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CRYSTAL STRUCTURE AND HIRSHFELD SURFACE ANALYSIS OF 7-((6-HYDROXY-2,5,5,8A-TETRAMETHYL-1,4,4A,5,6,7,8,8A-OCTAHYDRONAPHTHALEN-1-YL)METHOXY)-2H-CHROMEN-2-ONE ISOLATED FROM *FERULA PERSICA*: A NEW ENANTIOMORPH AT 100 K*

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From the underground part of *Ferula persica*, collected during the fruiting phase, an alcoholic extract was obtained, which, after removal of the solvent, was chromatographed on a column with neutral aluminium oxide (II degree of activity) and eluted with hexane, benzene, and their mixtures in a gradient of increasing polarity. As a result, 7-((6-Hydroxy-2,5,5,8a-tetramethyl-1,4,4a,5,6,7,8,8a-octahydronaphthalen-1-yl)methoxy)-2H-chromen-2-one (conferol – m.p. 137–138 °C), was isolated. For the confirmation of structure, we used X-Ray, NMR ¹H, ¹³C NMR, DEPT, COSY, HSQC, and HMBC methods. We note, that the crystal structure of the title compound, [systematic name; 7-((6-Hydroxy-2,5,5,8a-tetramethyl-1,4,4a,5,6,7,8,8a-octahydronaphthalen-1-yl)methoxy)-2H-chromen-2-one], C₂₄H₃₀O₄, has been reported previously at room temperature. The crystal structure was determined at higher precision at 100 K as a new enantiomorph. The title compound consists of two trans-fused cyclohexane rings, attached to the chromen-2-one moiety through an oxymethylene bridge. Cyclohexane rings adopt half-chair and chair conformations, respectively. The title compound is a new enantiomorph of the one reported. In the crystal, the molecules are joined by C-H···O hydrogen bonds along the c-axis, forming layers parallel to the (010) plane, while molecules are linked by O-H···O hydrogen bonds, which generate R22(6) motifs along the a-axis. The C-H···π and van der Waals interactions between these layers stabilize and maintain the structure. The Hirshfeld surface analysis indicates that the most important contributions to the crystal packing are from H···H (58.3%), O···H/H···O (21.8%) and C···H/H···C (19.7%) contacts.

Keywords: *Ferula persica*, extraction, chromatography, spectroscopy, crystal structure, enantiomorph, O-H···O hydrogen bonds, C-H···π interactions, redetermination, Hirshfeld surface analysis.

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Introduction

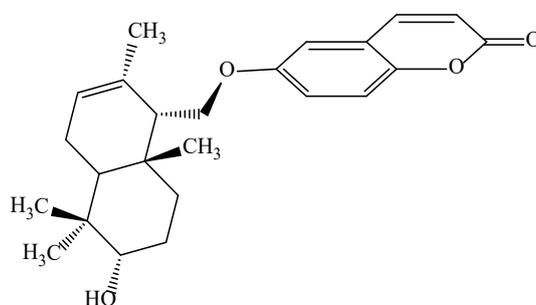
The genus *Ferula* belongs to the Apiaceae family, which comprises over 160 species and is spread throughout the world, including the Mediterranean region, Central Asia, Siberia, China, Afghanistan, Iran, North Africa and the Caucasus [1, 2]. This genus is considered a good source of biologically active compounds including sesquiterpene

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lactones and sesquiterpene coumarins [3, 4]. *F. persica* of four coumarins farnesiferol A, farnesiferone A, badrakemon, gummosin, and from the aerial part – badrakemon, farnesiferone A and farnesiferol A. and the configuration of badrakemin and gummosin was confirmed [5, 7]. A methanolic extract of the dried roots of *Ferula persica* were discovered four sesquiterpene coumarin glycosides, persicaosides A-D and two known phytosterol glucosides, sitosterol 3-O- β -glucoside and stigmaterol 3-O- β -glucoside [8]. Osthole, sitosterol, L-chimgin, L-chimganin were obtained from *F. persica* growing in Azerbaijan [9]. Luthiolin, apigenin, cynaroside, cosmosiin, quercetin, rutin were isolated from the aerial part of *F. persica* [10, 11]. Isolated sesquiterpene coumarins and polysulfides from *F. persica* have cytotoxic, antibacterial, antifungal [12], antileishmaniasis, chemopreventive action against cancer, and an inhibitory effect on lipoxygenase [13]. 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 folk medicine in the treatment of various health disorders, such as cough, asthma, toothache and gastroenteric problems [14–16]. These plants have been used for oleo-gum resin, plant extracts, and essential oils. Nevertheless, have been approved that the essential oils and extracts of different species of *Ferula* also can be used as natural food preservatives due to their antioxidant and antimicrobial activity [17, 18].

Herein, in the framework of our ongoing structural studies [19–23], we report the crystal structure and Hirshfeld surface analysis of the title compound, 7-((6-hydroxy-2,5,5,8a-tetramethyl-1,4,4a,5,6,7,8,8a-octahydronaphthalen-1-yl)methoxy)-2H-chromen-2-one extracted isolation and identification from *Ferula persica*.



Experimental part

The individuality of the isolated compounds was determined on plates "Silufol UV-254", melting point - on the device Stuart SMP10. The plates were developed using iodine vapor, UV lamp light at 254 nm and 365 nm. UV spectra were recorded on a Varian Cary 50 Scan spectrophotometer; IR spectra were recorded on a Bruker ALPHA IR-Fourier spectrometer; 300 MHz for ^1H and 75 MHz for ^{13}C nuclei. Solvent CDCl_3 , chemical shifts calibrated (^1H δ 7.27 ppm, ^{13}C δ 77.0). Chemical shifts are given on the δ scale (ppm). Internal standard TMS. X-Ray structure of single crystal (**1**) obtained using BRUKER APEX-II diffractometer.

Plant materials. *F. persica* plants were collected during the fruiting period on 20.07.2021 in the Republic of Azerbaijan (Jangi village, Gobustan district; 40° 30' 03.04" C 49° 15' 33.11" B 356 m a.s.l.).

Extraction and Isolation. Getting the sum of extractive substances. 2 kg of finely ground air-dried roots of the part were extracted three times with ethyl alcohol ($\geq 99\%$ Merck KGaA, EMD Millipore Corporation) for three days. The extract was filtered off, the alcohol was distilled off on a water bath using a rotary evaporator. Residue 146 g dark brown resin. Yield, 7.3%.

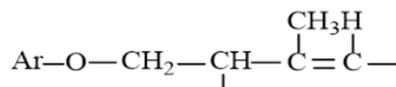
Chromatography sum of extractives. 70 g of the extractives was dissolved in a small amount of alcohol and chromatographed on a column filled with alumina oxide (neutral, grade II act. h = 1.20, d = 4.5 cm) in the ratio of the weight of the extract : weight with alumina oxide – 1 : 10. The volume of each fractions 150 ml. Eluated with hexane (20 fractions), a mixture of hexane and benzene in the ratio 8 : 2, 7 : 3, 6 : 4, 5 : 4, 1 : 1 (40 fractions).

Results and discussion

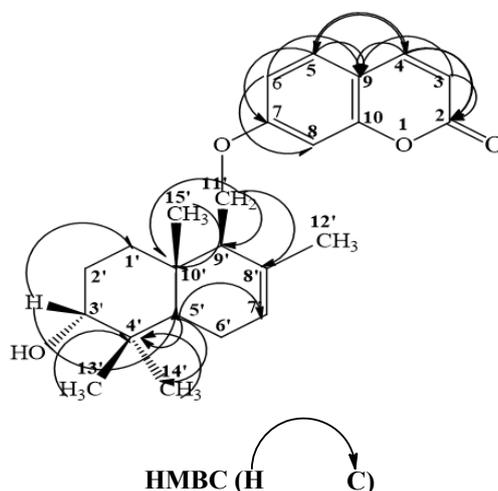
Chromatography and elution with a mixture of hexane and benzene in the ratio (7 : 3) in fractions revealed white crystals of composition $\text{C}_{24}\text{H}_{30}\text{O}_4$ with mp. 143–145 °C (from aqueous alcohol).

UV (λ_{max} , 212, 242, 258, 324 nm) and IR [ν_{max} , cm^{-1} 3479 (OH), 2922, 2855 (CH, CH_2 , CH_3), 1708 (CO- δ -lactone), 1608, 1555, 1507 (aromatic core)] spectral data, was allowing the test substance to be classified as a derivative of sesquiterpenoid coumarin.

The ^1H NMR spectrum of coumarin shows that it is a terpenoid ester of umbelliferone. In the region of δH 6.23–7.65 ppm there are signals from five protons of 7-hydroxy-substituted coumarin. ^1H NMR spectrum is observed from protons of tertiary methyl and vinyl methyl groups - singlets at δH 0.93, 0.94, 0.98, 1.70 ppm (3H each). The terpenoid residue $\text{C}_{15}\text{H}_{25}\text{O}$ contains three methyl groups at saturated quaternary carbon atoms δH (0.93, 0.94, 0.98 ppm 3H each). Signals in the olefinic proton at δH 5.55–5.56 ppm (1H) (broadened singlet) and vinylmethyl groups at δH 1.70 ppm. (3H) (broadened singlet) indicate the presence $\text{HC}=\text{C}-\text{CH}_3$ fragment in the the molecule. The terpenoid residue is attached to the hydroxy group of umbelliferone through a methylene group, two quartets centered at 4.18 and 4.03 ppm. with a general intensity of two proton units of 4.18 ppm. ($J_{\text{gem}} = 9.75$ Hz, $J_{\text{vic}} = 3.45$ Hz) and 4.03 ppm ($J_{\text{gem}} = 9.6$ Hz, $J_{\text{vic}} = 6$ Hz) due to the grouping of ArOCH_2- . The nature of the splitting of the signal of this group indicates that it is located at the secondary atom.



In HMBC ($^1\text{H} \rightarrow ^{13}\text{C}$) spectrum H-11'/C-8, 9, 10; H-3'/C-1', 5'; H-5'/C-4', 7', 14'; H-3/C-2', H-4/C-2, 5, 9; H-5/C-4, 7, 9; H-6/C-8, 9, H-13'/C-4', H-14'/C-4'; H-15'/C-10' give a correlation. In the $^1\text{H}-^1\text{H}$ COSY spectrum of H-3/H-4; H-4/H-3; H-5/H-6; H-6/H-5, H-11'a/H-11'b; H-5'/H-6' give a correlation.



The ^{13}C NMR spectrum, taken with complete suppression of spin-spin interaction with protons, reveals 24 singlet signals, which corresponds to the number of carbon atoms in the elemental composition of the compound. NMR and HSQC correlation data for compound are presented in Table 1.

X-Ray structural commentary

The title compound (Fig. 1) consists of two trans-fused cyclohexane rings (C10–C14/C19 and C14–C19), attached to the chromen-2-one moiety through an oxymethylene bridge. Cyclohexane rings C10–C14/C19 and C14–C19 adopt half-chair and chair conformations [the puckering parameters [24] are $Q_{\text{T}} = 0.5458$ (15) Å, $\theta = 49.18$ (17) $^\circ$ and $\varphi = 273.3$ (2) $^\circ$, and $Q_{\text{T}} = 0.5405$ (15) Å, $\theta = 9.33$ (16) $^\circ$ and $\varphi = 188.4$ (10) $^\circ$], respectively, with the equatorial hydroxyl at C16, and the C11–C12 olefinic bond. All of the bond angles and bond lengths were within the normal range.

In Figure 2, there is the overlap of the previous structure with room data solved and the last structure solved at 100K. The overlap images taken in the (100) and (010) planes show that both molecules have mirror symmetry of each other. Therefore, both molecules are enantiomorphs of each other.

Six compounds similar to the 1,2,3,4,4a,5,8,8a-octahydronaphthalene group are (E)-3-[(1R*,2S*,4aS*,8aR*)-2-(benzo[d][1,3]dioxol-5-yl)-1,2,4a,5,6,7,-8,8a-octahydronaphthalen-1-yl]-N-isobutylacrylamide} (**I**): [25], 3-(4-Bromophenyl)-1-[4-(4-bromophenyl)-3-butene-2-one-1-yl]-2-[3-(2,6,6-trimethyl-1-cyclohexene-1-yl)-2-propene-1-one-1-yl]-1,2,3,4,5,6,7,8-octahydro-8,8-dimethyl-naphthalene (**II**): [26], (+/-)-3 β -Bromo-2 β -methyl-4 $\alpha\beta$ H,8 $\alpha\alpha$ H-decahydronaphthalen-2 α -ol (**III**): [27], (2R,3R,5R)-2-[(2R,3aS,6aR)-2,3,3a,4,5,6a-Hexahydrofuro[2,3-b]furan-2-yl]-5-isopropenyl-2,3-dimethylcyclohexanone (**IV**): [28] (4aR,5S,7R)-5-isopropenyl-7,8,8-trimethyl-2,3,4,4a,-5,6,7,8-octahydronaphthalene-4a-carbonitrile (**V**): [28].

Table 1. ^1H (300 MHz) and ^{13}C (75 MHz) NMR Data of compound (CDCl_3 , δ , ppm (J/Hz))

Atom C	δ_{C}	δ_{H}	Atom C	δ_{C}	δ_{H}
2	161.27 (–C=O)		4'	37.20 (–C–)	
3	112.97 (–CH=)	6.25 (1H, d, J=9.6)	5'	43.38 (–CH–)	1.73 dd*
4	143.44 (–CH=)	7.64 (1H, d, J=9.3)	6'	23.21 (–CH ₂ –)	1.97 (2H, m)
5	128.69 (–CH=)	7.36 (1H, d, J=8.7)	7'	123.21 (–CH=)	5.56 (1H, br. s.)
6	113.18 (–CH=)	6.85 (1H, κ, J _{al} =2.4)	8'	132.51 (>C=)	
7	162.10 (>C=)		9'	53.52 (–CH–)	2.34 (1H, br. s.)
8	101.35 (–CH=)	6.82 (1H s)	10'	35.64 (–C–)	
9	155.94 (>C=)		11'a 11'b	67.08 (–CH ₂ O–)	4.18 (1H, κ, J _{gem} = 9.75, J _{vic} =3.45) 4.03 (1H, κ, J _{gem} = 9.6, J _{vic} = 6)
10	112.45 (>C=)		12'	22.36 (–CH ₃)	1.70 (3H, s)
1'	31.80 (–CH ₂ –)	1.75 (1H, m)	13'	28.09 (–CH ₃)	0.98 (3H, s)
2'	25.15 (–CH ₂ –)	1.69 (1H, m)	14'	21.74 (–CH ₃)	0.94 (3H, s)
3'	75.78 (–CH–)	3.49 (1H, br. s)	15'	14.81 (–CH ₃)	0.93 (3H, s)
		1.54 (br. s, OH)			

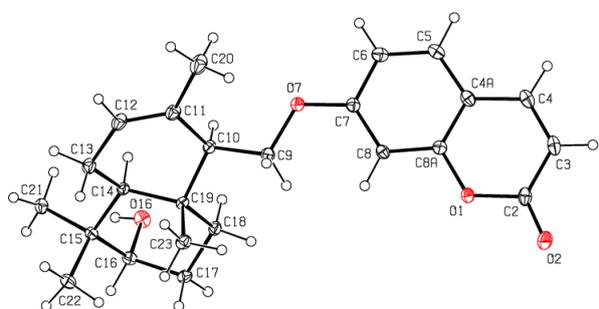


Fig. 1. The molecular structure of the title compound, showing the atom labelling and displacement ellipsoids drawn at the 30% probability level. For clarity, the minor disorder component is not shown

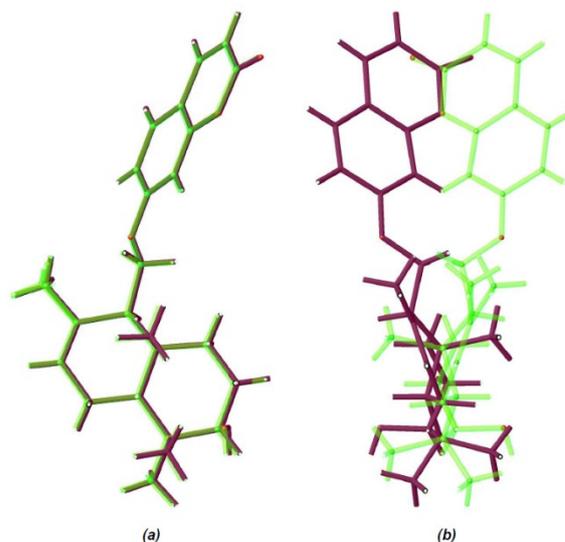


Fig. 2. Overlapping images of two molecules determined at room temperature (green) and 100 K (purple) in (a) (100) plane and (b) (010) plane

In the crystal of (**I**), molecules are linked by N-H \cdots O hydrogen bonds, forming chains propagating along [100]. The chains are linked by pairs of C-H \cdots O hydrogen bonds, involving inversion-related benzodioxole ring systems, forming ribbons lying parallel to (010). There are also C-H \cdots π interactions present within the ribbons, (**II**) is a structure of a new octahydronaphthalene. Disorder in the crystals arises from the coexistence of the two possible puckered conformations of the dimethylcyclohexene rings.

In (**III**), the trans-fused six-membered rings are chair-shaped but somewhat flattened.

(**IV**), and (**V**) are the molecular structures of two chiral cyclohexanones based on R-(–)-carvone. The six-membered ring in (**IV**) is in a chair conformation with the two fused five-membered rings of the furofuranlyl substituent in a *cis* configuration. Compound (**V**) contains a decalin group; one ring has the chair form whilst the other is in a half-boat conformation.

In the title compound studied at room temperature [29], space group: $P 2_1 2_1 2_1(19)$, $a = 6.1621(7) \text{ \AA}$, $b = 18.3914(19) \text{ \AA}$, $c = 18.621(2) \text{ \AA}$, $\alpha = 90.00^\circ$, $\beta = 90.00^\circ$, $\gamma = 90.00^\circ$, $V = 2110.3(4) \text{ \AA}^3$, $Z = 4$, $T = 273(2) \text{ K}$. Cyclohexane rings adopt chair and half-chair conformations, respectively. We determined the title structure which is a new enantiomorph at low temperature (100 K), to precise determination of unit cell parameters, unit volume and bond lengths and bond angles. Space group: $P 2_1 2_1 2_1(19)$, $a = 6.08556(3) \text{ \AA}$, $b = 18.20929(11) \text{ \AA}$, $c = 18.58004(11) \text{ \AA}$, $\alpha = 90^\circ$, $\beta = 90^\circ$, $\gamma = 90^\circ$, $V = 2058.92(2) \text{ \AA}^3$, $Z = 4$, $T = 100 \text{ K}$.

At 100 K, number of measured, independent and observed [$I > 2\sigma(I)$] reflections are 27476, 4382 and 4286, respectively. $R_{\text{int}} = 0.025$. $R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$ and S are 0.029, 0.078 and 1.03, respectively. At room temperature, number of measured, independent and observed [$I > 2\sigma(I)$] reflections are 12098, 2164 and 1680, respectively. $R_{\text{int}} = 0.046$. $R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$ and S are 0.040, 0.099 and 1.04, respectively. $\Delta\rho_{\text{max}}$ and $\Delta\rho_{\text{min}}$ are 0.16 and -0.21 e.\AA^{-3} , respectively. As a result, the values of the bond lengths and angles and hydrogen bonds etc. were also revealed more precisely at low temperature.

Supramolecular features and Hirshfeld surface analysis

In the crystal, the molecules are connected by C-H \cdots O hydrogen bonds along the c-axis, forming layers parallel to the (010) plane, while molecules are linked by O-H \cdots O hydrogen bonds, which generate $R^2_2(6)$ motifs along the a-axis [30], Table 1; Figs. 3, 4 and 5). The C-H \cdots π and van der Waals interactions between these layers stabilize and maintain the structure (Tables 1 and 3, Fig. 6).

Hirshfeld surfaces and two-dimensional fingerprint plots were produced using Crystal Explorer17.5 to quantify the intermolecular interactions in the crystal [31]. The Hirshfeld surface mapped over d_{norm} in the range -0.3269 to $+1.7684$ a.u. Figure 7 show the intermolecular contacts as red-colored spots, which indicate the O-H \cdots O and C-H \cdots O hydrogen bonds. The two-dimensional fingerprint plots are presented in Figure 8. The H \cdots H contacts comprise 58.3% of the total interactions. The O \cdots H/H \cdots O (21.8%) and C \cdots H/H \cdots C (19.7%) interactions also make significant contributions to the total Hirshfeld surface. The O \cdots C/C \cdots O contact contributes 0.2% of the total.

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

C2 \cdots H12	2.82	$3/2 - x, 1 - y, -1/2 + z$
O1 \cdots H16O	2.31	$1/2 + x, 1/2 - y, 1 - z$
O2 \cdots H3	2.41	$1/2 + x, 1/2 - y, -z$
H16O \cdots H22A	2.59	$-1 + x, y, z$
C3 \cdots H20A	3.06	$1 - x, -1/2 + y, 1/2 - z$
H13A \cdots H5	2.35	$1/2 - x, 1 - y, 1/2 + z$
H22A \cdots H16O	2.59	$1 + x, y, z$

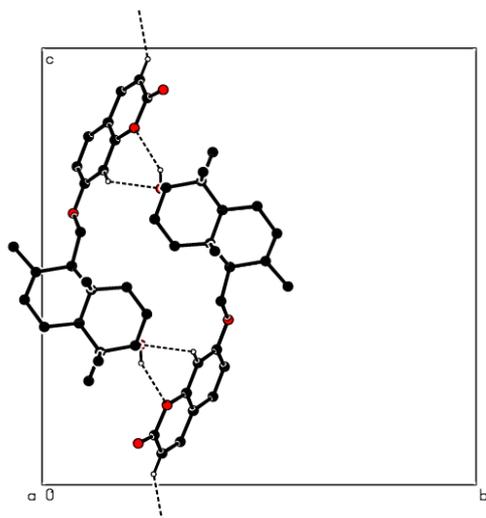


Fig. 3. View of the packing of the title compound with the O-H \cdots O and C-H \cdots O hydrogen bonds along the a-axis. H atoms not involved in hydrogen bonding have been omitted for clarity

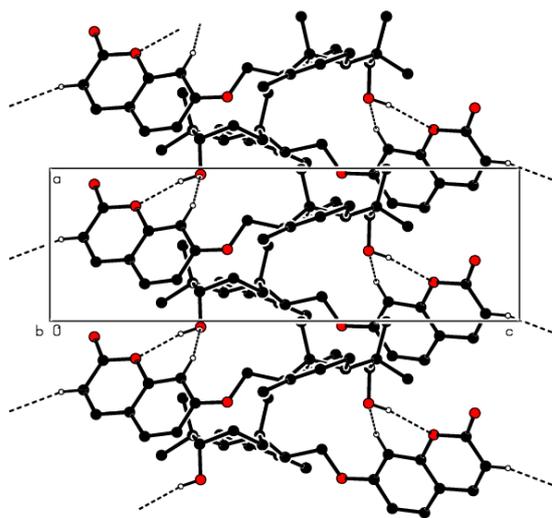


Fig. 4. View of the same interactions of the title compound in Figure 2 along the b-axis

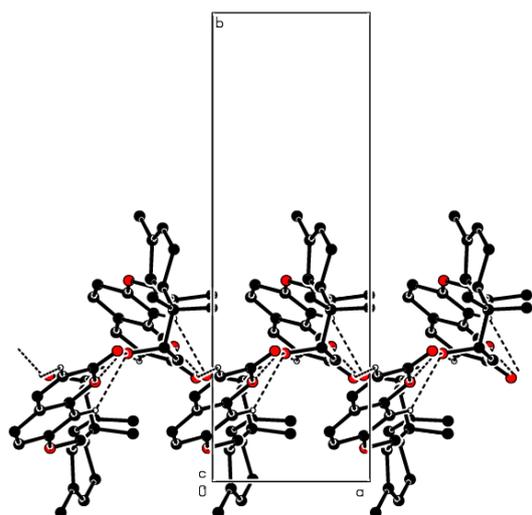


Fig. 5. View of the same interactions of the title compound in Figure 2 along the c-axis

Table 3. Hydrogen-bond geometry (\AA , $^\circ$)

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
O16–H16O \cdots O1i	0.83 (3)	2.31 (3)	3.0711 (15)	152 (3)
C3–H3 \cdots O2ii	0.95	2.41	3.2425 (19)	146
C8–H8 \cdots O16iii	0.95	2.55	3.2515 (18)	130
C12–H12 \cdots Cg1iv	0.95	2.65	3.4295 (17)	140
C16–H16 \cdots Cg2v	1.00	2.70	3.5913(16)	148

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

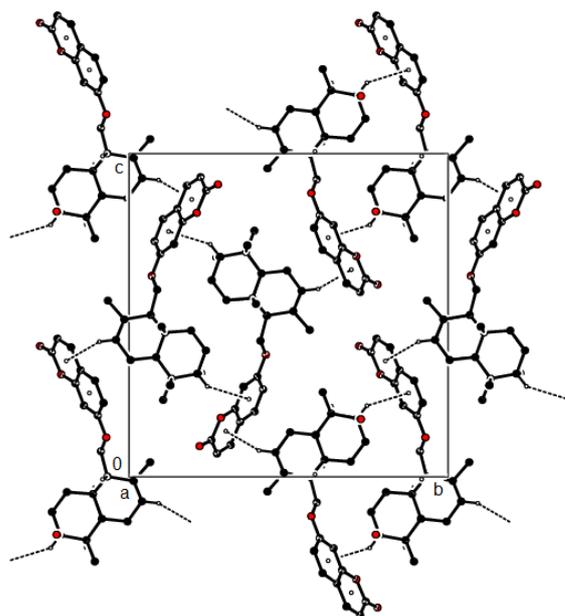


Fig. 6. View of the C-H \cdots π interactions of the title compound along the a-axis

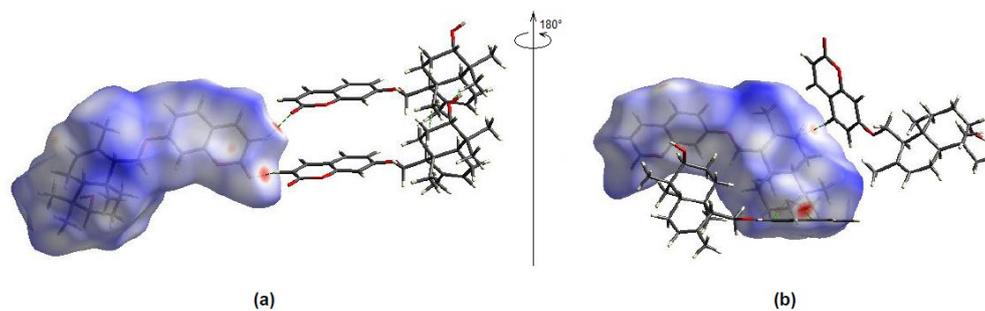


Fig. 7. (a) Front and (b) back sides of the three-dimensional Hirshfeld surface of the title compound mapped over d_{norm} , with a fixed colour scale of -0.3269 to $+1.7684$ a.u.

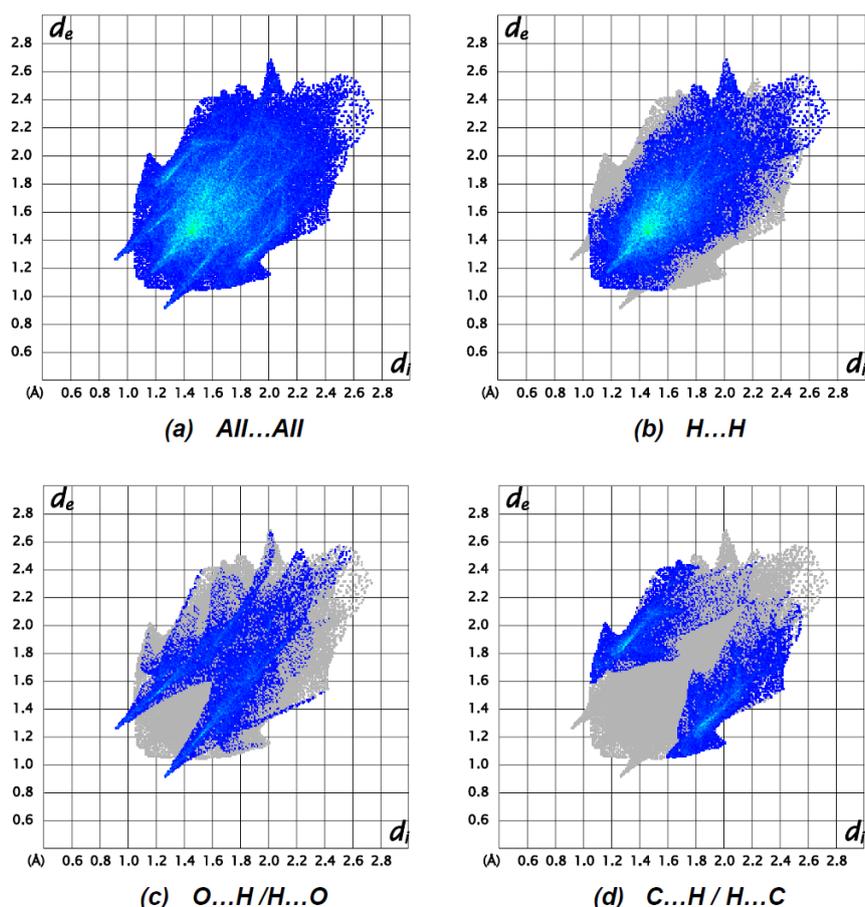


Fig. 8. The two-dimensional fingerprint plots of the title compound, showing (a) all interactions, and delineated into (b) H...H, (c) O...H/H...O and (d) C...H/H...C interactions. [d_e and d_i represent the distances from a point on the Hirshfeld surface to the nearest atoms outside (external) and inside (internal) the surface, respectively]

Crystal data, data collection and structure refinement details are summarized in Table 4. The H atoms of the OH group was located in a difference map, and refined freely [O16–H16 = 0.83 (3) Å]. All H atoms bound to C atoms were positioned geometrically and refined as riding with C–H = 0.95 (aromatic), 0.99 (methylene), 0.98 Å (methyl), and 1.00 Å (methine), with $U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{C})$ for methyl H atoms and $1.2U_{\text{eq}}(\text{C})$ for all others.

Table 4. Experimental details

Parameter	Meaning
<i>l</i>	<i>2</i>
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)	6.08556 (3), 18.20929 (11), 18.58004 (11)
V (Å ³)	2058.92 (2)
Z	4
Radiation type	Cu $K\alpha$
μ (mm ⁻¹)	0.66
Crystal size (mm)	0.10 × 0.05 × 0.03
Diffractometer	XtaLAB Synergy, Dualflex, HyPix
Absorption correction	Gaussian <i>CrysAlis PRO</i> 1.171.42.72a ([32] Rigaku OD, 2022) Numerical absorption correction based on gaussian integration over a multifaceted crystal model.
$T_{\text{min}}, T_{\text{max}}$	0.927, 1.000

End of table 4

1	2
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	27476, 4382, 4286
R_{int}	0.025
$(\sin \theta/\lambda)_{\text{max}}$ (\AA^{-1})	0.634
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.029, 0.078, 1.03
No. of reflections	4382
No. of parameters	261
H-atom treatment	H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{\text{max}}$, $\Delta\rho_{\text{min}}$ (e \AA^{-3})	0.31, -0.18
Absolute structure	Flack x determined using 1802 quotients [(I+)-(I-)]/[(I+)+(I-)] ([33] Parsons & Flack, 2004).

Computer programs: CrysAlis PRO 1.171.42.72a [32], SHELXT 2014/5 [34], SHELXL 2018/3 [35], ORTEP-3 for Windows [36], PLATON [37].

Conclusions

From the ethanolic extract of the roots of *Ferula persica* which is collected during the fruiting phase, $\text{C}_{24}\text{H}_{30}\text{O}_4$ (with m.p. 137–138 °C) was isolated by column chromatography (aluminium oxide as a stationary phase).

Via X-Ray, NMR ^1H , ^{13}C NMR, DEPT, COSY, HSQC, and HMBC methods the structure of 7-((6-Hydroxy-2,5,5,8a-tetramethyl-1,4,4a,5,6,7,8,8a-octahydronaphthalen-1-yl)methoxy)-2H-chromen-2-one (conferol) is confirmed.

The crystal structure of the 7-((6-Hydroxy-2,5,5,8a-tetramethyl-1,4,4a,5,6,7,8,8a-octahydronaphthalen-1-yl)methoxy)-2H-chromen-2-one has been reported previously at room temperature. In contrast to the literature, the crystal structure of the isolated compound determined with greater accuracy at 100 K as a new enantiomorph. The title compound consists of two trans-fused cyclohexane rings, attached to the chromen-2-one moiety through an oxymethylene bridge. Cyclohexane rings adopt half-chair and chair conformations, respectively. The title compound is a new enantiomorph of the one reported.

Supplementary Information

The electronic supplement to the article (DOI: <http://www.doi.org/10.14258/jcprm.20240413074s>) provides additional experimental material that reveals the main points set out in the article

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Conflict of Interest

The authors of this work declare that they have no conflicts of interest.

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