

1236 reflections  
149 parameters  
All H-atom parameters  
refined isotropically  
 $w = 1/[\sigma^2(F_o^2) + (0.0524P)^2]$   
where  $P = (F_o^2 + 2F_c^2)/3$

Atomic scattering factors  
from *International Tables  
for Crystallography* (1992,  
Vol. C, Tables 4.2.6.8 and  
6.1.1.4)

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: KH1026). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. Fractional atomic coordinates and equivalent isotropic displacement parameters ( $\text{\AA}^2$ )

$$U_{eq} = (1/3)\sum_i\sum_j U_{ij}a_i^*a_j^*a_i \cdot a_j$$

	x	y	z	$U_{eq}$
C1	-0.5088 (2)	0.1460 (2)	0.67035 (11)	0.0211 (3)
C2	-0.2908 (2)	0.1438 (2)	0.66390 (12)	0.0208 (3)
C3	-0.2116 (2)	0.3294 (2)	0.66479 (11)	0.0207 (3)
C4	0.0057 (2)	0.3258 (2)	0.66823 (11)	0.0213 (3)
O1	-0.6036 (2)	0.0667 (2)	0.60748 (8)	0.0312 (3)
O2	-0.57617 (15)	0.2294 (2)	0.74456 (8)	0.0282 (3)
O3	-0.2276 (2)	0.0498 (2)	0.57938 (9)	0.0295 (3)
O4	-0.2781 (2)	0.42691 (15)	0.58258 (9)	0.0292 (3)
O5	0.07300 (15)	0.2416 (2)	0.74383 (8)	0.0286 (3)
O6	0.1013 (2)	0.4000 (2)	0.60540 (9)	0.0334 (3)
N1	-0.0252 (2)	-0.2130 (2)	0.45011 (12)	0.0374 (4)
N2	0.0594 (3)	-0.2336 (2)	0.54541 (12)	0.0384 (4)

Table 2. Selected bond lengths ( $\text{\AA}$ ) and angles ( $^\circ$ )

C1—O1	1.236 (2)	C3—O4	1.413 (2)
C1—O2	1.275 (2)	C3—C4	1.530 (2)
C1—C2	1.537 (2)	C4—O6	1.220 (2)
C2—O3	1.416 (2)	C4—O5	1.293 (2)
C2—C3	1.524 (2)	N1—N2	1.422 (2)
O1—C1—O2	125.4 (1)	O4—C3—C2	111.4 (1)
O1—C1—C2	119.7 (2)	O4—C3—C4	111.4 (1)
O2—C1—C2	114.9 (1)	C2—C3—C4	110.4 (1)
O3—C2—C3	111.3 (1)	O6—C4—O5	125.0 (1)
O3—C2—C1	111.4 (1)	O6—C4—C3	121.5 (2)
C3—C2—C1	110.8 (1)	O5—C4—C3	113.5 (1)

Table 3. Hydrogen-bonding geometry ( $\text{\AA}$ ,  $^\circ$ )

D—H...A	D—H	H...A	D...A	D—H...A
O5—H5O...O2 <sup>i</sup>	1.15 (2)	1.33 (2)	2.471 (2)	175 (2)
O3—H3O...O6 <sup>ii</sup>	0.86 (2)	1.99 (2)	2.787 (2)	155 (2)
O4—H4O...O1 <sup>iii</sup>	0.82 (2)	2.08 (2)	2.835 (2)	154 (2)
N1—H1N1...O4 <sup>iii</sup>	0.92 (3)	2.02 (2)	2.828 (2)	146 (3)
N1—H2N1...O2 <sup>iv</sup>	0.92 (3)	2.39 (3)	2.857 (2)	112 (2)
N1—H2N1...O1 <sup>v</sup>	0.92 (3)	2.11 (3)	2.867 (2)	138 (2)
N1—H3N1...N2 <sup>vi</sup>	1.05 (3)	1.91 (3)	2.952 (2)	171 (3)
N2—H1N2...O3	0.85 (3)	2.29 (3)	2.997 (2)	141 (3)
N2—H2N2...O6 <sup>vii</sup>	0.85 (3)	2.24 (4)	2.930 (2)	140 (3)

Symmetry codes: (i)  $1 + x, y, z$ ; (ii)  $x - \frac{1}{2}, \frac{1}{2} - y, 1 - z$ ; (iii)  $\frac{1}{2} + x, \frac{1}{2} - y, 1 - z$ ; (iv)  $-\frac{1}{2} - x, -y, z - \frac{1}{2}$ ; (v)  $\frac{1}{2} + x, -\frac{1}{2} - y, 1 - z$ ; (vi)  $x - \frac{1}{2}, -\frac{1}{2} - y, 1 - z$ ; (vii)  $x, y - 1, z$ .

Data collection: XSCANS (Siemens, 1991). Cell refinement: XSCANS. Data reduction: XSCANS. Program(s) used to solve structure: SHELXS86 (Sheldrick, 1985). Program(s) used to refine structure: SHELXL93 (Sheldrick, 1993). Molecular graphics: SHELXTL/PC (Sheldrick, 1990). Software used to prepare material for publication: SHELXL93. Geometric calculations: PARST (Nardelli, 1983).

The authors would like to thank the Malaysian Government and the Universiti Sains Malaysia for research grant R&D No. 123-3417-2201. One of the authors (KS) thanks the Universiti Sains Malaysia for a Visiting Postdoctoral Research Fellowship.

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## Coumarin 311

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(Received 15 December 1994; accepted 21 April 1995)

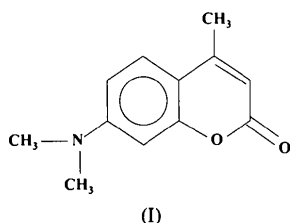
## Abstract

Coumarin 311, 7-(dimethylamino)-4-methyl-2H-1-benzopyran-2-one,  $C_{12}H_{13}NO_2$ , crystallizes in a triclinic crystal system with two molecules in the asymmetric unit. The molecules are planar and make an angle of  $10.41(2)^\circ$  between each other. The molecules are stacked in layers parallel to (103).

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### Comment

The title compound, (I), is a 7-aminocoumarin derivative and is used as laser dye. It gives laser emission around 453 nm in ethanol (Maeda, 1984). The crystal structure was determined as part of our structural studies on laser dyes.



The two molecules, *A* and *B*, in the asymmetric unit are shown in Fig. 1. The molecules are planar with a maximum deviation of 0.082 (2) Å for O11 in molecule *A* and 0.078 (1) Å for the same atom in *B*. The angle between the planes of the two molecules is 10.41 (2)°. The dihedral angle between the pyrone ring and the benzene ring is 1.23 (4)° for *A* and 2.01 (4)° for *B*. The dihedral angles between the benzene ring and the amino group are 1.3 (1) and 4.2 (1)° for *A* and *B*, respectively.

Bond lengths and angles in the coumarin ring system in both molecules are normal and are in good agreement with those observed in coumarin 485 [7-(dimethylamino)-4-(trifluoromethyl)-2*H*-1-benzopyran-2-one (Chinnakali, Sivakumar & Natarajan, 1990*b*)] and coumarin 480 [2,3,6,7-tetrahydro-9-methyl-1*H*,5*H*,11*H*-[1]benzopyrano[6,7,8-*ij*]quinolizin-11-one (Chinnakali, Sivakumar & Natarajan, 1990*a*)].

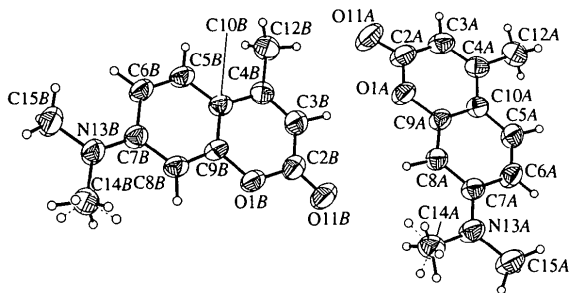


Fig. 1. Structure of coumarin 311 showing 50% probability displacement ellipsoids and the atom-numbering scheme.

The packing of the molecules viewed down the *b* axis (Fig. 2) shows that the molecules are stacked in layers parallel to (103), with an interlayer spacing of 3.428 Å. This distance is shorter than that observed in the packing of the rigidized aminocoumarins, which are normally stacked with an interlayer distance of *ca* 3.6 Å (Yip *et al.*, 1994).

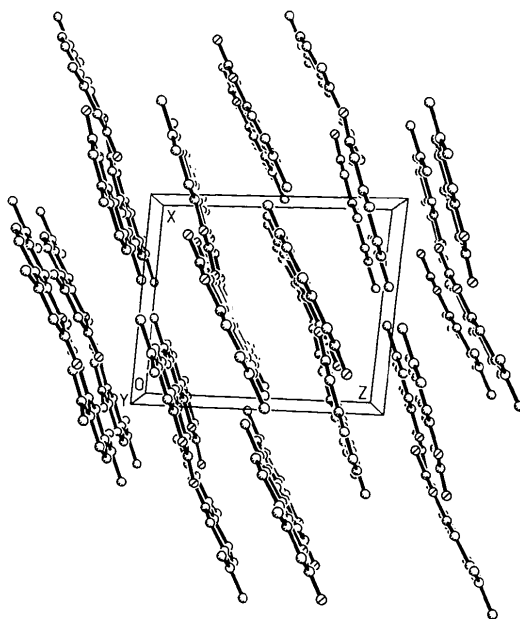


Fig. 2. Packing of the molecules viewed down the *b* axis.

### Experimental

The compound was purchased from Sigma Chemical Co., USA, and recrystallized from a mixture of methanol and dichloromethane by slow evaporation.

#### Crystal data

C<sub>12</sub>H<sub>13</sub>NO<sub>2</sub>  
*M<sub>r</sub>* = 203.23  
 Triclinic  
*P* $\bar{1}$   
*a* = 9.082 (2) Å  
*b* = 10.778 (2) Å  
*c* = 11.088 (3) Å  
 $\alpha$  = 76.93 (1)°  
 $\beta$  = 83.41 (1)°  
 $\gamma$  = 80.78 (1)°  
*V* = 1040.1 (4) Å<sup>3</sup>  
*Z* = 4  
*D<sub>x</sub>* = 1.298 Mg m<sup>-3</sup>

Mo *K*α radiation  
 $\lambda$  = 0.71073 Å  
 Cell parameters from 35 reflections  
 $\theta$  = 5–12.5°  
 $\mu$  = 0.089 mm<sup>-1</sup>  
*T* = 293 (2) K  
 Needle  
 0.50 × 0.42 × 0.22 mm  
 Yellow

#### Data collection

Siemens P4 four-circle diffractometer  
 $\theta$ -2 $\theta$  scans  
 Absorption correction: none  
 5556 measured reflections  
 4659 independent reflections  
 3397 observed reflections  
 [*I* > 2σ(*I*)]

*R*<sub>int</sub> = 0.0135  
 $\theta_{\text{max}}$  = 27.50°  
*h* = -10 → 10  
*k* = -13 → 13  
*l* = 0 → 14  
 3 standard reflections monitored every 97 reflections  
 intensity decay: none

#### Refinement

Refinement on *F*<sup>2</sup>  
*R*[*F*<sup>2</sup> > 2σ(*F*<sup>2</sup>)] = 0.0523  
*wR*(*F*<sup>2</sup>) = 0.1688

( $\Delta/\sigma$ )<sub>max</sub> < 0.001  
 $\Delta\rho_{\text{max}}$  = 0.192 e Å<sup>-3</sup>  
 $\Delta\rho_{\text{min}}$  = -0.269 e Å<sup>-3</sup>

$S = 1.116$   
 4659 reflections  
 352 parameters  
 All H-atom parameters  
 refined  
 $w = 1/[\sigma^2(F_o^2) + (0.1107P)^2 + 0.0148P]$   
 where  $P = (F_o^2 + 2F_c^2)/3$

Extinction correction: none  
 Atomic scattering factors  
 from *International Tables  
 for Crystallography* (1992,  
 Vol. C, Tables 4.2.6.8 and  
 6.1.1.4)

C3—C4—C10	118.97 (13)	118.60 (12)
C3—C4—C12	121.11 (14)	121.24 (13)
C10—C4—C12	119.91 (14)	120.15 (13)
C6—C5—C10	122.06 (13)	122.46 (12)
C5—C6—C7	121.37 (13)	121.20 (13)
N13—C7—C8	121.90 (12)	122.10 (12)
N13—C7—C6	120.80 (13)	120.70 (12)
C8—C7—C6	117.30 (12)	117.18 (12)
C9—C8—C7	119.74 (12)	119.95 (11)
O1—C9—C8	115.86 (11)	115.96 (11)
O1—C9—C10	120.79 (12)	120.52 (12)
C8—C9—C10	123.36 (12)	123.52 (12)
C5—C10—C9	116.16 (12)	115.67 (12)
C5—C10—C4	125.17 (13)	125.27 (12)
C9—C10—C4	118.67 (13)	119.04 (12)
C7—N13—C14	121.82 (12)	122.10 (12)
C7—N13—C15	120.49 (13)	121.29 (13)
C14—N13—C15	117.69 (13)	116.50 (13)

Table 1. Fractional atomic coordinates and equivalent isotropic displacement parameters ( $\text{\AA}^2$ )

$$U_{eq} = (1/3)\sum_i\sum_j U_{ij}a_i^*a_j^*a_i\cdot a_j$$

	x	y	z	$U_{eq}$
<b>Molecule A</b>				
O1A	0.16905 (11)	-0.01742 (10)	-0.22478 (10)	0.0585 (3)
C2A	0.2450 (2)	0.0880 (2)	-0.2487 (2)	0.0630 (4)
C3A	0.1622 (2)	0.20738 (15)	-0.22889 (15)	0.0596 (4)
C4A	0.0184 (2)	0.21790 (13)	-0.18247 (13)	0.0513 (3)
C5A	-0.2038 (2)	0.10152 (14)	-0.10307 (15)	0.0551 (4)
C6A	-0.2679 (2)	-0.00907 (15)	-0.0772 (2)	0.0573 (4)
C7A	-0.1874 (2)	-0.12559 (13)	-0.10129 (13)	0.0483 (3)
C8A	-0.0392 (2)	-0.12352 (13)	-0.15197 (12)	0.0471 (3)
C9A	0.02201 (15)	-0.01037 (13)	-0.17628 (12)	0.0453 (3)
C10A	-0.0568 (2)	0.10530 (12)	-0.15358 (12)	0.0466 (3)
O11A	0.37571 (15)	0.06928 (14)	-0.28473 (15)	0.0940 (5)
C12A	-0.0628 (2)	0.3431 (2)	-0.1585 (2)	0.0652 (4)
N13A	-0.25293 (15)	-0.23520 (12)	-0.07567 (13)	0.0607 (4)
C14A	-0.1737 (2)	-0.35507 (14)	-0.1011 (2)	0.0635 (4)
C15A	-0.4056 (2)	-0.2347 (2)	-0.0205 (2)	0.0724 (5)
<b>Molecule B</b>				
O1B	0.40315 (11)	-0.53610 (9)	-0.26603 (10)	0.0563 (3)
C2B	0.3285 (2)	-0.42335 (15)	-0.23725 (15)	0.0587 (4)
C3B	0.4021 (2)	-0.31119 (15)	-0.27519 (15)	0.0569 (4)
C4B	0.5364 (2)	-0.31127 (13)	-0.34120 (13)	0.0502 (3)
C5B	0.7445 (2)	-0.44296 (14)	-0.44737 (14)	0.0531 (4)
C6B	0.8063 (2)	-0.55687 (14)	-0.47725 (14)	0.0537 (4)
C7B	0.7357 (2)	-0.66893 (13)	-0.43499 (13)	0.0472 (3)
C8B	0.5989 (2)	-0.65671 (13)	-0.36280 (13)	0.0474 (3)
C9B	0.53933 (14)	-0.53976 (13)	-0.33533 (12)	0.0456 (3)
C10B	0.6087 (2)	-0.42904 (13)	-0.37473 (12)	0.0461 (3)
O11B	0.20594 (14)	-0.42934 (12)	-0.18230 (13)	0.0851 (4)
C12B	0.6089 (2)	-0.1915 (2)	-0.3812 (2)	0.0651 (4)
N13B	0.79847 (13)	-0.78265 (12)	-0.46583 (13)	0.0584 (3)
C14B	0.7273 (2)	-0.89738 (14)	-0.4276 (2)	0.0618 (4)
C15B	0.9438 (2)	-0.7958 (2)	-0.5319 (2)	0.0730 (5)

Table 2. Selected bond lengths ( $\text{\AA}$ ) and angles ( $^\circ$ )

	Molecule A	Molecule B
O1—C9	1.378 (2)	1.379 (2)
O1—C2	1.382 (2)	1.378 (2)
C2—O11	1.208 (2)	1.210 (2)
C2—C3	1.432 (2)	1.432 (2)
C3—C4	1.344 (2)	1.348 (2)
C4—C10	1.443 (2)	1.439 (2)
C4—C12	1.495 (2)	1.498 (2)
C5—C6	1.367 (2)	1.361 (2)
C5—C10	1.391 (2)	1.400 (2)
C6—C7	1.412 (2)	1.419 (2)
C7—N13	1.365 (2)	1.366 (2)
C7—C8	1.400 (2)	1.403 (2)
C8—C9	1.379 (2)	1.375 (2)
C9—C10	1.394 (2)	1.398 (2)
N13—C14	1.445 (2)	1.443 (2)
N13—C15	1.449 (2)	1.437 (2)
<hr/>		
C9—O1—C2	121.51 (11)	121.59 (10)
O11—C2—O1	115.96 (14)	116.18 (13)
O11—C2—C3	126.7 (2)	126.39 (14)
O1—C2—C3	117.36 (13)	117.43 (12)
C4—C3—C2	122.59 (13)	122.71 (13)

The structure was solved in the space group  $P1$  and refined in  $P\bar{1}$ . All the H atoms were located from difference Fourier maps. The methyl H atoms of C14 were found to be disordered with two possible sites rotated by  $60^\circ$  from each other. While the remaining H atoms were refined isotropically, the two sets of the C14 H atoms were refined with 0.5 site occupancy and allowed to ride on C14 with a common isotropic temperature factor.

Data collection: XSCANS (Siemens, 1994). Cell refinement: XSCANS. Data reduction: XSCANS. Program(s) used to solve structure: SHELXS86 (Sheldrick, 1985). Program(s) used to refine structure: SHELXL93 (Sheldrick, 1993). Molecular graphics: SHELXTL/PC (Sheldrick, 1990). Software used to prepare material for publication: SHELXL93. Geometric calculations: PARST (Nardelli, 1983).

The authors thank the Malaysian Government and the Universiti Sains Malaysia for research grant R&D No. 123-3417-2201. One of the authors (KS) thanks the Universiti Sains Malaysia for a Visiting Postdoctoral Research Fellowship.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: KH1033). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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