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Crystal structure and Hirshfeld surface analysis of 7-[(6-hydroxy-5,5,8a-trimethyl-2-methylenedeca-hydronaphthalen-1-yl)methoxy]-2*H*-chromen-2-one

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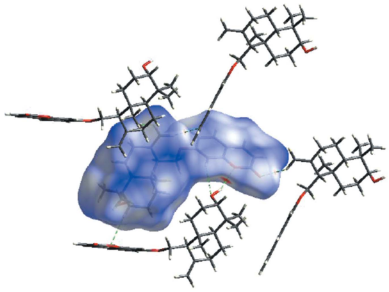
The title compound, $C_{24}H_{30}O_4$, consists of two *trans*-fused cyclohexane rings attached to a chromen-2-one moiety through an oxymethylene bridge. The cyclohexane rings adopt half-chair and chair conformations. In the crystal, molecules are linked by C—H···O and O—H···O hydrogen bonds, forming a triperiodic network. Furthermore, C—H···π interactions stabilize the crystal structure. A Hirshfeld surface analysis revealed that the most important contributions to the crystal packing are from H···H (56.9%), O···H/H···O (23.0%) and C···H/H···C (19.6%) contacts.

1. Chemical context

The genus *Ferula* belongs to the *Apiaceae* family, which comprises over 160 species and is spread throughout the world, including the Mediterranean region, Central Asia, Siberia, China, Afghanistan, Iran, North Africa and the Caucasus (Mir-Babayev & Houghton, 2002; Çelik *et al.*, 2023). This genus is rich in sesquiterpene coumarins. Extracts of *Ferula* species exhibit antimicrobial and estrogenic effects, and are natural plant-growth inhibitors and stimulants. Therefore, they have long been well known in popular medicine for the treatment of various health disorders, such as cough, asthma, toothache and gastroenteric problems (Salehi *et al.*, 2019; Tapera *et al.*, 2022; Lakhrissi *et al.*, 2022). These plants have been used for oleo-gum resin, plant extracts and essential oils. The extracts and essential oils of different species of *Ferula* can also be used as natural food preservatives due to their antioxidant and antimicrobial activity (Daneshniya *et al.*, 2021; Chalkha *et al.*, 2023).



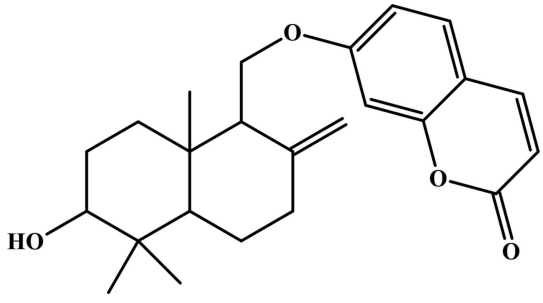


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Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
O16—H16O···O1^{i}	0.91 (7)	2.34 (7)	3.194 (4)	158 (6)
C6—H6···O2^{ii}	0.95	2.54	3.355 (6)	144
C8—H8···O16^{iii}	0.95	2.59	3.256 (5)	127
C16—H16···$Cg2^{iv}$	1.02 (5)	2.69 (5)	3.564 (5)	146 (4)
C21—H21A···$Cg1^{v}$	0.98	2.96	3.923 (6)	169

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

Herein, we report the molecular and crystal structure, and Hirshfeld surface analysis of the title compound, 7-[(6-hydroxy-5,5,8a-trimethyl-2-methylenedecahydronaphthalen-1-yl)-methoxy]-2*H*-chromen-2-one, extracted from *Ferula persica*.

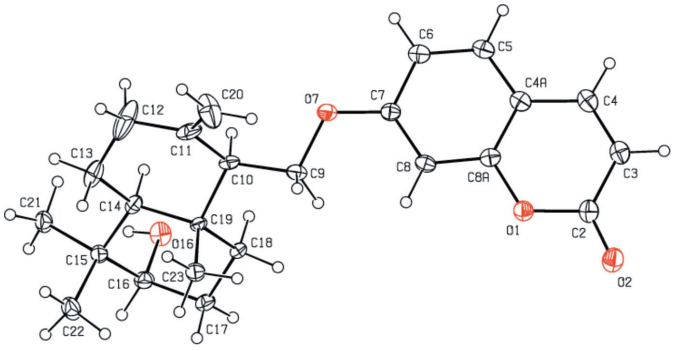

Figure 1
The molecular structure of the title compound, showing the atom labelling and displacement ellipsoids drawn at the 30% probability level.

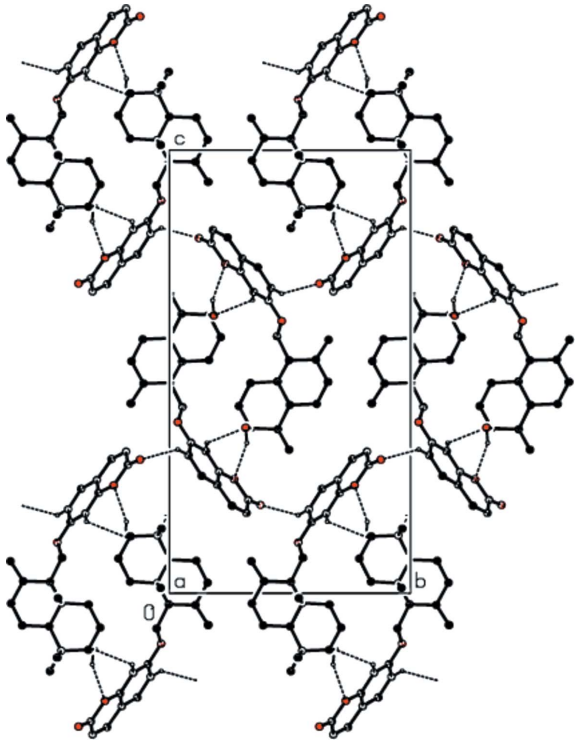

Figure 2
The crystal structure of the title compound in a view along [100]. O—H···O and C—H···O hydrogen bonds are indicated by dashed lines. H atoms not involved in hydrogen bonding have been omitted for clarity.

Table 2
Summary of short interatomic contacts (Å) in the title compound.

Atom pair	Distance	Symmetry code
O2···H4	2.66	$x-1, y, z$
O1···H16O	2.34	$x-\frac{1}{2}, -y+\frac{1}{2}, -z+1$
H4···H20B	2.51	$-x+1, y-\frac{1}{2}, -z+\frac{3}{2}$
O2···H20B	2.63	$-x, y-\frac{1}{2}, -z+\frac{3}{2}$
C4A···H21A	2.91	$-x+\frac{1}{2}, -y+1, z+\frac{1}{2}$
H12A···H20A	2.28	$x+\frac{1}{2}, -y+\frac{3}{2}, -z+1$

2. Structural commentary

The molecular structure of the title compound (Fig. 1) consists of two *trans*-fused cyclohexane rings (atoms C10–C14/C19 and C14–C19) attached to a chromen-2-one moiety through an oxymethylene bridge. The cyclohexane rings adopt half-chair and chair conformations, respectively. The puckering parameters (Cremer & Pople, 1975) are Q_T = 0.550 (5) Å, θ = 14.2 (5)° and φ = 292 (2)° for C10–C14/C19, and Q_T = 0.546 (5) Å, θ = 5.1 (5)° and φ = 199 (5)° for C14–C19. The bond lengths and angles within the title molecule are in agreement with those reported for similar compounds compiled in the *Database survey* section.

3. Supramolecular features and Hirshfeld surface analysis

In the crystal of the title compound, molecules are linked by C—H···O and O—H···O hydrogen bonds, forming a triperiodic network (Tables 1 and 2, and Figs. 2–4). Furthermore, C—H···π interactions consolidate the crystal structure (Table 1 and Figs. 5 and 6).

Hirshfeld surfaces and two-dimensional (2D) fingerprint plots were produced using *CrystalExplorer17.5* (Spackman *et al.*, 2021) to quantify the intermolecular interactions in the crystal. The Hirshfeld surface mapped over d_{norm} in the range

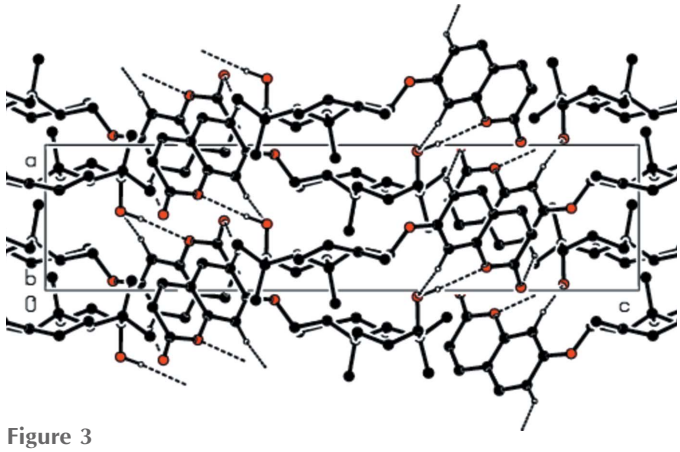

Figure 3
The crystal structure of the title compound in a view along [010]. O—H···O and C—H···O hydrogen bonds are indicated by dashed lines. H atoms not involved in hydrogen bonding have been omitted for clarity.

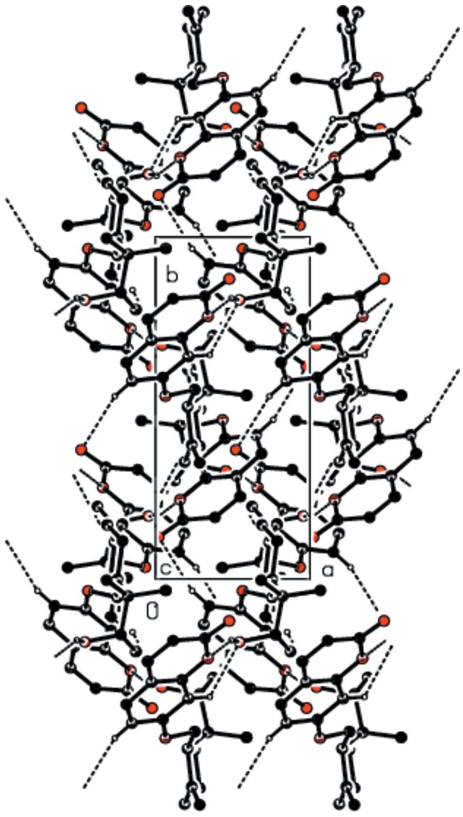

Figure 4
The crystal structure of the title compound in a view along [001]. O—H···O and C—H···O hydrogen bonds are indicated by dashed lines. H atoms not involved in hydrogen bonding have been omitted for clarity.

from −0.2594 to +1.5213 a.u. is shown in Fig. 7, where significant intermolecular contacts appear as red-coloured spots, which indicate the O—H···O and C—H···O hydrogen-bonding interactions. The 2D fingerprint plots are presented in Fig. 8. The H···H contacts comprise 56.9% of the total interactions. The O···H/H···O (23.0%) and C···H/H···C (19.6%) interactions also make significant contributions to the total Hirshfeld surface. The C···C and O···C/C···O contacts contribute 0.2% of the total interactions.

4. Database survey

Six compounds similar to the 1,2,3,4,4a,5,8,8a-octahydronaphthalene group were found from a search of the Cambridge Structural Database (updated 20 March 2023; Groom *et al.*, 2016): 7-[(6-hydroxy-2,5,5,8a-tetramethyl-1,4,4a,5,6,7,8,8a-octahydronaphthalen-1-yl)methoxy]-2*H*-chromen-2-one, (**I**) (Bashir *et al.*, 2014); (*E*)-3-[(1*R**,2*S**,4a*S**,8a*R**)-2- (benzo[*d*][1,3]dioxol-5-yl)-1,2,4a,5,6,7,8,8a-octahydronaphthalen-1-yl]-*N*-isobutylacrylamide, (**II**) (Lenta *et al.*, 2015); 3-(4-bromophenyl)-l-[4-(4-bromophenyl)-3-butene-2-one-1-yl]-2-[3-(2,6,6-trimethyl-1-cyclohexen-l-yl)-2-propene-1-one-1-yl]-l,2,3,4,5,6,7,8-octahydro-8,8-dimethylnaphtalene, (**III**) (Ginderow, 1996); (+/−)3β-bromo-2β-methyl-4aβ*H*,8aα*H*-decahydronaphthalen-2α-ol, (**IV**) (Fallon *et al.*, 1992); (2*R*,3*R*,5*R*)-2-[(2*R*,3a*S*,6a*R*)-2,3,3a,4,5,6a-hexahydrofuro[2,3-*b*]furan-2-yl]-5-isopropenyl-2,3-dimethylcyclohexanone, (**V**) (Ellis & Spek, 2000); (4a*R*,5*S*,7*R*)-5-isopropenyl-7,8,8-trimethyl-2,3,4,4a,5,6,7,8-octahydronaphthalene-4a-carbonitrile, (**VI**) (Ellis & Spek, 2000).

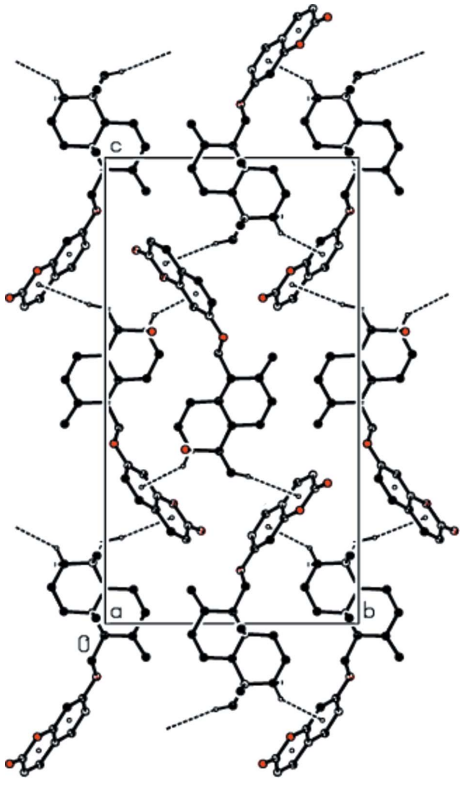

Figure 5
View along [100] of the C—H···π interactions (dashed lines) in the crystal of the title compound.

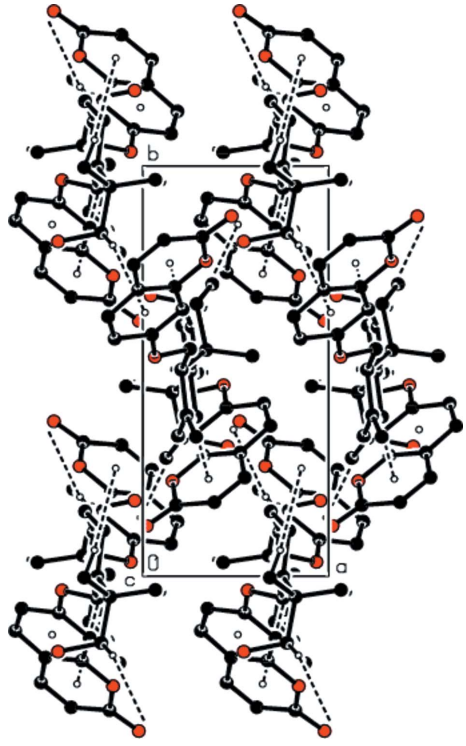

Figure 6
View along [001] of the C—H···π interactions (dashed lines) in the crystal of the title compound.

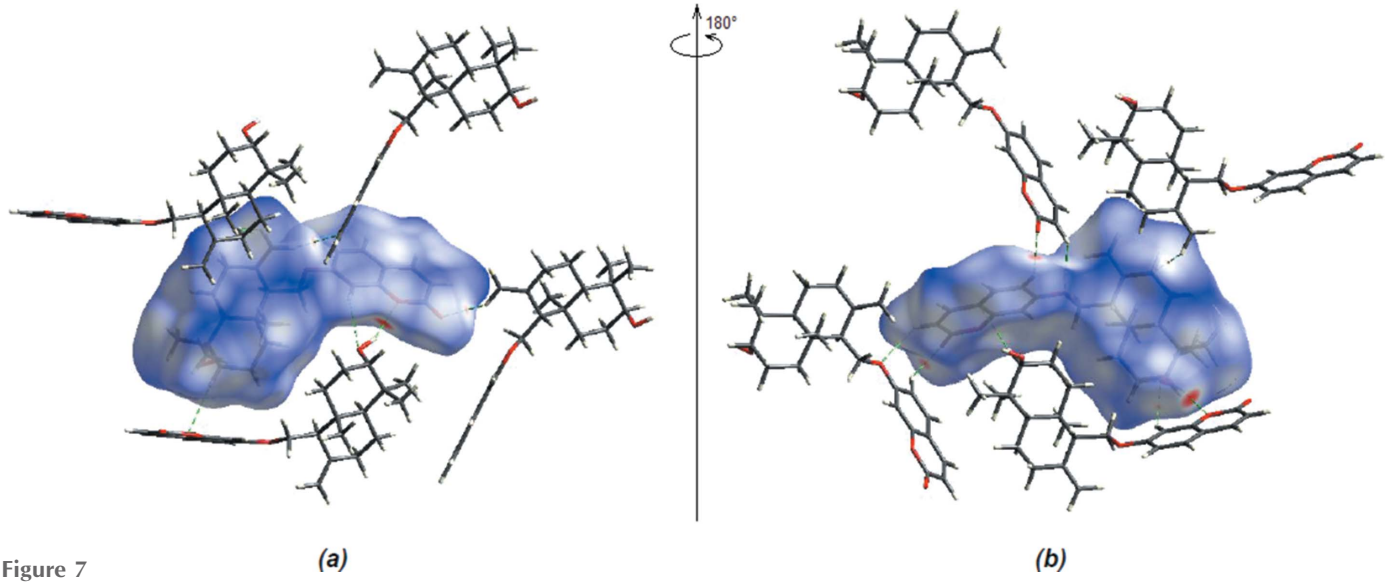

Figure 7
The (*a*) front and (*b*) back sides of the three-dimensional Hirshfeld surface of the title compound mapped over d_{norm}, with a fixed colour scale from −0.2594 to +1.5213 a.u.

In the crystal of (**I**), the molecules are connected by C—H···O and O—H···O hydrogen bonds, forming bands along [100] that are oriented parallel to (100). Additional C—H···π and van der Waals interactions result in a triperiodic network. In the crystal of (**II**), molecules are linked by N—H···O hydrogen bonds, forming chains propagating along [100]. The chains are linked by pairs of C—H···O hydrogen bonds, involving inversion-related benzodioxole ring systems, thus forming ribbons lying parallel to (010). There are also C—H···π interactions present within the ribbons. Disorder in the crystal of (**III**) arises from the co-existence of the two possible puckered conformations of the dimethylcyclohexene rings. In (**IV**), the *trans*-fused six-membered rings are chair-shaped but somewhat flattened. (**V**) and (**VI**) comprise the molecular structures of two chiral cyclohexanones based on *R*-(−)-carvone. The six-membered ring in (**V**) is in a chair conformation with the two fused five-membered rings of the furofuranyl substituent in a *cis* configuration. Compound (**VI**) contains a decalin group; one ring has the chair form, whilst the other is in a half-boat conformation.

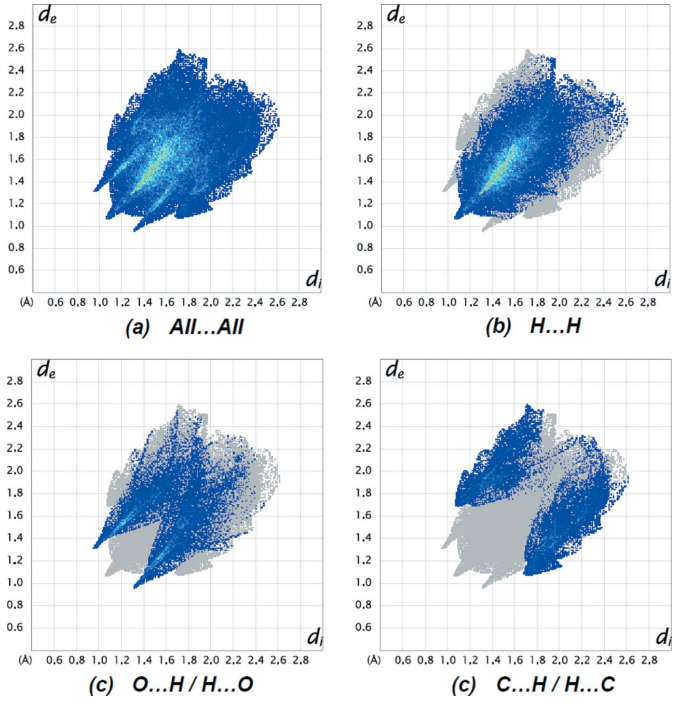

Figure 8
The 2D fingerprint plots of the title compound, showing (*a*) all interactions, and delineated into (*b*) H···H, (*c*) O···H/H···O and (*d*) C···H/H···C interactions. d_e and d_i represent the distances from a point on the Hirshfeld surface to the nearest atoms outside (external) and inside (internal) the surface, respectively.

5. Synthesis and crystallization

The title compound was isolated previously from *Ferula lehmanni* and fully characterized (Sagitdinova *et al.*, 1983). The present material was isolated from the roots of the *Ferula persica* herb by a similar method.

6. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 3. The H atoms of the –OH group and the methine H atom attached to C16 were located in a difference map, and refined freely [O16—H16O = 0.90 (6) Å and C16—H16 = 1.02 (5) Å]. The remaining H atoms bound to C atoms were positioned geometrically and refined as riding, with C—H = 0.95 (aromatic), 0.99 (methylene), 0.98 (methyl) and 1.00 Å (methine), with U_{iso}(H) = 1.5U_{eq}(C) for methyl H atoms and 1.2U_{eq}(C) for all others. Two reflections (590 and 2,15,0), for which I(obs) and I(calc) differed by more than 10σ, were removed from the refinement.

Table 3
Experimental details.

Crystal data	
Chemical formula	$C_{24}H_{30}O_4$
M_r	382.48
Crystal system, space group	Orthorhombic, $P2_12_12_1$
Temperature (K)	100
a, b, c (Å)	6.0526 (1), 13.3964 (4), 24.6848 (5)
V (Å^3)	2001.52 (8)
Z	4
Radiation type	Cu $K\alpha$
μ (mm^{-1})	0.68
Crystal size (mm)	0.20 × 0.14 × 0.09
Data collection	
Diffractometer	Rigaku XtaLAB Synergy Dualflex diffractometer with a HyPix detector
Absorption correction	Multi-scan (*CrysAlis PRO*; Rigaku OD, 2022)
T_{min}, T_{max}	0.664, 1.000
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	11367, 3918, 3687
R_{int}	0.034
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.633
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.062, 0.158, 1.12
No. of reflections	3918
No. of parameters	264
H-atom treatment	H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.43, −0.56
Absolute structure	Flack x determined using 1307 quotients $[(I^+) - (I^-)]/[(I^+) + (I^-)]$ (Parsons *et al.*, 2013)
Absolute structure parameter	−0.18 (12)

Computer programs: *CrysAlis PRO* (Rigaku OD, 2022), *SHELXT* (Sheldrick, 2015*a*), *SHELXL2018* (Sheldrick, 2015*b*), *ORTEP-3 for Windows* (Farrugia, 2012) and *PLATON* (Spek, 2020).

Acknowledgements

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supporting information

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Crystal structure and Hirshfeld surface analysis of 7-[(6-hydroxy-5,5,8a-trimethyl-2-methylenedecahydronaphthalen-1-yl)methoxy]-2*H*-chromen-2-one

Elvin G. Karimli, Victor N. Khrustalev, Mehmet Akkurt, Ali N. Khalilov, Ajaya Bhattarai, Adila N. Aleskerova and İbrahim G. Mamedov

Computing details

Data collection: *CrysAlis PRO* (Rigaku OD, 2022); cell refinement: *CrysAlis PRO* (Rigaku OD, 2022); data reduction: *CrysAlis PRO* (Rigaku OD, 2022); program(s) used to solve structure: SHELXT (Sheldrick, 2015a); program(s) used to refine structure: *SHELXL2018* (Sheldrick, 2015b); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 2012); software used to prepare material for publication: *PLATON* (Spek, 2020).

7-[(6-Hydroxy-5,5,8a-trimethyl-2-methylenedecahydronaphthalen-1-yl)methoxy]-2*H*-chromen-2-one

Crystal data

$C_{24}H_{30}O_4$
M_r = 382.48
Orthorhombic, $P2_12_12_1$
a = 6.0526 (1) Å
b = 13.3964 (4) Å
c = 24.6848 (5) Å
V = 2001.52 (8) Å^3
Z = 4
$F(000)$ = 824
D_x = 1.269 Mg m^{-3}
Cu $K\alpha$ radiation, λ = 1.54184 Å
Cell parameters from 7116 reflections
θ = 3.7–77.5°
μ = 0.68 mm^{-1}
T = 100 K
Prism, colourless
0.20 × 0.14 × 0.09 mm

Data collection

Rigaku XtaLAB Synergy Dualflex diffractometer with a HyPix detector
Radiation source: micro-focus sealed X-ray tube
φ and ω scans
Absorption correction: multi-scan (CrysAlis PRO; Rigaku OD, 2022)
T_{min} = 0.664, T_{max} = 1.000
11367 measured reflections
3918 independent reflections
3687 reflections with $I > 2\sigma(I)$
R_{int} = 0.034
θ_{max} = 77.5°, θ_{min} = 3.6°
h = −7→7
k = −16→16
l = −25→31

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.062
$wR(F^2)$ = 0.158
S = 1.12
3918 reflections
264 parameters
0 restraints
Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0424P)^2 + 2.9169P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.43 e Å^{-3}
$\Delta\rho_{min}$ = −0.56 e Å^{-3}

Absolute structure: Flack *x* determined using 1307 quotients [(I+)-(I-)]/[(I+)+(I-)] (Parsons *et al.*, 2013)
Absolute structure parameter: −0.18 (12)

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 (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
O1	0.1603 (5)	0.2319 (2)	0.74312 (12)	0.0304 (7)
C2	0.1791 (8)	0.1730 (4)	0.78982 (18)	0.0318 (10)
O2	0.0215 (6)	0.1226 (3)	0.80119 (13)	0.0405 (8)
C3	0.3855 (8)	0.1803 (4)	0.81926 (18)	0.0327 (10)
H3	0.405572	0.141392	0.851048	0.039*
C4	0.5488 (8)	0.2406 (3)	0.80275 (17)	0.0319 (10)
H4	0.682740	0.243074	0.822775	0.038*
C4A	0.5245 (7)	0.3017 (3)	0.75521 (17)	0.0277 (9)
C5	0.6868 (7)	0.3667 (3)	0.73537 (18)	0.0290 (9)
H5	0.822769	0.373281	0.754240	0.035*
C6	0.6516 (8)	0.4212 (3)	0.68878 (18)	0.0301 (9)
H6	0.762016	0.465638	0.675843	0.036*
C7	0.4511 (7)	0.4107 (3)	0.66054 (17)	0.0266 (9)
O7	0.4349 (5)	0.4653 (2)	0.61394 (11)	0.0286 (7)
C8	0.2858 (7)	0.3495 (3)	0.67958 (17)	0.0265 (9)
H8	0.147774	0.344923	0.661441	0.032*
C8A	0.3277 (7)	0.2944 (3)	0.72632 (17)	0.0267 (9)
C9	0.2455 (7)	0.4463 (3)	0.57989 (17)	0.0277 (9)
H9A	0.110286	0.474519	0.596468	0.033*
H9B	0.224179	0.373572	0.574977	0.033*
C10	0.2921 (6)	0.4964 (3)	0.52545 (17)	0.0250 (8)
H10	0.451638	0.483852	0.517389	0.030*
C11	0.2644 (8)	0.6075 (3)	0.5269 (2)	0.0365 (11)
C12	0.3107 (17)	0.6579 (4)	0.4766 (2)	0.080 (3)
H12A	0.472930	0.661076	0.471769	0.096*
H12B	0.255956	0.727365	0.479326	0.096*
C13	0.2122 (9)	0.6113 (4)	0.4270 (2)	0.0433 (12)
H13A	0.279824	0.641920	0.394525	0.052*
H13B	0.051974	0.625974	0.426186	0.052*
C14	0.2453 (7)	0.4992 (3)	0.42470 (17)	0.0261 (8)
H14	0.409159	0.490292	0.426388	0.031*
C15	0.1770 (7)	0.4497 (4)	0.36979 (16)	0.0289 (9)
C16	0.2232 (7)	0.3372 (4)	0.37214 (18)	0.0298 (9)
O16	0.4562 (5)	0.3160 (3)	0.37334 (13)	0.0353 (7)
C17	0.1259 (8)	0.2879 (3)	0.42204 (17)	0.0309 (9)

H17A	0.164982	0.216149	0.422058	0.037*
H17B	−0.037133	0.293255	0.420808	0.037*
C18	0.2109 (7)	0.3362 (3)	0.47414 (16)	0.0240 (8)
H18A	0.372883	0.326882	0.476324	0.029*
H18B	0.143699	0.301914	0.505609	0.029*
C19	0.1583 (6)	0.4484 (3)	0.47759 (15)	0.0209 (8)
C20	0.2133 (13)	0.6603 (5)	0.5719 (2)	0.067 (2)
H20A	0.207618	0.731071	0.570476	0.080*
H20B	0.183005	0.626517	0.604933	0.080*
C21	0.3154 (8)	0.4964 (4)	0.32367 (17)	0.0347 (10)
H21A	0.259824	0.563489	0.315693	0.052*
H21B	0.303667	0.454693	0.291171	0.052*
H21C	0.470411	0.500524	0.334907	0.052*
C22	−0.0672 (8)	0.4665 (5)	0.3545 (2)	0.0424 (13)
H22A	−0.162189	0.430585	0.380056	0.064*
H22B	−0.093601	0.441732	0.317737	0.064*
H22C	−0.100894	0.538043	0.356051	0.064*
C23	−0.0900 (7)	0.4630 (4)	0.48846 (17)	0.0300 (10)
H23A	−0.175588	0.428509	0.460426	0.045*
H23B	−0.125252	0.534374	0.487810	0.045*
H23C	−0.127311	0.435337	0.524059	0.045*
H16	0.162 (9)	0.307 (4)	0.338 (2)	0.031 (13)*
H16O	0.508 (11)	0.321 (5)	0.339 (3)	0.054 (17)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0258 (14)	0.0338 (17)	0.0317 (15)	0.0023 (14)	0.0012 (13)	0.0019 (13)
C2	0.033 (2)	0.030 (2)	0.032 (2)	0.008 (2)	0.0034 (19)	0.0009 (17)
O2	0.0323 (18)	0.045 (2)	0.0446 (18)	0.0045 (17)	0.0059 (15)	0.0103 (15)
C3	0.038 (2)	0.028 (2)	0.032 (2)	0.009 (2)	−0.0008 (18)	−0.0023 (18)
C4	0.037 (2)	0.027 (2)	0.032 (2)	0.011 (2)	−0.004 (2)	−0.0058 (17)
C4A	0.028 (2)	0.0245 (19)	0.030 (2)	0.0069 (18)	−0.0001 (17)	−0.0082 (16)
C5	0.025 (2)	0.024 (2)	0.038 (2)	0.0066 (18)	−0.0055 (18)	−0.0080 (18)
C6	0.026 (2)	0.027 (2)	0.037 (2)	0.0019 (19)	−0.0011 (18)	−0.0053 (17)
C7	0.026 (2)	0.025 (2)	0.0289 (19)	0.0002 (18)	−0.0003 (17)	−0.0042 (16)
O7	0.0243 (14)	0.0292 (16)	0.0324 (15)	−0.0032 (13)	−0.0040 (12)	0.0000 (12)
C8	0.0198 (19)	0.030 (2)	0.030 (2)	0.0021 (18)	−0.0015 (16)	−0.0048 (17)
C8A	0.0255 (19)	0.023 (2)	0.0311 (19)	0.0017 (18)	0.0023 (17)	−0.0055 (16)
C9	0.0219 (18)	0.032 (2)	0.0296 (19)	−0.0049 (18)	−0.0031 (16)	−0.0035 (18)
C10	0.0158 (17)	0.024 (2)	0.035 (2)	−0.0048 (16)	0.0009 (16)	−0.0031 (17)
C11	0.030 (2)	0.020 (2)	0.060 (3)	−0.0065 (19)	−0.001 (2)	−0.005 (2)
C12	0.169 (9)	0.020 (2)	0.051 (3)	−0.009 (4)	0.036 (5)	0.001 (2)
C13	0.052 (3)	0.027 (2)	0.051 (3)	0.006 (2)	0.013 (3)	0.015 (2)
C14	0.0222 (18)	0.023 (2)	0.033 (2)	0.0005 (17)	0.0040 (17)	0.0071 (17)
C15	0.0196 (17)	0.038 (2)	0.029 (2)	−0.0034 (19)	−0.0017 (16)	0.0098 (19)
C16	0.031 (2)	0.031 (2)	0.027 (2)	−0.0076 (19)	−0.0012 (18)	−0.0027 (17)
O16	0.0343 (16)	0.0376 (18)	0.0341 (16)	0.0092 (15)	0.0053 (15)	−0.0005 (14)

C17	0.036 (2)	0.024 (2)	0.032 (2)	−0.0095 (19)	−0.0024 (19)	−0.0003 (18)
C18	0.028 (2)	0.0175 (19)	0.0271 (19)	−0.0058 (17)	−0.0028 (17)	0.0025 (15)
C19	0.0153 (16)	0.0184 (19)	0.0289 (18)	−0.0030 (16)	−0.0006 (15)	0.0033 (16)
C20	0.118 (6)	0.040 (3)	0.042 (3)	0.021 (4)	−0.024 (4)	−0.011 (2)
C21	0.027 (2)	0.047 (3)	0.030 (2)	0.000 (2)	0.0008 (18)	0.013 (2)
C22	0.025 (2)	0.065 (4)	0.038 (2)	0.001 (2)	−0.0041 (19)	0.018 (2)
C23	0.0196 (19)	0.038 (3)	0.032 (2)	−0.0009 (19)	0.0011 (16)	0.0049 (19)

Geometric parameters (Å, º)

O1—C8A	1.378 (5)	C13—H13A	0.9900
O1—C2	1.401 (5)	C13—H13B	0.9900
C2—O2	1.202 (6)	C14—C19	1.563 (5)
C2—C3	1.449 (7)	C14—C15	1.565 (6)
C3—C4	1.340 (7)	C14—H14	1.0000
C3—H3	0.9500	C15—C16	1.534 (7)
C4—C4A	1.438 (6)	C15—C22	1.542 (6)
C4—H4	0.9500	C15—C21	1.545 (6)
C4A—C8A	1.392 (6)	C16—O16	1.439 (5)
C4A—C5	1.401 (7)	C16—C17	1.516 (6)
C5—C6	1.379 (6)	C16—H16	1.02 (5)
C5—H5	0.9500	O16—H16O	0.90 (6)
C6—C7	1.407 (6)	C17—C18	1.529 (6)
C6—H6	0.9500	C17—H17A	0.9900
C7—O7	1.367 (5)	C17—H17B	0.9900
C7—C8	1.376 (6)	C18—C19	1.539 (6)
O7—C9	1.444 (5)	C18—H18A	0.9900
C8—C8A	1.393 (6)	C18—H18B	0.9900
C8—H8	0.9500	C19—C23	1.539 (5)
C9—C10	1.528 (6)	C20—H20A	0.9500
C9—H9A	0.9900	C20—H20B	0.9500
C9—H9B	0.9900	C21—H21A	0.9800
C10—C11	1.498 (6)	C21—H21B	0.9800
C10—C19	1.570 (5)	C21—H21C	0.9800
C10—H10	1.0000	C22—H22A	0.9800
C11—C20	1.352 (8)	C22—H22B	0.9800
C11—C12	1.443 (8)	C22—H22C	0.9800
C12—C13	1.496 (9)	C23—H23A	0.9800
C12—H12A	0.9900	C23—H23B	0.9800
C12—H12B	0.9900	C23—H23C	0.9800
C13—C14	1.516 (6)		
C8A—O1—C2	122.0 (4)	C13—C14—C15	114.7 (4)
O2—C2—O1	116.4 (4)	C19—C14—C15	116.8 (3)
O2—C2—C3	127.2 (4)	C13—C14—H14	104.3
O1—C2—C3	116.4 (4)	C19—C14—H14	104.3
C4—C3—C2	121.6 (4)	C15—C14—H14	104.3
C4—C3—H3	119.2	C16—C15—C22	109.1 (4)

C2—C3—H3	119.2	C16—C15—C21	109.1 (4)
C3—C4—C4A	121.1 (4)	C22—C15—C21	106.3 (3)
C3—C4—H4	119.5	C16—C15—C14	109.6 (3)
C4A—C4—H4	119.5	C22—C15—C14	113.8 (4)
C8A—C4A—C5	117.7 (4)	C21—C15—C14	108.9 (4)
C8A—C4A—C4	117.7 (4)	O16—C16—C17	106.1 (4)
C5—C4A—C4	124.6 (4)	O16—C16—C15	111.9 (4)
C6—C5—C4A	120.8 (4)	C17—C16—C15	112.8 (4)
C6—C5—H5	119.6	O16—C16—H16	107 (3)
C4A—C5—H5	119.6	C17—C16—H16	111 (3)
C5—C6—C7	119.6 (4)	C15—C16—H16	107 (3)
C5—C6—H6	120.2	C16—O16—H16O	108 (4)
C7—C6—H6	120.2	C16—C17—C18	111.6 (3)
O7—C7—C8	123.7 (4)	C16—C17—H17A	109.3
O7—C7—C6	115.2 (4)	C18—C17—H17A	109.3
C8—C7—C6	121.2 (4)	C16—C17—H17B	109.3
C7—O7—C9	116.9 (3)	C18—C17—H17B	109.3
C7—C8—C8A	117.8 (4)	H17A—C17—H17B	108.0
C7—C8—H8	121.1	C17—C18—C19	113.0 (3)
C8A—C8—H8	121.1	C17—C18—H18A	109.0
O1—C8A—C4A	121.2 (4)	C19—C18—H18A	109.0
O1—C8A—C8	115.9 (4)	C17—C18—H18B	109.0
C4A—C8A—C8	122.9 (4)	C19—C18—H18B	109.0
O7—C9—C10	106.7 (3)	H18A—C18—H18B	107.8
O7—C9—H9A	110.4	C18—C19—C23	109.6 (3)
C10—C9—H9A	110.4	C18—C19—C14	108.0 (3)
O7—C9—H9B	110.4	C23—C19—C14	114.8 (3)
C10—C9—H9B	110.4	C18—C19—C10	109.6 (3)
H9A—C9—H9B	108.6	C23—C19—C10	108.7 (3)
C11—C10—C9	113.2 (4)	C14—C19—C10	106.1 (3)
C11—C10—C19	111.5 (4)	C11—C20—H20A	120.0
C9—C10—C19	112.8 (3)	C11—C20—H20B	120.0
C11—C10—H10	106.2	H20A—C20—H20B	120.0
C9—C10—H10	106.2	C15—C21—H21A	109.5
C19—C10—H10	106.2	C15—C21—H21B	109.5
C20—C11—C12	120.5 (5)	H21A—C21—H21B	109.5
C20—C11—C10	124.4 (5)	C15—C21—H21C	109.5
C12—C11—C10	115.0 (5)	H21A—C21—H21C	109.5
C11—C12—C13	115.6 (5)	H21B—C21—H21C	109.5
C11—C12—H12A	108.4	C15—C22—H22A	109.5
C13—C12—H12A	108.4	C15—C22—H22B	109.5
C11—C12—H12B	108.4	H22A—C22—H22B	109.5
C13—C12—H12B	108.4	C15—C22—H22C	109.5
H12A—C12—H12B	107.5	H22A—C22—H22C	109.5
C12—C13—C14	113.1 (5)	H22B—C22—H22C	109.5
C12—C13—H13A	109.0	C19—C23—H23A	109.5
C14—C13—H13A	109.0	C19—C23—H23B	109.5
C12—C13—H13B	109.0	H23A—C23—H23B	109.5

C14—C13—H13B	109.0	C19—C23—H23C	109.5
H13A—C13—H13B	107.8	H23A—C23—H23C	109.5
C13—C14—C19	110.8 (4)	H23B—C23—H23C	109.5
C8A—O1—C2—O2	178.7 (4)	C11—C12—C13—C14	−46.2 (9)
C8A—O1—C2—C3	−0.8 (6)	C12—C13—C14—C19	54.0 (6)
O2—C2—C3—C4	−179.1 (5)	C12—C13—C14—C15	−171.2 (5)
O1—C2—C3—C4	0.4 (6)	C13—C14—C15—C16	178.7 (4)
C2—C3—C4—C4A	0.7 (7)	C19—C14—C15—C16	−49.3 (4)
C3—C4—C4A—C8A	−1.4 (6)	C13—C14—C15—C22	−58.8 (5)
C3—C4—C4A—C5	179.9 (4)	C19—C14—C15—C22	73.2 (5)
C8A—C4A—C5—C6	0.0 (6)	C13—C14—C15—C21	59.5 (5)
C4—C4A—C5—C6	178.7 (4)	C19—C14—C15—C21	−168.5 (4)
C4A—C5—C6—C7	−0.7 (6)	C22—C15—C16—O16	166.2 (4)
C5—C6—C7—O7	−177.9 (4)	C21—C15—C16—O16	50.5 (5)
C5—C6—C7—C8	2.3 (6)	C14—C15—C16—O16	−68.7 (4)
C8—C7—O7—C9	−8.5 (6)	C22—C15—C16—C17	−74.2 (4)
C6—C7—O7—C9	171.7 (4)	C21—C15—C16—C17	170.1 (3)
O7—C7—C8—C8A	177.1 (4)	C14—C15—C16—C17	50.9 (5)
C6—C7—C8—C8A	−3.1 (6)	O16—C16—C17—C18	65.8 (5)
C2—O1—C8A—C4A	0.1 (6)	C15—C16—C17—C18	−57.1 (5)
C2—O1—C8A—C8	−179.2 (4)	C16—C17—C18—C19	58.7 (5)
C5—C4A—C8A—O1	179.8 (4)	C17—C18—C19—C23	72.6 (4)
C4—C4A—C8A—O1	1.0 (6)	C17—C18—C19—C14	−53.1 (4)
C5—C4A—C8A—C8	−0.9 (6)	C17—C18—C19—C10	−168.2 (3)
C4—C4A—C8A—C8	−179.7 (4)	C13—C14—C19—C18	−176.3 (4)
C7—C8—C8A—O1	−178.2 (4)	C15—C14—C19—C18	49.9 (4)
C7—C8—C8A—C4A	2.5 (6)	C13—C14—C19—C23	61.2 (5)
C7—O7—C9—C10	−166.8 (3)	C15—C14—C19—C23	−72.6 (5)
O7—C9—C10—C11	−75.9 (4)	C13—C14—C19—C10	−58.9 (4)
O7—C9—C10—C19	156.3 (3)	C15—C14—C19—C10	167.3 (3)
C9—C10—C11—C20	2.8 (7)	C11—C10—C19—C18	174.2 (4)
C19—C10—C11—C20	131.3 (6)	C9—C10—C19—C18	−57.1 (4)
C9—C10—C11—C12	179.0 (5)	C11—C10—C19—C23	−66.1 (5)
C19—C10—C11—C12	−52.6 (7)	C9—C10—C19—C23	62.6 (5)
C20—C11—C12—C13	−138.1 (6)	C11—C10—C19—C14	57.8 (4)
C10—C11—C12—C13	45.6 (9)	C9—C10—C19—C14	−173.5 (3)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O16—H16*O*···O1^{i}	0.91 (7)	2.34 (7)	3.194 (4)	158 (6)
C6—H6···O2^{ii}	0.95	2.54	3.355 (6)	144
C8—H8···O16^{iii}	0.95	2.59	3.256 (5)	127
C16—H16···*Cg*2^{iv}	1.02 (5)	2.69 (5)	3.564 (5)	146 (4)
C21—H21*A*···*Cg*1^{v}	0.98	2.96	3.923 (6)	169

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