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7-Ethylamino-4-(trifluoromethyl)coumarin

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Key indicators

Single-crystal X-ray study
T = 296 K
Mean σ(C–C) = 0.005 Å
Disorder in main residue
R factor = 0.062
wR factor = 0.168
Data-to-parameter ratio = 17.2

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

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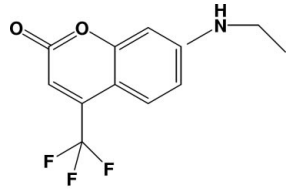
The title compound, $C_{12}H_{10}F_3NO_2$, also known as coumarin 500, exhibits a planar coumarin ring system with an ethyl group essentially perpendicular to it. The molecules form layers, which are stacked along the *c* axis.

Comment

The title compound, (I), a laser dye aminocoumarin compound, has been found to be very useful with various laser pump sources, exhibiting a lasing maximum in the range 470–522 nm (Bos, 1981; Alcock *et al.*, 1978; Eschrich & Morgan, 1985; Gunthals & Nibler, 1979). Bond lengths and angles in the rigid coumarin ring system (Fig. 1) are similar to those observed in the structurally related compounds: 7-amino-4-trifluoromethylcoumarin (coumarin 151 or 490; Selladurai & Subramanian, 1992), 7-dimethylamino-4-trifluoromethylcoumarin polymorphs (coumarin 152A and 152B; Jasinski & Paight, 1994; Chinnakali *et al.*, 1990), and 7-diethylaminocoumarin (coumarin 466; Yufit *et al.*, 1991), 7-ethylamino-6-methyl-4-trifluoromethylcoumarin (coumarin 307; Chinnakali *et al.*, 1992*a*), and 7-diethylamino-4-trifluoromethylcoumarin (Chinnakali *et al.*, 1992*b*). However, (I) exhibits disorder of atoms C14 and C15, which has been modelled with a restrained C14—C15 bond length of 1.52 Å in both components. The benzene and pyrone rings are planar (r.m.s. deviations = 0.0012 and 0.0137 Å) and the angle between the least-squares planes of the rings is 0.65 (18)°.



(I)

The amino N atom, N13, trifluoromethyl carbon, C12, and carboxyl oxygen, O11, are all coplanar with the coumarin ring, with deviations of −0.013 (4), 0.069 (5) and 0.096 (4) Å, respectively. The ethyl group is essentially perpendicular to the plane of the coumarin ring [C7—N13—C14*A*—C15*A* = −84.8 (7)° and C7—N13—C14*B*—C15*B* = −166.9 (13)°].

A packing diagram of the structure (Fig. 2) indicates that the molecules form layers, which are stacked along the *c* axis, with the parallel planes of the coumarin rings inclined at an angle to the axis and a closest contact interlayer spacing of 3.64 (2) Å. Intermolecular hydrogen-bonding interactions are detailed in Table 1.

In three of the structurally related aminocoumarin systems mentioned earlier (coumarin 151, 152A and 152B, and 307)

crystallization also occurs in the triclinic space group $P\overline{1}$, while coumarin 466 crystallizes in the space group $P2_1/n$. In all of these systems, the molecules stack in parallel layers, as does the title compound.

Experimental

Commercial coumarin 500 (Exiton Inc.) was crystallized by slow evaporation from acetonitrile.

Crystal data

$C_{12}H_{10}F_3NO_2$
$M_r = 257.21$
Triclinic, $P\overline{1}$
$a = 10.567\ (6)$ Å
$b = 11.856\ (2)$ Å
$c = 4.6321\ (11)$ Å
$\alpha = 93.303\ (18)^\circ$
$\beta = 100.25\ (3)^\circ$
$\gamma = 99.14\ (3)^\circ$
$V = 561.6\ (4)$ Å^3
$Z = 2$
$D_x = 1.521$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 20 reflections
$\theta = 30.2$–39.4°
$\mu = 0.14$ mm^{-1}
$T = 296\ (2)$ K
Needle, colorless
0.90 × 0.30 × 0.10 mm

Data collection

Rigaku AFC-6*S* diffractometer
$2\theta/\omega$ scans
Absorption correction: ψ scan (North *et al.*, 1968)
$T_{min} = 0.887$, $T_{max} = 0.987$
3292 measured reflections
3126 independent reflections
965 reflections with $I > 2\sigma(I)$
$R_{int} = 0.061$
$\theta_{max} = 30.0^\circ$
$h = 0 \rightarrow 13$
$k = -16 \rightarrow 16$
$l = -6 \rightarrow 6$
3 standard reflections
every 150 reflections
intensity decay: 2.2%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.062$
$wR(F^2) = 0.168$
$S = 0.90$
3126 reflections
182 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0477P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.20$ e Å^{-3}
$\Delta\rho_{min} = -0.19$ e Å^{-3}

Table 1
Hydrogen-bonding geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
N13—H13···O11^{i}	0.96	2.19	3.124 (4)	163
C8—H8A···O1^{i}	0.93	2.51	3.401 (4)	161

Symmetry code: (i) $1-x, 1-y, 2-z$.

The H atoms on C3, C5, C6, C8 and N13 were included in their calculated positions as riding atoms. Distance and angle restraints were applied to the ethyl H atoms on the disordered C14 and C15 atoms; refined occupancy factors were 0.725 (8) and 0.275 (8), respectively.

Data collection: *MSC/AFC Diffractometer Control Software* (Molecular Structure Corporation, 1998); cell refinement: *MSC/AFC Diffractometer Control Software*; data reduction: *TEXSAN* (Molecular Structure Corporation, 1998); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *TEXSAN*; software used to prepare material for publication: *TEXSAN*.

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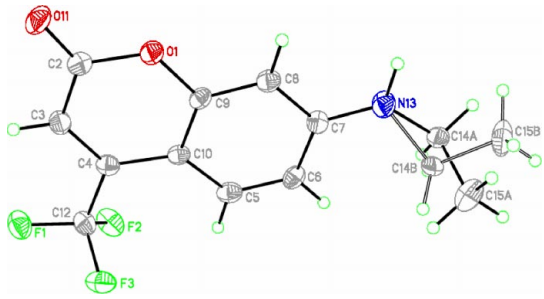

Figure 1
*ORTEP*II (Johnson, 1976) drawing of the title compound, showing ellipsoids at the 50% probability level and the atomic numbering scheme.

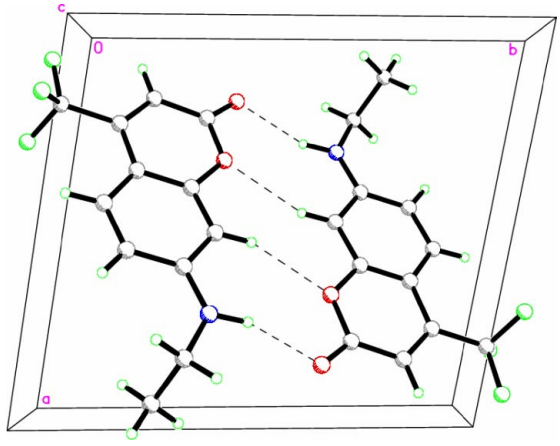

Figure 2
*ORTEP*II (Johnson, 1976) packing diagram of the title compound, viewed down the *c* axis. Only one component of the disorder is shown.

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