causes the formation of centrosymmetric dimers. Other contacts are consistent with van der Waals interactions.

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Structure of Visnagin, 4-Methoxy-7-methyl-5H-furo[3,2-g][1]benzopyran-5-one

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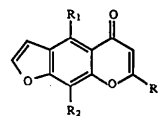
AND A. M. ABD EL-RAHMAN

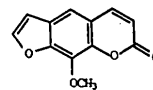Physics Department, Faculty of Science, Ain Shams University, Cairo, Egypt

Abstract. $C_{13}H_{10}O_4$, $M_r = 230.22$, orthorhombic, $P2_12_12_1$, $a = 7.0870$ (6), $b = 9.917$ (1), $c = 14.884$ (2) Å, $V = 1046$ (1) Å^3, $D_x = 1.462$ Mg m^{-3} for $Z = 4$, $\mu = 0.118$ mm^{-1}, $F(000) = 480$, $T = 294$ K, diffractometer data, $\lambda(\text{Mo }K\alpha) = 0.71069$ Å, 1109 data, 817 with $I > 3\sigma(I)$, $R = 0.031$. The furobenzopyranone skeleton of the molecule is planar with r.m.s. deviation 0.032 Å. The methoxy methyl group is 1.16 Å out of plane. There are no short intermolecular contacts.

Introduction. Ammi visnaga, a perennial herbaceous plant that belongs to the family Umbelliferae, grows in the wastelands of the eastern Mediterranean and in the Nile delta. It is one of the more important Egyptian medicinal plants. The seeds have been used since ancient times as a diuretic and as an aid in passing urethral stones. Furobenzopyranones extracted from the seeds, namely visnagin (I), khellin (II) and khellol glucoside (III), have been the object of extensive chemical, pharmacological and clinical research (Huttrer & Dale, 1951). In this paper the structure of visnagin is reported and compared with the structures of khellin, butocrolol hydrochloride (IV) and xanthotoxin (V).

(I):	$R_1 = OCH_3$, $R_2 = H$, $R_3 = CH_3$
(II):	$R_1 = R_2 = OCH_3$, $R_3 = CH_3$
(III):	$R_1 = OCH_3$, $R_2 = H$, $R_3 = CH_2$-β-glucosyloxy
(IV):	$R_1 = OH$, $R_2 = O$-CH_2-$CH(OH)$-CH_2-$\overset{+}{N}H_2$-$C(CH_3)_3$ Cl$^-$

(V)

Experimental. Pale yellow needles from ethanol–water, $0.35 \times 0.30 \times 0.22$ mm crystal, Enraf–Nonius CAD-4 diffractometer, Mo radiation with incident-beam

graphite monochromator, cell parameters from 25 reflections automatically centered in the range $9 \cdot 9 < \theta < 14 \cdot 7°$, 2θ–θ scan at variable θ speed of 16·48–2·06° min^{-1}, θ scan range = $(1 \cdot 5 + 0 \cdot 35 \tan\theta)°$, peak obtained from the central 2/3 of the overall scan, backgrounds from the two outer 1/6 regions of the scan, θ range for data collection 1–25°, index range $h = 0{\rightarrow}8$, $k = 0{\rightarrow}11$, $l = 0{\rightarrow}16$, six standards measured at 200 reflection intervals, average and maximum standard intensity variations of 1·0 and 4·0%, 1163 total data measured, 1109 unique data, 817 data with $I > 3\sigma(I)$, no absorption correction. All crystallographic calculations performed with the *TEXSAN* (Molecular Structure Corporation, 1986) program system on a DEC MicroVax II. Structure solved with the *MITHRIL* direct-methods link, refinement by full-matrix least squares with anisotropic temperature factors for C and O and isotropic terms for H; the ring-linked H atoms were originally placed at positions defined by the C-atom geometry, methyl H atoms were located in a difference electron density map, quantity minimized $= \sum w(F_o - F_c)^2$, $w = 1/\sigma^2(F)$, refinement included a correction for secondary isotropic extinction (Zachariasen, 1968), final S, R and weighted R values 1·88, 0·031 and 0·040, maximum parameter shift/e.s.d. 0·11, minimum and maximum values in the final difference electron density map $-0 \cdot 137$ and 0·140 e Å^{-3}. Atomic coordinates are listed in Table 1.*

Discussion. An *ORTEP* drawing is given in Fig. 1; bond lengths and angles are listed in Table 2. The three-ring framework is essentially planar with r.m.s. deviation 0·032 Å for the 13 atoms. The methoxy carbon is 1·16 Å out of this plane to provide steric clearance between the methyl group and neighboring furobenzopyranone atoms; the torsion angle between the present methyl position and one in which the methyl group would be in the three-ring plane is 88·1 (4)°. A similar out-of-plane distance of 1·329 (2) Å was reported for the C4-linked CH$_3$O methyl group in khellin (II) (Carpy, Hickel & Leger, 1979).

Bond lengths in (I) are normal and within experimental error of the values reported for (II) (Carpy *et al.*, 1979; Beale, 1973), (VI) (Leger, Carpy, Hickel & Laguerre, 1981) and the benzofuran portion of (V) (Stemple & Watson, 1972). Bond angles in the compounds are also similar with the exception of the exocyclic angles at C4 and C5 in (I), (II) and (IV). In the case of (I) and (II) the C4 substituent is methoxy and the C4 and C5 angles indicate that there is substantial steric repulsion between O4—CH$_3$ and O5.

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* Lists of structure factors, anisotropic temperature factors and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 44578 (9 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. *Fractional coordinates, equivalent isotropic temperature factors* (Å^2) *and e.s.d.'s* (*in parentheses*)

$$B_{eq} = \tfrac{4}{3}[\beta(1,1)a^2 + \ldots + 2\beta(2,3)bc\cos\alpha].$$

	x	y	z	B_{eq}
O1	0·2255 (4)	−0·2098 (2)	0·2667 (1)	3·8 (1)
O4	0·2085 (3)	0·1488 (2)	0·0702 (1)	3·4 (1)
O5	0·2569 (4)	0·4061 (2)	0·1459 (1)	4·5 (1)
O8	0·3069 (3)	0·2260 (2)	0·3905 (1)	3·3 (1)
C2	0·1948 (6)	−0·2504 (3)	0·1781 (2)	3·9 (2)
C3	0·1825 (5)	−0·1457 (3)	0·1227 (2)	3·4 (1)
C3A	0·2071 (5)	−0·0261 (3)	0·1768 (2)	2·8 (1)
C4A	0·2529 (4)	0·2002 (3)	0·2291 (2)	2·6 (1)
C4	0·2192 (5)	0·1097 (3)	0·1584 (2)	2·7 (1)
C5	0·2735 (5)	0·3484 (3)	0·2184 (2)	3·0 (1)
C6	0·3146 (5)	0·4212 (3)	0·3000 (2)	3·3 (1)
C7	0·3282 (5)	0·3615 (3)	0·3798 (2)	3·2 (1)
C8A	0·2736 (4)	0·1460 (3)	0·3164 (2)	2·7 (1)
C9	0·2640 (5)	0·0099 (3)	0·3356 (2)	3·2 (1)
C9A	0·2340 (5)	−0·0722 (3)	0·2643 (2)	3·0 (1)
C10	0·3667 (7)	0·4280 (4)	0·4670 (2)	4·5 (2)
C11	0·0298 (7)	0·2003 (5)	0·0419 (3)	4·5 (2)

Table 2. *Intramolecular distances* (Å) *and angles* (°) *with e.s.d.'s in parentheses*

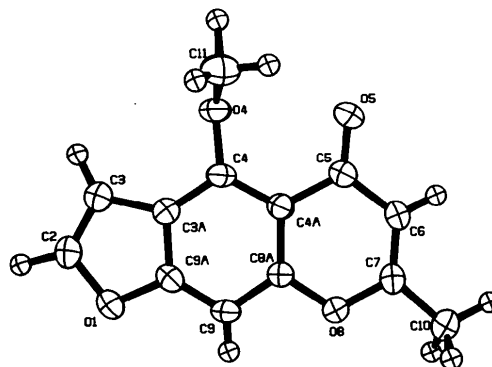|			
O1—C9A	1·367 (3)	C3A—C9A	1·393 (4)
O1—C2	1·397 (4)	C4A—C4	1·403 (4)
O4—C4	1·372 (3)	C4A—C8A	1·415 (4)
O4—C11	1·429 (5)	C4A—C5	1·486 (4)
O5—C5	1·227 (4)	C5—C6	1·442 (4)
O8—C7	1·361 (3)	C6—C7	1·331 (4)
O8—C8A	1·378 (3)	C7—C10	1·481 (4)
C2—C3	1·329 (5)	C8A—C9	1·382 (4)
C3—C3A	1·445 (4)	C9—C9A	1·355 (4)
C3A—C4	1·377 (4)		
C9A—O1—C2	105·7 (2)	O5—C5—C6	121·8 (3)
C4—O4—C11	115·6 (3)	O5—C5—C4A	123·1 (3)
C7—O8—C8A	119·6 (2)	C6—C5—C4A	115·1 (3)
C3—C2—O1	111·8 (3)	C7—C6—C5	122·9 (3)
C2—C3—C3A	106·7 (3)	C6—C7—O8	122·3 (3)
C4—C3A—C9A	119·8 (3)	C6—C7—C10	126·7 (3)
C4—C3A—C3	134·5 (3)	O8—C7—C10	111·0 (3)
C9A—C3A—C3	105·6 (3)	O8—C8A—C9	113·9 (2)
C4—C4A—C8A	117·6 (2)	O8—C8A—C4A	122·3 (2)
C4—C4A—C5	124·7 (3)	C9—C8A—C4A	123·8 (2)
C8A—C4A—C5	117·6 (3)	C9A—C9—C8A	115·6 (2)
O4—C4—C3A	117·6 (2)	C9—C9A—O1	125·8 (3)
O4—C4—C4A	123·1 (2)	C9—C9A—C3A	123·9 (3)
C3A—C4—C4A	119·2 (2)	O1—C9A—C3A	110·3 (2)

Fig. 1. *ORTEP* drawing. The C and O atoms are drawn as 50% probability ellipsoids; H atoms as spheres with B values of 1·5 Å^2.

In (IV), however, the C4 substituent is hydroxy and the angle spreading found in (I) and (II) has all but vanished. These differences are probably less the result of the steric properties of the O-methyl substituents in (I) and (II) than of the intramolecular H···O5 link observed in (IV). In (I), for example, the O4···O5 distance of 2·810 (3) Å is close to the usual 2·8 Å O···O contact distance, whereas the corresponding 2·56 (1) Å value in (IV) is substantially shorter.

The crystal packing is ordinary. There are no intermolecular contacts less than the sum of the appropriate van der Waals radii.

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Structures of 1H-Indole-3-ethylene-3'-methoxysalicylaldimine (3'-Meo-salTPA) and 3-[3'-Azapentyl-3'-en-4'-(2''-hydroxyphenyl)]indole (OHAPH-TPA)

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Abstract. 3'-Meo-salTPA, systematic name 2-[2-(1H-indol-3-yl)ethyliminomethyl]-6-methoxyphenol, $C_{18}H_{18}N_2O_2$, $M_r = 294·34$, triclinic, $P\bar{1}$, $a = 11·932$ (2), $b = 11·590$ (3), $c = 11·401$ (4) Å, $\alpha = 89·94$ (2), $\beta = 101·44$ (2), $\gamma = 101·24$ (2)°, $V = 1514$ (1) Å^3, $Z = 4$, $D_x = 1·29$, $D_m = 1·30$ Mg m^{-3}, $F(000) = 624$, $\lambda(\text{Mo } K\alpha) = 0·71069$ Å, $\mu = 0·92$ mm^{-1}, room temperature, final $R = 0·0505$, $wR = 0·0597$ for 1809 observed reflections. OHAPH-TPA, systematic name 2-{1-[2-(1H-indol-3-yl)ethylimino]ethyl}phenol, $C_{18}H_{18}N_2O$, $M_r = 278·36$, monoclinic, $P2_1/a$, $a = 10·018$ (2), $b = 14·669$ (4), $c = 10·015$ (2) Å, $\beta = 90·54$ (2)°, $V = 1472$ (1) Å^3, $Z = 4$, $D_x = 1·26$, $D_m = 1·29$ Mg m^{-3}, $F(000) = 592$, $\lambda(\text{Mo } K\alpha) = 0·71069$ Å, $\mu = 0·44$ mm^{-1}, final $R = 0·0962$, $wR = 0·1174$ for 1490 observed reflections. The two structures are compared and some structural features are emphasized. The C7A–C8A bond length for both compounds is shorter than a normal aromatic C–C bond. Both structures also show intramolecular and intermolecular hydrogen bonds, the first between the phenolic H and the N of the imino group and the second between the indolic N–H and the phenolic O of the other molecule.

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