

## 3-(Benzoxazol-2-yl)cromem-2-one

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## Key indicators

Single-crystal X-ray study

$T = 293\text{ K}$

Mean  $\sigma(\text{C}-\text{C}) = 0.003\text{ \AA}$

$R$  factor = 0.055

w $R$  factor = 0.120

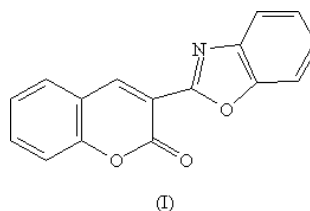
Data-to-parameter ratio = 16.4

For details of how these key indicators were  
 automatically derived from the article, see  
<http://journals.iucr.org/e>.

The coumarin and benzoxazole ring systems in the title compound,  $\text{C}_{16}\text{H}_9\text{NO}_3$ , are planar. The angle between them is  $5.24(8)^\circ$ . The crystal structure is stabilized by intermolecular  $\text{C}-\text{H}\cdots\text{O}$  and  $\text{C}-\text{H}\cdots\text{N}$  attractive interactions.

## Comment

Coumarin derivatives are known to be a very interesting class of natural and synthetic compounds, which afford extensive and diverse applications (O'Kennedy & Thornes, 1997; Krasovitskii, 1988). These compounds exhibit strong fluorescence in the visible region and their properties vary according to the substituents on the coumarin ring (Novak & Kovac, 2000; Machado & Miranda, 2001).



The title compound, (I) (Luan *et al.*, 2002), shows good photochemical stability, high-fluorescence quantum yields ( $\Phi_f$ ) in solvents of different polarities, and generates singlet oxygen with a relatively low quantum efficiency (Machado *et al.*, 2002). These characteristics make it suitable for use as a laser dye in the range between 438 and 450 nm.

X-ray analysis of (I) was undertaken as a part of studies to elucidate the relationships between the characteristics of the functional dyes containing the coumarin skeletons and their molecular structures.

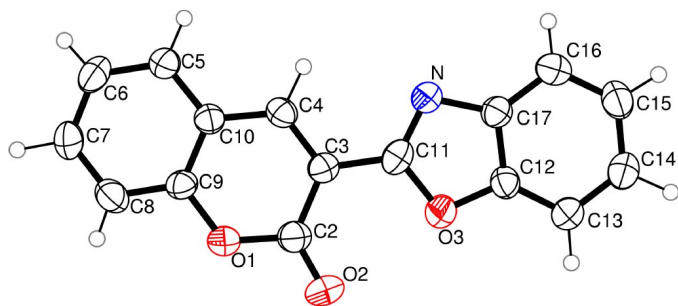
An ORTEP-3 (Farrugia, 1997) drawing of (I) is shown in Fig. 1, and selected geometric parameters presented in Table 1. The angle between the benzoxazole and the coumarin moieties is  $5.24(8)^\circ$ , showing some deviation from planarity, as observed for other coumarin derivatives (Dhaneshwar *et al.*, 1988; Chinnakali *et al.*, 1990). The two ring systems are each planar.

The coumarin skeleton geometry can be compared with that of unsubstituted coumarin (Gavuzzo *et al.*, 1974), of coumarin-3-carboxylic acid (Dobson & Gerkin, 1996) and of 3-(bromoacetyl)coumarin (Vasudevan *et al.*, 1991). The double-bond character of C3–C4 is observed in all these compounds. The C2–C3 and C4–C10 bonds, adjacent to the double bond, are systematically longer than 1.40 Å in these four molecules. The angles C8–C9–O1 and C5–C10–C4 at the junction of the two rings are, respectively, smaller and greater than  $120^\circ$ . The angles around C2 follow the same behavior in all the compounds.

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**Figure 1**  
An ORTEP-3 view (Farrugia, 1997) of (I), showing 50% probability displacement ellipsoids.

Compared with unsubstituted coumarin (Gavuzzo *et al.*, 1974), the decrease in the C2–C3–C4 bond angle by 2° and increase in C3–C4–C10 by the same amount may be due to the benzoxazole group substituted at C3. The bond distances and angles in the benzoxazole moiety are within normally expected ranges. The bond length C3–C11 is in good agreement, within experimental error, with those observed in 3-(2-benzimidazolyl)-7-(diethylamino)coumarin (Chinnakali *et al.*, 1990) and 3-(2-benzothiazolyl)-7-(diethylamino)coumarin (Jasinski & Paight, 1995).

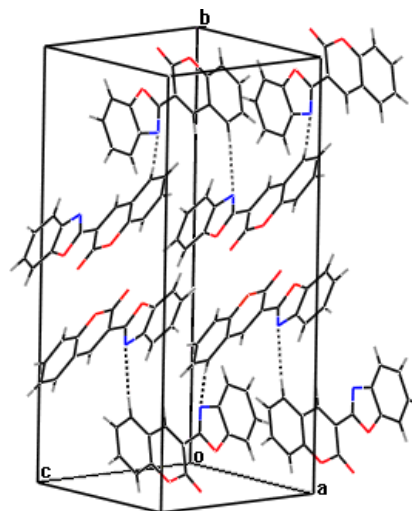
In this crystal structure the molecule is linked to inversion- and glide-related neighbours by several directionally specific C–H···O and C–H···N interactions (Table 2). The  $Csp^2-H\cdots O$  interaction links the molecules in an infinite zigzag in the [102] direction, while the  $Csp^2-H\cdots N$  produces an infinite zigzag in the [001] direction (Fig. 2). The shape and disposition of molecules in the structure and the rather high density (1.491 Mg m<sup>-3</sup>; values for analogous molecules typically lie in the range 1.34–1.42 Mg m<sup>-3</sup>) suggest that these close approaches are produced by attractive interactions rather than as a consequence of minimizing repulsive effects elsewhere in the molecule. Similar behavior is observed for the 7-acetoxycoumarin (Sarma & Desiraju, 1987) and coumarin 3-carboxylic acid (Dobson & Gerkin, 1996).

## Experimental

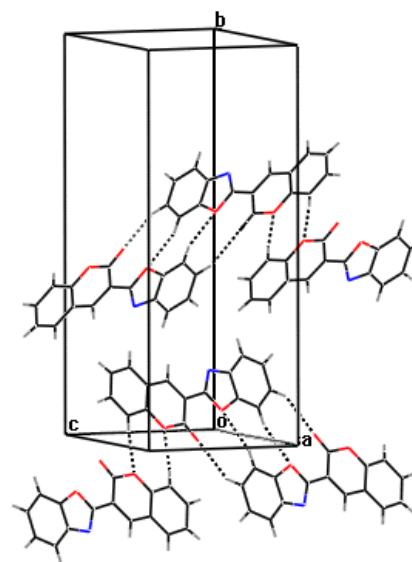
The title compound was prepared and purified according to Luan *et al.* (2002). Suitable single crystals were grown from chloroform/methanol (1:1 *v*) by slow evaporation.

### Crystal data

C <sub>16</sub> H <sub>9</sub> NO <sub>3</sub>	$D_x = 1.491 \text{ Mg m}^{-3}$
$M_r = 263.24$	Mo $K\alpha$ radiation
Monoclinic, $P2_1/c$	Cell parameters from 25 reflections
$a = 7.493 (5) \text{ \AA}$	$\theta = 2.1\text{--}30^\circ$
$b = 21.276 (5) \text{ \AA}$	$\mu = 0.11 \text{ mm}^{-1}$
$c = 8.024 (5) \text{ \AA}$	$T = 293 (2) \text{ K}$
$\beta = 113.508 (5)^\circ$	Prism, yellow
$V = 1173.0 (11) \text{ \AA}^3$	$0.25 \times 0.20 \times 0.10 \text{ mm}$
$Z = 4$	



(a)



(b)

**Figure 2**

View of the (a) C–H···N and (b) C–H···O intermolecular interactions in the crystal structure of (I).

### Data collection

Enraf–Nonius Turbo-CAD-4 diffractometer	$\theta_{\max} = 30.0^\circ$
Non-profiled $\omega/2\theta$ scans	$h = 0 \rightarrow 10$
Absorption correction: none	$k = -29 \rightarrow 0$
3638 measured reflections	$l = -11 \rightarrow 10$
3409 independent reflections	3 standard reflections
1232 reflections with $I > 2\sigma(I)$	frequency: 120 min
$R_{\text{int}} = 0.022$	intensity decay: 6%

### Refinement

Refinement on $F^2$	Only coordinates of H atoms refined
$R[F^2 > 2\sigma(F^2)] = 0.055$	$w = 1/[\sigma^2(F_o^2) + (0.0479P)^2]$
$wR(F^2) = 0.120$	where $P = (F_o^2 + 2F_c^2)/3$
$S = 0.80$	$(\Delta/\sigma)_{\max} = 0.001$
3409 reflections	$\Delta\rho_{\max} = 0.23 \text{ e \AA}^{-3}$
208 parameters	$\Delta\rho_{\min} = -0.19 \text{ e \AA}^{-3}$

**Table 1**

Selected geometric parameters (Å, °).

O1—C9	1.374 (2)	C3—C4	1.356 (3)
O1—C2	1.381 (3)	C3—C11	1.460 (3)
O2—C2	1.203 (2)	C4—C10	1.420 (3)
C2—C3	1.462 (3)	C9—C10	1.392 (3)
O2—C2—O1	116.2 (2)	C3—C4—C10	122.0 (2)
O2—C2—C3	127.6 (2)	O1—C9—C8	117.7 (2)
O1—C2—C3	116.19 (19)	C5—C10—C4	123.8 (2)
C4—C3—C2	120.0 (2)		
C4—C3—C11—N	6.5 (3)	C4—C3—C11—O3	−173.7 (2)
C2—C3—C11—N	−173.4 (2)	C2—C3—C11—O3	6.4 (3)

**Table 2**

Intermolecular interaction geometry (Å, °).

$D-H \cdots A$	$D-H$	$H \cdots A$	$D \cdots A$	$D-H \cdots A$
C5—H5 <sup>i</sup> ···N <sup>i</sup>	0.97 (2)	2.66 (2)	3.626 (3)	175.9 (17)
C14—H14 <sup>ii</sup> ···O2 <sup>ii</sup>	1.02 (2)	2.53 (2)	3.283 (3)	130.7 (15)
C13—H13 <sup>iii</sup> ···O3 <sup>iii</sup>	0.97 (2)	2.65 (2)	3.572 (3)	160.0 (17)
C8—H8 <sup>iii</sup> ···O1 <sup>iii</sup>	0.88 (2)	2.61 (2)	3.487 (3)	172 (2)

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

H atoms were located by difference Fourier synthesis; the positional parameters have been refined with  $U_{iso}$  set to 1.2 times the value of  $U_{eq}$  of the atom to which they are attached. The final refined C—H distances ranged from 0.88 (2) to 1.02 (2) Å, with a mean value of 0.96 (2) Å.

Data collection: *CAD-4 EXPRESS Software* (Enraf-Nonius, 1994); cell refinement: *CAD-4 EXPRESS Software*; data reduction: *XCAD4* (Harms & Wocadlo, 1995); program(s) used to solve structure: *SIR97* (Altomare *et al.*, 1999); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3*

for Windows (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

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