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The Crystal Structures of O,O-Dimethylalpinumisoflavone and 5-O-Methyl-4'-O-(3-methyl-but-2-en-1-yl)alpinumisoflavone

The petrol extract of the rootbark of Milletia Thonningii obtained by column chromatography afforded sixteen different crystalline samples to be isolated. The crystal structures of two of these compounds, O,O-Dimethylalpinumisoflavone (I) and 5-O-Methyl-4'-O-(3-methyl-but-2-en-1-yl)alpinumisoflavone (II) are being reported here. (II) has two independent molecules in the asymmetric unit and differs from (I) in a longer side chain attached to C(15) of the phenyl ring. The structural features of the three molecules in the title compounds are reported and compared. The derivatives, being subject of this article are the first reported crystal structures where the isoflavone fragment is fused to a further six membered ring that results in a tricyclic ring system. The benzopyrone fragments are planar. The dihedral angles between the benzopyrone fragment and the phenyl ring being 55.38(6)° for (I) and 44.75(15)° /44.64(15)° for the respective independent molecules of (II) are within the range of values observed for similar structures.

Keywords: crystal structure

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Introduction

Plant and herbal medicine continues to play an important role in the drug and health delivery system in many developing countries. This is more so in countries where the plant regime is rich in diversity and the income levels of the majority of its rural folk are low to patronise modern drugs and medicines. Unfortunately, these are areas where many diseases are still endemic.

Milletia thonningii tree, a legumnosae indigenous in West and Central Africa, has medicinal and chemical values that needs to be exploited. In the subregions, the plant is used as a laxative, a blood purifier, dewormer, an analgesic and for the treatment of diarrhoea (IRVINE, 1961; ABBIW, 1990). The leaves are toxic to the *Bullinus* snail, the vector for schistomiasis (ABBIW, 1990).

The petrol extract of the rootbark of *M. Thonningii obtained* by column chromatography afforded sixteen different crystalline samples to be isolated. The crystal structures of two of these compounds are being reported here. The chemical identity of the two title compounds, using ^{1}HNMR and IR, had earlier been established by one of us (ASOMANING et al., 1994). The x-ray analyses were carried out to establish their structures and spatial geometries. This forms part of an ongoing project to fully characterize the chemical identity and structures of all the isolated compounds in this plant.

Table 1: Crystal data, details of data collections and structure determinations

	I	II
Formula	$C_{22}H_{20}O_5$	$C_{26}H_{26}O_5$
M_r	364.38	418.47
a (Å)	9.183(4)	19.889(3)
b (Å)	19.044(10)	19.122(3)
c (Å)	10.739(3)	12.007(2)
β (°)	101.70(3)	-
V (Å ^{3})	1839.0(14)	4566.5(13)
Z	4	8
Space group	$P2_1/n$	$P2_1cn$
D*x* (Mg. m^{-3})	1.316	1.217
F(000)	768	1776
Radiation [λ/Å]	CuKα[1.5418]	CuKα[1.5418]
μ (mm^{-1})	0.764	0.679
Crystal size (mm)	0.62 x 0.60 x 0.30	0.48 x 0.38 x 0.28
Diffractometer	STOE 4-Circle	STOE 4-Circle
No. of reflections measured[1)]	3555	4771
No. of independent reflections	3279	4201
h_{min} , h_{max}	-10,10	0,23
k_{min} , k_{max}	-2,22	-22,3
l_{min}, l_{max}	-12,12	0,14
θ_{max}(°)	67.13	67.04
R_{int}	0.007	0.027
Refinement	on F^2	on F^2
No. of parameters refined	249	572
No. of reflections	2813 [|Fo| > 2σ(Fo)]	3104 [|Fo| >2σ(Fo)]
*w*R(F^2) (overall)	0.1150	0.1319
*w*R(F^2) (obs)	0.1098	0.1161
R (overall)	0.0449	0.0642
R (obs)	0.0380	0.0408
Mean shift / esd	0.005	0.003
Electron density (e Å $^{-3}$) maximum	0.187	0.294
Electron density (e Å $^{-3}$) minimum	-0.200	-0.145

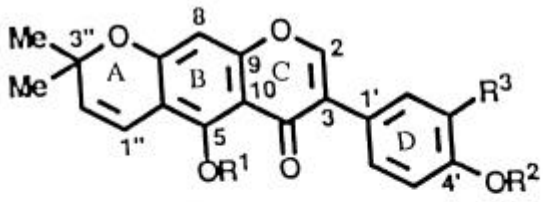
[1)] For (I) a quadrant and for (II) an octant of the limiting sphere were measured (with a few symmetry related ones). A few reflections with $l < 0$ occured for (I) to avoid collision problem of the diffractometer.

[*] Additional material to this paper can be ordered refering to the CCDC nos.141628 and 141629, the names of the authors and the journal citation from the Cambridge Crystallographic Data Centre, 12 Union Road, Cambridge CB2 1EZ, UK.

Experimental and Structure Determination

The rootbark of *M. Thonningii* was collected in July 1998, cut into pieces, air dried in the shade for three weeks and then pulverized. A similar extraction procedure as outlined earlier (ASOMANING et al, 1994) was followed. 2.4 *kg* of the pulverized rootbark was eluted with

petroleum-ether (60-80 °C) for a total of 38 hours. This yielded 130 g of the crude extract. 65 g of this was subjected to column chromatographic separation on silca gel using eluents of increasing polarity gave rise to many fractions. Further purification by TLC afforded 16 different crystalline samples to be obtained among them the two title compounds, O,O-Dimethylalpinumisoflavone (I) mp 138-140 °C and 5-O-Methyl-4'-O-(3-methyl-but-2-en-1-yl)alpinumisoflavone (II) mp 108-110 °C. They both gave colorless crystals (see Scheme 1).



O,O–Dimethylalpinumisoflavone (**I**) 5–O–Methyl–4'–O–(3–methyl–but–2–en –1–yl)alpinumisoflavone (**II**)

	R1	R2	R3
O,O–Dimethylalpinumisoflavone	Me	Me	H
5–O–Methyl–4'–O–(3–methyl–but–2–en–1–yl)alpinumisoflavone	Me	$CH_2CH=C(Me)_2$	H

Scheme 1

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

Ueq = (1/3)ΣΣU_{ij} a_i*a_j*$\boldsymbol{a}_i$ $\boldsymbol{a}_j$

(**I**)

	x	y	z	Ueq
O(1)	0.3140(1)	0.5918(1)	0.0648(1)	0.061(1)
C(2)	0.2294(2)	0.6408(1)	-0.0247(2)	0.063(1)
C(3)	0.2446(2)	0.6214(1)	-0.1595(2)	0.067(1)
C(4)	0.2634(2)	0.5555(1)	-0.1908(2)	0.060(1)
C(5)	0.2797(2)	0.5012(1)	-0.0937(1)	0.051(1)
C(6)	0.3089(2)	0.5228(1)	0.0343(2)	0.051(1)
C(7)	0.3452(2)	0.4756(1)	0.1322(1)	0.053(1)
C(8)	0.3427(2)	0.4054(1)	0.1026(1)	0.049(1)
O(9)	0.3789(1)	0.3616(1)	0.2055(1)	0.059(1)
C(10)	0.3647(2)	0.2921(1)	0.1860(2)	0.057(1)
C(11)	0.3147(2)	0.2608(2)	0.0740(1)	0.050(1)
C(12)	0.3002(2)	0.1836(1)	0.0690(1)	0.050(1)
C(13)	0.4146(2)	0.1411(1)	0.1304(2)	0.056(1)
C(14)	0.4005(2)	0.0692(1)	0.1320(2)	0.058(1)
C(15)	0.2695(2)	0.0381(1)	0.0710(1)	0.054(1)
C(16)	0.1534(2)	0.0798(1)	0.0085(2)	0.059(1)
C(17)	0.1689(2)	0.1512(1)	0.0070(2)	0.058(2)

C(18)	0.2757(2)	0.3040(1)	-0.0415(1)	0.050(1)
C(19)	0.3033(2)	0.3791(1)	-0.0219(1)	0.047(1)
C(20)	0.2792(2)	0.4302(1)	-0.1200(1)	0.048(1)
C(21)	0.2978(2)	0.7113(1)	0.0147(2)	0.084(1)
C(22)	0.0673(2)	0.6367(1)	-0.0141(2)	0.084(1)
O(23)	0.2509(1)	0.4119(1)	-0.2460(1)	0.058(1)
C(24)	0.3688(2)	0.3779(1)	-0.2903(2)	0.066(1)
O(25)	0.2215(2)	0.2776(1)	-0.1456(1)	0.070(1)
O(26)	0.2437(1)	-0.0322(1)	0.0677(1)	0.068(1)
C(27)	0.3631(2)	-0.0767(1)	0.1236(2)	0.079(1)

(**II**)

	x	y	z	Ueq
O(101)	0.3801(2)	0.1273(2)	-0.1110(3)	0.102(1)
C(102)	0.4288(2)	0.0829(2)	-0.0580(4)	0.090(1)
C(103)	0.4005(2)	0.0301(2)	0.0164(4)	0.093(1)
C(104)	0.3358(2)	0.0129(2)	0.0157(4)	0.090(1)
C(105)	0.2903(2)	0.0478(2)	-0.0604(3)	0.069(1)
C(106)	0.3152(2)	0.1034(2)	-0.1235(4)	0.074(1)
C(107)	0.2748(2)	0.1397(2)	-0.1965(3)	0.074(1)
C(108)	0.2087(2)	0.1187(2)	-0.2092(3)	0 .061(1)
O(109)	0.1730(1)	0.1559(1)	-0.2858(2)	0.073(1)
C(110)	0.1071(2)	0.1411(2)	-0.3010(3)	0.067(1)
C(111)	0.0728(2)	0.0915(2)	- 0.2464(3)	0.061(1)
C(112)	0.0000(2)	0.0829(2)	- 0.2684(3)	0.061(1)
C(113)	-0.0426(2)	0.1403(2)	- 0.2749(3)	0.067(1)
C(114)	-0.1107(2)	0.1331(2)	-0.2971(3)	0.074(1)
C(115)	-0.1377(2)	0.0671(2)	-0.3111(3)	0.071(1)
O(115)	-0.2050(2)	0.0519(2)	-0.3287(3)	0.097(1)
C(116)	-0.0960(2)	0.0098(2)	-0.3061(4)	0.082(1)
C(117)	-0.0285(2)	0.0175(2)	-0.2850(3)	0.073(1)
C(118)	0.1085(2)	0.0467(2)	-0.1655(3)	0.066(1)
C(119)	0.1799(2)	0.0641(2)	-0.1487(3)	0.059(1)
C(120)	0.2230(2)	0.0294(2)	-0.0727(3)	0.062(1)
C(121)	0.4638(5)	0.0451(5)	-0.1548(8)	0.179(4)
C(122)	0.4806(5)	0.1297(4)	-0.0057(9)	0.184(5)
O(123)	0.2003(2)	-0.0257(1)	-0.0105(2)	0.076(1)
C(124)	0.1655(3)	-0.0063(3)	0.0880(4)	0.100(2)
O(125)	0.0803(2)	-0.0015(2)	-0.1184(3)	0.100(1)
C(126)	-0.2485(2)	0.1055(3)	-0.3660(4)	0.092(1)
C(127)	-0.3179(2)	0.0763(3)	-0.3722(4)	0.099(1)
C(128)	-0.3424(2)	0.0304(3)	-0.4437(4)	0.093(2)
C(129)	-0.2974(1)	-0.0011(4)	-0.5326(6)	0.145(2)
C(130)	-0.4133(3)	0.0061(5)	-0.4405(7)	0.152(3)
O(201)	-0.3092(2)	0.2461(2)	-0.1914(3)	0.100(1)

C(202)	-0.3448(3)	0.3037(3)	-0.2442(5)	0.098(2)
C(203)	-0.2996(3)	0.3582(3)	-0.2888(5)	0.103(2)
C(204)	-0.2356(3)	0.3635(3)	-0.2601(4)	0.094(2)
C(205)	-0.2058(2)	0.3138(2)	-0.1840(3)	0.077(1)
C(206)	-0.2465(2)	0.2563(2)	-0.1502(3)	0.077(1)
C(207)	-0.2223(2)	0.2062(2)	-0.0791(3)	0.077(1)
C(208)	-0.1563(2)	0.2115(2)	-0.0436(3)	0.072(1)
O(209)	-0.1355(2)	0.1578(1)	0.0229(3)	0.085(1)
C(210)	-0.0710(2)	0.1566(2)	0.0556(4)	0.076(1)
C(211)	-0.0239(2)	0.2037(2)	0.0309(3)	0.066(1)
C(212)	0.0455(2)	0.1957(2)	0.0746(3)	0.066(1)
C(213)	0.0557(2)	0.1766(2)	0.1845(3)	0.067(1)
C(214)	0.1199(2)	0.1694(2)	0.2277(3)	0.072(1)
C(215)	0.1755(2)	0.1817(2)	0.1601(3)	0.073(1)
O(215)	0.2410(2)	0.1784(2)	0.1947(3)	0.092(1)
C(216)	0.1655(2)	0.1994(2)	0.0503(3)	0.075(1)
C(217)	0.1017(2)	0.2066(2)	0.0084(3)	0.074(1)
C(218)	-0.0422(2)	0.2650(2)	-0.0363(3)	0.067(1)
C(219)	-0.1127(2)	0.2663(2)	-0.0740(3)	0.066(1)
C(220)	-0.1404(2)	0.3177(2)	-0.1447(3)	0.071(1)
C(221)	-0.3887(4)	0.3335(4)	-0.1546(8)	0.153(3)
C(222)	-0.3868(5)	0.2674(4)	-0.3316(8)	0.198(5)
O(223)	-0.1023(2)	0.3736(2)	-0.1811(2)	0.083(1)
C(224)	-0.0922(3)	0.4282(2)	-0.1008(5)	0.102(2)
O(225)	-0.0007(2)	0.3106(2)	-0.0578(3)	0.083(1)
C(226)	0.2543(2)	0.1603(3)	0.3050(4)	0.096(1)
C(227)	0.3283(3)	0.1660(3)	0.3219(6)	0.105(2)
C(228)	0.3609(3)	0.1706(3)	0.4125(6)	0.103(2)
C(229)	0.4363(3)	0.1730(4)	0.4176(9)	0.160(3)
C(230)	0.3289(4)	0.1771(7)	0.5195(7)	0.206(5)

Crystals with dimensions 0.62 x 0.60 x 0.30 mm for (**I**) and 0.48 x 0.38 x 0.28 mm for (**II**) were used for all the X-ray investigations on a STOE four circle difractometer using Ni-filtered CuKα radiation. Crystal data and details of the intensity data collection are summarized in Table 1. The unit cell constants were determined by least square refinement of 49 reflections in a 2θ range of 18-80^{o} for (**I**) and 70 reflections in a 2q range of 50-80 ^{o} for (**II**). Repeated measurements of three standard reflections monitored every 90 minutes revealed no significant decay during the data collection for both crystals. The intensity data were corrected for Lorentz and polarization factors but not for absorption. The atomic scattering factors were taken from International Tables for Crystallography, Vol. C.

Both structures were solved by routine application of direct methods (program S*ir* 92, ALTOMARE, 1994), least square refinements with anisotropic displacements for C and O and isotropic displacement parameters for H atoms. Riding model constraints were adopted in improving the H atoms refinements using SHELXL97 (SHELDRICK, 1997). Details of the refinements are included in Table 1 and final atomic parameters in Table 2. Software used to prepare material for publication: PLATON (SPEK, 1990).

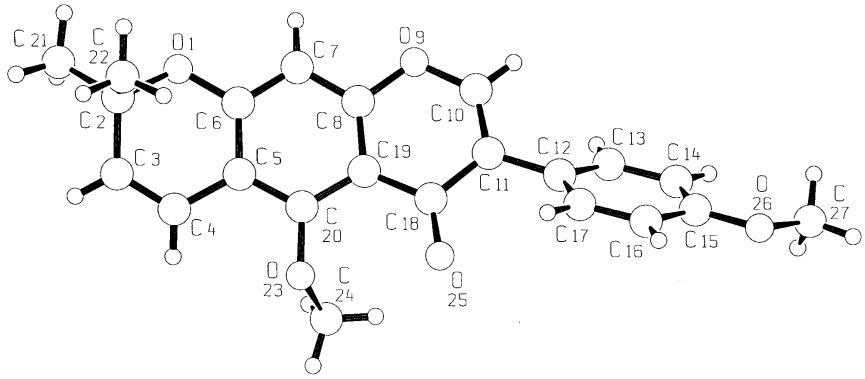


Fig. 1: Molecular structure and atom numbering scheme for the title compound I, SCHAKAL drawing (KELLER, 1988).

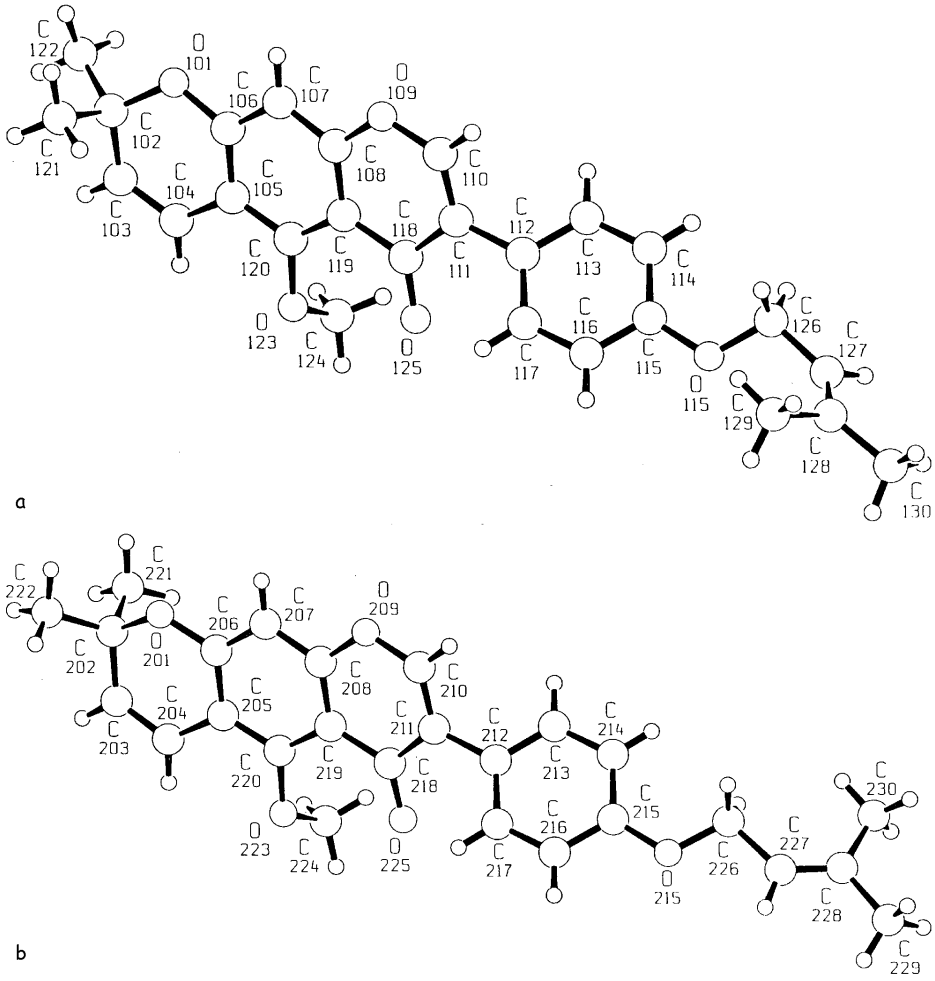


Fig. 2: Molecular structures and atom numbering schemes for the title compound **IIa** and **IIb,** SCHAKAL drawing (KELLER, 1988).

Results and Discussion

The molecular structures of the title compounds O,O-Dimethylalpinumisoflavone (**I**) and 5-O-Methyl-4'-O-(3-methyl-but-2-en-1-yl)alpinumisoflavone (**II**) with their atomic numbering scheme, are shown in Figs. 1 and 2. The title compound **II** consists of two independent molecules in the asymmetric unit and their atoms have been labelled **IIa** and **IIb** accordingly in Fig 2. A graphical superimposition of the three molecules is shown in Fig. 3. A selection of torsion angles is shown in Table 3. A summary of the ring conformations in the title compounds is seen in Table 4.

Compound **II** differs from **I** in a longer side chain attached to C(15) of the phenyl ring being methoxy for **I** and O-3-methyl-but-en-1-yl for **II** containing a C=C double bond C(26)-C(27). Common to both compounds is the isoflavone molecular fragment consisting mainly of the six membered rings B/C and D, where the benzopyrone part of the molecule is fused with the ring A to form the tricyclic ring system A/B/C. The graphical superposition of the three molecules **I**, **IIa** and **IIb** as seen in Fig. 3 shows the geometries of the 6-membered ring systems A/B/C to be very similar. Bond lengths and angles in this region agree within 3σ and need no further discussion. The benzopyrone fragment B/C is planar (see Table 4) which holds also for ring D but not for ring A. This six membered ring is in a half chair conformation (Table 4) in all the three molecules with C(2) and C(3) atoms as the out-of-plane atoms. The Cremer-Pople puckering amplitude Q (CREMER & POPLE, 1975; LUGER & BÜLOW, 1983) indicates a slightly higher puckering of the ring A in **I** than in **IIa** or **IIb**.

Table 3: Selected Torsion Angles (°) in **I**, and in analogous positions in **IIa** and **IIb**

	I	IIa	IIb
C(19)-C(20)-O(23)-C(24)	-65.89(19)	-83.2(4)	-77.3(5)
C(14)-C(15)-O(26)-C(27)	-4.4(2)	1.1(7)	18.7(6)
C(15)-O(15)-C(26)-C(27)	-	-177.1(4)	-175.8(4)
O(15)-C(26)-C(27)-C(28)	-	-70.9(7)	161.8(6)
C(26)-C(27)-C(28)-C(30)	-	1.8(9)	-5.6(11)

Table 4. Analysis of Rings Geometries in (**I**) and (**II**), a) Cremer & Pople Puckering Parameters of Ring A

Compound	Ring	Q, q_2[A]	ϕ, φ_2[°]	θ[°]	Type[1]
I	A	0.352(2)	39.6(3)	68.0(3)	H
IIa	A	0.181(5)	27.5(19)	63.2(16)	H
IIb	A	0.190(5)	28.2(18)	62.3(15)	H

[1]Type : H = Half-Chair Conformer

b) Least-Squares Planes Calculation Results for rings B/C and D

	Ring	σ*	Ring	σ*
I	B/C	0.07(1)	D	0.002(2)
IIa	B/C	0.01(3)	D	0.005(4)
IIb	B/C	0.03(4)	D	0.005(3)

σ* is the average deviation of contributing atoms from the least squares plane

The methoxy group O(23)-C(24) has a gauche arrangement with respect to the torsion C(19)-C(20)-O(23)-C24) in all the three cases giving rise to a short intramolecular contact (~2.3 Å) between one of the protons of the C(24) methyl groups and the carbonyl oxygen O(25). The dihedral angle between the benzopyrone ring B/C and the phenyl ring D is 55.38(6)° for **I** and 44.98(15)° /44.53(14)° for **IIa/b**. The somewhat lower values for **II**, by virtue of the possibility of a longer side chain may show a less flapping tendency(higher potential barrier to free rotation) than **I**. These dihedral angles are nevertheless, in the range observed in the previously determined isoflavone structures from 32° to 75° (LAKSHMI, KUMAR, SENTHILSELVAN & SUBRAMANIAN, 1996; SHOJA , 1992 a,b; BRETON, PRECIGOUX, COURSEILLE & HOSPITAL, 1975; KANEDA, IITAKA & SHIBATA, 1973; ACHARYA, PURANIK, TAVALE & ROW, 1986; BÖCSKEL, SIMON, VARGA & HERMECZ, 1996; MAZUREK *et al*., 1998). Despite the biochemical activity of flavones and isoflavones few are listed in the Cambridge data file. The derivatives being subject of this article are the first ones where the isoflavone fragment is fused to a further six membered ring that results in the ring system A/B/C.

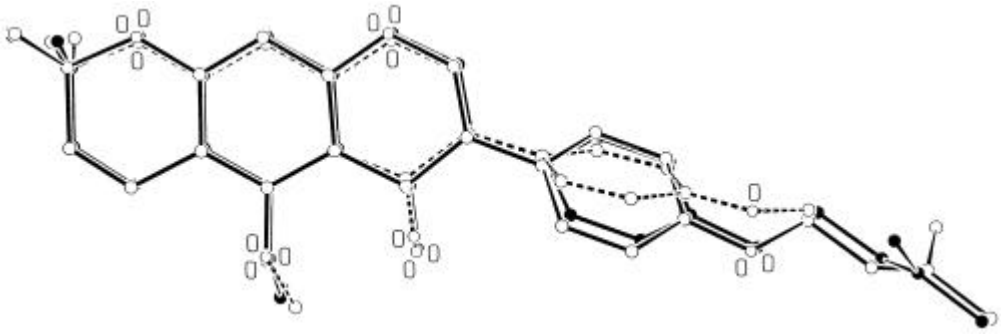


Fig. 3: Graphical superposition of title molecules of **I** (open dashed lines)**,** of **IIa** (closed solid line) and of **IIb (open solid lines**), drawing generated with SCHAKAL.

The observed C=C double bond for C(26)-C(27) being 1.30(1)Å (averaged for **IIa**/**b**) is somewhat shorter than in aurmillone (ACHARYA, PURANIK, TAVALE & ROW, 1986)- an isoflavone with identical moiety. The C=C double bond length in aurmillone is 1.334(8) Å. Notably different are the conformations around the butenyl moiety for the two independent molecules of **II**. With respect to the bond C(26)-C(27) the torsion angles O(15)-C(26)-C(27)-C(28) being -70.9(7)° for the **IIa**, but 161.8(6)° for **IIb** indicate gauche and trans arrangements at this site, being the only major conformational differences between the two independent molecules of **II**.

In the crystal lattices of **I** and **II** the molecular units are linked by weak C-H...O intermolecular contacts. The shortest of these contacts in compound **I** is C(14)-H(14)...O(23)' (the primed atom generated by ½+x, ½-y, ½+z). In the crystal packing of compound **II**, the carbonyl and the methoxy oxygen atoms accept protons to form short intermolecular contacts which are slightly stronger than those for the molecular crystals of **I**. The shortest contacts are observed for C(203)-H(203)...O(123)´(x, 3/2-y, 1/2+z) to be 2.44 Å and 2.55Å for C(122)-H(12E)...O(125)´ (1/2+x, 1-y, 1+z).

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