C1a—C14a	1.322 (5)	C14—C15	1.518 (5)
C1a—C22a	1.489 (6)	C15—C15a	1.610 (5)
C2—C3	1.412 (5)	C15—C16	1.513 (5)
C2—C7	1.427 (5)	C15a—C16a	1.537 (6)
C3—C4	1.350 (5)	C16a—C21a	1.555 (5)
C4—C5	1.387 (6)	C16—C17	1.367 (5)
C5—C6	1.378 (5)	C16—C21	1.410 (5)
C6—C7	1.397 (5)	C17—C18	1.388 (6)
C7—C8	1.457 (4)	C18—C19	1.380 (5)
C8—C9	1.414 (5)	C19—C20	1.366 (5)
C8—C13	1.414 (5)	C20—C21	1.385 (5)
C9—C10	1.374 (5)	C21—C22	1.509 (5)
C10—C11	1.380 (6)	C21a—C22a	1.534 (6)
C11—C12	1.367 (5)	C22a—C22	1.612 (5)

C2—C1—C14	121.5 (3)	C1—C14—C15	117.1 (3)
C2—C1—C22	122.7 (3)	C13—C14—C15	122.9 (3)
C14—C1—C22	115.8 (3)	C14—C15—C15a	113.5 (3)
C14a—C1a—C22a	117.7 (3)	C14—C15—C16	107.8 (3)
C1—C2—C3	123.1 (3)	C15a—C15—C16	112.8 (3)
C1—C2—C7	120.0 (3)	C14a—C15a—C15	112.5 (3)
C3—C2—C7	116.9 (3)	C14a—C15a—C16a	108.2 (3)
C2—C3—C4	122.8 (4)	C15—C15a—C16a	114.3 (3)
C3—C4—C5	120.3 (3)	C15a—C16a—C21a	112.4 (3)
C4—C5—C6	119.2 (4)	C15—C16—C17	125.0 (3)
C5—C6—C7	121.9 (4)	C15—C16—C21	115.0 (3)
C2—C7—C6	118.9 (3)	C17—C16—C21	119.9 (3)
C2—C7—C8	118.6 (3)	C16—C17—C18	120.4 (3)
C6—C7—C8	122.6 (3)	C17—C18—C19	119.7 (3)
C7—C8—C9	121.3 (3)	C18—C19—C20	120.3 (4)
C7—C8—C13	119.9 (3)	C19—C20—C21	120.9 (3)
C9—C8—C13	118.7 (3)	C16—C21—C20	118.7 (3)
C8—C9—C10	121.1 (4)	C16—C21—C22	116.5 (3)
C9—C10—C11	120.0 (4)	C20—C21—C22	124.7 (3)
C10—C11—C12	120.9 (3)	C16a—C21a—C22a	112.8 (3)
C11—C12—C13	120.8 (3)	C1a—C22a—C21a	107.9 (3)
C8—C13—C12	118.4 (3)	C1a—C22a—C22	112.7 (3)
C8—C13—C14	120.0 (3)	C21a—C22a—C22	114.6 (3)
C12—C13—C14	121.6 (3)	C1—C22—C21	108.1 (3)
C1a—C14a—C15a	119.1 (4)	C1—C22—C22a	113.0 (3)
C1—C14—C13	119.9 (3)	C21—C22—C22a	112.9 (3)

C1—C22—C22a—C1a	−2.3	C21—C22—C22a—C21a	−3.3
C14—C15—C15a—C14a	1.6	C16—C15—C15a—C16a	2.9

Plane (1)	Plane (2)	Dihedral angle
C1-C14	C16-C21	53.9
C15a, C16a, C21a, C22a	C1a, C14a, C15a, C22a	52.6

Crystals were grown from a solution of dichloromethane and methanol by slow evaporation. A suitable single crystal was attached to the end of a glass fiber using fast-drying epoxy glue. The intensity data were corrected for empirical absorption (ψ scans) (North, Phillips & Mathews, 1968), Lorentz and polarization effects and secondary extinction. The structure was solved by direct methods and subsequent difference Fourier maps. All C atoms were refined anisotropically. The H atoms were located on difference electron density maps or generated in chemically reasonable positions and were not refined. All calculations were performed using a PDP-11 minicomputer and Enraf-Nonius *SDP-Plus* software (B. A. Frenz & Associates, Inc., 1983).

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6,7,8,9-Tetrahydro-4-methyl-2*H*-pyrano-[3,2-g]quinolin-2-one

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Abstract

In the title compound, also known as coumarin 339, the coumarin moiety is planar and coplanar with the methyl group at C4 [C2—C3—C4—C12 = 179.9 (2) and C9—C10—C4—C12 = 178.4 (2)°]. The structure is stabilized by extensive intermolecular C—O$\cdots$H hydrogen bonding.

Comment

The title compound, a laser dye, has been used in novel polymeric environments (Jones & Ragman, 1990), as well as in singlet excitation-energy correlation chemiluminescence reactions (Tod, Farinotti, Mahuzier & Gaury, 1989). The benzene and pyrone rings are planar (χ^2 = 24.5 and 88.2) and the dihedral angle between the rings is 1.57°.
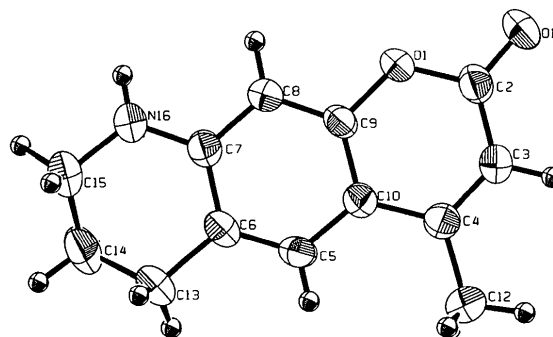

Fig. 1. *ORTEP* (Johnson, 1976) drawing (50% probability ellipsoids) and atomic numbering scheme.

Experimental

Crystal data

$C_{13}H_{13}NO_2$	Cell parameters from 24
$M_r = 215.25$	reflections
Triclinic	$\theta = 46.39 - 50.01°$
$P\bar{1}$	$\mu = 0.0853$ mm^{-1}
$a = 9.094$ (2) Å	$T = 296.1$ K
$b = 9.078$ (2) Å	Prism
$c = 6.967$ (2) Å	$1.00 \times 0.90 \times 0.40$ mm
$\alpha = 101.25$ (2)°	Clear
$\beta = 106.99$ (2)°	Crystal source: Eastman
$\gamma = 76.48$ (2)°	Kodak Chemical Co.,
$V = 529.8$ (2) Å^3	Rochester, NY 14650,
$Z = 2.00$	USA
$D_x = 1.349$ Mg m^{-3}	
Mo $K\alpha$ radiation	
$\lambda = 0.7107$ Å	

Data collection

Rigaku AFC-6S diffractometer	$R_{int} = 0.024$
	$\theta_{max} = 27.5°$
ω scans with profile analysis	$h = 0 \rightarrow 12$
Absorption correction:	$k = -12 \rightarrow 12$
empirical	$l = -9 \rightarrow 9$
$T_{min} = 0.909$, $T_{max} = 1.000$	3 standard reflections monitored every 150
2587 measured reflections	reflections
2433 independent reflections	intensity variation: 1.57%
1681 observed reflections	
$[I > 3.00\sigma(I)]$	

Refinement

Refinement on F	$\Delta\rho_{max} = 0.53$ e Å^{-3}
Final $R = 0.0612$	$\Delta\rho_{min} = -0.36$ e Å^{-3}
$wR = 0.0707$	Extinction correction:
$S = 2.749$	Zachariasen type 2
1681 reflections	Gaussian isotropic
198 parameters	(Zachariasen, 1963)
Only coordinates of H atoms refined	Extinction coefficient: 0.25439×10^{-4}
$w = 4F_o^2/\sigma^2(F_o^2)$	Atomic scattering factors
$(\Delta/\sigma)_{max} = 0.0237$	from International Tables for X-ray Crystallography (1974, Vol. IV)

Table 1. *Fractional atomic coordinates and equivalent isotropic thermal parameters* (Å^2)

$$U_{eq} = \tfrac{1}{3}\Sigma_i\Sigma_j U_{ij}a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	U_{eq}
O1	0.2868 (2)	1.1418 (2)	0.9201 (2)	0.0484
O11	0.2013 (2)	1.3290 (2)	1.1365 (2)	0.0587
N16	0.5042 (3)	0.7284 (2)	0.4978 (3)	0.0560
C2	0.1821 (3)	1.2728 (3)	0.9589 (3)	0.0445
C3	0.0635 (3)	1.3281 (3)	0.7905 (3)	0.0433
C4	0.0491 (3)	1.2553 (3)	0.5987 (3)	0.0400
C5	0.1624 (3)	1.0308 (3)	0.3720 (3)	0.0420
C6	0.2729 (3)	0.9033 (3)	0.3455 (3)	0.0420
C7	0.3926 (3)	0.8559 (2)	0.5159 (3)	0.0405
C8	0.3928 (3)	0.9401 (3)	0.7069 (3)	0.0433
C9	0.2786 (3)	1.0659 (2)	0.7252 (3)	0.0378
C10	0.1598 (3)	1.1183 (2)	0.5618 (3)	0.0385
C12	-0.0775 (4)	1.3172 (4)	0.4260 (4)	0.0547
C13	0.2733 (4)	0.8106 (3)	0.1399 (4)	0.0578
C14	0.3566 (4)	0.6487 (4)	0.1606 (5)	0.0737
C15	0.5138 (4)	0.6380 (3)	0.3044 (4)	0.0566

Table 2. *Geometric parameters* (Å, °)

O1—C2	1.375 (3)	C5—C6	1.365 (3)
O1—C9	1.387 (2)	C5—C10	1.407 (3)
O11—C2	1.221 (2)	C6—C7	1.416 (3)
N16—C7	1.362 (3)	C6—C13	1.513 (3)
N16—C15	1.450 (3)	C7—C8	1.399 (3)
C2—C3	1.424 (3)	C8—C9	1.366 (3)
C3—C4	1.354 (3)	C9—C10	1.393 (3)
C4—C10	1.438 (3)	C13—C14	1.501 (4)
C4—C12	1.497 (3)	C14—C15	1.481 (4)
C2—O1—C9	121.8 (2)	N16—C7—C6	121.3 (2)
C7—N16—C15	122.8 (2)	N16—C7—C8	119.7 (2)
O1—C2—O11	115.6 (2)	C6—C7—C8	118.9 (2)
O1—C2—C3	117.4 (2)	C7—C8—C9	119.6 (2)
O11—C2—C3	127.0 (2)	O1—C9—C8	115.8 (2)
C2—C3—C4	122.7 (2)	O1—C9—C10	120.6 (2)
C3—C4—C10	118.9 (2)	C8—C9—C10	123.6 (2)
C3—C4—C12	121.0 (2)	C4—C10—C5	126.0 (2)
C10—C4—C12	120.1 (2)	C4—C10—C9	118.6 (2)
C6—C5—C10	123.4 (2)	C5—C10—C9	115.4 (2)
C5—C6—C7	119.1 (2)	C6—C13—C14	111.0 (2)
C5—C6—C13	122.5 (2)	C13—C14—C15	113.0 (3)
C7—C6—C13	118.4 (2)		

	$C-O\cdots H$	$O\cdots H$	$C-O\cdots H$
C2=O11$\cdots$H16^i		2.31 (3)	129.55 (3)
C2=O11$\cdots$H3^{ii}		2.54 (3)	111.91 (3)
C2=O11$\cdots$H14B^{iii}		2.55 (4)	113.01 (9)
C2=O11$\cdots$H12C^{iv}		2.84 (3)	118.64 (8)
C2—O1$\cdots$H8^i		2.39 (3)	117.18 (6)
C2—O1$\cdots$H15B^v		2.83 (4)	86.32 (6)
C2—O1$\cdots$H13A^{iv}		2.92 (3)	128.53 (2)

Symmetry code: (i) $-x + 1, -y + 2, -z + 2$; (ii) $-x, -y + 3, -z + 2$; (iii) $x, y + 1, z + 1$; (iv) $x, y, z + 1$; (v) $-x + 1, -y + 2, -z + 1$.

Crystals were grown from acetonitrile by slow evaporation. The data were scanned at 8.0° min^{-1} (1.42 + 0.30tan θ)°. Weak reflections $[I < 10.0\sigma(I)]$ were rescanned (maximum of two rescans) and the counts accumulated to ensure accurate counting statistics. Lp correction was applied but no decay correction. H atoms were given calculated positions (C—H = 0.95 Å) and assigned isotropic thermal parameters of 1.5 × B_{eq} of the associated C atom; anisotropic thermal parameters were assigned for non-H atoms. The weighting scheme was based on counting statistics and included a factor ($p = 0.03$) to down-weight the intense reflections; $\sigma^2(F^2) = S^2(C + 4B) + (pF_o^2)^2$ where $S =$ scan count, $B =$ normalized background. Plots of $\Sigma w(|F_o| - |F_c|)^2$ versus $|F_o|$, the reflection order in data collection, sin θ/λ and various classes of indices showed no unusual trends. All calculations were performed on a VAXstation 3520 minicomputer using *TEXSAN* (Molecular Structure Corporation, 1985) software. Atomic positions were obtained from the direct-methods program *MITHRIL* (Gilmore, 1984). The enantiomorphs are indistinguishable from the X-ray data.

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Structure of 2-(2-Hydroxyethyl)-1-*p*-tolyl-4,5,6,7-tetrahydro-3(1*H*)-isoindolone

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Abstract

The flexible cyclohexene ring of the isoindole moiety, as indicated by the positional disorder of the C5 and C6 atoms, assumes two kinds of half-chair conformation, $^4H_5$ or $^5H_4$, at an equilibrium ratio of 68:32. The planar pyrrole ring forms dihedral angles of 76.45 (3) and 86.21 (9)° with the best planes through the 1-*p*-tolyl and 2-ethyl moieties, respectively. Both moieties occupy a *cis* position with respect to the pyrrole ring [N2—C1—C12—C13 = 98.9 (2), C1—N2—C8—C9 = 82.0 (2)°].

Comment

The title compound (4) was synthesized with pharmacological aims (Stájer *et al.*, 1992, unpublished

results), because it is known that similar structures, including different isoindoles condensed with heterocycles, have a potentially anorexigenic effect (Orzalesi *et al.*, 1978).

(4)
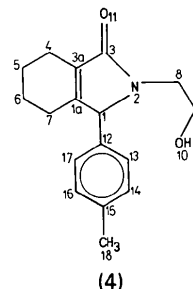
cis-2-(*p*-Toluoyl)-1-cyclohexanecarboxylic acid (1) was refluxed with ethanolamine in toluene, in the presence of *p*-toluenesulfonic acid as catalyst and 9b-*p*-tolyl-2,3,5a,6,7,8,9,9a-octahydrooxazolo[2,3-*a*]-isoindol-5(9b*H*)-one (2) was obtained. Under similar conditions in xylene, (1) afforded 1-*p*-tolyl-1*H*-3,4,7,8,9,10-hexahydro-2,5-benzoxazocin-6(5*H*)-one (3) which could also be prepared from (2) by heating in xylene. (2) may undergo transformation to give (3) or 2-(2-hydroxyethyl)-1-*p*-tolyl-4,5,6,7-tetrahydroisoindol-3(1*H*)-one (4). The isomeric structures of (3) and (4) cannot be differentiated by means of NMR because the signals are merged and the splits are similar. The X-ray data prove structure (4), which may be formed from (2) by simple hydrolysis, but the ring transformation to (3) and subsequent hydrolysis can also be supposed.
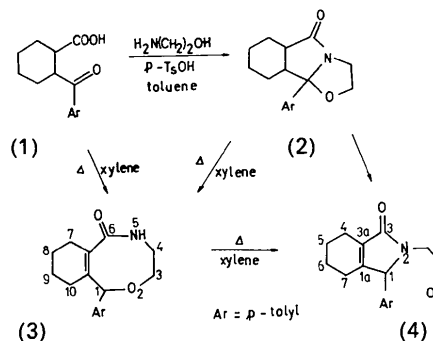
The structure was refined for the two most probable positions of atoms C5 and C6, given in Table 1, which indicates the conformational disorder (Fig. 1). The puckering parameters (Cremer & Pople, 1975) of cyclohexene ring conformations C [C1a—C3a—C4—C5—C6—C7: $Q = 0.484$ (5) Å, $\varphi = 32.4$ (8), $\theta = 130.0$ (6)°] and C' [C1a—C3a—C4—C5'—C6'—C7: $Q = 0.46$ (1) Å, $\varphi = 211(2)°$, $\theta = 49$ (2)°] show that it adopts two kinds of half-chair form, $^4H_5$ and $^5H_4$, respectively (Boeyens, 1978). The increased motion of the atomic displacements of the peripheral atoms