the design of new drugs and in assessing the activity of medicinal substances (Jóźwiak *et al.*, 2001). Most often, the increase in lipophilicity increases the biological activity of compounds as a result of the affinity of substances with biological membranes and better permeability (Dołowy, 2009). However, a further increase in lipophilicity results in greater affinity for lipids and hinders the transport of compound molecules through the aqueous phase. That is why it is important to choose substances with optimal hydrophobic and hydrophilic properties and partition coefficient $\log P$ (Dołowy, 2009).

The experimental lipophilicity ($\log P$) of compounds **I** and **II** was determined using the RP–TLC method. The values of $\log P$ obtained are 2.95 and 3.98, respectively for **I** and **II**, the difference being due to the different, bulky substituent at the C2 position of the pyran ring. The theoretical values of lipophilicity (miLog P) also show the same trend, the value for compound **I** is lower (miLog P = 3.14) than that for compound **II** (miLog P = 4.70). This is in agreement with the values

previously reported for similar arylidenochromanone/flavanone derivatives (Adamus-Grabicka *et al.*, 2018). The theoretical values of lipophilicity were calculated using the online *Molinspiration Cheminformatics* software (<http://www.molinspiration.com>). According to the ‘rule of five’ proposed by Lipinski *et al.* (2001), compounds **I** and **II** may be potential anti-cancer drugs, the most important parameters according to Lipinski being the $\log P$ value ($\log P < 5$) and molar mass (< 500 Da).

6. Hirshfeld surface analysis and lipophilicity index versus C...H contact

As the Hirshfeld surface (HS) analysis may provide useful descriptors for QSAR study (Kupcewicz, *et al.*, 2016) and the lipophilicity parameter in biologically active compounds is associated with the contribution of intermolecular interactions to the Hirshfeld surface (Małeczka & Budzisz, 2014), we generated the Hirshfeld surfaces (Hirshfeld, 1977; Spackman & Jayatilaka, 2009) using the *CrystalExplorer* program (Turner *et al.*, 2017) for chromone and flavanone derivatives for which the lipophilicity parameters are available, *i.e.* compound **I**, **II**, 3-(4-chlorobenzylidene)-2-phenyl-2,3-dihydro-4*H*-chromen-4-one (**III**; Kupcewicz *et al.*, 2013), (*E*)-3-(4-*N,N*-diethylaminobenzylidene)chroman-4-one (**IV**; Adamus-Grabicka *et al.*, 2018) and (*E*)-3-(4-*N,N*-diethylaminobenzylidene)-2-phenylchroman-4-one (**V**; Adamus-Grabicka *et al.*, 2018).

The Hirshfeld surfaces were mapped over d_{norm} (Fig. 6). The red, white and blue regions visible on the d_{norm} surfaces

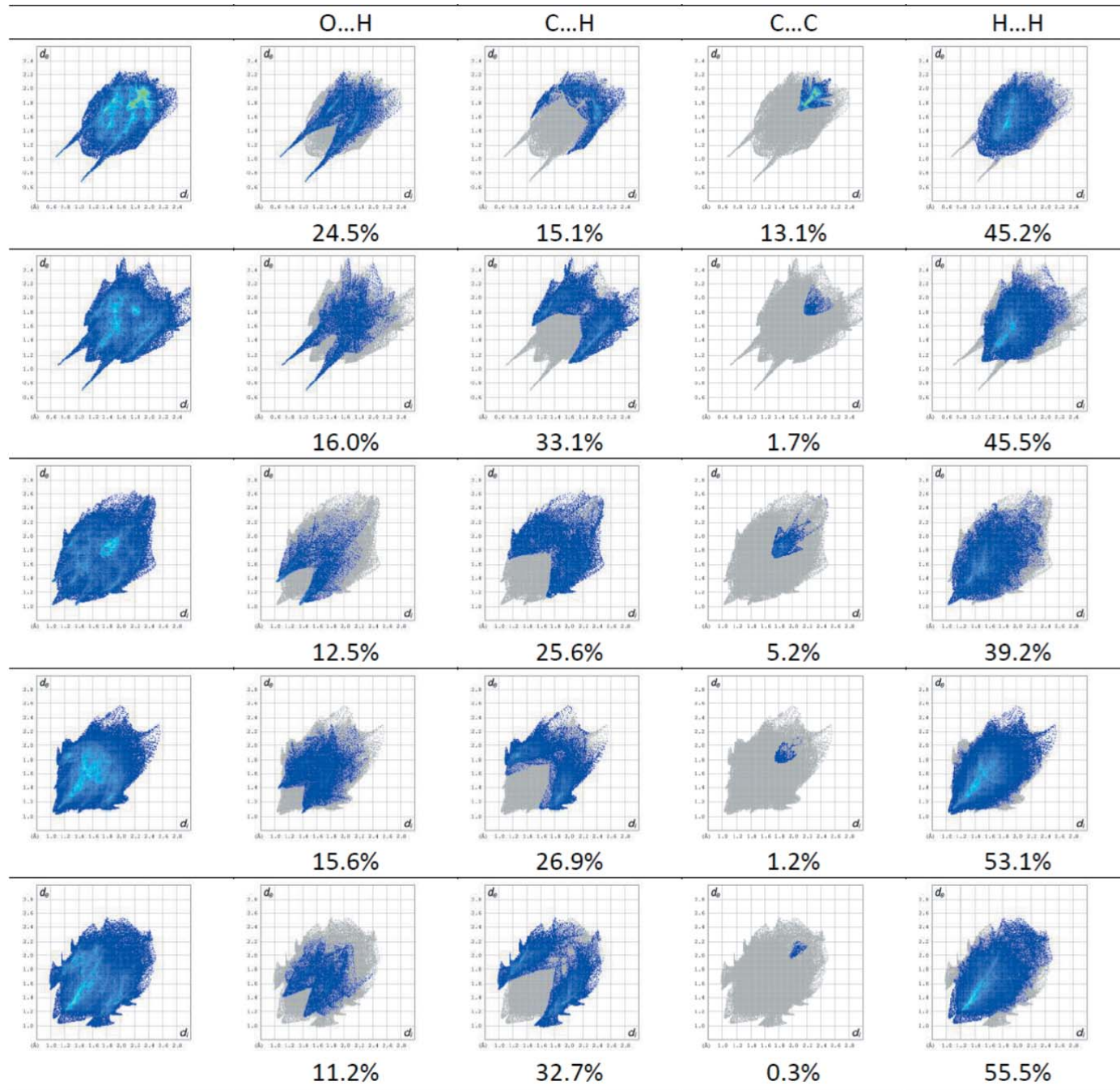


Figure 7

Fingerprint plots of the title compounds; full Hirshfeld surface (left) and delineated into H...O, H...C, C...C, and H...H contacts, showing the percentage contributions of the contacts to the total Hirshfeld surface area of the molecules. First row: compound **I**; second row: compound **II**; third row: 3-(4-chlorobenzylidene)-2-phenyl-2,3-dihydro-4*H*-chromen-4-one (Kupcewicz *et al.*, 2013); fourth row: (*E*)-3-(4-*N,N*-diethylaminobenzylidene)chroman-4-one (Adamus-Grabicka *et al.*, 2018); fifth row: (*E*)-3-(4-*N,N*-diethylaminobenzylidene)-2-phenylchroman-4-one (Adamus-Grabicka *et al.*, 2018).

indicate contacts with distances shorter, longer and equal to the van der Waals radii. The decomposition of the HS into 2D fingerprint plots for particular contacts is presented in Fig. 7, together with the relative percentage of contributions of different contacts. The dominant interaction in all derivatives is the H...H interaction. The contribution to the Hirshfeld surface is in the range 39.2–55.5% for **III** and **V**. Comparing

the C...C contacts, we can observe a large spread of percentage contribution ranging from 0.3% for **V** to 13.1% for compound **I**. This is also reflected in the presence of π - π stacking interactions observed in compound **I** (Table 2).

As in our previous studies (Małeczka *et al.*, 2014; Kupcewicz *et al.*, 2013), we found a relationship between the log*P* value and the fraction of the Hirshfeld surface covered by different

Table 4
Experimental details.

| | I | II |
|---|--|--|
| Crystal data | | |
| Chemical formula | C ₁₆ H ₁₂ O ₃ | C ₂₂ H ₁₆ O ₃ |
| <i>M_r</i> | 252.27 | 328.37 |
| Crystal system, space group | Monoclinic, <i>P</i> 2 ₁ / <i>c</i> | Triclinic, <i>P</i> $\bar{1}$ |
| Temperature (K) | 120 | 120 |
| <i>a</i> , <i>b</i> , <i>c</i> (Å) | 3.8510 (2), 22.2541 (11), 13.7837 (9) | 5.3969 (6), 11.6576 (16), 12.944 (2) |
| α , β , γ (°) | 90, 96.766 (5), 90 | 91.992 (12), 98.282 (10), 97.568 (10) |
| <i>V</i> (Å ³) | 1173.04 (11) | 797.68 (19) |
| <i>Z</i> | 4 | 2 |
| Radiation type | Mo <i>K</i> α | Mo <i>K</i> α |
| μ (mm ⁻¹) | 0.10 | 0.09 |
| Crystal size (mm) | 0.4 × 0.2 × 0.1 | 0.8 × 0.2 × 0.05 |
| Data collection | | |
| Diffractometer | STOE IPDS 2T | STOE IPDS 2T |
| Absorption correction | — | — |
| No. of measured, independent and observed [<i>I</i> > 2 σ (<i>I</i>)] reflections | 7027, 2413, 1618 | 6849, 3281, 1804 |
| <i>R</i> _{int} | 0.050 | 0.077 |
| (<i>sin</i> θ / λ) _{max} (Å ⁻¹) | 0.628 | 0.628 |
| Refinement | | |
| <i>R</i> [<i>F</i> ² > 2 σ (<i>F</i> ²)], <i>wR</i> (<i>F</i> ²), <i>S</i> | 0.048, 0.115, 1.03 | 0.068, 0.200, 0.94 |
| No. of reflections | 2413 | 3281 |
| No. of parameters | 173 | 228 |
| H-atom treatment | H-atom parameters constrained | H-atom parameters constrained |
| $\Delta\rho_{\max}$, $\Delta\rho_{\min}$ (e Å ⁻³) | 0.19, -0.19 | 0.23, -0.29 |

Computer programs: *X-AREA* and *X-RED32* (Stoe & Cie, 2002), *SHELXT* (Sheldrick, 2015a), *SHELXL2014/7* (Sheldrick, 2015b) and *pubCIF* (Westrip, 2010).

intermolecular interactions. The increase of log*P* corresponds in fact to increasing the C··H contribution in the Hirshfeld surface. Furthermore, for compounds **I–V**, the contribution of the O··H interaction in the Hirshfeld surface is inversely proportional to the value of log*P*.

7. Synthesis and crystallization

The synthesis of compounds **I** and **II** is based on the condensation of chromanone or flavanone with an aryl aldehyde in the presence of piperidine (Fig. 8). Compound **I** was prepared according to a slightly modified procedure with respect to that described in the literature (Levai & Schag, 1979). A mechanically stirred mixture of chroman-4-one (0.01 mol), *p*-methoxybenzaldehyde (0.01 mol) and five drops of piperidine was heated at 413 K in an oil bath for four h. The progress of the synthesis was controlled by thin layer chromatography (TLC) using toluene/methanol (9:1 *v/v*) as eluent. After cooling the reaction mixture was left for 24 h at room temperature. The solidified product was filtered and crystal-

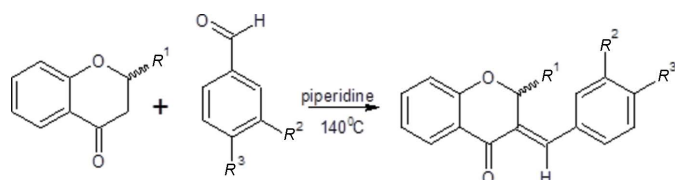


Figure 8
Scheme of the synthesis of compounds **I** and **II**. *R*¹ = H/Ph, *R*² = H/OH, *R*³ = OH/H, respectively for compound **I** and **II**.

lized from methanol. Compound **I** was obtained as a yellow powder. The isolated solid was further recrystallized by slow evaporation at room temperature of an acetone solution. Yield: 64%, M.p.: 501–502.5 K. MS (ESI⁺): *m/z* 253.3 C₁₆H₁₂O₃ [*M*+H]⁺. IR (KBr): 3126 (O–H), 2809 (C–H_{aromat}), 1652 (C=O), 1608, 1578 (C=C), 1164 (C–O–C), 751 (C–H). ¹H NMR (600 MHz, DMSO-*d*₆) δ (ppm): 5.42 (1H, *s*, =CH), 6.86–7.86 (8H, *m*, C–H_{aromat}), 7.87 (2H, *d*, *J*_{AB} = 18 Hz C2–H), 10.12 (1H, *s*, OH). Analysis calculated for C₁₆H₁₂O₃ (*M* = 252.23 g mol⁻¹) % C: 76.18; % H: 4.81; % O: 19.01. Found % C: 75.3; % H: 5.01; % O: 19.69.

Compound **II** was synthesized according to the procedure described by Pijewska *et al.*, (1993). A mixture of 2-phenylchroman-4-one (0.01 mol), 3-hydroxybenzaldehyde (0.01 mol) and five drops of piperidine was heated under reflux in an oil bath with mechanical stirring. The reaction proceeded at 413 K for 5 h. The progress of the reaction was controlled by TLC (eluent: toluene/methanol, 9:1 *v/v*). After cooling at room temperature, the mixture was dissolved in methanol. After 24 h compound **II** precipitated as a light-cream fine crystalline powder and was purified by crystallization from methanol. Crystal suitable for X-ray analysis were obtained by slow evaporation of an ethanol solution at room temperature. Yield: 52.4%. M.p.: 482–483 K. MS (ESI⁺): *m/z* 329.2 C₂₂H₁₆O₃ [*M*+H]⁺. IR (KBr): 3297 (O–H), 3054 (C–H_{aromat}), 2351 (C–H_{aliph}), 1663 (C=O), 1608, 1590, 1504 (C=C), 1141 (C–O–C), 757 (C–H). ¹H NMR (600 MHz, DMSO-*d*₆) δ (ppm): 6.57 (1H, *s*, C2–H), 5.69 (1H, *s*, =CH), 6.89–7.91 (14H, *m*, CH_{aromat}), 8.12 (1H, *s*, OH). Analysis calculated For C₂₂H₁₆O₃ (*M* = 328.19 g mol⁻¹) %C: 80.51;

%H: 4.87; % O: 14.62. Found %C: 79.99; %H: 5.11; % O: 14.90.

8. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 4. All hydrogen atoms were fixed geometrically at calculated positions (O–H = 0.84 Å, C–H = 0.95–0.99 Å) and refined as riding model with $U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{O})$ or $1.2U_{\text{eq}}(\text{C})$. A rotating model was used for the hydroxy groups.

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supporting information

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Crystal structures of (*E*)-3-(4-hydroxybenzylidene)chroman-4-one and (*E*)-3-(3-hydroxybenzylidene)-2-phenylchroman-4-one

Kamil Suchojad, Anna Dołęga, Angelika Adamus-Grabicka, Elżbieta Budzisz and Magdalena Małecka

Computing details

For both structures, data collection: *X-AREA* (Stoe & Cie, 2002); cell refinement: *X-AREA* (Stoe & Cie, 2002); data reduction: *X-AREA* (Stoe & Cie, 2002), *X-RED32* (Stoe & Cie, 2002); program(s) used to solve structure: SHELXT (Sheldrick, 2015a); program(s) used to refine structure: *SHELXL2014/7* (Sheldrick, 2015b); software used to prepare material for publication: *SHELXL2014/7* (Sheldrick, 2015b), *pubCIF* (Westrip, 2010).

(*E*)-3-(4-Hydroxybenzylidene)chroman-4-one (I)

Crystal data

$C_{16}H_{12}O_3$

$M_r = 252.27$

Monoclinic, $P2_1/c$

$a = 3.8510$ (2) Å

$b = 22.2541$ (11) Å

$c = 13.7837$ (9) Å

$\beta = 96.766$ (5)°

$V = 1173.04$ (11) Å³

$Z = 4$

$F(000) = 528$

$D_x = 1.428$ Mg m⁻³

Melting point: 220 K

Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å

Cell parameters from 219 reflections

$\theta = 4.1$ – 28.9 °

$\mu = 0.10$ mm⁻¹

$T = 120$ K

Needle, light-yellow

$0.4 \times 0.2 \times 0.1$ mm

Data collection

STOE IPDS 2T

diffractometer

Radiation source: GeniX Mo, 0.05 x 0.05 mm²

microfocus

Detector resolution: 6.67 pixels mm⁻¹

rotation method, ω scans

7027 measured reflections

2413 independent reflections

1618 reflections with $I > 2\sigma(I)$

$R_{int} = 0.050$

$\theta_{max} = 26.5$ °, $\theta_{min} = 3.7$ °

$h = -4 \rightarrow 4$

$k = -27 \rightarrow 27$

$l = -16 \rightarrow 17$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.048$

$wR(F^2) = 0.115$

$S = 1.02$

2413 reflections

173 parameters

0 restraints

Primary atom site location: difference Fourier map

Secondary atom site location: difference Fourier map

Hydrogen site location: inferred from neighbouring sites

H-atom parameters constrained

$$w = 1/[\sigma^2(F_o^2) + (0.0546P)^2 + 0.2287P]$$

where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} < 0.001$

$$\Delta\rho_{\max} = 0.19 \text{ e } \text{\AA}^{-3}$$

$$\Delta\rho_{\min} = -0.19 \text{ e } \text{\AA}^{-3}$$

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 (\AA^2)

| | <i>x</i> | <i>y</i> | <i>z</i> | $U_{\text{iso}}^*/U_{\text{eq}}$ |
|-----|-------------|-------------|--------------|----------------------------------|
| O3 | 0.5127 (4) | 0.21588 (6) | 0.68339 (10) | 0.0282 (4) |
| H3 | 0.5952 | 0.1823 | 0.6701 | 0.042* |
| O4 | -0.1988 (4) | 0.39218 (6) | 0.15977 (10) | 0.0304 (4) |
| O1 | 0.2599 (4) | 0.50810 (6) | 0.36926 (10) | 0.0252 (4) |
| C9 | 0.1347 (5) | 0.52849 (8) | 0.27851 (14) | 0.0217 (4) |
| C17 | 0.3626 (5) | 0.25884 (8) | 0.42639 (15) | 0.0218 (4) |
| H17 | 0.4012 | 0.2441 | 0.3639 | 0.026* |
| C3 | 0.0393 (5) | 0.40641 (8) | 0.32620 (14) | 0.0207 (4) |
| C14 | 0.2335 (5) | 0.29994 (8) | 0.60754 (15) | 0.0216 (4) |
| H14 | 0.1846 | 0.3135 | 0.6698 | 0.026* |
| C11 | 0.0949 (5) | 0.34735 (8) | 0.34238 (15) | 0.0219 (4) |
| H11 | 0.0614 | 0.3229 | 0.2856 | 0.026* |
| C10 | -0.0381 (5) | 0.49118 (8) | 0.20685 (15) | 0.0217 (4) |
| C12 | 0.1982 (5) | 0.31486 (8) | 0.43307 (14) | 0.0200 (4) |
| C13 | 0.1298 (5) | 0.33398 (8) | 0.52539 (15) | 0.0219 (4) |
| H13 | 0.0104 | 0.3709 | 0.5318 | 0.026* |
| C8 | 0.2011 (6) | 0.58826 (9) | 0.25768 (15) | 0.0256 (5) |
| H8 | 0.3229 | 0.6133 | 0.3061 | 0.031* |
| C15 | 0.4089 (5) | 0.24590 (8) | 0.59954 (15) | 0.0208 (4) |
| C7 | 0.0889 (6) | 0.61086 (9) | 0.16621 (16) | 0.0277 (5) |
| H7 | 0.1330 | 0.6518 | 0.1523 | 0.033* |
| C4 | -0.0782 (5) | 0.42671 (9) | 0.22612 (14) | 0.0216 (4) |
| C2 | 0.0920 (6) | 0.45481 (8) | 0.40230 (15) | 0.0227 (4) |
| H2A | -0.1381 | 0.4662 | 0.4220 | 0.027* |
| H2B | 0.2363 | 0.4387 | 0.4607 | 0.027* |
| C6 | -0.0882 (6) | 0.57486 (9) | 0.09380 (16) | 0.0283 (5) |
| H6 | -0.1649 | 0.5909 | 0.0311 | 0.034* |
| C5 | -0.1499 (6) | 0.51545 (9) | 0.11493 (15) | 0.0249 (5) |
| H5 | -0.2708 | 0.4906 | 0.0661 | 0.030* |
| C16 | 0.4693 (5) | 0.22483 (8) | 0.50797 (15) | 0.0217 (4) |
| H16 | 0.5830 | 0.1874 | 0.5018 | 0.026* |

Atomic displacement parameters (\AA^2)

| | U^{11} | U^{22} | U^{33} | U^{12} | U^{13} | U^{23} |
|----|------------|------------|------------|------------|------------|------------|
| O3 | 0.0407 (9) | 0.0236 (7) | 0.0200 (8) | 0.0065 (7) | 0.0022 (7) | 0.0031 (6) |

| | | | | | | |
|-----|-------------|-------------|-------------|-------------|-------------|-------------|
| O4 | 0.0459 (10) | 0.0235 (7) | 0.0198 (8) | -0.0049 (7) | -0.0044 (7) | -0.0004 (6) |
| O1 | 0.0305 (8) | 0.0209 (7) | 0.0231 (8) | -0.0046 (6) | -0.0020 (6) | 0.0006 (6) |
| C9 | 0.0221 (11) | 0.0231 (10) | 0.0200 (11) | 0.0031 (8) | 0.0023 (8) | 0.0014 (8) |
| C17 | 0.0266 (11) | 0.0229 (10) | 0.0160 (10) | -0.0013 (8) | 0.0025 (8) | -0.0018 (8) |
| C3 | 0.0204 (10) | 0.0223 (9) | 0.0190 (11) | -0.0003 (8) | 0.0015 (8) | 0.0008 (8) |
| C14 | 0.0257 (11) | 0.0199 (9) | 0.0192 (11) | -0.0036 (8) | 0.0030 (8) | -0.0029 (7) |
| C11 | 0.0229 (11) | 0.0242 (10) | 0.0179 (11) | -0.0002 (8) | -0.0003 (8) | -0.0023 (8) |
| C10 | 0.0240 (11) | 0.0200 (9) | 0.0212 (11) | 0.0025 (8) | 0.0036 (9) | 0.0006 (8) |
| C12 | 0.0205 (10) | 0.0215 (9) | 0.0178 (10) | -0.0019 (8) | 0.0009 (8) | -0.0008 (8) |
| C13 | 0.0218 (10) | 0.0192 (10) | 0.0245 (11) | -0.0005 (8) | 0.0015 (8) | -0.0001 (8) |
| C8 | 0.0271 (12) | 0.0228 (10) | 0.0276 (12) | -0.0012 (8) | 0.0052 (9) | -0.0029 (8) |
| C15 | 0.0228 (10) | 0.0198 (9) | 0.0190 (10) | -0.0023 (8) | -0.0010 (8) | 0.0035 (8) |
| C7 | 0.0345 (13) | 0.0200 (10) | 0.0303 (13) | 0.0021 (9) | 0.0111 (10) | 0.0028 (8) |
| C4 | 0.0235 (11) | 0.0234 (10) | 0.0169 (11) | 0.0013 (8) | -0.0014 (8) | -0.0001 (8) |
| C2 | 0.0280 (11) | 0.0203 (9) | 0.0193 (11) | -0.0016 (8) | 0.0000 (9) | 0.0005 (8) |
| C6 | 0.0326 (12) | 0.0275 (11) | 0.0256 (12) | 0.0066 (9) | 0.0070 (9) | 0.0055 (9) |
| C5 | 0.0285 (12) | 0.0250 (10) | 0.0211 (11) | 0.0024 (8) | 0.0024 (9) | -0.0008 (8) |
| C16 | 0.0249 (11) | 0.0169 (9) | 0.0231 (11) | 0.0006 (8) | 0.0024 (9) | -0.0005 (8) |

Geometric parameters (Å, °)

| | | | |
|-------------|-------------|-------------|-------------|
| O3—C15 | 1.354 (2) | C11—H11 | 0.9500 |
| O3—H3 | 0.8400 | C10—C5 | 1.398 (3) |
| O4—C4 | 1.242 (2) | C10—C4 | 1.471 (3) |
| O1—C9 | 1.364 (2) | C12—C13 | 1.396 (3) |
| O1—C2 | 1.450 (2) | C13—H13 | 0.9500 |
| C9—C8 | 1.391 (3) | C8—C7 | 1.379 (3) |
| C9—C10 | 1.397 (3) | C8—H8 | 0.9500 |
| C17—C16 | 1.378 (3) | C15—C16 | 1.392 (3) |
| C17—C12 | 1.406 (3) | C7—C6 | 1.394 (3) |
| C17—H17 | 0.9500 | C7—H7 | 0.9500 |
| C3—C11 | 1.346 (3) | C2—H2A | 0.9900 |
| C3—C4 | 1.471 (3) | C2—H2B | 0.9900 |
| C3—C2 | 1.501 (3) | C6—C5 | 1.381 (3) |
| C14—C13 | 1.382 (3) | C6—H6 | 0.9500 |
| C14—C15 | 1.390 (3) | C5—H5 | 0.9500 |
| C14—H14 | 0.9500 | C16—H16 | 0.9500 |
| C11—C12 | 1.458 (3) | | |
| C15—O3—H3 | 109.5 | C7—C8—H8 | 120.3 |
| C9—O1—C2 | 115.95 (15) | C9—C8—H8 | 120.3 |
| O1—C9—C8 | 116.97 (18) | O3—C15—C14 | 117.16 (18) |
| O1—C9—C10 | 122.55 (17) | O3—C15—C16 | 122.95 (18) |
| C8—C9—C10 | 120.40 (18) | C14—C15—C16 | 119.89 (18) |
| C16—C17—C12 | 121.80 (19) | C8—C7—C6 | 121.30 (19) |
| C16—C17—H17 | 119.1 | C8—C7—H7 | 119.4 |
| C12—C17—H17 | 119.1 | C6—C7—H7 | 119.4 |
| C11—C3—C4 | 118.72 (18) | O4—C4—C10 | 120.57 (18) |

| | | | |
|-------------|-------------|-------------|-------------|
| C11—C3—C2 | 125.41 (18) | O4—C4—C3 | 123.19 (18) |
| C4—C3—C2 | 115.86 (16) | C10—C4—C3 | 116.22 (17) |
| C13—C14—C15 | 120.37 (19) | O1—C2—C3 | 113.35 (16) |
| C13—C14—H14 | 119.8 | O1—C2—H2A | 108.9 |
| C15—C14—H14 | 119.8 | C3—C2—H2A | 108.9 |
| C3—C11—C12 | 130.40 (19) | O1—C2—H2B | 108.9 |
| C3—C11—H11 | 114.8 | C3—C2—H2B | 108.9 |
| C12—C11—H11 | 114.8 | H2A—C2—H2B | 107.7 |
| C5—C10—C9 | 118.78 (18) | C5—C6—C7 | 118.8 (2) |
| C5—C10—C4 | 120.86 (18) | C5—C6—H6 | 120.6 |
| C9—C10—C4 | 120.22 (18) | C7—C6—H6 | 120.6 |
| C13—C12—C17 | 117.70 (18) | C6—C5—C10 | 121.3 (2) |
| C13—C12—C11 | 124.65 (18) | C6—C5—H5 | 119.4 |
| C17—C12—C11 | 117.57 (17) | C10—C5—H5 | 119.4 |
| C14—C13—C12 | 120.83 (18) | C17—C16—C15 | 119.31 (18) |
| C14—C13—H13 | 119.6 | C17—C16—H16 | 120.3 |
| C12—C13—H13 | 119.6 | C15—C16—H16 | 120.3 |
| C7—C8—C9 | 119.48 (19) | | |

Hydrogen-bond geometry (Å, °)

| <i>D</i> —H... <i>A</i> | <i>D</i> —H | H... <i>A</i> | <i>D</i> ... <i>A</i> | <i>D</i> —H... <i>A</i> |
|----------------------------|-------------|---------------|-----------------------|-------------------------|
| C11—H11...O3 ⁱ | 0.95 | 2.55 | 3.264 (2) | 132 |
| C11—H11...O3 ⁱⁱ | 0.95 | 2.52 | 3.194 (2) | 129 |
| O3—H3...O4 ⁱⁱⁱ | 0.84 | 1.85 | 2.6852 (19) | 172 |
| C2—H2A...O1 ^{iv} | 0.99 | 2.53 | 3.397 (3) | 147 |
| C11—H11...O4 | 0.95 | 2.45 | 2.818 (2) | 103 |

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

(*E*)-3-(3-Hydroxybenzylidene)-2-phenylchroman-4-one (II)*Crystal data*

C₂₂H₁₆O₃
M_r = 328.37
 Triclinic, *P* $\bar{1}$
a = 5.3969 (6) Å
b = 11.6576 (16) Å
c = 12.944 (2) Å
 α = 91.992 (12)°
 β = 98.282 (10)°
 γ = 97.568 (10)°
V = 797.68 (19) Å³
Z = 2

F(000) = 344
D_x = 1.367 Mg m⁻³
 Melting point: 210 K
 Mo *K* α radiation, λ = 0.71073 Å
 Cell parameters from 3650 reflections
 θ = 3.5–29.5°
 μ = 0.09 mm⁻¹
T = 120 K
 Plate, colourless
 0.8 × 0.2 × 0.05 mm

Data collection

STOE IPDS 2T
 diffractometer
 Radiation source: GeniX Mo, 0.05 × 0.05 mm²
 microfocus
 Detector resolution: 6.67 pixels mm⁻¹

rotation method, ω scans
 6849 measured reflections
 3281 independent reflections
 1804 reflections with $I > 2\sigma(I)$
R_{int} = 0.077

$\theta_{\max} = 26.5^\circ$, $\theta_{\min} = 3.5^\circ$
 $h = -6 \rightarrow 6$

$k = -13 \rightarrow 14$
 $l = -16 \rightarrow 16$

Refinement

Refinement on F^2
 Least-squares matrix: full
 $R[F^2 > 2\sigma(F^2)] = 0.068$
 $wR(F^2) = 0.200$
 $S = 0.94$
 3281 reflections
 228 parameters
 0 restraints
 Primary atom site location: difference Fourier
 map

Secondary atom site location: difference Fourier
 map
 Hydrogen site location: inferred from
 neighbouring sites
 H-atom parameters constrained
 $w = 1/[\sigma^2(F_o^2) + (0.1159P)^2]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} < 0.001$
 $\Delta\rho_{\max} = 0.23 \text{ e } \text{\AA}^{-3}$
 $\Delta\rho_{\min} = -0.29 \text{ e } \text{\AA}^{-3}$

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 (\AA^2)

| | <i>x</i> | <i>y</i> | <i>z</i> | $U_{\text{iso}}^*/U_{\text{eq}}$ |
|-----|-------------|--------------|--------------|----------------------------------|
| O1 | 0.1107 (4) | 0.13471 (17) | 0.24184 (16) | 0.0423 (6) |
| O4 | 0.7889 (4) | 0.30522 (17) | 0.39197 (16) | 0.0432 (6) |
| O3 | -0.1593 (4) | 0.6939 (2) | 0.44060 (17) | 0.0503 (6) |
| H3 | -0.0545 | 0.6964 | 0.4956 | 0.075* |
| C10 | 0.5289 (5) | 0.1266 (2) | 0.3391 (2) | 0.0365 (7) |
| C17 | 0.1145 (5) | 0.5660 (2) | 0.3820 (2) | 0.0390 (7) |
| H17 | 0.1940 | 0.5611 | 0.4518 | 0.047* |
| C9 | 0.2888 (5) | 0.0736 (3) | 0.2913 (2) | 0.0386 (7) |
| C3 | 0.3849 (5) | 0.3163 (3) | 0.2945 (2) | 0.0378 (7) |
| C7 | 0.3920 (6) | -0.1113 (3) | 0.3446 (2) | 0.0453 (8) |
| H7 | 0.3455 | -0.1925 | 0.3474 | 0.054* |
| C5 | 0.6977 (6) | 0.0575 (3) | 0.3885 (2) | 0.0405 (7) |
| H5 | 0.8608 | 0.0925 | 0.4207 | 0.049* |
| C4 | 0.5884 (5) | 0.2528 (3) | 0.3452 (2) | 0.0370 (7) |
| C15 | -0.1803 (6) | 0.6487 (3) | 0.2598 (2) | 0.0431 (8) |
| H15 | -0.3077 | 0.6973 | 0.2454 | 0.052* |
| C16 | -0.0743 (5) | 0.6358 (3) | 0.3619 (2) | 0.0401 (7) |
| C11 | 0.3763 (5) | 0.4240 (3) | 0.3311 (2) | 0.0385 (7) |
| H11 | 0.5114 | 0.4544 | 0.3843 | 0.046* |
| C12 | 0.1891 (5) | 0.5030 (2) | 0.3014 (2) | 0.0378 (7) |
| C8 | 0.2210 (6) | -0.0459 (3) | 0.2943 (2) | 0.0431 (7) |
| H8 | 0.0587 | -0.0818 | 0.2620 | 0.052* |
| C13 | 0.0823 (6) | 0.5165 (3) | 0.1989 (3) | 0.0442 (8) |
| H13 | 0.1332 | 0.4755 | 0.1427 | 0.053* |
| C14 | -0.0998 (6) | 0.5905 (3) | 0.1791 (2) | 0.0440 (8) |
| H14 | -0.1694 | 0.6010 | 0.1090 | 0.053* |



| | | | | |
|-----|------------|-------------|-------------|-------------|
| C2 | 0.2058 (5) | 0.2476 (2) | 0.2083 (2) | 0.0376 (7) |
| H2 | 0.0587 | 0.2908 | 0.1899 | 0.045* |
| C21 | 0.3266 (5) | 0.2321 (3) | 0.1100 (2) | 0.0387 (7) |
| C22 | 0.2451 (7) | 0.1373 (3) | 0.0408 (2) | 0.0517 (9) |
| H22 | 0.1084 | 0.0819 | 0.0531 | 0.062* |
| C6 | 0.6322 (6) | -0.0602 (3) | 0.3915 (2) | 0.0440 (8) |
| H6 | 0.7491 | -0.1062 | 0.4251 | 0.053* |
| C26 | 0.5228 (6) | 0.3134 (3) | 0.0894 (2) | 0.0493 (8) |
| H26 | 0.5783 | 0.3797 | 0.1357 | 0.059* |
| C23 | 0.3612 (8) | 0.1222 (3) | -0.0467 (3) | 0.0632 (11) |
| H23 | 0.3042 | 0.0566 | -0.0938 | 0.076* |
| C24 | 0.5596 (7) | 0.2024 (4) | -0.0654 (3) | 0.0593 (10) |
| H24 | 0.6417 | 0.1913 | -0.1244 | 0.071* |
| C25 | 0.6382 (6) | 0.2991 (3) | 0.0022 (3) | 0.0558 (9) |
| H25 | 0.7716 | 0.3556 | -0.0114 | 0.067* |

Atomic displacement parameters (Å²)

| | U^{11} | U^{22} | U^{33} | U^{12} | U^{13} | U^{23} |
|-----|-------------|-------------|-------------|-------------|-------------|--------------|
| O1 | 0.0412 (11) | 0.0325 (12) | 0.0520 (13) | 0.0005 (9) | 0.0064 (10) | 0.0075 (10) |
| O4 | 0.0394 (11) | 0.0355 (12) | 0.0525 (13) | 0.0035 (10) | 0.0017 (10) | -0.0001 (10) |
| O3 | 0.0553 (13) | 0.0463 (14) | 0.0510 (13) | 0.0193 (11) | 0.0022 (10) | 0.0025 (11) |
| C10 | 0.0432 (16) | 0.0317 (17) | 0.0372 (15) | 0.0082 (13) | 0.0115 (13) | 0.0044 (12) |
| C17 | 0.0413 (16) | 0.0267 (16) | 0.0469 (17) | 0.0024 (13) | 0.0014 (13) | 0.0043 (13) |
| C9 | 0.0412 (16) | 0.0344 (17) | 0.0416 (16) | 0.0037 (13) | 0.0122 (13) | 0.0036 (13) |
| C3 | 0.0397 (15) | 0.0375 (17) | 0.0372 (16) | 0.0058 (13) | 0.0083 (13) | 0.0043 (13) |
| C7 | 0.0550 (19) | 0.0309 (17) | 0.0522 (19) | 0.0020 (15) | 0.0195 (15) | 0.0020 (14) |
| C5 | 0.0438 (16) | 0.0377 (18) | 0.0419 (16) | 0.0075 (14) | 0.0108 (13) | 0.0049 (13) |
| C4 | 0.0406 (16) | 0.0363 (17) | 0.0343 (15) | 0.0048 (14) | 0.0077 (13) | -0.0015 (12) |
| C15 | 0.0397 (16) | 0.0345 (17) | 0.0538 (18) | 0.0054 (13) | 0.0007 (14) | 0.0081 (14) |
| C16 | 0.0428 (16) | 0.0294 (16) | 0.0478 (18) | 0.0044 (13) | 0.0063 (14) | 0.0033 (13) |
| C11 | 0.0410 (16) | 0.0328 (17) | 0.0426 (16) | 0.0055 (13) | 0.0082 (13) | 0.0057 (13) |
| C12 | 0.0383 (15) | 0.0275 (16) | 0.0469 (17) | 0.0018 (13) | 0.0053 (13) | 0.0053 (13) |
| C8 | 0.0455 (16) | 0.0343 (18) | 0.0504 (18) | 0.0023 (14) | 0.0134 (14) | 0.0009 (14) |
| C13 | 0.0481 (17) | 0.0372 (18) | 0.0479 (17) | 0.0061 (14) | 0.0079 (14) | 0.0074 (14) |
| C14 | 0.0477 (17) | 0.0373 (18) | 0.0473 (18) | 0.0074 (14) | 0.0055 (14) | 0.0087 (14) |
| C2 | 0.0374 (15) | 0.0285 (16) | 0.0463 (17) | 0.0015 (12) | 0.0063 (13) | 0.0070 (13) |
| C21 | 0.0391 (15) | 0.0376 (17) | 0.0390 (16) | 0.0077 (13) | 0.0016 (13) | 0.0051 (13) |
| C22 | 0.064 (2) | 0.041 (2) | 0.0476 (19) | 0.0048 (17) | 0.0052 (16) | -0.0032 (15) |
| C6 | 0.0511 (18) | 0.0351 (18) | 0.0492 (18) | 0.0124 (14) | 0.0117 (15) | 0.0086 (14) |
| C26 | 0.0465 (18) | 0.058 (2) | 0.0433 (18) | 0.0043 (16) | 0.0078 (15) | 0.0034 (16) |
| C23 | 0.087 (3) | 0.057 (2) | 0.046 (2) | 0.023 (2) | 0.0006 (19) | -0.0052 (17) |
| C24 | 0.059 (2) | 0.079 (3) | 0.0456 (19) | 0.032 (2) | 0.0082 (17) | 0.0074 (19) |
| C25 | 0.0512 (19) | 0.070 (3) | 0.0469 (19) | 0.0052 (18) | 0.0094 (15) | 0.0139 (18) |

Geometric parameters (Å, °)

| | | | |
|-------------|-----------|-------------|-----------|
| O1—C9 | 1.369 (3) | C15—H15 | 0.9500 |
| O1—C2 | 1.453 (3) | C11—C12 | 1.475 (4) |
| O4—C4 | 1.234 (3) | C11—H11 | 0.9500 |
| O3—C16 | 1.370 (4) | C12—C13 | 1.391 (4) |
| O3—H3 | 0.8400 | C8—H8 | 0.9500 |
| C10—C5 | 1.397 (4) | C13—C14 | 1.393 (4) |
| C10—C9 | 1.405 (4) | C13—H13 | 0.9500 |
| C10—C4 | 1.460 (4) | C14—H14 | 0.9500 |
| C17—C12 | 1.392 (4) | C2—C21 | 1.526 (4) |
| C17—C16 | 1.388 (4) | C2—H2 | 1.0000 |
| C17—H17 | 0.9500 | C21—C22 | 1.381 (4) |
| C9—C8 | 1.396 (4) | C21—C26 | 1.388 (4) |
| C3—C11 | 1.335 (4) | C22—C23 | 1.387 (5) |
| C3—C4 | 1.492 (4) | C22—H22 | 0.9500 |
| C3—C2 | 1.499 (4) | C6—H6 | 0.9500 |
| C7—C8 | 1.380 (4) | C26—C25 | 1.381 (4) |
| C7—C6 | 1.396 (4) | C26—H26 | 0.9500 |
| C7—H7 | 0.9500 | C23—C24 | 1.381 (5) |
| C5—C6 | 1.374 (4) | C23—H23 | 0.9500 |
| C5—H5 | 0.9500 | C24—C25 | 1.383 (5) |
| C15—C14 | 1.378 (4) | C24—H24 | 0.9500 |
| C15—C16 | 1.383 (4) | C25—H25 | 0.9500 |
| | | | |
| C9—O1—C2 | 115.9 (2) | C7—C8—H8 | 120.3 |
| C16—O3—H3 | 109.5 | C9—C8—H8 | 120.3 |
| C5—C10—C9 | 118.8 (3) | C12—C13—C14 | 119.7 (3) |
| C5—C10—C4 | 121.1 (3) | C12—C13—H13 | 120.1 |
| C9—C10—C4 | 119.8 (2) | C14—C13—H13 | 120.1 |
| C12—C17—C16 | 120.9 (3) | C15—C14—C13 | 121.0 (3) |
| C12—C17—H17 | 119.5 | C15—C14—H14 | 119.5 |
| C16—C17—H17 | 119.5 | C13—C14—H14 | 119.5 |
| O1—C9—C8 | 117.0 (3) | O1—C2—C3 | 111.0 (2) |
| O1—C9—C10 | 122.8 (3) | O1—C2—C21 | 109.4 (2) |
| C8—C9—C10 | 120.2 (3) | C3—C2—C21 | 112.3 (2) |
| C11—C3—C4 | 118.3 (3) | O1—C2—H2 | 108.0 |
| C11—C3—C2 | 127.3 (3) | C3—C2—H2 | 108.0 |
| C4—C3—C2 | 114.4 (2) | C21—C2—H2 | 108.0 |
| C8—C7—C6 | 121.1 (3) | C22—C21—C26 | 118.9 (3) |
| C8—C7—H7 | 119.5 | C22—C21—C2 | 121.1 (3) |
| C6—C7—H7 | 119.5 | C26—C21—C2 | 120.1 (3) |
| C6—C5—C10 | 121.2 (3) | C21—C22—C23 | 120.6 (3) |
| C6—C5—H5 | 119.4 | C21—C22—H22 | 119.7 |
| C10—C5—H5 | 119.4 | C23—C22—H22 | 119.7 |
| O4—C4—C10 | 123.2 (3) | C5—C6—C7 | 119.3 (3) |
| O4—C4—C3 | 121.2 (3) | C5—C6—H6 | 120.3 |
| C10—C4—C3 | 115.6 (3) | C7—C6—H6 | 120.3 |

| | | | |
|-------------|-----------|-------------|-----------|
| C14—C15—C16 | 119.6 (3) | C25—C26—C21 | 120.7 (3) |
| C14—C15—H15 | 120.2 | C25—C26—H26 | 119.6 |
| C16—C15—H15 | 120.2 | C21—C26—H26 | 119.6 |
| O3—C16—C15 | 118.4 (2) | C24—C23—C22 | 120.1 (3) |
| O3—C16—C17 | 121.8 (3) | C24—C23—H23 | 120.0 |
| C15—C16—C17 | 119.8 (3) | C22—C23—H23 | 120.0 |
| C3—C11—C12 | 129.9 (3) | C23—C24—C25 | 119.7 (3) |
| C3—C11—H11 | 115.1 | C23—C24—H24 | 120.2 |
| C12—C11—H11 | 115.1 | C25—C24—H24 | 120.2 |
| C17—C12—C13 | 118.9 (2) | C26—C25—C24 | 120.0 (4) |
| C17—C12—C11 | 117.1 (3) | C26—C25—H25 | 120.0 |
| C13—C12—C11 | 124.1 (3) | C24—C25—H25 | 120.0 |
| C7—C8—C9 | 119.4 (3) | | |

Hydrogen-bond geometry (Å, °)

| <i>D—H...A</i> | <i>D—H</i> | <i>H...A</i> | <i>D...A</i> | <i>D—H...A</i> |
|---------------------------|------------|--------------|--------------|----------------|
| O3—H3...O4 ⁱ | 0.84 | 1.89 | 2.728 (3) | 172 |
| C17—H17...O4 ⁱ | 0.95 | 2.49 | 3.184 (4) | 130 |
| C6—H6...O3 ⁱⁱ | 0.95 | 2.45 | 3.265 (4) | 143 |
| C11—H11...O4 | 0.95 | 2.43 | 2.807 (3) | 103 |

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