

ФАРМАЦЕВТИЧЕСКАЯ ХИМИЯ

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

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*An ethanol extract was obtained from the underground part of *Ferula persica* Wild., collected during the fruiting phase, which, after the solvent removal, was chromatographed on a column with neutral "Alumina" (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. To confirm the structure, we used X-Ray, NMR ¹H, ¹³C NMR, DEPT, COSY, HSQC, and HMBC methods. It has been previously mentioned about crystalline structure of the given compound C₂₄H₃₀O₄, isolated from *F. persica* roots at room temperature. Unlike literature data in this study crystalline structure was determined with higher precision at 100 K (-173 °C). The compound being investigated consists of octahydronaphthalene fragment, attached to the chromen-2-one fragment through an oxymethylene bridge. The title compound is a new enantiomorph of the one reported previously. In the crystal the molecules are connected by C—H...O hydrogen bonds along the c-axis forming the 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 contribution 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, crystalline structure, enantiomorph, O—H...O hydrogen bonds, C—H...π interactions, redetermination, Hirshfeld surface analysis.

INTRODUCTION

The genus *Ferula* L. 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 lactones and sesquiterpene coumarins [3, 4].

The scientific literature mentions information about the isolation and identification of four coumarins farnesiferol A, farnesiferon A, badrakemon, gummosin from the underground part of *Ferula persica* (*F. persica*), and badrakemon, farnesiferon A and farnesiferol A from the aerial parts of it.

Moreover, the configuration of badrakemin and gummosin was confirmed [5–7].

In a methanolic extract of the dried roots of *F. persica* were discovered four sesquiterpene coumarin glycosides, persicaosides A–D and two known phytosterol glucosides; sitosterol 3-O- β -glucoside and stigmasterol 3-O- β -glucoside [8]. Osthole, sitosterol, L-chimgin, L-chimganin were obtained from *F. persica* growing in Azerbaijan [9]. Luteolin, apigenin, cynaroside, cosmoisin, 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, it has been approved that the essential oils and extracts of different species of *Ferula* can also 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 have also studied the 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 which has been isolated and then identified from the roots of *F. persica*.

MATERIAL AND METHODS

The purity of the isolated compounds was determined by using TLC plates “Silufol UV-254”, by measuring melting point via the device Stuart SMP10. The spots of the plates were detected by using iodine vapor, UV lamp light at 254 nm and 365 nm. As a mobile phase hexane: benzene (8:2) were used. 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 are given on the δ scale (ppm). Internal standard TMS. X-Ray structure of a single

crystal (**1**) obtained by using a 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 was 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. The residue is 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 aluminium oxide – 1 : 10 eluent hexane : benzene (8 : 2).

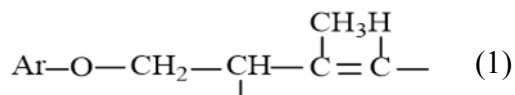
RESULTS AND DISCUSSION

Chromatographic isolation 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. 137–138 °C (from aqueous EtOH 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 allowed 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. Protons of three of tertiary methyl and one vinylmethyl groups - singlets at δH 0,93, 0,94, 0,98. 1,70 ppm (3H each) have been detected from the ^1H NMR spectrum. 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 for 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 of the $\text{HC}=\text{C}-\text{CH}_3$ fragments in 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. ($J_{gem}=9,75$ Hz, $J_{vic}=3,45$ Hz) and 4,03 ppm ($J_{gem}=9,6$ Hz, $J_{vic}=6$ 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. Then the terpenoid part connection contains a fragment (fotmula 1).



In HMBC (1H ^{13}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 (figure 1). In the 1H - 1H 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 (figure 1).

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).

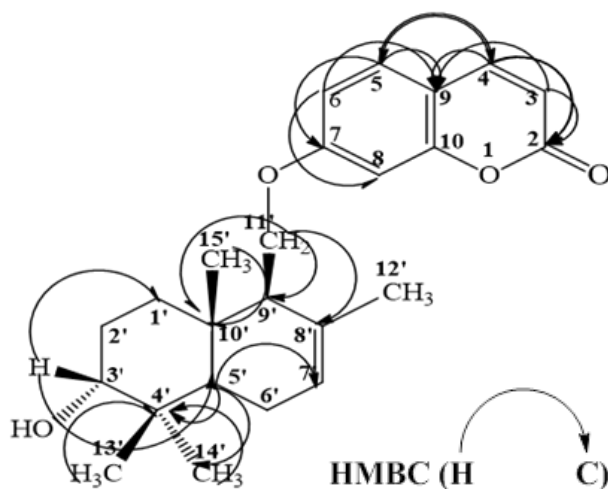


Figure 1. – Structures of compound conferol

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

HSQC					
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 (1H, m)
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 (2H, m)	13'	28,09 (-CH ₃)	0,98 (3H, s)
2'	25,15 (-CH ₂ -)	1,69 (2H, 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)			

The title compound (figure 2) consists of octahydronaphthalene rings (C10–C14/C19 and C14–C19), attached to the chromen-2-one moiety through an oxymethylene bridge. Octahydronaphthalene rings C10–C14/C19 and C14–C19 adopt half-chair and chair conformations for puckering parameters [24]

are $Q_T = 0,5458 (15) \text{ \AA}$, $\theta = 49,18 (17)^\circ$ and $\phi = 273,3 (2)^\circ$, and $Q_T = 0,5405 (15) \text{ \AA}$, $\theta = 9,33 (16)^\circ$ and $\phi = 188,4 (10)^\circ$, 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.

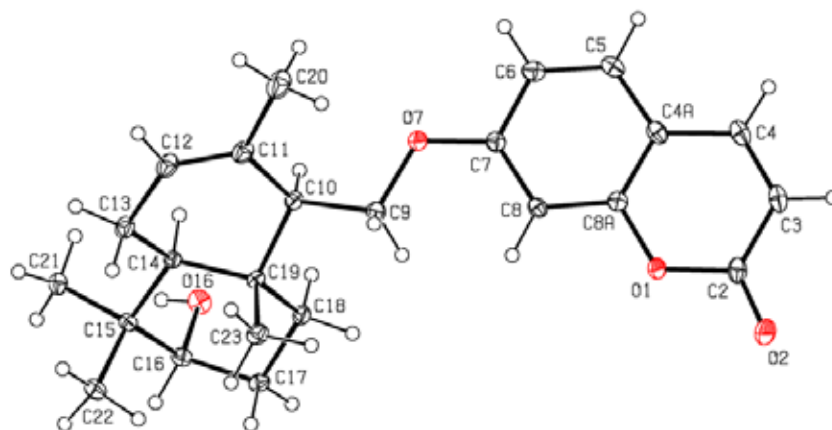


Figure 2. – The molecular structure of the title compound, showing the atom labelling and displacement ellipsoids drawn at the 30% probability level. For clarity, the minor disorder component is not shown

In (figure 3), there is the overlap of the previous structure with room data solved and the last structure solved at 100 K. 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.

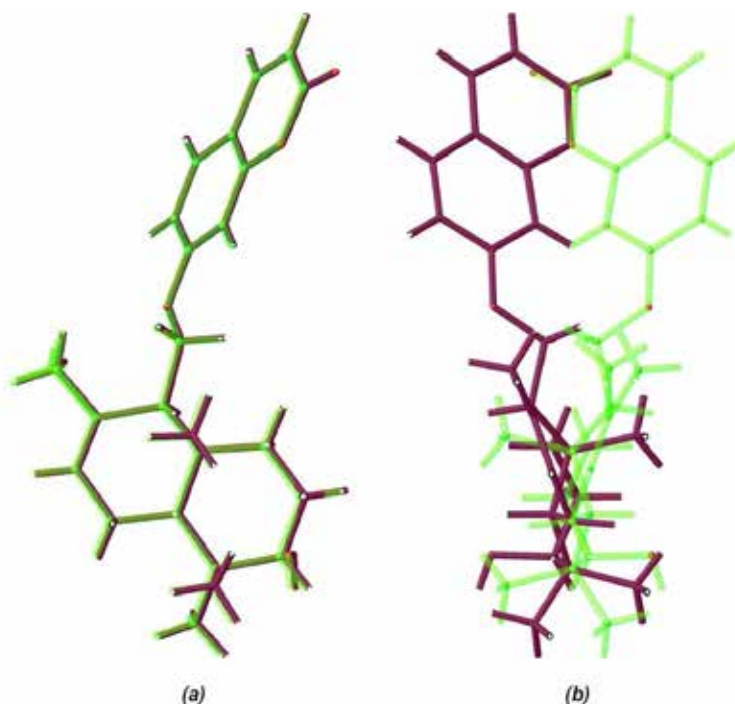


Figure 3. – Overlapping images of two molecules determined at room temperature (green) and 100K (purple) in (a) (100) plane and (b) (010) plane

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],(+/-)-3β-Bromo-2β-methyl-4αβ H,8αH-decahydronaphthalen-2α-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]).

In the crystal of (I), 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, forming ribbons lying parallel to (010). There are also C—H···π interactions present within the ribbons (II) in the 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: $P 2_12_12_1(19)$, $a = 6,1621(7) \text{ \AA}$, $b = 18,3914(19) \text{ \AA}$, $c = 18,621(2) \text{ \AA}$, $\alpha = 90,00^\circ$, $\beta = 90,00^\circ$, $\gamma = 90,00^\circ$, $V = 2110,3(4) \text{ \AA}^3$, $Z = 4$, $T =$

273(2) 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: $P 2_12_12_1(19)$, $a = 6,08556(3) \text{ \AA}$, $b = 18,20929(11) \text{ \AA}$, $c = 18,58004(11) \text{ \AA}$, $\alpha = 90^\circ$, $\beta = 90^\circ$, $\gamma = 90^\circ$, $V = 2058,92(2) \text{ \AA}^3$, $Z = 4$, $T = 100 \text{ K}$.

At 100K, 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 $e.\text{\AA}^{-3}$, 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···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 [30], (table 2, figures 4–6).

The C—H···π and van der Waals interactions between these layers stabilize and maintain the structure (tables 2 and 3, figure 7).

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 de norm in the range -0,3269 to +1,7684 a.u. (figure 8) shows the intermolecular contacts as red-colored spots, which indicate the O—H···O and C—H···O hydrogen bonds.

Table 2. – Hydrogen-bond geometry (\AA , $^\circ$)

D—H···A	D—H	H···A	D···A	D—H···A
O16—H16O···O1i	0,83(3)	2,31(3)	3,0711(15)	152(3)
C3—H3···O2 ⁱⁱ	0,95	2,41	3,2425(19)	146
C8—H8···O16 ⁱⁱⁱ	0,95	2,55	3,2515(18)	130
C12—H12···Cg1 ^{iv}	0,95	2,65	3,4295(17)	140
C16—H16···Cg2 ^v	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) $-x, y+1/2, -z+3/2$.

The two-dimensional fingerprint plots are presented in figure 9. 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.

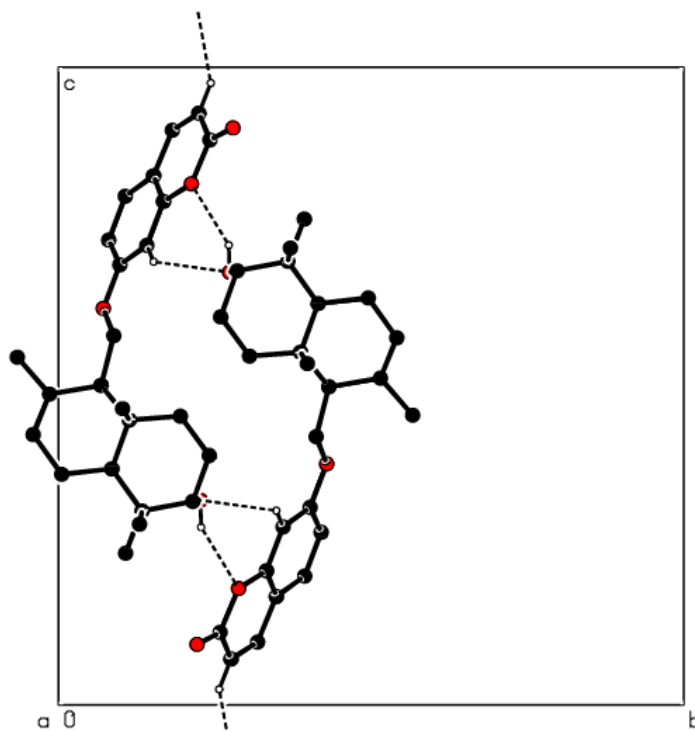


Figure 4. – View of the packing of the title compound with the O—H \cdots O and C—H \cdots O hydrogen bonds along the a-axis. H atoms not involved in hydrogen bonding have been omitted for clarity

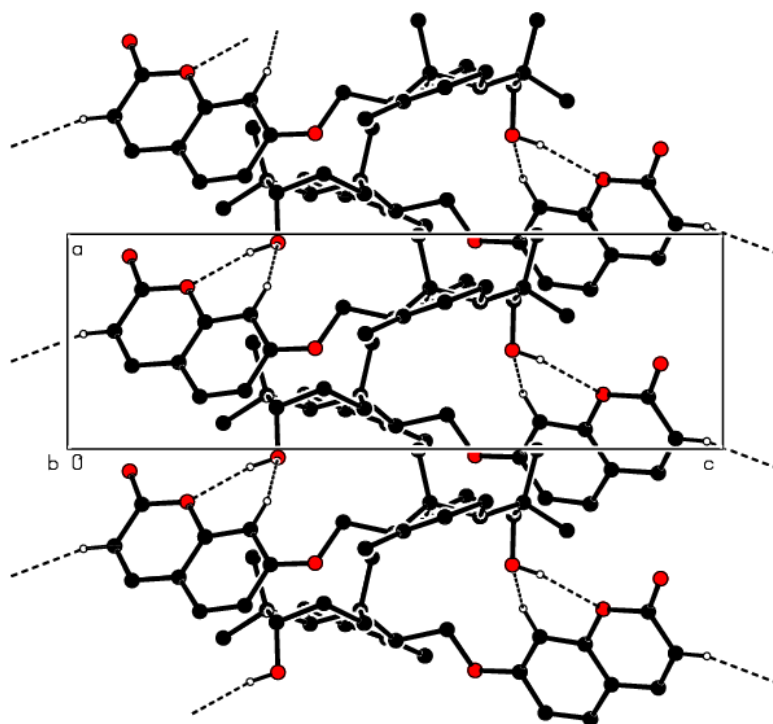


Figure 5. – View of the same interactions of the title compound in figure 3 along the b-axis

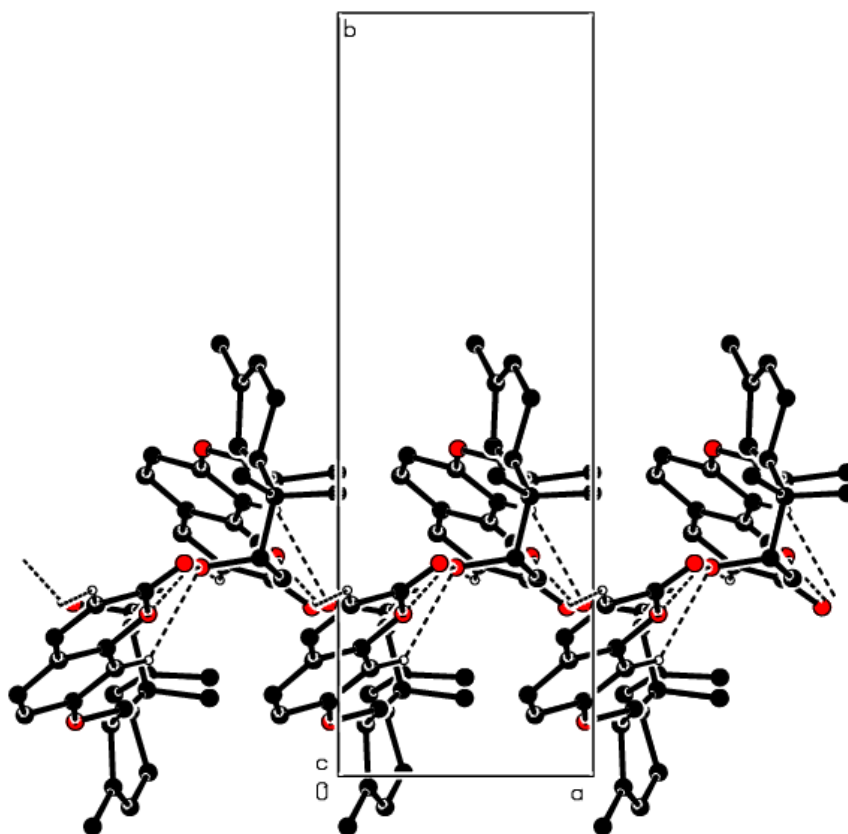


Figure 6. – View of the same interactions of the title compound in figure 3 along the c-axis

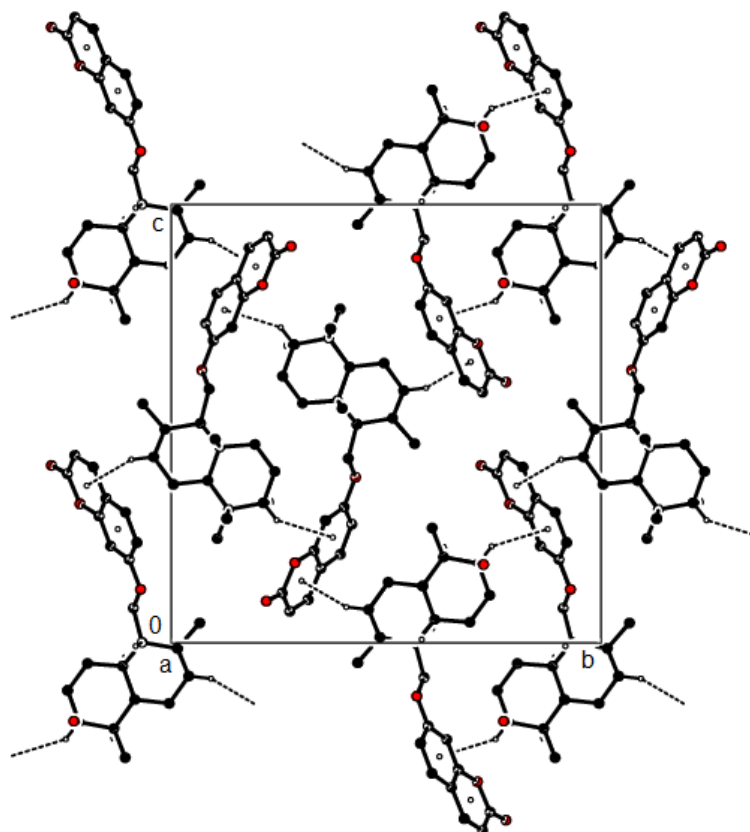
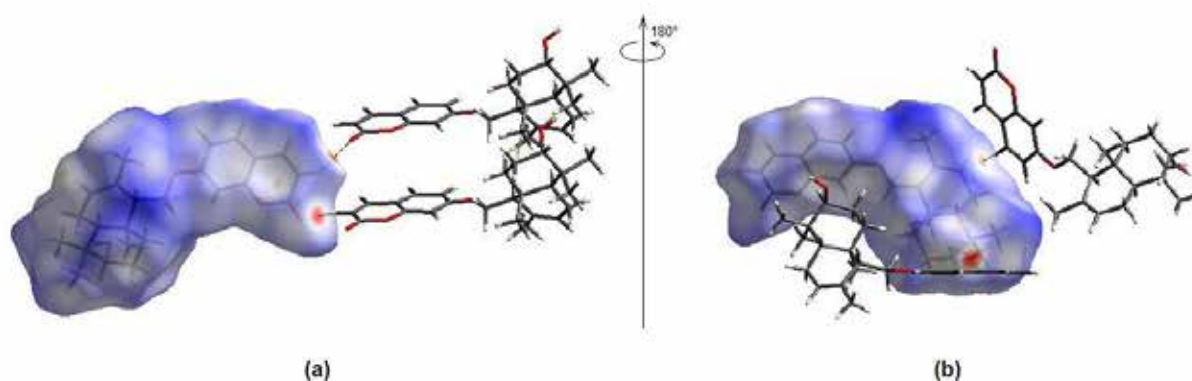
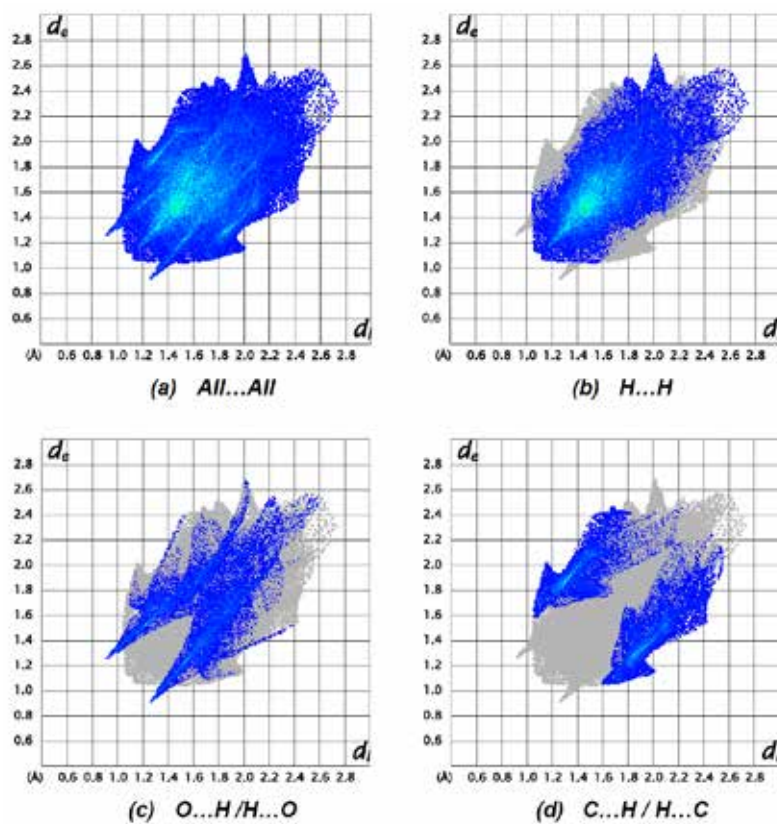


Figure 7. – View of the C—H··· π interactions of the title compound along the a-axis

Table 3. – Summary of short interatomic contacts (Å) in the title compound

C2...H12	2,82	$3/2 - x, 1 - y, -1/2 + z$
O1...H16O	2,31	$1/2 + x, 1/2 - y, 1 - z$
O2...H3	2,41	$1/2 + x, 1/2 - y, -z$
H16O...H22A	2,59	$-1 + x, y, z$
C3...H20A	3,06	$1 - x, -1/2 + y, 1/2 - z$
H13A...H5	2,35	$1/2 - x, 1 - y, 1/2 + z$
H22A...H16O	2,59	$1 + x, y, z$

Figure 8. – (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.Figure 9. – 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 atom 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

Chemical formula	C ₂₄ H ₃₀ O ₄
Mr	382,48
Crystal system, space group	Orthorhombic, P212121
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 α
μ (mm ⁻¹)	0,66
Crystal size (mm)	0,10 × 0,05 × 0,03
Diffractometer	XtaLAB Synergy, Dualflex, HyPix
Absorption correction	Gaussian CrysAlis PRO 1.171.42.72a ([32] Rigaku OD, 2022) Numerical absorption correction based on gaussian integration over a multifaceted crystal model
Tmin, Tmax	0,927, 1,000
No. of measured, independent and observed [I > 2 σ (I)] reflections	27476, 4382, 4286
R _{int}	0,025
(sin θ / λ) _{max} (Å ⁻¹)	0,634
R[F ₂ > 2 σ (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 are treated by a mixture of independent and constrained refinement
$\Delta\rho_{\text{max}}$, $\Delta\rho_{\text{min}}$ (e Å ⁻³)	0,31, -0,18
Absolute structure	Flack x is determined using 1802 quotients [(I ⁺)-(I ⁻)]/[(I ⁺)+(I ⁻)] ([33] Parsons & Flack, 2004).

Computer programs: CrysAlis PRO 1.171.42.72a ([32] Rigaku OD, 2022), [34] SHELXT 2014/5 (Sheldrick, 2015a), [35] SHELXL 2018/3 (Sheldrick, 2015b), ORTEP-3 for Windows [36] (Farrugia, 2012), PLATON [37] (Spek, 2020).

CONCLUSIONS

From the ethanolic extract of the roots of *F. persica* collected during the fruiting phase, C₂₄H₃₀O₄ (with m.p. 137–138 °C) was isolated by column chromatography (aluminium oxide as a stationary phase).

Via X-Ray, NMR ¹H, ¹³C 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.

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РЕЗЮМЕ

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Ю. Б. Керимов, С. Д. Ибадуллаева,
А. Н. Алескерова

**КРИСТАЛЛИЧЕСКАЯ СТРУКТУРА
И АНАЛИЗ ПОВЕРХНОСТИ
ХИРШФЕЛЬДА 7-((6-ГИДРОКСИ-
2,5,5,8А-ТЕТРАМЕТИЛ-1,4,4А,5,6,7,8,8А-
ОКТАГИДРОНАФТАЛИН-1-ИЛ)
МЕТОКСИ)-2Н-ХРОМЕН-2-ОНА,
ВЫДЕЛЕННОГО ИЗ КОРНЕЙ FERULA
PERSICA: НОВЫЙ ЭНАНТИОМОРФ
ПРИ 100 К**

Из подземной части *Ferula persica* Wild., собранной в фазу плодоношения, получали этанольный экстракт, который после удаления растворителя хроматографировали на колонке с нейтральным «оксидом алюминия» (II степень активности) и элюировали гексаном, бензолом и их смесями с постепенно возрастающей полярностью. В результате был выделен 7-((6-гидрокси-2,5,5,8а-тетраметил-1,4,4а,5,6,7,8,8 а-октагидронафталин-1-ил) метокси)-2Н-хромен-2-он (конферол – т.пл. 137–138 °С). Для подтверждения его структуры использовали методы рентгенографии, ЯМР ¹H, ЯМР ¹³C, DEPT, COSY, HSQC и HMBC. Ранее сообщалось о кристаллической структуре названного соединения C₂₄H₃₀O₄, изолированного из корней *F. persica* при комнатной температуре. В отличие от литературных данных, в этом исследовании кристаллическая структура определена с более высокой точностью при 100 К (-173 °С). Исследуемое соединение состоит из фрагмента октагидронафталина, присоединенного к фрагменту хромен-2-он через оксиметиленовый мостик. Титульное соединение является новым энантиомером соединения, о котором сообщалось ранее. В кристалле молекулы соединены водородными связями С—Н···О вдоль оси с, образуя слои, параллельные плоскости (010), в то время как молекулы связаны водородными связями О—Н···О, которые генерируют структур-

ные R22(6) вдоль оси а. Взаимодействия С—Н···π и ван-дер-Ваальса между этими слоями стабилизируют и поддерживают структуру. Анализ поверхности Хиршфельда показывает, что наиболее важный вклад в упаковку кристаллов вносят контакты Н···Н (58,3%), О···Н/Н···О (21,8%) и С···Н/Н···С (19,7%).

Ключевые слова: *Ferula persica*, экстракция, хроматография, спектроскопия, кристаллическая структура, энантиомер, водородные связи О—Н···О, С—Н···π взаимодействия, переопределение, анализ поверхности Хиршфельда.

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