Structure of 7-Ethylamino-6-methyl-4-trifluoromethylcoumarin

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Abstract. $C_{13}H_{12}F_3NO_2$, $M_r = 271.2$, triclinic, $P\bar{1}$, $a = 5.029$ (2), $b = 7.479$ (2), $c = 17.073$ (5) Å, $\alpha = 97.98$ (2), $\beta = 95.54$ (3), $\gamma = 103.62$ (3)°, $V = 612.4$ (4) Å^3, $Z = 2$, $D_m = 1.463$, $D_x = 1.471$ g cm^{-3}, $\lambda(Mo\ K\alpha) = 0.71069$ Å, $\mu = 1.23$ cm^{-1}, $F(000) = 280$, $T = 298$ K, final R value is 0.041 for 2047 observed reflections with $|F_o| \geq 6\sigma(|F_o|)$. The N—C($sp^2$) bond length is 1.356 (2) Å. The N and C atoms of the ethylamino group deviate by <0.15 Å from the plane of the aromatic ring. Short intramolecular contacts, C(3)···F(17) 2.668 (3) Å [H(3)···F(17) 2.39 (2) Å, C(3)—H(C3)···F(17) 98 (1)°], C(5)···F(18) 3.074 (3) and C(5)···F(19) 3.077 (3) Å exist in the structure. The crystal structure is stabilized by intermolecular N—H···O hydrogen bonds with N(12)—H(N12) 0.79 (3), H(N12)···O(11)′ 2.36 (3), N(12)···O(11)′ ($x - 1, y + 1$, z) 3.105 (3) Å and N(12)—H(N12)···O(11)′ 155 (2)°.

Experimental. Compound from Exciton Chemicals Co., USA, greenish-yellow needle-shaped crystals from a mixture of aqueous ethanol and chloroform, density measured by flotation. Crystal dimensions $0.3 \times 0.4 \times 0.2$ mm. Enraf–Nonius CAD-4 diffractometer, graphite-monochromated Mo $K\alpha$ radiation, cell dimensions determined from 25 2θ angles in the range $9.7 < 2\theta < 18.9°$. Intensities measured up to $2\theta = 56°$, hkl range: $h = 0$ to 6, $k = -9$ to 9 and $l =$

Fig. 1. Atomic numbering scheme.

Table 1. *Fractional atomic coordinates and equivalent isotropic temperature factors for the non-*H *atoms*

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

	x	y	z	$U_{eq}(\text{Å}^2)$
O(1)	0.1982 (2)	−0.2853 (1)	0.1776 (1)	0.0465 (4)
C(2)	0.3045 (3)	−0.4032 (2)	0.2190 (1)	0.0448 (5)
C(3)	0.2662 (3)	−0.3936 (2)	0.3022 (1)	0.0438 (5)
C(4)	0.1306 (3)	−0.2748 (2)	0.3369 (1)	0.0375 (5)
C(5)	−0.1274 (3)	−0.0242 (2)	0.3220 (1)	0.0370 (4)
C(6)	−0.2227 (3)	0.0890 (2)	0.2755 (1)	0.0366 (4)
C(7)	−0.1721 (3)	0.0781 (2)	0.1941 (1)	0.0383 (4)
C(8)	−0.0281 (3)	−0.0500 (2)	0.1641 (1)	0.0423 (5)
C(9)	0.0597 (3)	−0.1635 (2)	0.2126 (1)	0.0372 (5)
C(10)	0.0182 (3)	−0.1544 (2)	0.2927 (1)	0.0355 (5)
O(11)	0.4227 (3)	−0.5037 (2)	0.1830 (1)	0.0644 (5)
N(12)	−0.2596 (3)	0.1914 (2)	0.1475 (1)	0.0473 (5)
C(13)	−0.1953 (4)	0.1986 (2)	0.0667 (1)	0.0502 (6)
C(14)	−0.3237 (5)	0.3344 (3)	0.0294 (1)	0.0628 (7)
C(15)	−0.3768 (3)	0.2252 (2)	0.3096 (1)	0.0448 (5)
C(16)	0.1044 (3)	−0.2677 (2)	0.4245 (1)	0.0470 (5)
F(17)	0.2203 (3)	−0.3874 (2)	0.4569 (1)	0.0762 (6)
F(18)	−0.1585 (2)	−0.3066 (2)	0.4377 (1)	0.0635 (4)
F(19)	0.2242 (2)	−0.0983 (2)	0.4659 (1)	0.0718 (5)

−22 to 22, ω–2θ scans, three standard reflections ($\bar{2}04$, $\bar{3}3\bar{1}$, 331) monitored every 150 measurements showed no significant change. 2662 unique reflections measured, 2047 observed with $|F_o| \geq 6\sigma(|F_o|)$, Lp corrections but no absorption correction. Structure solved by direct methods using *SHELXS*86 (Sheldrick, 1986), no solution in space group $P\bar{1}$, attempts with $P1$ gave the correct solution with two centrosymmetrically related molecules. Origin shifted to that centre of symmetry, full-matrix least-squares refinement on F, using *SHELX*76 (Sheldrick, 1976), for space group $P\bar{1}$. H atoms located on a difference Fourier map, anisotropic thermal parameters for non-H and isotropic for H atoms, final $R = 0.041$, $wR = 0.053$, $w = 1.0/[\sigma^2(|F_o|) + 0.00351|F_o|^2]$, $S = 1.10$ for 220 parameters, $(\Delta/\sigma)_{max} = 0.002$, final difference map featureless with maximum and minimum peak heights 0.20 and -0.22 e Å^{-3}; no correction for secondary extinction, atomic scattering factors for all atoms as in *SHELX*76 (Sheldrick, 1976), geometrical calculations using *PARST* (Nardelli, 1983). The atomic numbering scheme is
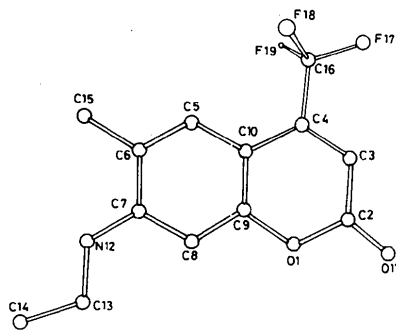
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Table 2. *Bond lengths (Å) and bond angles (°)*

O(1)—C(2)	1.373 (2)	C(6)—C(15)	1.507 (2)
O(1)—C(9)	1.378 (2)	C(7)—C(8)	1.402 (2)
C(2)—C(3)	1.446 (2)	C(7)—N(12)	1.356 (2)
C(2)—O(11)	1.204 (2)	C(8)—C(9)	1.379 (2)
C(3)—C(4)	1.350 (2)	C(9)—C(10)	1.398 (2)
C(4)—C(10)	1.430 (2)	N(12)—C(13)	1.452 (2)
C(4)—C(16)	1.509 (2)	C(13)—C(14)	1.504 (3)
C(5)—C(6)	1.371 (2)	C(16)—F(17)	1.323 (2)
C(5)—C(10)	1.416 (2)	C(16)—F(18)	1.333 (2)
C(6)—C(7)	1.432 (2)	C(16)—F(19)	1.333 (2)

C(2)—O(1)—C(9)	122.3 (2)	O(1)—C(9)—C(8)	115.7 (2)
O(1)—C(2)—O(11)	117.1 (2)	C(8)—C(9)—C(10)	122.8 (2)
O(1)—C(2)—C(3)	116.8 (2)	O(1)—C(9)—C(10)	121.5 (2)
C(3)—C(2)—O(11)	126.1 (2)	C(5)—C(10)—C(9)	116.4 (2)
C(3)—C(4)—C(16)	118.6 (2)	C(4)—C(10)—C(9)	116.9 (2)
C(3)—C(4)—C(10)	121.3 (2)	C(4)—C(10)—C(5)	126.7 (2)
C(10)—C(4)—C(16)	120.1 (2)	C(7)—N(12)—C(13)	123.1 (2)
C(6)—C(5)—C(10)	122.7 (2)	N(12)—C(13)—C(14)	111.2 (2)
C(5)—C(6)—C(15)	120.6 (2)	C(4)—C(16)—F(19)	111.3 (2)
C(5)—C(6)—C(7)	119.3 (2)	C(4)—C(16)—F(18)	112.1 (2)
C(7)—C(6)—C(15)	120.1 (2)	C(4)—C(16)—F(17)	112.6 (2)
C(6)—C(7)—N(12)	120.3 (2)	F(18)—C(16)—F(19)	106.5 (2)
C(6)—C(7)—C(8)	118.7 (2)	F(17)—C(16)—F(19)	107.1 (2)
C(8)—C(7)—N(12)	120.9 (2)	F(17)—C(16)—F(18)	106.9 (2)
C(7)—C(8)—C(9)	120.0 (2)		

shown in Fig. 1. The final fractional atomic coordinates and the equivalent isotropic temperature factors for the non-H atoms are given in Table 1; the bond lengths and bond angles involving these atoms are listed in Table 2.*

* List of structure factors, anisotropic thermal parameters, H-atom coordinates, distances and angles involving H atoms, torsion angles, least-squares planes and intermolecular distances less than 3.5 Å have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54491 (19 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: MU0271]

Related literature. The title compound, a laser dye also known as coumarin 503, has been found to give laser emissions around 503 nm in alcohols (Drexhage & Reynolds, 1974; Reynolds & Drexhage, 1975). The structural study was performed as part of our program on the structural aspects of aminocoumarin laser dyes. The structural details of related aminocoumarin dyes have been published elsewhere (Messager & Delugeard, 1974; Chinnakali, Sivakumar & Natarajan, 1989, 1990; Chinnakali, Selladurai, Sivakumar, Subramanian & Natarajan, 1990).

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Structure of 1-(3-Deoxy-3-phenylseleno-2,5-di-*O*-pivaloyl-β-D-xylofuranosyl)uracil

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Abstract. $C_{25}H_{23}N_2O_7Se$, $M_r = 542.43$, monoclinic, C2, $a = 21.078$ (1), $b = 7.036$ (2), $c = 19.603$ (1) Å, $\beta = 109.05$ (1)°, $V = 2748.1$ (7) Å^3, $Z = 4$, $D_x = 1.311$ Mg m^{-3}, λ(Cu $K\alpha_1$) = 1.54050 Å, $\mu = 2.238$ mm^{-1}, $F(000) = 1108$, $T = 295$ K, final $R = 0.037$ for 2191 reflections. The sugar conformation and puckering parameters are ^2E with $P = -15.8°$ and $\psi_m = 40.3°$. The N-glycosidic torsion angle χ has a value of 142.6 (4)° in the *anti* range. The C4'—C5' side-chain conformation is $-ap$ with $\gamma = -175.7$ (6)°. The conformation parameters are in accordance with the IUPAC–IUB Joint Commission