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(±)-Byak-angelicol. A Natural Furanocoumarin

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Abstract. $C_{17}H_{16}O_6$, monoclinic, space group $P2_1/c$; eight molecules in a unit cell; $a=11{\cdot}415$ (4), $b=9{\cdot}378$ (4), $c=28{\cdot}81$ (2) Å, $\beta=104{\cdot}56$ (8)°. This racemic natural product was isolated by Pinar (1975, personal communication) from the plant *Magydaris panacifolia*, an endemic Umbellifera of the Iberian peninsula.

Introduction. All 2930 unique reflexions with $\sin\theta/\lambda < 0{\cdot}481$ (no higher scatter was observed) were collected from a yellow crystal of edge $\sim 0{\cdot}3$ mm. A fully automated four-circle diffractometer and monochromated Mo $K\alpha$ radiation were used. 1517 reflexions were considered as observed. The two molecules per asymmetric unit give 46 non-hydrogen atoms with 33 reflexions per atom and produce high standard deviations on the atomic parameters after anisotropic refinement.

Phasing of the 230 largest normalized structure factors (E) was accomplished with *MULTAN* (Main, Woolfson, Declercq & Germain, 1974). The E map giving the best solution located 35 atoms, which gave phases for the 1517 observed reflexions to obtain the remaining atoms of both molecules. Positional and anisotropic thermal parameters were corrected by a least-squares procedure, which converged to an R of

Table 1. *Atomic fractional coordinates of the two independent molecules of* (±)-*byak-angelicol*

For the oxygen atoms the averaged least-squares e.s.d.'s are 10 for x, 14 for y and 4 for z. For the carbon atoms the values are 16, 21 and 6 respectively. Coordinates and standard deviations are multiplied by 10^4.

	x	y	z		x	y	z
O(1)	3548	3280	1164	O(1′)	3111	5385	5745
C(2)	2496	2559	1172	C(2′)	2115	5388	5347
O(2)	2515	1763	1501	O(2′)	2186	4607	5012
C(3)	1421	2856	789	C(3′)	1114	6334	5348
C(4)	1447	3850	441	C(4′)	1200	7236	5733
C(5)	2645	5635	98	C(5′)	2319	8128	6541
O(5)	1625	5901	−260	O(5′)	1381	9020	6508
C(6)	3739	6342	144	C(6′)	3337	8015	6924
C(7)	4759	5933	520	C(7′)	4241	7003	6897
C(8)	4724	4879	856	C(8′)	4193	6078	6494
O(8)	5754	4419	1172	O(8′)	5075	5093	6522
C(9)	3615	4281	825	C(9′)	3192	6259	6132
C(10)	2551	4587	454	C(10′)	2270	7217	6140
O(11)	5733	6677	493	O(11′)	5177	7025	7295
C(12)	5354	7589	90	C(12′)	4916	8066	7601
C(13)	4142	7419	−130	C(13′)	3825	8713	7404
C(14)	1602	5111	−698	C(14′)	1288	9920	6909
C(15)	6046	5110	1648	C(15′)	5888	5438	6211
C(16)	7146	4438	1923	C(16′)	6909	4465	6366
C(17)	8114	5193	2277	C(17′)	8157	4856	6334
O(18)	8256	4851	1796	O(18′)	7822	4836	6794
C(19)	8911	4206	2659	C(19′)	8932	3613	6270
C(20)	8181	6762	2388	C(20′)	8500	6317	6193

Table 2. *Anisotropic temperature factors* (× 10^2) *expressed as* $\exp(-2\pi^2\sum U_{ij}a_i^*a_j^*h_ih_j)$

Average standard deviation is 1.

	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}		U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
O(1)	4	7	4	0	1	0	O(1′)	4	6	4	0	2	−1
C(2)	8	4	3	0	1	0	C(2′)	6	8	4	−2	1	1
O(2)	7	7	7	1	3	2	O(2′)	8	10	4	1	0	−3
C(3)	5	7	6	1	2	0	C(3′)	4	3	9	−1	4	1
C(4)	4	6	6	0	3	−2	C(4′)	4	3	6	0	2	0
C(5)	4	5	4	2	2	0	C(5′)	2	9	3	0	1	2
O(5)	3	7	4	1	0	0	O(5′)	5	8	5	3	0	1
C(6)	4	6	5	0	2	−2	C(6′)	5	3	5	1	3	0
C(7)	4	5	3	0	1	−2	C(7′)	5	4	5	−1	1	0
C(8)	2	5	4	0	1	−2	C(8′)	3	6	4	0	2	0
O(8)	3	8	4	2	0	−2	O(8′)	4	7	5	2	2	1
C(9)	6	4	2	1	1	0	C(9′)	3	2	4	1	1	−1
C(10)	3	5	4	0	0	−2	C(10′)	4	4	3	−2	1	0
O(11)	3	5	6	−2	1	−1	O(11′)	4	6	4	0	0	0
C(12)	8	6	5	1	3	−1	C(12′)	6	7	2	−1	1	−1
C(13)	7	6	4	0	3	−1	C(13′)	6	5	4	−2	0	−1
C(14)	7	8	2	1	1	−3	C(14′)	9	8	6	3	2	−3
C(15)	3	10	4	2	0	−1	C(15′)	4	12	4	3	3	1
C(16)	5	13	4	−1	3	−2	C(16′)	4	12	5	0	2	1
C(17)	4	9	2	−2	2	−1	C(17′)	4	9	4	1	1	0
O(18)	4	11	4	−1	1	0	O(18′)	5	10	4	0	1	−1
C(19)	9	11	6	0	1	3	C(19′)	8	9	8	4	3	−1
C(20)	11	6	8	−1	5	−1	C(20′)	9	6	6	−1	4	2

8·9 %.* The X-RAY 70 system (Stewart, Kundell & Baldwin, 1970) was used for crystallographic calculations. Although the reflexions/parameter ratio is encouraging for isotropic refinement, anisotropic refinement was preferred because it produced better agreement between the geometries of both independent molecules. The atomic coordinates of both molecules are given in Table 1 and the temperature factors in Table 2. Fig. 1 is a perspective drawing of one molecule. For the reason given above, mean standard deviations on bond distances and angles are 0·05 Å and 1·5°. Nevertheless, the average differences between corresponding bond distances and angles for both independent molecules are 0·018 Å and 1·9° respectively. These low figures justify the average values taken in Fig. 2 for the geometry of both molecules. No intermolecular distance less than 3·2 Å was found.

Discussion. To the author's knowledge this is the first furanocoumarin crystal structure reported. The three rings (main plane) of both molecules are almost coplanar, the atoms deviating less than 0·05 Å from the mean plane. Substituents on C(5) and C(8) seem to adapt to the crystal packing. In one molecule C(15) is 1·01 above the main plane and C(14) is 1·41 Å below. However in the second molecule (Fig. 2) C(15) is 1·15 below the plane and C(14) is only 0·08 above, being 3·10 Å from C(13). This C(14) position in the second molecule causes a significant opening of the bond angle at O(5) to 121° (113° for the first molecule). The molecular geometry presents no significant discrepancies from the usual values. In particular, the geometry of the coumarin ring system does not deviate from that found in several other coumarin compounds (Valente, Trager & Jensen, 1975).

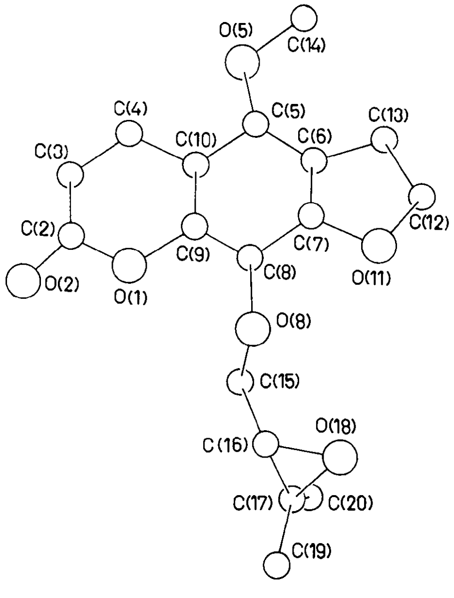

Fig. 1. A perspective drawing of one molecule of byak-angelicol.

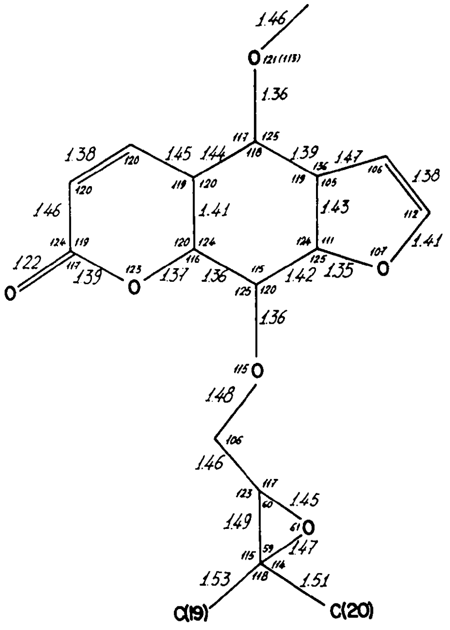

Fig. 2. Averaged bond distances and angles for byak-angelicol. Angles C(16)–C(17)–C(20) and O(18)–C(17)–C(19) are 126 and 112° respectively.

References

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* A list of structure factors has been deposited with the British Library Lending Division as Supplementary Publication No. SUP 31848 (15 pp., 1 microfiche). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 13 White Friars, Chester CH1 1NZ, England.