dans le Tableau 1,* le Tableau 2 donne les longueurs et les angles des liaisons pour les atomes non-hydrogène.

Au vu de ces résultats, et en dépit du faible nombre de données disponibles dû à la petite taille de l'échantillon, on peut remarquer une bonne homogénéité dans la géométrie globale du motif [dispersion des liaisons C—C aromatiques: 1,383 (7) ± 0,007 Å pour le cycle C1 à C6, 1,386 (7) ± 0,008 Å pour le cycle C10 à C15] et en particulier la géométrie caractéristique de la partie 'munchnone' intermédiaire entre les deux formes limites (2a) et (2b) du schéma: ce cycle est parfaitement plan du fait de son caractère conjugué important. Cependant cette conjugaison ne s'étend pas entièrement aux cycles aromatiques [angle dièdre 22,5 (9)° avec le cycle C1 à C6 et 29·8 (9)° avec le cycle C10 à C15].

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3-(2-Hydroxy-5-methylbenzyl)-6-methylcoumarin

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Abstract. $C_{18}H_{16}O_3$, $M_r = 280.32$, monoclinic, $P2_1/a$, $a = 7.324$ (1), $b = 28.326$ (2), $c = 7.304$ (1) Å, $\beta = 107.31$ (1)°, $V = 1446.6$ (3) Å^3, $Z = 4$, $D_m = 1.302$, $D_x = 1.287$ g cm^{-3}, λ(Cu $K\alpha$) = 1.5418 Å, $\mu = 6.64$ cm^{-1}, $F(000) = 592$, $T = 297$ K, final $R = 0.058$ for 1969 observed reflections. The plane of the phenyl ring makes an angle of 113.02 (6)° with the plane of the coumarin moiety.

Introduction. Coumarin and its derivatives are used in oral anticoagulation therapy (Cole, Minifee & Wolma, 1988; Greenfield, 1988). 3-Substituted coumarins exhibit a variety of pharmacological activities. The chemical structure of the title compound has been reported as (I) from spectral data (IR and NMR) (Gopal & Rajagopalan, 1987) but our investigations reveal the structure as (II).

(I) (II)
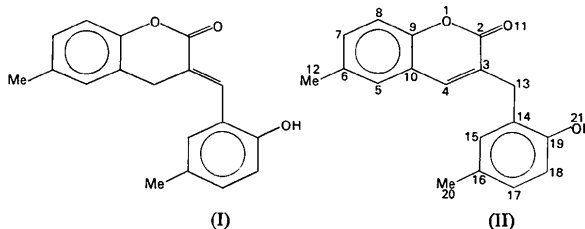
Experimental. Colourless rectangular crystals from a mixture of aqueous ethanol and chloroform, D_m by flotation method, Enraf–Nonius CAD-4 diffractometer, graphite-monochromated Cu $K\alpha$ radiation, crystal dimensions $0.3 \times 0.3 \times 0.2$ mm, cell parameters from 2θ angles of 25 reflections in the range $35 \leq \theta \leq 45°$, $2\theta_{max} = 130°$. Intensities measured in the range $h = -8$ to 8, $k = 0$ to 33 and $l = 0$ to 8, ω–2θ scan technique; three standard reflections monitored every 100 measurements showed no significant change, 2294 unique reflections measured, 1969 observed with $I \geq 3\sigma(I)$, Lp but no absorption corrections, structure solved by direct methods using *SHELXS*86 (Sheldrick, 1986), full-matrix least-squares refinement on F using *SHELX*76 (Sheldrick, 1976), H atoms initially located from difference synthesis and then refined isotropically, anisotropic thermal parameters for non-H atoms; final $R = 0.058$, $wR = 0.071$, $w = 1/[\sigma^2(F_o) + 0.0158F_o^2]$, $S = 1.56$, max. $(\Delta/\sigma) = 0.005$; no significant features in final ΔF syntheses with max. and min. peak heights 0.23 and -0.34 e Å^{-3}; no correction for secondary extinction, atomic scattering factors for all atoms as in *SHELX*76 (Sheldrick, 1976), geometrical calculations using *PARST* (Nardelli, 1982).*

Discussion. The final atomic coordinates and equivalent isotropic temperature factors are given in Table 1. Bond lengths, valence angles and some selected torsion angles are listed in Table 2. Bond lengths and valence angles in this structure agree well with those of related structures (Shimizu, Kashino & Haisa, 1975).

The non-H atoms of the pyrone and benzene rings are in each case coplanar to ± 0.010 (2) Å. The dihedral angle between these planes is 0.9 (6)°, so that the coumarin moiety is also essentially planar. C(12) deviates from the plane of the coumarin moiety by 0.062 (3) and C(13) by 0.034 (2) Å. The phenyl-ring C atoms are coplanar to ± 0.018 (3) Å; C(20) lies in this plane [deviation -0.012 (3) Å] while O(21) deviates from it by -0.042 (3) Å. The phenyl ring makes a dihedral angle of 113.02 (6)° with the plane of the coumarin moiety.

In structure (I) proposed from the spectral data C(4) is sp^3 hybridized while C(13) participates in a double bond with C(3) (Gopal & Rajagopalan, 1987). However, our results show that C(13) is sp^3 hybridized and that C(3)—C(4) [1.349 (3) Å] is a double bond in conformity with structure (II).

Table 1. *Positional parameters and equivalent isotropic thermal parameters*

$$B_{eq} = 8/3\pi^2(U_{11} + U_{22} + U_{33}).$$

|	x	y	z	B_{eq}(Å^2)
O(1)	0.2645 (2)	0.5264 (1)	0.2285 (2)	5.57 (4)
C(2)	0.2601 (2)	0.5745 (1)	0.2329 (3)	5.22 (6)
C(3)	0.2500 (2)	0.6003 (1)	0.0576 (3)	4.85 (5)
C(4)	0.2436 (2)	0.5758 (1)	−0.1025 (3)	4.98 (5)
C(5)	0.2315 (3)	0.4980 (1)	−0.2701 (3)	5.32 (6)
C(6)	0.2323 (3)	0.4496 (1)	−0.2637 (3)	5.33 (6)
C(7)	0.2485 (3)	0.4272 (1)	−0.0897 (3)	5.32 (6)
C(8)	0.2584 (3)	0.4531 (1)	0.0732 (3)	5.66 (6)
C(9)	0.2548 (2)	0.5016 (1)	0.0625 (3)	4.76 (5)
C(10)	0.2432 (2)	0.5254 (1)	−0.1069 (3)	4.59 (6)
O(11)	0.2700 (2)	0.5924 (1)	0.3862 (2)	6.66 (5)
C(12)	0.2168 (4)	0.4198 (1)	−0.4393 (3)	6.92 (8)
C(13)	0.2442 (2)	0.6536 (1)	0.0660 (3)	5.41 (6)
C(14)	0.0499 (2)	0.6729 (1)	0.0578 (3)	4.58 (5)
C(15)	−0.0873 (3)	0.6787 (1)	−0.1188 (3)	4.79 (5)
C(16)	−0.2660 (3)	0.6981 (1)	−0.1397 (3)	5.09 (6)
C(17)	−0.3053 (3)	0.7128 (1)	0.0279 (3)	5.28 (6)
C(18)	−0.1737 (3)	0.7071 (1)	0.2049 (3)	5.41 (6)
C(19)	0.0030 (3)	0.6864 (1)	0.2231 (3)	5.09 (5)
C(20)	−0.4108 (4)	0.7044 (1)	−0.3319 (4)	7.49 (8)
O(21)	0.1261 (2)	0.6810 (1)	0.4033 (2)	7.07 (5)
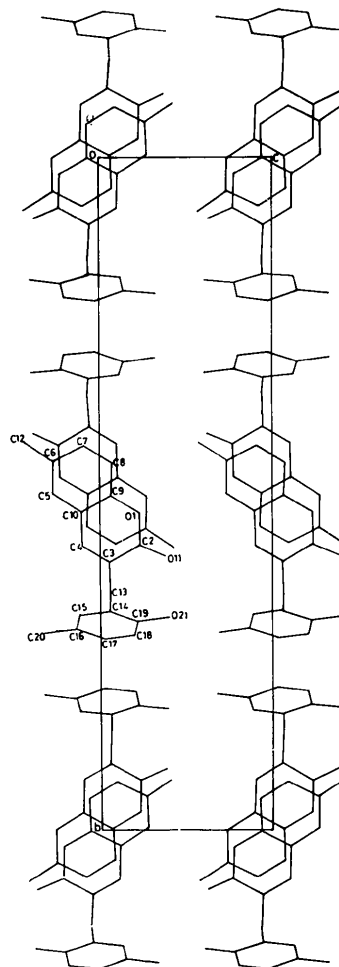
Fig. 1. Molecular packing of the title compound in the unit cell viewed down the a axis.

Table 2. *Bond lengths* (Å), *bond angles* (°) *and selected torsion angles involving non-*H *atoms* (°)

O(1)—C(2)	1·363 (3)	C(8)—C(9)	1·376 (4)
O(1)—C(9)	1·384 (3)	C(9)—C(10)	1·389 (3)
C(2)—C(3)	1·457 (3)	C(13)—C(14)	1·509 (2)
C(2)—O(11)	1·211 (3)	C(14)—C(15)	1·389 (3)
C(3)—C(4)	1·348 (3)	C(14)—C(19)	1·403 (3)
C(3)—C(13)	1·512 (4)	C(15)—C(16)	1·385 (3)
C(4)—C(10)	1·428 (4)	C(16)—C(17)	1·402 (3)
C(5)—C(6)	1·372 (4)	C(16)—C(20)	1·497 (3)
C(5)—C(10)	1·403 (3)	C(17)—C(18)	1·373 (3)
C(6)—C(7)	1·394 (3)	C(18)—C(19)	1·391 (3)
C(6)—C(12)	1·511 (3)	C(19)—O(21)	1·365 (2)
C(7)—C(8)	1·382 (3)		

C(2)—O(1)—C(9)	122·1 (1)	C(5)—C(10)—C(9)	117·4 (2)
O(1)—C(2)—O(11)	116·4 (2)	C(4)—C(10)—C(9)	117·8 (2)
O(1)—C(2)—C(3)	118·4 (2)	C(4)—C(10)—C(5)	124·8 (2)
C(3)—C(2)—O(11)	125·1 (3)	C(3)—C(13)—C(14)	113·4 (2)
C(2)—C(3)—C(13)	117·4 (2)	C(13)—C(14)—C(19)	122·3 (2)
C(2)—C(3)—C(4)	118·9 (2)	C(13)—C(14)—C(15)	119·5 (2)
C(4)—C(3)—C(13)	123·7 (2)	C(15)—C(14)—C(19)	118·2 (2)
C(3)—C(4)—C(10)	122·2 (2)	C(14)—C(15)—C(16)	123·2 (2)
C(6)—C(5)—C(10)	121·7 (2)	C(15)—C(16)—C(20)	122·2 (2)
C(5)—C(6)—C(12)	122·1 (2)	C(15)—C(16)—C(17)	117·0 (2)
C(5)—C(6)—C(7)	118·9 (2)	C(17)—C(16)—C(20)	120·7 (2)
C(7)—C(6)—C(12)	118·9 (2)	C(16)—C(17)—C(18)	121·2 (2)
C(6)—C(7)—C(8)	120·8 (2)	C(17)—C(18)—C(19)	120·8 (2)
C(7)—C(8)—C(9)	119·1 (2)	C(14)—C(19)—C(18)	119·4 (2)
O(1)—C(9)—C(8)	117·5 (1)	C(18)—C(19)—O(21)	117·9 (2)
C(8)—C(9)—C(10)	122·0 (2)	C(14)—C(19)—O(21)	122·6 (2)
O(1)—C(9)—C(10)	120·5 (2)		

C(2)—C(3)—C(13)—C(14)	−78·3 (2)	C(3)—C(13)—C(14)—C(15)	−83·2 (3)
C(4)—C(3)—C(13)—C(14)	100·7 (3)	C(3)—C(13)—C(14)—C(19)	98·3 (3)

The packing of the molecules in the unit cell viewed down the *a* axis is shown in Fig. 1. The crystal structure is stabilized by van der Waals interactions; the shortest non-hydrogen contact [3·432 (3) Å] is between C(20) and O(21) transformed to $x - 1, y, z - 1$.

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Carbon Tetrachloride–Thiourea (1/3) Adduct at 170 K

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Abstract. $CCl_4.3CH_4N_2S$, $M_r = 382.17$, trigonal, $R\bar{3}$, $a = 15.539$ (4), $c = 12.529$ (9) Å, $V = 2620.0$ (20) Å³, $Z = 18$, $D_m = 1.42$ (2) (298 K), $D_x = 1.433$ g cm⁻³ (170 K), λ(Mo $K\alpha$) = 0.71069 Å, $\mu = 10.17$ cm⁻¹, $F(000) = 1164$, $T = 170$ K, $R = 0.043$ for 640 observed ($F > 6.0\sigma_F$) reflections. The structure of the adduct has been solved and a model for the disordered carbon tetrachloride guest molecule has been developed. The adduct is isostructural with other non-aromatic adducts of this type. The carbon of the disordered carbon tetrachloride molecule is clearly co-planar with the three thiourea S atoms that point

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