The C—C distances in the phenyl rings for the title compound, the non-perfluorinated parent compound, and perfluorobenzoic acid are compared in Table 3. This reveals no systematic trends, suggesting perfluorination has little if any effect on the intraannular distances. The average ring distances (averaged over all positions) were also indistinguishable for the three compounds.

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Structure of 7-Methoxy-8-(3-methyl-2-butenyl)coumarin

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Abstract. $C_{15}H_{16}O_3$, $M_r = 244{\cdot}28$, triclinic, $P\bar{1}$, $a = 7{\cdot}488$ (1), $b = 9{\cdot}643$ (2), $c = 10{\cdot}894$ (3) Å, $\alpha = 117{\cdot}34$ (2), $\beta = 87{\cdot}97$ (2), $\gamma = 108{\cdot}32$ (2)°, $V = 657{\cdot}8$ (3) Å^3, $Z = 2$, $D_x = 1{\cdot}233$ Mg m^{-3}, $\lambda(\mathrm{Cu}\,K\alpha) = 1{\cdot}54178$ Å, $\mu = 0{\cdot}61$ mm^{-1}, $F(000) = 260$, room temperature, $R = 0{\cdot}045$ for 1616 observed reflections. The coumarin ring system is almost planar and the methoxy C atom is slightly rotated out of the coumarin mean plane. The 3-methyl-2-butenyl substituent plane makes an angle of 73·6 (3)° with the phenyl ring.

Introduction. Coumarin compounds occur frequently in nature and, similarly to flavonoids, make everyday nutrients of edible plants. A wide spectrum of biological activity of coumarin compounds is known, *e.g.* antithrombotic effect, vasodilating effect on coronary vessels, tonic influence on capillary blood vessels, reduction in blood pressure, antispastic and photosensitizing effect. The mechanism of coumarin biological activity is, for the most part, unknown. This paper presents results of the structural study of the title compound extracted from *Peucedanum ostruthium* L. Koch which shows a weak bacteriostatic effect on Gram-positive bacteria, stimulating influence on respiratory system and immunosuppressive properties (Cisowski, 1987).

Experimental. Crystals grown from methyl alcohol by slow evaporation; crystal system and approximate cell dimensions determined from oscillation and Weissenberg photographs; crystal specimen 0·40 × 0·13 × 0·60 mm, Syntex $P2_1$ four-circle diffractometer, graphite-monochromated Cu $K\alpha$ radiation, θ–2θ scan mode, background and intensity of reflections determined by peak-profile analysis (Lehmann & Larsen, 1974; Jaskólski, 1982); accurate cell parameters refined from setting angles of 15 reflections with $9 \le \theta \le 26°$; max. $(\sin\theta)/\lambda = 0{\cdot}528$ Å^{-1}, $0 \le h \le 9$, $-11 \le k \le 11$, $-13 \le l \le 13$, 2017 unique reflections measured, 1616 considered as observed $[I \ge 2\sigma(I)]$. Lp correction but no absorption correction. Structure solved by direct methods with *MULTAN*78 (Main, Hull, Lessinger, Germain, Declercq & Woolfson, 1978) and *DIRDIF* (Beurskens *et al.*, 1981); the best set of phases enabled location of 16 out of 17 non-H atoms. After two cycles of isotropic least squares a difference synthesis showed the position of missing C(3). Structure refined by full-matrix least squares with *SHELX*76 (Sheldrick, 1976), function minimized $\sum w(|F_o| - |F_c|)^2$, $w^{-1} = \sigma^2(F_o) + 0{\cdot}0002F_o^2$, $\sigma(F_o)$ based on counting statistics.

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Non-H atoms refined anisotropically, all H atoms placed in their theoretical positions and then refined isotropically; empirical isotropic extinction parameter x used to correct F_c according to $F_c' = F_c(1 - xF_c^2/\sin\theta)$, x converged at 0·0637 (5). Three reflections, 100, 011 and $0\bar{2}3$, with high F_c/F_o ratios possibly due to extinction, removed. Final $R = 0{\cdot}045$, $wR = 0{\cdot}068$, $S = 3{\cdot}70$, largest peak in final ΔF map 0·14, largest hole $-0{\cdot}18$ e Å^{-3}. Atomic scattering factors from *International Tables for X-ray Crystallography* (1974). Other computer programs used: *ORTEP* (Johnson, 1976).

Discussion. Final positional parameters and U_{eq} for non-H atoms are given in Table 1.* Fig. 1 shows a stereoview of the molecule, bond lengths and bond angles are listed in Table 2.

The coumarin ring system is nearly planar, with no atomic deviation greater than 0·022 (3) Å from its least-squares plane; the angle between both strictly planar rings is equal to 0·8 (3)°. The benzene ring has equivalent bonds which are within 3σ of the average value of 1·395 Å. The C(2)—O(2) bond, 1·208 (3) Å, has a normal value for a carbonyl group; the double-bond character for C(3)—C(4) of 1·330 (4) Å appears to be retained as for coumarin, 1·344 (5) Å (Gavuzzo, Mazza & Giglio, 1974). The two bonds, C(2)—C(3) and C(4)—C(10), with distances 1·448 (4) and 1·427 (3) Å, respectively, are consistent with the values 1·448 (5) and 1·431 (5) Å in coumarin. The mean value of the two C—O bonds in the heterocyclic ring is 1·375 (2) Å and is in agreement with the C(sp^2)—O(sp^2) distance. The angles C(8)—C(9)—O(1) and C(4)—C(10)—C(5) at the junction of the two rings are smaller and greater than 120°, respectively, and cause the approach of O(1) to C(8) and the removal of C(4) from C(5). The angles around C(2) agree with those of coumarin [117·3 (3), 117·2 (3) and 125·6 (3)°]. The methoxy atom O(11) is coplanar with the benzene ring, while the methyl C(12) is slightly rotated to a position 0·036 (5) Å out of the ring plane. In this case of an almost coplanar methoxy group, the orbitals of the O electron pairs have the greatest overlap with the π orbitals of the aromatic ring system. The 3-methyl-2-butenyl substituent is almost planar, C(14) is 0·010 (2) and C(17) −0·022 (3) Å out of the plane. The angle between the substituent plane and benzene ring is equal to 73·6 (3)°. With this orientation of the substituent there are two short intramolecular contacts: H(131)···O(11) and H(132)···O(1), 2·41 (2) and 2·46 (2) Å, respectively. The double-bond character of C(14)—C(15), 1·327 (3) Å, is retained. In the crystal structure no short intermolecular contacts have been observed. The 3-methyl-2-butenyl substituent has a strong hindering effect and, in consequence, the

Table 1. *Final fractional coordinates and equivalent isotropic thermal parameters* (Å^2)

$$U_{eq} = (U_{11}U_{22}U_{33})^{1/3}.$$

	x	y	z	U_{eq}
C(8)	0·9383 (2)	0·4446 (2)	0·6265 (2)	0·0565 (6)
O(1)	1·0053 (2)	0·2054 (1)	0·4724 (1)	0·0614 (4)
C(9)	0·8714 (2)	0·2813 (2)	0·5223 (2)	0·0576 (6)
C(7)	0·8043 (2)	0·5232 (2)	0·6780 (2)	0·0681 (7)
C(10)	0·6794 (3)	0·1956 (2)	0·4681 (2)	0·0663 (7)
C(6)	0·6123 (3)	0·4406 (3)	0·6259 (2)	0·0780 (8)
C(5)	0·5517 (3)	0·2802 (3)	0·5238 (2)	0·0753 (9)
O(11)	0·8760 (2)	0·6828 (2)	0·7805 (1)	0·0797 (5)
C(15)	1·2476 (2)	0·6224 (2)	0·9383 (2)	0·0580 (6)
O(2)	1·0879 (3)	−0·0093 (2)	0·3330 (2)	0·0933 (6)
C(14)	1·1879 (2)	0·5071 (2)	0·8067 (2)	0·0533 (6)
C(13)	1·1448 (2)	0·5320 (2)	0·6864 (2)	0·0547 (6)
C(12)	0·7482 (5)	0·7710 (4)	0·8415 (4)	0·112 (1)
C(4)	0·6301 (3)	0·0287 (3)	0·3593 (2)	0·0787 (9)
C(2)	0·9590 (3)	0·0450 (3)	0·3675 (2)	0·0745 (7)
C(17)	1·2781 (4)	0·5752 (4)	1·0482 (3)	0·091 (1)
C(16)	1·2881 (5)	0·8025 (3)	0·9904 (3)	0·0833 (9)
C(3)	0·7609 (4)	−0·0427 (3)	0·3105 (3)	0·0833 (9)

Table 2. *Bond lengths* (Å) *and bond angles* (°) *with e.s.d.'s in parentheses*

O(1)—C(2)	1·373 (3)	C(8)—C(9)	1·387 (3)
C(2)—O(2)	1·208 (3)	C(9)—O(1)	1·376 (2)
C(2)—C(3)	1·448 (4)	C(7)—O(11)	1·360 (3)
C(3)—C(4)	1·330 (4)	O(11)—C(12)	1·424 (4)
C(4)—C(10)	1·427 (3)	C(8)—C(13)	1·508 (2)
C(10)—C(9)	1·401 (3)	C(13)—C(14)	1·499 (3)
C(10)—C(5)	1·394 (3)	C(14)—C(15)	1·327 (3)
C(5)—C(6)	1·360 (4)	C(15)—C(16)	1·485 (3)
C(6)—C(7)	1·395 (3)	C(15)—C(17)	1·509 (4)
C(7)—C(8)	1·394 (3)		
O(1)—C(2)—C(3)	117·0 (2)	C(6)—C(7)—C(8)	121·2 (2)
O(1)—C(2)—O(2)	116·5 (2)	O(11)—C(7)—C(6)	124·0 (2)
O(2)—C(2)—C(3)	126·5 (2)	O(11)—C(7)—C(8)	114·9 (2)
C(2)—C(3)—C(4)	121·2 (2)	C(7)—C(8)—C(9)	116·9 (2)
C(3)—C(4)—C(10)	121·5 (2)	C(5)—C(10)—C(4)	125·1 (2)
C(4)—C(10)—C(9)	117·4 (2)	C(7)—C(8)—C(13)	121·1 (1)
C(10)—C(9)—O(1)	120·7 (1)	C(9)—C(8)—C(13)	122·0 (1)
C(9)—O(1)—C(2)	122·3 (1)	C(8)—C(13)—C(14)	111·5 (1)
O(1)—C(9)—C(8)	116·2 (1)	C(13)—C(14)—C(15)	127·2 (2)
C(8)—C(9)—C(10)	123·1 (2)	C(14)—C(15)—C(16)	124·7 (2)
C(9)—C(10)—C(5)	117·5 (2)	C(14)—C(15)—C(17)	120·5 (2)
C(10)—C(5)—C(6)	121·0 (2)	C(16)—C(15)—C(17)	114·8 (2)
C(5)—C(6)—C(7)	120·3 (2)	C(7)—O(11)—C(12)	118·6 (2)

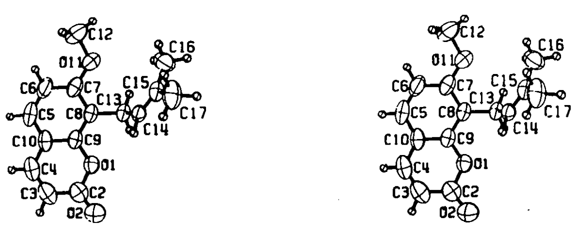

Fig. 1. Stereodrawing of the molecule.

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* Lists of structure factors, anisotropic thermal parameters, torsion angles, H-atom parameters, bond lengths and angles involving H atoms and least-squares-planes data have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51278 (14 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

stacking of molecules is not possible which is the mode of packing of flat coumarin derivatives (Gaultier & Hauw, 1965; Stemple & Watson, 1972; Lai & Marsh, 1974; Ueno & Saito, 1977; Bravic & Bideau, 1978; Bideau, Bravic & Desvergne, 1979; Dall'Acqua, Benetollo & Bombieri, 1981).

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Structure d'Aryl-3 Diphényl-1,4*t* (*E*-Aryl-2 ethényl)-4*c* Cyclohexènes (Aryl = Phényl ou α-Thiényl)

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Abstract. $C_{32}H_{28}$, $M_r = 412{\cdot}57$, monoclinic, $P2_1/c$, $a = 9{\cdot}858\,(3)$, $b = 11{\cdot}951\,(2)$, $c = 20{\cdot}133\,(3)$ Å, $\beta = 100{\cdot}72\,(2)°$, $V = 2351{\cdot}6\,(2)$ Å^3, $Z = 4$, $D_x = 1{\cdot}163$ Mg m^{-3}, $\mu = 3{\cdot}3$ mm^{-1}, λ(Mo $K\alpha$) = 0·71069 Å, $F(000) = 880$, $T = 295$ K, $R = 0{\cdot}053$ for 1245 observed reflections. $C_{28}H_{24}S_2$, $M_r = 424{\cdot}63$, triclinic, $P\overline{1}$, $a = 9{\cdot}944\,(7)$, $b = 11{\cdot}137\,(4)$, $c = 12{\cdot}095\,(4)$ Å, $\alpha = 67{\cdot}58\,(4)$, $\beta = 85{\cdot}73\,(5)$, $\gamma = 66{\cdot}10\,(4)°$, $V = 1126{\cdot}9\,(10)$ Å^3, $Z = 2$, $D_x = 1{\cdot}251$ Mg m^{-3}, λ(Mo $K\alpha$) = 0·71069 Å, $\mu = 20{\cdot}6$ mm^{-1}, $F(000) = 448$, $T = 295$ K, $R = 0{\cdot}060$ for 2962 observed reflections. Both compounds present the same configurational and conformational features with the 3-aryl group being pseudoaxial, a position which limits its steric constraints with the vicinal substituents. For the first compound, the cyclohexene ring adopts a sofa conformation which allows the remaining *gauche* interaction to be reduced. The less intense intramolecular nonbonded interactions of the second compound do not require such a conformational adaptation; therefore, the

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