Table 2. *Bond lengths* (Å) *and angles* (°) *in the* $[B_{10}H_{13}]^-$ *anion*

B(1)—B(2)	1·783 (5)	B(3)—B(8)	1·758 (5)
B(1)—B(3)	1·780 (5)	B(4)—B(8)	1·754 (5)
B(1)—B(4)	1·745 (5)	B(4)—B(9)	1·736 (5)
B(1)—B(5)	1·780 (5)	B(4)—B(10)	1·786 (5)
B(1)—B(10)	1·731 (5)	B(5)—B(6)	1·783 (5)
B(2)—B(3)	1·765 (5)	B(5)—B(10)	1·848 (5)
B(2)—B(5)	1·798 (5)	B(6)—B(7)	1·768 (5)
B(2)—B(6)	1·707 (5)	B(7)—B(8)	2·003 (5)
B(2)—B(7)	1·767 (5)	B(8)—B(9)	1·788 (5)
B(3)—B(4)	1·756 (5)	B(9)—B(10)	1·657 (5)
B(3)—B(7)	1·734 (5)		

B(2)—B(1)—B(3)	59·39 (19)	B(8)—B(4)—B(9)	61·63 (21)
B(2)—B(1)—B(5)	60·61 (20)	B(9)—B(4)—B(10)	56·13 (20)
B(3)—B(1)—B(4)	59·74 (19)	B(1)—B(5)—B(2)	59·78 (20)
B(4)—B(1)—B(10)	61·81 (20)	B(1)—B(5)—B(10)	56·98 (19)
B(5)—B(1)—B(10)	63·48 (21)	B(2)—B(5)—B(6)	56·93 (20)
B(1)—B(2)—B(3)	60·20 (19)	B(2)—B(6)—B(5)	61·99 (21)
B(1)—B(2)—B(5)	59·61 (20)	B(2)—B(6)—B(7)	61·10 (21)
B(3)—B(2)—B(7)	58·80 (19)	B(2)—B(7)—B(3)	60·54 (20)
B(5)—B(2)—B(6)	61·08 (21)	B(2)—B(7)—B(6)	57·74 (20)
B(6)—B(2)—B(7)	61·16 (21)	B(3)—B(7)—B(8)	55·57 (19)
B(1)—B(3)—B(2)	60·41 (19)	B(3)—B(8)—B(4)	59·98 (20)
B(1)—B(3)—B(4)	59·16 (19)	B(3)—B(8)—B(7)	54·43 (18)
B(2)—B(3)—B(7)	60·66 (20)	B(4)—B(8)—B(9)	58·68 (21)
B(4)—B(3)—B(8)	59·89 (20)	B(4)—B(9)—B(8)	59·69 (21)
B(7)—B(3)—B(8)	70·01 (21)	B(4)—B(9)—B(10)	63·46 (22)
B(1)—B(4)—B(3)	61·10 (20)	B(1)—B(10)—B(4)	59·48 (20)
B(1)—B(4)—B(10)	58·71 (20)	B(1)—B(10)—B(5)	59·55 (20)
B(3)—B(4)—B(8)	60·13 (20)	B(4)—B(10)—B(9)	60·40 (21)

readily understood *via* an extended Huckel molecular orbital calculation (Howell, Rossi, Wallace, Haraki & Hoffmann, 1977) on a model of $[B_{10}H_{13}]^-$ derived from the structure of $B_{10}H_{14}$ by μ-H(9,10) deprotonation. The highest occupied molecular orbital of this model is localized on, and bonding between, B(5) and B(6). A minor contribution on B(10) is in phase with that on B(5). Protonation causes partial deoccupation of this orbital (Mitchell & Welch, 1987) thereby lengthening both B(5)—B(6) and B(5)—B(10).

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Structure of *cis*-Bis(pentafluorophenyl)vinylene Bis(pentafluorobenzoate)

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Abstract. $C_{28}F_{20}O_4$, $M_r = 780·31$, triclinic, $P\bar{1}$, $a = 8·200\,(1)$, $b = 9·899\,(2)$, $c = 18·160\,(3)$ Å, $\alpha = 75·75\,(1)$, $\beta = 81·40\,(1)$, $\gamma = 73·47\,(1)°$, $V = 1363·5\,(4)$ Å^3, $Z = 2$, $D_x = 1·90$ g cm^{-3}, λ(Cu $K\alpha$) = 1·5418 Å, $\mu = 19·9$ cm^{-1}, $F(000) = 760$, $T = 300$ K, final $R = 0·0485$, $wR = 0·0621$ for 3699 independent reflections. The compound contains no H atoms. The benzoate phenyl rings of the title compound are rotated 36·2 (14) and 36·4 (5)° relative to the carboxyl group whereas in the non-perfluorinated parent compound these rings are coplanar with the carboxyl groups [torsion angles 6·7 (8) and 0·3 (2)°]. The average torsion angle about the double bond is also larger in the title compound [11·4 (23) *vs* 6·6 (30)°].

Introduction. In our investigations of the electrochemistry of fluorinated compounds, we have discovered that the reduction of decafluorobenzil in the presence of pentafluorobenzoyl chloride produces a mixture of *cis*- and *trans*-bis(pentafluorophenyl)vinylene bis(pentafluorobenzoate). Similar behavior is observed in the parent diphenylvinylene dibenzoate

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Table 1. *Atomic coordinates* (× 10^4) *and equivalent isotropic displacement parameters* ($Å^2 \times 10^3$) *for* $C_{28}F_{28}O_4$

	x	*y*	*z*	U_{eq}*
C(1*a*)	−2409 (3)	6422 (3)	7380 (1)	42 (1)
C(2*a*)	−3876 (3)	6547 (3)	8587 (1)	46 (1)
C(3*a*)	−3172 (3)	5532 (3)	7072 (1)	41 (1)
C(4*a*)	−2783 (3)	4031 (3)	7321 (2)	50 (1)
C(5*a*)	−3490 (4)	3178 (3)	7050 (2)	55 (1)
C(6*a*)	−4633 (4)	3806 (3)	6497 (2)	58 (1)
C(7*a*)	−5035 (3)	5276 (3)	6233 (2)	52 (1)
C(8*a*)	−4328 (3)	6112 (3)	6517 (1)	43 (1)
C(9*a*)	−3625 (3)	6268 (3)	9410 (1)	44 (1)
C(10*a*)	−2438 (4)	5085 (3)	9777 (2)	53 (1)
C(11*a*)	−2242 (4)	4855 (3)	10536 (2)	59 (1)
C(12*a*)	−3238 (4)	5811 (3)	10964 (2)	57 (1)
C(13*a*)	−4423 (4)	6983 (4)	10618 (2)	60 (1)
C(14*a*)	−4606 (4)	7204 (3)	9856 (2)	55 (1)
C(1*b*)	−1640 (3)	7410 (3)	6972 (1)	42 (1)
C(2*b*)	446 (3)	8161 (3)	7408 (1)	43 (1)
C(3*b*)	−1225 (3)	7661 (3)	6139 (1)	43 (1)
C(4*b*)	−144 (3)	6595 (3)	5795 (1)	41 (1)
C(5*b*)	272 (3)	6847 (3)	5021 (2)	51 (1)
C(6*b*)	−409 (4)	8164 (4)	4571 (2)	60 (1)
C(7*b*)	−1488 (4)	9239 (3)	4896 (2)	63 (1)
C(8*b*)	−1877 (4)	8985 (3)	5665 (2)	52 (1)
C(9*b*)	648 (3)	9169 (3)	7853 (1)	42 (1)
C(10*b*)	2034 (3)	9769 (3)	7667 (1)	48 (1)
C(11*b*)	2311 (4)	10658 (3)	8086 (2)	56 (1)
C(12*b*)	1210 (4)	10963 (3)	8702 (2)	52 (1)
C(13*b*)	−178 (3)	10401 (3)	8898 (2)	48 (1)
C(14*b*)	−449 (3)	9515 (3)	8479 (1)	43 (1)
O(1*a*)	−2388 (2)	6168 (2)	8174 (1)	48 (1)
O(2*a*)	−5215 (3)	7055 (3)	8317 (1)	81 (1)
O(1*b*)	−1242 (2)	8334 (2)	7347 (1)	49 (1)
O(2*b*)	1558 (3)	7315 (2)	7137 (1)	61 (1)
F(1*a*)	−1644 (2)	3395 (2)	7842 (1)	72 (1)
F(2*a*)	−3068 (3)	1749 (2)	7304 (1)	80 (1)
F(3*a*)	−5326 (3)	2993 (2)	6224 (1)	88 (1)
F(4*a*)	−6116 (2)	5887 (2)	5689 (1)	77 (1)
F(5*a*)	−4773 (2)	7541 (2)	6248 (1)	58 (1)
F(6*a*)	−1471 (3)	4098 (2)	9397 (1)	88 (1)
F(7*a*)	−1064 (3)	3710 (2)	10867 (1)	98 (1)
F(8*a*)	−3048 (3)	5607 (2)	11696 (1)	83 (1)
F(9*a*)	−5363 (3)	7931 (3)	11030 (1)	97 (1)
F(10*a*)	−5736 (3)	8388 (2)	9541 (1)	93 (1)
F(1*b*)	530 (2)	5300 (2)	6216 (1)	56 (1)
F(2*b*)	1352 (2)	5793 (2)	4703 (1)	69 (1)
F(3*b*)	7 (3)	8402 (3)	3817 (1)	93 (1)
F(4*b*)	−2120 (3)	10538 (2)	4454 (1)	97 (1)
F(5*b*)	−2942 (3)	10046 (2)	5971 (1)	79 (1)
F(6*b*)	3117 (2)	9532 (2)	7064 (1)	68 (1)
F(7*b*)	3647 (3)	11233 (3)	7889 (1)	90 (1)
F(8*b*)	1472 (3)	11811 (2)	9115 (1)	79 (1)
F(9*b*)	−1263 (2)	10709 (2)	9496 (1)	68 (1)
F(10*b*)	−1797 (2)	8959 (2)	8710 (1)	62 (1)

* Equivalent isotropic *U* defined as one third of the trace of the orthogonalized U_{ij} tensor.

Table 2. *Bond lengths* (Å) *and angles* (°) *for* $C_{28}F_{28}O_4$

C(1*a*)—C(3*a*)	1·462 (4)	C(1*a*)—C(1*b*)	1·325 (4)
C(1*a*)—O(1*a*)	1·403 (3)	C(2*a*)—C(9*a*)	1·487 (4)
C(2*a*)—O(1*a*)	1·340 (3)	C(2*a*)—O(2*a*)	1·193 (3)
C(3*a*)—C(4*a*)	1·397 (4)	C(3*a*)—C(8*a*)	1·391 (4)
C(4*a*)—C(5*a*)	1·363 (5)	C(4*a*)—F(1*a*)	1·342 (3)
C(5*a*)—C(6*a*)	1·384 (4)	C(5*a*)—F(2*a*)	1·333 (3)
C(6*a*)—C(7*a*)	1·373 (4)	C(6*a*)—F(3*a*)	1·325 (5)
C(7*a*)—C(8*a*)	1·363 (5)	C(7*a*)—F(4*a*)	1·338 (3)
C(8*a*)—F(5*a*)	1·337 (3)	C(9*a*)—C(10*a*)	1·386 (3)
C(9*a*)—C(14*a*)	1·381 (4)	C(10*a*)—C(11*a*)	1·367 (4)
C(10*a*)—F(6*a*)	1·337 (3)	C(11*a*)—C(12*a*)	1·376 (4)
C(11*a*)—F(7*a*)	1·339 (3)	C(12*a*)—C(13*a*)	1·365 (4)
C(12*a*)—F(8*a*)	1·322 (3)	C(13*a*)—C(14*a*)	1·371 (4)
C(13*a*)—F(9*a*)	1·339 (4)	C(14*a*)—F(10*a*)	1·330 (3)
C(1*b*)—C(3*b*)	1·476 (3)	C(1*b*)—O(1*b*)	1·395 (4)
C(2*b*)—C(9*b*)	1·484 (4)	C(2*b*)—O(1*b*)	1·364 (3)
C(2*b*)—O(2*b*)	1·187 (3)	C(3*b*)—C(4*b*)	1·383 (3)
C(3*b*)—C(8*b*)	1·388 (3)	C(4*b*)—C(5*b*)	1·373 (4)
C(4*b*)—F(1*b*)	1·332 (3)	C(5*b*)—C(6*b*)	1·369 (4)
C(5*b*)—F(2*b*)	1·350 (3)	C(6*b*)—C(7*b*)	1·371 (4)
C(6*b*)—F(3*b*)	1·339 (3)	C(7*b*)—C(8*b*)	1·361 (4)
C(7*b*)—F(4*b*)	1·342 (3)	C(8*b*)—F(5*b*)	1·339 (3)
C(9*b*)—C(10*b*)	1·389 (4)	C(9*b*)—C(14*b*)	1·388 (3)
C(10*b*)—C(11*b*)	1·378 (5)	C(10*b*)—F(6*b*)	1·325 (3)
C(11*b*)—C(12*b*)	1·367 (4)	C(11*b*)—F(7*b*)	1·337 (4)
C(12*b*)—C(13*b*)	1·368 (4)	C(12*b*)—F(8*b*)	1·331 (4)
C(13*b*)—C(14*b*)	1·373 (5)	C(13*b*)—F(9*b*)	1·335 (3)
C(14*b*)—F(10*b*)	1·340 (3)		

C(3*a*)—C(1*a*)—C(1*b*)	125·7 (2)	C(3*a*)—C(1*a*)—O(1*a*)	118·2 (2)
C(1*b*)—C(1*a*)—O(1*a*)	116·0 (3)	C(9*a*)—C(2*a*)—O(1*a*)	111·3 (2)
C(9*a*)—C(2*a*)—O(2*a*)	125·2 (2)	O(1*a*)—C(2*a*)—O(2*a*)	123·5 (2)
C(1*a*)—C(3*a*)—C(4*a*)	121·8 (2)	C(1*a*)—C(3*a*)—C(8*a*)	122·7 (2)
C(4*a*)—C(3*a*)—C(8*a*)	115·5 (3)	C(3*a*)—C(4*a*)—C(5*a*)	123·0 (3)
C(3*a*)—C(4*a*)—F(1*a*)	118·7 (3)	C(5*a*)—C(4*a*)—F(1*a*)	118·3 (2)
C(4*a*)—C(5*a*)—C(6*a*)	119·4 (3)	C(4*a*)—C(5*a*)—F(2*a*)	120·6 (3)
C(6*a*)—C(5*a*)—F(2*a*)	120·0 (3)	C(5*a*)—C(6*a*)—C(7*a*)	119·4 (3)
C(5*a*)—C(6*a*)—F(3*a*)	120·2 (3)	C(7*a*)—C(6*a*)—F(3*a*)	120·4 (3)
C(6*a*)—C(7*a*)—C(8*a*)	120·3 (3)	C(6*a*)—C(7*a*)—F(4*a*)	119·7 (3)
C(8*a*)—C(7*a*)—F(4*a*)	120·0 (3)	C(3*a*)—C(8*a*)—C(7*a*)	122·5 (2)
C(3*a*)—C(8*a*)—F(5*a*)	119·4 (3)	C(7*a*)—C(8*a*)—F(5*a*)	118·2 (2)
C(2*a*)—C(9*a*)—C(10*a*)	123·5 (2)	C(2*a*)—C(9*a*)—C(14*a*)	120·3 (2)
C(10*a*)—C(9*a*)—C(14*a*)	116·2 (2)	C(9*a*)—C(10*a*)—C(11*a*)	122·1 (3)
C(9*a*)—C(10*a*)—F(6*a*)	120·3 (2)	C(11*a*)—C(10*a*)—F(6*a*)	117·6 (2)
C(10*a*)—C(11*a*)—C(12*a*)	120·2 (2)	C(10*a*)—C(11*a*)—F(7*a*)	120·2 (3)
C(12*a*)—C(11*a*)—F(7*a*)	119·5 (3)	C(11*a*)—C(12*a*)—C(13*a*)	119·0 (3)
C(11*a*)—C(12*a*)—F(8*a*)	120·7 (2)	C(13*a*)—C(12*a*)—F(8*a*)	120·3 (3)
C(12*a*)—C(13*a*)—C(14*a*)	120·3 (3)	C(12*a*)—C(13*a*)—F(9*a*)	118·9 (3)
C(14*a*)—C(13*a*)—F(9*a*)	120·7 (2)	C(9*a*)—C(14*a*)—C(13*a*)	122·2 (2)
C(9*a*)—C(14*a*)—F(10*a*)	119·8 (2)	C(13*a*)—C(14*a*)—F(10*a*)	118·0 (3)
C(1*a*)—C(1*b*)—C(3*b*)	125·2 (3)	C(1*a*)—C(1*b*)—O(1*b*)	118·2 (2)
C(3*b*)—C(1*b*)—O(1*b*)	116·5 (2)	C(9*b*)—C(2*b*)—O(1*b*)	110·1 (2)
C(9*b*)—C(2*b*)—O(2*b*)	126·6 (3)	O(1*b*)—C(2*b*)—O(2*b*)	123·3 (3)
C(1*b*)—C(3*b*)—C(4*b*)	121·6 (2)	C(1*b*)—C(3*b*)—C(8*b*)	121·6 (2)
C(4*b*)—C(3*b*)—C(8*b*)	116·9 (2)	C(3*b*)—C(4*b*)—C(5*b*)	121·3 (2)
C(3*b*)—C(4*b*)—F(1*b*)	120·0 (2)	C(5*b*)—C(4*b*)—F(1*b*)	118·7 (2)
C(4*b*)—C(5*b*)—C(6*b*)	120·2 (3)	C(4*b*)—C(5*b*)—F(2*b*)	120·0 (2)
C(6*b*)—C(5*b*)—F(2*b*)	119·9 (2)	C(5*b*)—C(6*b*)—C(7*b*)	119·8 (3)
C(5*b*)—C(6*b*)—F(3*b*)	119·8 (3)	C(7*b*)—C(6*b*)—F(3*b*)	120·4 (3)
C(6*b*)—C(7*b*)—C(8*b*)	119·6 (3)	C(6*b*)—C(7*b*)—F(4*b*)	119·5 (3)
C(8*b*)—C(7*b*)—F(4*b*)	120·9 (3)	C(3*b*)—C(8*b*)—C(7*b*)	122·2 (3)
C(3*b*)—C(8*b*)—F(5*b*)	119·0 (2)	C(7*b*)—C(8*b*)—F(5*b*)	118·7 (2)
C(2*b*)—C(9*b*)—C(10*b*)	119·6 (2)	C(2*b*)—C(9*b*)—C(14*b*)	123·5 (3)
C(10*b*)—C(9*b*)—C(14*b*)	116·8 (3)	C(9*b*)—C(10*b*)—C(11*b*)	121·5 (2)
C(9*b*)—C(10*b*)—F(6*b*)	120·8 (3)	C(11*b*)—C(10*b*)—F(6*b*)	117·6 (3)
C(10*b*)—C(11*b*)—C(12*b*)	119·9 (3)	C(10*b*)—C(11*b*)—F(7*b*)	120·3 (3)
C(12*b*)—C(11*b*)—F(7*b*)	119·8 (3)	C(11*b*)—C(12*b*)—C(13*b*)	120·1 (3)
C(11*b*)—C(12*b*)—F(8*b*)	120·4 (3)	C(13*b*)—C(12*b*)—F(8*b*)	119·4 (2)
C(12*b*)—C(13*b*)—C(14*b*)	119·7 (2)	C(12*b*)—C(13*b*)—F(9*b*)	120·2 (3)
C(14*b*)—C(13*b*)—F(9*b*)	120·1 (3)	C(9*b*)—C(14*b*)—C(13*b*)	121·9 (3)
C(9*b*)—C(14*b*)—F(10*b*)	120·8 (3)	C(13*b*)—C(14*b*)—F(10*b*)	117·2 (2)
C(1*a*)—O(1*a*)—C(2*a*)	118·0 (2)	C(1*b*)—O(1*b*)—C(2*b*)	116·8 (2)

system (Guirado, Barba, Manzanera & Velasco, 1982; Cheek & Horine, 1984). Since the perfluorinated derivatives have not previously been prepared, it was considered necessary to establish unambiguously the configurations of these compounds. An X-ray structure of the parent (non-fluorinated) *cis* isomer has appeared (Cantrell & Silverton, 1979) affording an opportunity for an interesting comparison of the effects of fluorine substitution on molecular geometry. This is the first structure reported for an ester of perfluorobenzoic acid.

Experimental. The title compound was prepared by electrolysis of a mixture of decafluorobenzil (Chambers, Clark & Spring, 1972) and pentafluorobenzoyl chloride (1:2 molar ratio) in acetonitrile/0·1 *M* tetraethylammonium perchlorate. The electrolysis was carried out at a potential 100 mV negative to the peak potential for decafluorobenzil (Cheek & Horine, 1984). Following electrolysis, the solvent was evaporated on a rotary evaporator, and the residue extracted with benzene. Both *cis* and *trans* isomers of the title compound were prepared in the electrolysis (1·7:10 *trans*:*cis*); however, the *cis* isomer crystallized preferentially from benzene. Further purification was achieved by recrystallization from *n*-hexane. The clear, colorless crystal used in this study, 0·42 × 0·66 × 0·92 mm, was obtained by slow evaporation of solvent from a solution of the *cis* isomer in hexane. Automated Nicolet *R*3*m* diffractometer with incident-beam monochromator. 25 centered reflections within $50 \leq 2\theta \leq 60°$ used for determining lattice parameters. $[(\sin\theta)/\lambda]_{\max} = 0{\cdot}55\,\text{Å}^{-1}$, range of *hkl*: $0 \leq h \leq 9$, $-10 \leq k \leq 10$, $-19 \leq l \leq 19$. Standards 2,0,10, 0,4,10, 160 monitored every 60 reflections with random variation of 3·3% over data collection, $\theta/2\theta$ mode, scan width $[2\theta(K\alpha_1) - 1{\cdot}0]$ to $[2\theta(K\alpha_2) + 1{\cdot}0]°$, scan rate a function of count rate (4° min^{-1} minimum, 15° min^{-1} maximum), 4013 reflections measured, 3699 unique, $R_{\text{int}} = 0{\cdot}017$, 3404 observed with $F_o > 3\sigma(|F_o|)$. Data corrected for Lorentz, polarization and absorption effects (azimuthal scans), 0·960/0·611 maximum/minimum transmission factors. Structure solved by direct methods. The least-squares refinement used program *SHELXTL* (Sheldrick, 1980). $\sum w(|F_o| - |F_c|)^2$ minimized where $w = 1/[\sigma^2(|F_o|) + g(F_o)^2]$, $g = 0{\cdot}000200$. Secondary-extinction parameter $p = 0{\cdot}144\,(6)$ in $F_c^* = F_c/[1{\cdot}0 + 0{\cdot}002p(F_o)^2/\sin 2\theta]^{0.25}$. There were 470 parameters refined including atom coordinates and anisotropic thermal parameters for all atoms. $(\Delta/\sigma)_{\max} = 0{\cdot}005$, $R = 0{\cdot}0485$, $wR = 0{\cdot}0621$, $S = 3{\cdot}22$ for all reflections. The structure was initially refined with 3404 observed reflections $[F_o > 3\sigma(|F_o|)]$. This gave $R = 0{\cdot}0465$, $wR = 0{\cdot}0634$ and $S = 3{\cdot}44$. The crystal suffered from severe extinction, which probably accounts for the high goodness of fit. This was evident in the analysis of the variance as a function of $\sin\theta$ (the low-angle reflections gave substantially poorer agreements than the high-angle reflections). Various weighting schemes gave no improvement. The final refinement reported herein utilized all data. This had no effect on atomic parameters (no shifts greater than 1σ relative to the conventional refinement) but did lower *wR*, *S* and some of the standard deviations. Final difference Fourier map excursions 0·28 and −0·40 e Å^{-3}. Atomic scattering factors from *International Tables for X-ray Crystallography* (1974).* Atom numbering for Table 1, atom coordinates, and Table 2, bond distances and bond angles, follows that shown in Fig. 1.

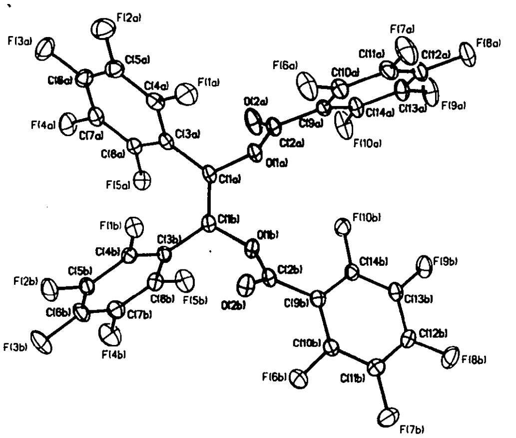

Fig. 1. Molecular structure of the title compound (30% probability thermal ellipsoids).

Table 3. *Comparison of phenyl* C—C *distances* (Å) *for three compounds*

Compound	C(1)—C(2)^a	C(2)—C(3)	C(3)—C(4)	Reference
Title compound	1·388 (5)^b	1·369 (6)	1·372 (6)	This work
Diphenylvinylene dibenzoate	1·388 (6)	1·385 (8)	1·374 (9)	Cantrell & Silverton (1979)
Perfluorobenzoic acid	1·392 (3)	1·384 (2)	1·371 (1)	Benghiat & Leiserowitz (1972)

Notes: (*a*) 1 refers to the substituted carbon of the phenyl ring; C(1)—C(2) is averaged over all such distances in the molecule. (*b*) Standard deviations are for the sample rather than the mean.

Discussion. The structural analysis confirms the chemical tests (GTC, unpublished results) which indicated the *cis* geometry about the double bond. The most surprising feature is the large rotation of the pentafluorobenzoate phenyl rings relative to the carboxyl moieties. In the hydrogenated parent compound these are essentially coplanar. By comparison this angle is 29·8° in pentafluorobenzoic acid (Benghiat & Leiserowitz, 1972) whereas most benzoates are planar (DSD, unpublished results). The origin of this nonplanarity is uncertain. The van der Waals radius of fluorine (1·50–1·60 Å) is only slightly larger than that for hydrogen (1·20–1·45 Å) (Huheey, 1983). Given a longer covalent radius for fluorine (0·71 *vs* 0·37 Å), this twisting may be from increased steric repulsions. Photoelectron data (Brundle, Robin & Kuebler, 1972) show that the π orbitals of perfluorobenzene are shifted by about 0·50 e V to more negative (*i.e.* stable) values relative to those of benzene. If this causes a significant mismatch in the carboxyl and phenyl π-orbital energies, delocalization of the phenyl ring and carboxyl group electrons could be greatly diminished, giving less incentive for them to be coplanar. This question is under further investigation.

The phenyl rings bonded to the alkene carbons [C(1*a*) and C(1*b*)] are rotated by 29·4 and 47·1° relative to the plane containing the double bond. In the parent (non-fluorinated) compound, the corresponding angles are 49·0 and 59·1°. The substantial variation suggests a shallow energy well for rotation of this phenyl ring.

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* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51505 (16 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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. $C_{15}H_{16}O_3$, $M_r = 244·28$, triclinic, $P\bar{1}$, $a = 7·488$ (1), $b = 9·643$ (2), $c = 10·894$ (3) Å, $\alpha = 117·34$ (2), $\beta = 87·97$ (2), $\gamma = 108·32$ (2)°, $V = 657·8$ (3) Å^3, $Z = 2$, $D_x = 1·233$ Mg m^{-3}, λ(Cu $K\alpha$) = 1·54178 Å, $\mu = 0·61$ mm^{-1}, $F(000) = 260$, room temperature, $R = 0·045$ 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 $9 \leq \theta \leq 26°$; max. $(\sin\theta)/\lambda = 0·528$ Å^{-1}, $0 \leq h \leq 9$, $-11 \leq k \leq 11$, $-13 \leq l \leq 13$, 2017 unique reflections measured, 1616 considered as observed $[I \geq 2\sigma(I)]$. 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 $\sum w(|F_o| - |F_c|)^2$, $w^{-1} = \sigma^2(F_o) + 0·0002F_o^2$, $\sigma(F_o)$ based on counting statistics.

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