Bausch, M. J., David, B., Dobrowolski, P., Guadalupe-Fasano, C., Gustowski, R., Selmarten, D., Prasad, V., Vaughn, A. & Wang, L.-H. (1991). *J. Org. Chem.* **56**, 5643–5651.
Belaj, F. (1992). *Acta Cryst.* C**48**, 1088–1090.
Jackson, D. E., Bycroft, B. W. & King, T. J. (1988). *J. Comput.-Aided Mol. Design*, **2**, 321–328.
Johnson, C. K. (1965). *ORTEP*. Report ORNL-3794. Oak Ridge National Laboratory, Tennessee, USA.
Kolb, V. M., Dworkin, J. P. & Miller, S. L. (1994). *J. Mol. Evol.* **38**, 549–557.
Molecular Structure Corporation (1985). *TEXSAN. TEXRAY Structure Analysis Package*. MSC, 3200 Research Forest Drive, The Woodlands, TX 77381, USA.
Molecular Structure Corporation (1988). *MSC/AFC Diffractometer Control Software*. MSC, 3200 Research Forest Drive, The Woodlands, TX 77381, USA.
Sheldrick, G. M. (1985). *SHELXS86. Crystallographic Computing* 3, edited by G. M. Sheldrick, C. Krüger & R. Goddard, pp. 175–189. Oxford Univ. Press.
Zachariasen, W. H. (1963). *Acta Cryst.* **16**, 1139–1144.

Acta Cryst. (1994). C**50**, 1928–1930

7-Dimethylamino-4-trifluoromethylcoumarin, a New Polymeric Form

JERRY P. JASINSKI AND ERNEST S. PAIGHT†

Chemistry Department, Keene State College,
229 Main Street, Keene, New Hampshire 03431, USA

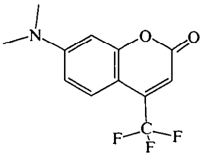
(*Received* 21 *October* 1992; *accepted* 5 *April* 1994)

Abstract

This molecule represents a new polymorph of the title laser-dye aminocoumarin compound, $C_{12}H_{10}F_3NO_2$. Torsion angles in the trifluoro moiety and intermolecular packing effects indicate significant structural differences when compared to its polymorphic analog. The crystal structure is stabilized by an intermolecular C—H···O contact of 3.324 (4) Å.

Comment

The title compound (I), also known as coumarin 152 (Eastman Kodak Co.) or coumarin 485 (Exiton Chemical Co.), is a fluorinated aminocoumarin and is used as a laser dye in the blue–green (Drexhage, 1973) as well as in the ultraviolet spectral regions (Eschrich & Morgan, 1985). Its structural features, including bond lengths and bond angles, closely resemble those of its polymorphic counterpart within experimental error (Chinnakali, Sivakumar & Natarajan, 1990), except for the torsion angles involving the F atoms [C3—C4—C12—F12*A* = 3.5 (4), C3—C4—C12—F12*B* = −11.64 (3), C3—C4—C12—F12*C* = 123.9 (3)°]. The C3—C4 bond of 1.340 (4) Å is clearly double in nature. The independent C—F bond lengths of 1.331 (3), 1.333 (4) and 1.328 (4) Å are normal (Krishnamurthy & Vijayan, 1979; Chinnakali, Sivakumar & Natarajan, 1992). Since this polymorph crystallizes in a different space group, $P2_1/a$, its packing characteristics are somewhat different. In addition, excited-state charge transfer from the amino (donor) to the carboxyl (acceptor) group (Masilamani, Chandrasekar, Sivaram, Sivasankar & Natarajan, 1986) is most likely influenced by the trifluoromethyl group in a somewhat different fashion (Guo & Feng, 1987).



(I)

There exists an intermolecular hydrogen bond of the type C—H···O [C3···O11^i 3.324 (4), C3—H3 0.91 (3), H3···O11^i 2.42 (4) Å, C3—H3···O11^i 168 (3)°; symmetry code: (i) $1 - x$, $-y$, $1 - z$] (Taylor & Kennard, 1982).

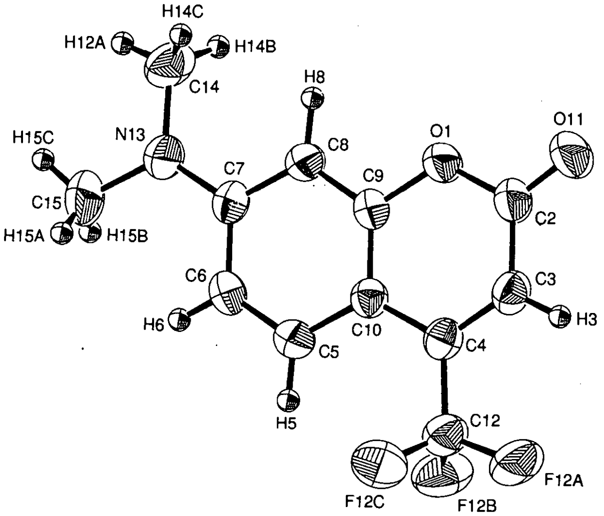


Fig. 1. *ORTEP* (Johnson, 1965) drawing of the title molecule with 50% probability ellipsoids and atomic numbering scheme.

Experimental

Crystals of the title compound were grown from ethanol by slow evaporation and were supplied by Eastman Kodak Co., Rochester, NY 14650, USA.

---

† Present address: Pfizer Inc., Central Research Division, Eastern Point Road, Groton, CT 06340, USA.

Crystal data

$C_{12}H_{10}F_3NO_2$
$M_r = 257.21$
Monoclinic
$P2_1/a$
$a = 7.2796\ (7)$ Å
$b = 15.755\ (2)$ Å
$c = 9.834\ (1)$ Å
$\beta = 91.087\ (9)^\circ$
$V = 1127.7\ (2)$ Å^3
$Z = 4$
$D_x = 1.515$ Mg m^{-3}

Mo $K\alpha$ radiation
$\lambda = 0.7107$ Å
Cell parameters from 25 reflections
$\theta = 22.7$–25.0°
$\mu = 1.289$ cm^{-1}
$T = 298.0$ K
Prism
$0.80 \times 0.75 \times 0.35$ mm
Clear yellow prism

Data collection

AFC-6*S* diffractometer
$\omega/2\theta$ scans
Absorption correction: empirical (ψ scans)
$T_{\min} = 0.876$, $T_{\max} = 1.000$
2073 measured reflections
2240 independent reflections
1460 observed reflections [$I > 3.00\sigma(I)$]

$R_{\text{int}} = 0.0328$
$\theta_{\max} = 25^\circ$
$h = 0 \rightarrow 9$
$k = 0 \rightarrow 19$
$l = -12 \rightarrow 12$
3 standard reflections monitored every 150 reflections
intensity variation: 0.99%

Refinement

Refinement on F
$R = 0.0445$
$wR = 0.0654$
$S = 2.888$
1460 reflections
204 parameters
All H-atom parameters refined (isotropic)
$w = 4F_o^2/\sigma^2(F_o^2)$
$(\Delta/\sigma)_{\max} = 0.0067$

$\Delta\rho_{\max} = 0.29$ e Å^{-3}
$\Delta\rho_{\min} = -0.32$ e Å^{-3}
Extinction correction: Zachariasen (1963), type 2 Gaussian isotropic
Extinction coefficient: $2.833\ (7) \times 10^{-5}$
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

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

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

	x	y	z	U_{eq}
F12*A*	0.8721 (3)	0.1460 (1)	0.3714 (2)	0.0809
F12*B*	0.7789 (3)	0.2475 (1)	0.2441 (2)	0.0767
F12*C*	0.9335 (3)	0.1456 (2)	0.1603 (2)	0.0803
O1	0.3010 (3)	0.0326 (1)	0.1646 (2)	0.0470
O11	0.3097 (3)	−0.0220 (2)	0.3702 (2)	0.0760
N13	0.2466 (4)	0.1362 (2)	−0.2863 (2)	0.0538
C2	0.3848 (4)	0.0229 (2)	0.2895 (3)	0.0506
C3	0.5550 (4)	0.0677 (2)	0.3135 (3)	0.0452
C4	0.6277 (4)	0.1168 (2)	0.2171 (3)	0.0402
C5	0.6007 (4)	0.1755 (2)	−0.0231 (3)	0.0455
C6	0.5059 (4)	0.1789 (2)	−0.1437 (3)	0.0471
C7	0.3401 (4)	0.1332 (2)	−0.1651 (3)	0.0408
C8	0.2761 (4)	0.0855 (2)	−0.0564 (3)	0.0407
C9	0.3752 (3)	0.0828 (2)	0.0638 (3)	0.0371
C10	0.5400 (3)	0.1259 (2)	0.0865 (3)	0.0375
C12	0.8030 (4)	0.1637 (2)	0.2484 (3)	0.0522
C14	0.0726 (5)	0.0927 (3)	−0.3046 (4)	0.0628
C15	0.3179 (7)	0.1817 (3)	−0.4013 (4)	0.0687

Table 2. *Selected geometric parameters* (Å, °)

F12*A*—C12	1.331 (3)	C3—C4	1.340 (4)
F12*B*—C12	1.333 (4)	C4—C10	1.431 (3)
F12*C*—C12	1.328 (4)	C4—C12	1.501 (4)
O1—C2	1.369 (3)	C5—C6	1.362 (4)
O1—C9	1.385 (3)	C5—C10	1.409 (4)
O11—C2	1.203 (4)	C6—C7	1.418 (4)
N13—C7	1.362 (3)	C7—C8	1.394 (4)
N13—C14	1.448 (4)	C8—C9	1.374 (4)
N13—C15	1.444 (4)	C9—C10	1.393 (4)
C2—C3	1.441 (4)		
C2—O1—C9	122.1 (2)	N13—C7—C8	121.4 (3)
C7—N13—C14	120.9 (3)	C6—C7—C8	117.1 (2)
C7—N13—C15	121.4 (3)	C7—C8—C9	119.9 (3)
C14—N13—C15	117.7 (3)	O1—C9—C8	115.3 (2)
O1—C2—O11	117.2 (3)	O1—C9—C10	120.7 (2)
O1—C2—C3	117.4 (2)	C8—C9—C10	124.0 (2)
O11—C2—C3	125.4 (3)	C4—C10—C5	126.9 (2)
C2—C3—C4	121.1 (3)	C4—C10—C9	117.6 (2)
C3—C4—C10	121.1 (3)	C5—C10—C9	115.4 (2)
C3—C4—C12	119.0 (3)	F12*A*—C12—F12*B*	106.4 (2)
C10—C4—C12	119.9 (2)	F12*A*—C12—F12*C*	106.6 (3)
C6—C5—C10	121.8 (3)	F12*A*—C12—C4	112.9 (3)
C5—C6—C7	121.6 (3)	F12*B*—C12—F12*C*	106.6 (3)
N13—C7—C6	121.4 (3)	F12*B*—C12—C4	111.7 (3)

Weak reflections [$I < 10.0\sigma(I)$] were rescanned (maximum of two rescans) and the counts accumulated to assure good counting statistics. Lp correction was applied, but no decay correction. The weighting scheme was based on counting statistics and included a factor ($p = 0.03$) to down-weight intense reflections. Plots of $\Sigma w(|F_o| - |F_c|)^2$ *versus* $|F_o|$, reflection order in data collection, $\sin\theta/\lambda$, and various classes of indices showed no unusual trends.

Data collection and cell refinement: *MSC/AFC Diffractometer Control Software* (Molecular Structure Corporation, 1988). All calculations were performed on a VAXstation 3520 minicomputer using the *TEXSAN* (Molecular Structure Corporation, 1985) software package. Atomic positions were obtained from the direct-methods program *MITHRIL* (Gilmore, 1984). The entaniomorphs are indistinguishable.

Lists of structure factors, anisotropic displacement parameters and H-atom coordinates have been deposited with the IUCr (Reference: CD1035). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

References

Chinnakali, K., Sivakumar, K. & Natarajan, S. (1990). *Acta Cryst.* C**46**, 833–835.

Chinnakali, K., Sivakumar, K. & Natarajan, S. (1992). *Acta Cryst.* C**48**, 386–387.

Drexhage, K. H. (1973). In *Dye Lasers*, *Topics in Applied Physics*, edited by F. P. Schaefer, Vol. 1, ch. 4. New York: Springer-Verlag.

Eschrich, T. C. & Morgan, T. L. (1985). *Appl. Opt.* **24**, 937–939.

Krishnamurthy, H. M. & Vijayan, M. (1979). *Acta Cryst.* B**35**, 262–263.

Gilmore, C. J. (1894). *J. Appl. Cryst.* **17**, 42–46.

Guo, C. & Feng, Y. B. (1987). *J. Chem. Soc. Faraday Trans.* 1, **83**, 2533–2539.

Johnson, C. K. (1965). *ORTEP*. Report ORNL-3794. Oak Ridge National Laboratory, Tennessee, USA.

Masilamani, V., Chandrasekar, V., Sivaram, B., Sivasankar, B. & Natarajan, S. (1986). *Opt. Commun.* **59**, 203–207.

Molecular Structure Corporation (1985). *TEXSAN. TEXRAY Structure Analysis Package*. MSC, 3200 Research Forest Drive, The Woodlands, TX 77381 USA.

Molecular Structure Corporation (1988). *MSC/AFC Diffractometer Control Software*. MSC, 3200 Research Forest Drive, The Woodlands, TX 77381, USA.

Taylor, R. & Kennard, O. (1982). *J. Am. Chem. Soc.* **104**, 5063–5070.

Zachariasen, W. H. (1963). *Acta Cryst.* **16**, 1139–1144.

Acta Cryst. (1994). **C50**, 1930–1931

trans-5-Nitro-2-furanacrylic Acid

TOMASZ A. OLSZAK, OSWALD M. PEETERS,
NORBERT M. BLATON AND CAMIEL J. DE RANTER

*Laboratorium voor Analytische Chemie en
Medicinale Fysicochemie, Faculteit Farmaceutische
Wetenschappen, Katholieke Universiteit Leuven,
Van Evenstraat 4, B-3000 Leuven, Belgium*

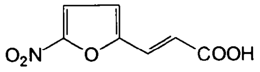
(*Received* 8 *December* 1993; *accepted* 4 *January* 1994)

Abstract

The molecules of the title compound, $C_7H_5NO_5$, are linked through hydrogen bonds to form a dimeric structure.

Comment

A perspective view showing the atomic numbering scheme and hydrogen bonds is given in Fig. 1. The crystal is composed of well ordered molecules of *trans*-5-nitro-2-furanacrylic acid, (I), in a form similar to the one observed in crystals of 2-(2′-dimethylaminovinyl)-5-nitrofuran (Kusa, Polynova, Porai-Koshits, Kovach & Vegkh, 1979). Both compounds are *trans* isomers with respect to the C21═C22 double bond. The molecule of the title compound is almost planar with no deviation from planarity greater than 0.05 Å. An examination of the torsion angles shows no deviation greater than 5° from either 0 or 180°.



(I)

The planar C3═C2—C21═C22 group shows no significant conjugation between the double bonds. The bond orders are 1.6, 1.2 and 2.0 for C3═C2, C2—C21 and C21═C22, respectively (Burke-Laing & Laing, 1976).

The molecules, related by an inversion, are linked into dimers by hydrogen bonds [O231···O232^{i} 1.76 (3) Å, O231—H231···O232^{i} 177 (2)°; symmetry code: (i) $1-x$, $-y$, $2-z$]. The atoms involved in the hydrogen bonds form a planar eight-membered ring with deviations from planarity not greater than 0.02 Å. The ring makes a dihedral angle of 0.52 (3)° with the plane of the whole molecule.

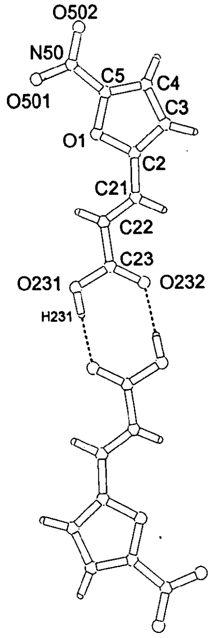


Fig. 1. The atomic numbering scheme and hydrogen bonds.

Experimental

The title compound was obtained from American Cyanamid Company (Pearl River, New York). Crystals were grown from an ethanol–acetonitrile (3:1) solution.

Crystal data

$C_7H_5NO_5$
$M_r = 183.12$
Monoclinic
$P2_1/n$
$a = 5.154\ (4)$ Å
$b = 14.253\ (5)$ Å
$c = 10.400\ (5)$ Å
$\beta = 99.61\ (6)°$
$V = 753.3\ (8)$ Å^3
$Z = 4$
$D_x = 1.615$ Mg m^{-3}

Mo $K\alpha$ radiation
$\lambda = 0.71069$ Å
Cell parameters from 31 reflections
$\theta = 10.15$–$13.56°$
$\mu = 0.1320$ mm^{-1}
$T = 293$ K
Block
0.20 × 0.20 × 0.10 mm
Light brown

Data collection

Stoe Stadi-4 four-circle diffractometer
ω scans
Absorption correction: none
3631 measured reflections
1738 independent reflections
1075 observed reflections [$I > 2.0\sigma(I)$]

$R_{\rm int} = 0.03$
$\theta_{\rm max} = 27.5°$
$h = -7 \rightarrow 7$
$k = -19 \rightarrow 19$
$l = 0 \rightarrow 14$
4 standard reflections
frequency: 60 min
intensity variation: <3.0%

Acta Crystallographica Section C
ISSN 0108-2701 ©1994