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8-(2-Bromo-4-methylanilino)-9-hydroxy-4-methyl-7,8,9,10-tetrahydro-7,8-benzocoumarin

K. Chinnakali, H.-K. Fun, K. Sriraghavan and V. T. Ramakrishnan

Abstract

In the title molecule, $C_{21}H_{20}BrNO_3$, the coumarin moiety is planar and the tetrahydrobenzene ring adopts a half-chair conformation. The planes of the coumarin and phenyl rings form a dihedral angle of 50.97 (8)° between them. The crystal structure is stabilized by O—H···O hydrogen bonds involving carbonyl and hydroxyl O atoms.

Comment

The coumarin derivatives are found in natural products and exhibit antifungal and anticoagulant properties (Parrish, Fitzpatrick, Tanenbaum & Pathak, 1974). The β-aminoalcohol sequence plays an important role in organic and in medicinal chemistry (Goodman & Gilman, 1980). Specifically, the β-amino alcohol subunit has been of particular value in the study of acetylcholine metabolism in intact nerve-terminal preparations (Rogers *et al.*, 1989). The crystal structure determination of the title compound, (I), one of the above derivatives, was performed in order to elucidate its molecular conformation.

In the coumarin moiety C3—C4 and C5—C6 bonds show double bond character; steric interactions cause the widening of C3—C2—O11, C4—C10—C5 and narrowing of O1—C2—O11, O1—C9—C8 angles from 120°. Similar features are observed in other coumarin derivatives (Chinnakali, Fun, Sriraghavan & Ramakrishnan, 1997*a*, 1997*b*; Kumar, Chinnakali, Sivakumar, Fun & Sriraghavan, 1997). The mean value of C—C lengths in the phenyl ring [1.389 (5) Å], the N—C bond distances and C(sp^3)-C(sp^3) bond lengths in the tetrahydrobenzene ring agree with the reported values (Allen *et al.*, 1987). The coumarin moiety is planar within ±0.013 (2)Å and atoms C13 and C16 are coplanar with it. The sum of the bond angles around N18 indicates that it is in a pyramidal configuration. The tetrahydrobenzene ring adopts a half-chair conformation with C14 and C15 deviating from the C7—C8—C13—C16 plane by 0.517 (4) and −0.328 (4) Å, respectively; the asymmetry parameter ΔC_2(C7—C8) is 0.031 (2) (Nardelli, 1983). The phenyl ring plane forms a dihedral angle of 50.97 (8)° with the coumarin ring plane. In the crystal, the screw axis related molecules are linked by O—H·O hydrogen bonds involving hydroxyl and carbonyl O atoms (Table 2).

Experimental

Ring opening of 4-methyl-7,10-dihydro-7,8-benzocoumarin-8,9-oxide (1.14 g, 5.0 mmol) with 2-bromo-4-methylaniline (1.11 g, 6.0 mmol) furnished equimolar amounts of two regioisomeric compounds in a combined yield of 80–90% (Sriraghavan, 1997). Single crystals of the title compound (one of the isomers), were grown by slow evaporation from a solution of the compound in chloroform/methanol (1:1).

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Refinement

The structure was solved by direct methods and refined by full-matrix least- squares techniques. Except H atoms of C25, all other H atoms were located from a difference Fourier map and refined isotropically. A rotating group refinement (AFIX 137) was carried out for the methyl group C25.

Computing details

Data collection: *XSCANS* (Siemens, 1994); cell refinement: *XSCANS* (Siemens, 1994); data reduction: *XSCANS* (Siemens, 1994); program(s) used to solve structure: *SHELXTL*PC (Sheldrick, 1990); program(s) used to refine structure: *SHELXL93* (Sheldrick, 1993); molecular graphics: *SHELXTL*PC (Sheldrick, 1990); software used to prepare material for publication: *SHELXL93* (Sheldrick, 1993) *PARST* (Nardelli, 1995).

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Crystal data

$C_{21}H_{20}BrNO_3$	$V = 1880.6\ (5)$ Å^3
$M_r = 414.29$	$Z = 4$
Monoclinic, $P2_1/c$	Mo $K\alpha$
$a = 19.8970\ (18)$ Å	$\mu = 2.21$ mm^{-1}
$b = 11.878\ (2)$ Å	$T = 293\ (2)$ K
$c = 8.0208\ (12)$ Å	0.64 × 0.42 × 0.38 mm
$\beta = 97.204\ (10)^o$	

Data collection

Siemens P4 diffractometer	1879 reflections with $I > 2\sigma(I)$
Absorption correction: empirical (using intensity measurements) ψ scans (Siemens, 1994)	$R_{int} = 0.026$
$T_{min} = 0.296$, $T_{max} = 0.432$	3 standard reflections
5654 measured reflections	every 97 reflections
4284 independent reflections	intensity decay: <3%

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.044$	304 parameters
$wR(F^2) = 0.123$	All H-atom parameters refined
$S = 0.83$	$\Delta\rho_{max} = 0.51$ e Å^{-3}
4282 reflections	$\Delta\rho_{min} = -0.50$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °)

C3—C4	1.345 (5)	C15—O17	1.424 (4)

C5—C6	1.354 (5)	N18—C19	1.384 (4)
C14—N18	1.465 (4)		
O11—C2—O1	115.7 (3)	C8—C9—O1	115.3 (3)
O11—C2—C3	126.9 (3)	C5—C10—C4	124.7 (3)
C13—C7—C8—C16	1.2 (5)	C13—C14—C15—C16	−69.5 (4)
C8—C7—C13—C14	−22.2 (5)	C7—C8—C16—C15	−14.4 (5)
C7—C13—C14—C15	54.8 (4)	C14—C15—C16—C8	48.5 (4)

Table 2
Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O17—H17···O11^{i}	0.77 (4)	2.03 (4)	2.776 (4)	163 (4)
N18—H18···Br	0.84 (3)	2.67 (3)	3.073 (3)	111 (2)
N18—H18···O17	0.84 (3)	2.22 (3)	2.675 (4)	114 (2)

Symmetry codes: (i) −*x*+1, *y*−1/2, −*z*+1/2.

Acknowledgements

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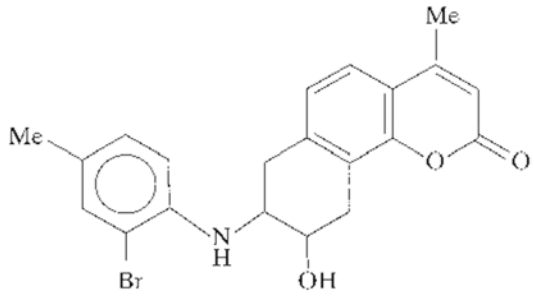
Scheme 1



supplementary materials

8-(2-Bromo-4-methylanilino)-9-hydroxy-4-methyl-7,8,9,10-tetrahydro-7,8- benzocoumarin

Crystal data

$C_{21}H_{20}BrNO_3$	F_{000} = 848
M_r = 414.29	D_x = 1.463 Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: -P 2ybc	Cell parameters from 43 reflections
a = 19.8970 (18) Å	θ = 5.5–12.6º
b = 11.878 (2) Å	μ = 2.21 mm^{-1}
c = 8.0208 (12) Å	T = 293 (2) K
β = 97.204 (10)º	Parallelepiped, colourless
V = 1880.6 (5) Å^3	0.64 × 0.42 × 0.38 mm
Z = 4	

Data collection

Siemens P4 diffractometer	R_{int} = 0.026
Radiation source: fine-focus sealed tube	θ_{max} = 27.5º
Monochromator: graphite	θ_{min} = 2.0º
T = 293(2) K	h = −25→25
$\theta/2\theta$ scans	k = −15→1
Absorption correction: empirical (using intensity measurements) ψ scans (Siemens, 1994)	l = −1→10
T_{min} = 0.296, T_{max} = 0.432	3 standard reflections
5654 measured reflections	every 97 reflections
4284 independent reflections	intensity decay: <3%
1879 reflections with $I > 2\sigma(I)$	

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: difference Fourier map
$R[F^2 > 2\sigma(F^2)]$ = 0.044	All H-atom parameters refined
$wR(F^2)$ = 0.123	Calculated $w = 1/[\sigma^2(F_o^2) + (0.0597P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$?
S = 0.83	$\Delta\rho_{max}$ = 0.51 e Å^{-3}
4282 reflections	$\Delta\rho_{min}$ = −0.50 e Å^{-3}
304 parameters	Extinction correction: none
Primary atom site location: structure-invariant direct methods	

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement on F^2 for ALL reflections except for 2 with very negative F^2 or flagged by the user for potential systematic errors. Weighted *R*-factors *wR* and all goodnesses of fit S are based on F^2, conventional *R*-factors *R* are based on F, with F set to zero for negative F^2. The observed criterion of $F^2 > 2\mathrm{sigma}(F^2)$ is used only for calculating observed *R*-factor *etc*. and is not relevant to the choice of reflections for refinement. *R*-factors based on F^2 are statistically about twice as large as those based on F, and *R*-factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
Br	0.16979 (2)	−0.03046 (3)	0.49627 (7)	0.0949 (2)
O1	0.43772 (10)	0.5801 (2)	0.3165 (3)	0.0578 (6)
C2	0.4790 (2)	0.6676 (3)	0.2841 (5)	0.0639 (10)
C3	0.4552 (2)	0.7801 (3)	0.3107 (6)	0.0709 (12)
C4	0.3949 (2)	0.8017 (3)	0.3631 (5)	0.0601 (10)
C5	0.2879 (2)	0.7170 (3)	0.4485 (6)	0.0671 (11)
C6	0.2507 (2)	0.6245 (3)	0.4734 (5)	0.0629 (11)
C7	0.27412 (15)	0.5157 (3)	0.4471 (4)	0.0492 (8)
C8	0.33778 (14)	0.5022 (2)	0.3938 (4)	0.0464 (8)
C9	0.37494 (14)	0.5985 (3)	0.3702 (4)	0.0491 (8)
C10	0.3521 (2)	0.7074 (3)	0.3946 (5)	0.0542 (9)
O11	0.53234 (12)	0.6414 (2)	0.2363 (4)	0.0892 (9)
C12	0.3716 (3)	0.9207 (3)	0.3846 (9)	0.0790 (15)
C13	0.2307 (2)	0.4156 (3)	0.4773 (6)	0.0560 (10)
C14	0.2504 (2)	0.3118 (3)	0.3811 (5)	0.0499 (9)
C15	0.3261 (2)	0.2936 (3)	0.4325 (5)	0.0487 (8)
C16	0.3656 (2)	0.3871 (3)	0.3608 (6)	0.0501 (9)
O17	0.34392 (13)	0.1852 (2)	0.3763 (4)	0.0671 (8)
N18	0.21404 (14)	0.2105 (2)	0.4234 (4)	0.0527 (7)
C19	0.1494 (2)	0.1872 (3)	0.3460 (4)	0.0485 (8)
C20	0.1094 (2)	0.2650 (3)	0.2503 (5)	0.0600 (10)
C21	0.0440 (2)	0.2408 (4)	0.1783 (6)	0.0697 (11)
C22	0.0150 (2)	0.1368 (4)	0.1997 (6)	0.0781 (12)
C23	0.0534 (2)	0.0581 (4)	0.2972 (6)	0.0770 (13)
C24	0.1192 (2)	0.0821 (3)	0.3671 (5)	0.0605 (10)
C25	−0.0573 (2)	0.1112 (5)	0.1225 (7)	0.120 (2)
H25A	−0.0653 (7)	0.0316 (6)	0.126 (5)	0.181*
H25B	−0.0638 (6)	0.136 (3)	0.0078 (17)	0.181*
H25C	−0.0886 (2)	0.150 (3)	0.185 (3)	0.181*
H3	0.487 (2)	0.838 (4)	0.295 (5)	0.107 (15)*
H5	0.2701 (16)	0.792 (3)	0.464 (4)	0.072 (11)*

H6	0.2117 (16)	0.633 (3)	0.520 (4)	0.062 (10)*
H12A	0.324 (3)	0.937 (4)	0.295 (6)	0.123 (16)*
H12B	0.402 (2)	0.957 (4)	0.361 (6)	0.090 (16)*
H12C	0.359 (2)	0.937 (4)	0.491 (6)	0.091 (17)*
H13A	0.2336 (18)	0.406 (3)	0.586 (5)	0.073 (14)*
H13B	0.178 (2)	0.439 (4)	0.435 (5)	0.109 (14)*
H14	0.2452 (14)	0.324 (3)	0.261 (4)	0.049 (9)*
H15	0.3360 (14)	0.297 (3)	0.556 (4)	0.051 (10)*
H16A	0.4123 (17)	0.382 (3)	0.411 (4)	0.061 (10)*
H16B	0.3618 (15)	0.375 (3)	0.233 (5)	0.062 (11)*
H17	0.382 (2)	0.179 (4)	0.363 (6)	0.105 (17)*
H18	0.2394 (15)	0.154 (3)	0.436 (4)	0.053 (10)*
H20	0.1279 (15)	0.320 (3)	0.222 (4)	0.045 (9)*
H21	0.0247 (16)	0.287 (3)	0.089 (4)	0.059 (11)*
H23	0.030 (2)	−0.005 (4)	0.320 (6)	0.100 (14)*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Br	0.0900 (3)	0.0405 (2)	0.1589 (5)	−0.0013 (2)	0.0340 (3)	0.0108 (3)
O1	0.0446 (11)	0.0376 (11)	0.093 (2)	−0.0084 (10)	0.0148 (12)	0.0013 (13)
C2	0.053 (2)	0.049 (2)	0.090 (3)	−0.012 (2)	0.006 (2)	0.002 (2)
C3	0.063 (2)	0.041 (2)	0.109 (4)	−0.017 (2)	0.013 (2)	0.008 (2)
C4	0.067 (2)	0.034 (2)	0.076 (3)	−0.005 (2)	−0.002 (2)	0.001 (2)
C5	0.057 (2)	0.037 (2)	0.107 (3)	0.009 (2)	0.013 (2)	−0.004 (2)
C6	0.047 (2)	0.045 (2)	0.098 (3)	0.003 (2)	0.016 (2)	−0.007 (2)
C7	0.044 (2)	0.038 (2)	0.065 (2)	0.0012 (14)	0.007 (2)	0.000 (2)
C8	0.041 (2)	0.0362 (15)	0.061 (2)	0.0004 (12)	0.003 (2)	−0.001 (2)
C9	0.040 (2)	0.041 (2)	0.065 (2)	−0.0028 (14)	0.005 (2)	0.001 (2)
C10	0.053 (2)	0.037 (2)	0.070 (3)	−0.0028 (15)	−0.002 (2)	0.003 (2)
O11	0.0538 (14)	0.0572 (15)	0.163 (3)	−0.0104 (12)	0.040 (2)	0.004 (2)
C12	0.084 (3)	0.031 (2)	0.122 (5)	−0.003 (2)	0.013 (4)	0.002 (3)
C13	0.047 (2)	0.045 (2)	0.078 (3)	−0.003 (2)	0.017 (2)	−0.002 (2)
C14	0.045 (2)	0.037 (2)	0.070 (3)	−0.0032 (13)	0.014 (2)	0.005 (2)
C15	0.047 (2)	0.035 (2)	0.065 (3)	−0.0020 (14)	0.010 (2)	−0.004 (2)
C16	0.041 (2)	0.0332 (15)	0.077 (3)	−0.0012 (14)	0.011 (2)	−0.001 (2)
O17	0.0547 (15)	0.0337 (12)	0.117 (2)	0.0019 (11)	0.027 (2)	−0.0020 (13)
N18	0.050 (2)	0.0388 (14)	0.072 (2)	−0.0085 (13)	0.017 (2)	0.001 (2)
C19	0.047 (2)	0.047 (2)	0.055 (2)	−0.005 (2)	0.019 (2)	−0.009 (2)
C20	0.052 (2)	0.054 (2)	0.077 (3)	−0.014 (2)	0.018 (2)	0.003 (2)
C21	0.053 (2)	0.083 (3)	0.073 (3)	−0.004 (2)	0.008 (2)	0.005 (3)
C22	0.051 (2)	0.095 (3)	0.089 (3)	−0.021 (2)	0.011 (2)	−0.012 (3)
C23	0.067 (3)	0.063 (2)	0.108 (4)	−0.026 (2)	0.035 (3)	−0.018 (3)
C24	0.056 (2)	0.042 (2)	0.087 (3)	−0.009 (2)	0.025 (2)	−0.008 (2)
C25	0.066 (3)	0.147 (5)	0.147 (5)	−0.043 (3)	0.005 (3)	−0.029 (4)

Geometric parameters (Å, °)

Br—C24	1.901 (4)	C9—C10	1.393 (4)

O1—C2	1.370 (4)	C13—C14	1.531 (5)
O1—C9	1.389 (3)	C14—N18	1.465 (4)
C2—O11	1.214 (4)	C14—C15	1.526 (4)
C2—C3	1.442 (5)	C15—O17	1.424 (4)
C3—C4	1.345 (5)	C15—C16	1.515 (4)
C4—C10	1.448 (4)	N18—C19	1.384 (4)
C4—C12	1.505 (5)	C19—C20	1.386 (5)
C5—C6	1.354 (5)	C19—C24	1.405 (4)
C5—C10	1.404 (5)	C20—C21	1.387 (5)
C6—C7	1.399 (4)	C21—C22	1.384 (6)
C7—C8	1.396 (4)	C22—C23	1.385 (6)
C7—C13	1.506 (5)	C22—C25	1.525 (5)
C8—C9	1.388 (4)	C23—C24	1.387 (5)
C8—C16	1.510 (4)		
C2—O1—C9	121.6 (3)	C7—C13—C14	111.5 (3)
O11—C2—O1	115.7 (3)	N18—C14—C15	108.7 (3)
O11—C2—C3	126.9 (3)	N18—C14—C13	112.3 (3)
O1—C2—C3	117.3 (3)	C15—C14—C13	106.5 (3)
C4—C3—C2	123.0 (3)	O17—C15—C16	112.4 (3)
C3—C4—C10	118.4 (3)	O17—C15—C14	108.6 (3)
C3—C4—C12	121.0 (4)	C16—C15—C14	109.5 (3)
C10—C4—C12	120.6 (4)	C8—C16—C15	112.2 (3)
C6—C5—C10	121.0 (3)	C19—N18—C14	121.2 (3)
C5—C6—C7	121.9 (3)	N18—C19—C20	123.5 (3)
C8—C7—C6	119.0 (3)	N18—C19—C24	120.8 (3)
C8—C7—C13	121.2 (3)	C20—C19—C24	115.7 (3)
C6—C7—C13	119.7 (3)	C19—C20—C21	122.4 (4)
C9—C8—C7	117.8 (3)	C22—C21—C20	121.2 (4)
C9—C8—C16	120.6 (3)	C21—C22—C23	117.6 (4)
C7—C8—C16	121.6 (3)	C21—C22—C25	120.9 (5)
C8—C9—O1	115.3 (3)	C23—C22—C25	121.4 (4)
C8—C9—C10	123.9 (3)	C22—C23—C24	121.0 (4)
O1—C9—C10	120.8 (3)	C23—C24—C19	122.0 (4)
C9—C10—C5	116.4 (3)	C23—C24—Br	119.0 (3)
C9—C10—C4	118.9 (3)	C19—C24—Br	119.0 (3)
C5—C10—C4	124.7 (3)		
C9—O1—C2—O11	−179.3 (3)	C8—C7—C13—C14	−22.2 (5)
C9—O1—C2—C3	1.0 (5)	C6—C7—C13—C14	158.0 (4)
O11—C2—C3—C4	179.5 (4)	C7—C13—C14—N18	173.6 (3)
O1—C2—C3—C4	−0.8 (6)	C7—C13—C14—C15	54.8 (4)
C2—C3—C4—C10	0.2 (6)	N18—C14—C15—O17	46.2 (4)
C2—C3—C4—C12	−178.4 (5)	C13—C14—C15—O17	167.4 (3)
C10—C5—C6—C7	0.0 (6)	N18—C14—C15—C16	169.3 (3)
C5—C6—C7—C8	0.1 (6)	C13—C14—C15—C16	−69.5 (4)
C5—C6—C7—C13	179.8 (4)	C9—C8—C16—C15	166.3 (3)
C6—C7—C8—C9	0.3 (5)	C7—C8—C16—C15	−14.4 (5)
C13—C7—C8—C9	−179.4 (4)	O17—C15—C16—C8	169.3 (3)
C6—C7—C8—C16	−179.1 (4)	C14—C15—C16—C8	48.5 (4)

C13—C7—C8—C16	1.2 (5)	C15—C14—N18—C19	−159.1 (3)
C7—C8—C9—O1	−179.7 (3)	C13—C14—N18—C19	83.3 (4)
C16—C8—C9—O1	−0.3 (5)	C14—N18—C19—C20	−12.9 (5)
C7—C8—C9—C10	−0.8 (5)	C14—N18—C19—C24	169.9 (3)
C16—C8—C9—C10	178.6 (4)	N18—C19—C20—C21	−178.0 (3)
C2—O1—C9—C8	178.3 (3)	C24—C19—C20—C21	−0.6 (5)
C2—O1—C9—C10	−0.6 (5)	C19—C20—C21—C22	0.4 (6)
C8—C9—C10—C5	0.9 (5)	C20—C21—C22—C23	0.8 (6)
O1—C9—C10—C5	179.8 (3)	C20—C21—C22—C25	179.1 (4)
C8—C9—C10—C4	−178.9 (3)	C21—C22—C23—C24	−1.6 (6)
O1—C9—C10—C4	0.0 (5)	C25—C22—C23—C24	−179.9 (4)
C6—C5—C10—C9	−0.5 (6)	C22—C23—C24—C19	1.4 (6)
C6—C5—C10—C4	179.3 (4)	C22—C23—C24—Br	−178.8 (3)
C3—C4—C10—C9	0.2 (5)	N18—C19—C24—C23	177.1 (3)
C12—C4—C10—C9	178.8 (4)	C20—C19—C24—C23	−0.3 (5)
C3—C4—C10—C5	−179.6 (4)	N18—C19—C24—Br	−2.6 (4)
C12—C4—C10—C5	−1.0 (6)	C20—C19—C24—Br	180.0 (3)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O17—H17···O11^{i}	0.77 (4)	2.03 (4)	2.776 (4)	163 (4)
N18—H18···Br	0.84 (3)	2.67 (3)	3.073 (3)	111 (2)
N18—H18···O17	0.84 (3)	2.22 (3)	2.675 (4)	114 (2)

Symmetry codes: (i) −*x*+1, *y*−1/2, −*z*+1/2.