

Table 1. Coordinates ( $\times 10^4$ ) for non-H atoms and  $U_{eq}$  or  $U_{iso}$  values ( $\text{\AA}^2 \times 10^3$ ) with *e.s.d.*'s in parentheses
$$U_{eq} = \frac{1}{3} \sum_j U_{ij} a_i^* a_j^* a_i \cdot a_j$$

	x	y	z	$U_{eq}/U_{iso}$
Sn	5000†	-2968 (2)	7500†	71 (1)
N†	6409 (6)	3321 (16)	4782 (4)	81 (4)
O(2)	5435 (5)	-3296 (9)	6459 (3)	87 (3)
O(1)	5239 (4)	-724 (10)	6965 (3)	73 (2)
O(3)	6330 (7)	5018 (13)	4836 (4)	116 (4)
O(4)	6698 (5)	2585 (11)	4390 (3)	97 (3)
C(1)†	5444 (6)	-1566 (6)	6505 (4)	59 (4)
C(2)	5688 (6)	-293 (14)	6060 (4)	58 (3)
C(3)	5602 (7)	1680 (15)	6096 (4)	68 (4)
C(4)	5831 (7)	2847 (16)	5689 (4)	69 (3)
C(5)	6161 (6)	2055 (18)	5234 (4)	62 (3)
C(6)	6237 (7)	112 (16)	5177 (4)	66 (4)
C(7)	5991 (6)	-1022 (15)	5586 (4)	63 (3)
C(8)	6254 (9)	-4062 (21)	7893 (6)	116 (5)
C(9)	6214 (14)	-6213 (32)	7944 (9)	182 (7)
C(10)	7012 (14)	-7043 (35)	8182 (9)	194 (8)
C(11)	7034 (15)	-8784 (37)	8443 (11)	215 (9)

† Invariant by symmetry.

‡ Chemical identity of these atoms established from infrared spectroscopic data.

Table 2. Bond lengths ( $\text{\AA}$ ) and valency angles ( $^\circ$ ) with *e.s.d.*'s in parentheses

Sn—C(8)	2.11 (1)	C(1)—O(1)	1.32 (1)
Sn—O(1)	2.089 (7)	C(1)—O(2)	1.22 (1)
Sn—O(2)	2.645 (7)	C(2)—C(7)	1.38 (1)
C(1)—C(2)	1.48 (1)	C(2)—C(3)	1.40 (1)
C(3)—C(4)	1.35 (1)	C(4)—C(5)	1.39 (1)
C(5)—C(6)	1.38 (1)	C(6)—C(7)	1.36 (1)
N—O(3)	1.21 (1)	N—O(4)	1.22 (1)
N—C(5)	1.49 (1)	C(8)—C(9)	1.52 (2)
C(9)—C(10)	1.38 (2)	C(10)—C(11)	1.36 (3)
C(8)—Sn—C(8')	137.1 (6)	O(1)—Sn—C(8)	104.4 (4)
O(1)—Sn—O(2)	54.2 (2)	O(1)—Sn—C(8')	107.7 (4)
O(1)—Sn—O(2')	135.9 (2)	O(2)—Sn—C(8)	86.6 (4)
O(2)—Sn—O(2')	170.0 (2)	O(2)—Sn—C(8')	89.7 (2)
O(1)—Sn—O(1')	81.7 (3)	O(1)—C(1)—O(2)	121.2 (9)
Sn—O(1)—C(1)	104.0 (6)	C(1)—C(2)—C(3)	121.2 (9)
Sn—O(2)—C(1)	80.5 (6)	C(1)—C(2)—C(7)	120.8 (10)
O(1)—C(1)—C(2)	115.7 (9)	C(2)—C(3)—C(4)	121.1 (9)
O(2)—C(1)—C(2)	123.0 (9)	C(4)—C(5)—C(6)	121.6 (10)
C(3)—C(4)—C(5)	118.8 (11)	C(6)—C(7)—C(2)	122.1 (10)
C(5)—C(6)—C(7)	118.2 (9)	O(3)—N—O(4)	124.2 (10)
C(5)—N—O(3)	118.1 (10)	C(5)—N—O(4)	117.7 (10)
C(6)—C(5)—N	119.1 (10)	Sn—C(8)—C(9)	110.2 (12)
C(8)—C(9)—C(10)	113.8 (21)	C(9)—C(10)—C(11)	119.0 (24)
C(4)—C(5)—N	119.3 (11)	C(3)—C(2)—C(7)	118.1 (9)

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## Structure of 7-Amino-4-trifluoromethylcoumarin

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**Abstract.**  $C_{10}H_6F_3NO_2$ ,  $M_r = 229.1$ , triclinic,  $P\bar{1}$ ,  $a = 478.7$  (4)  $\text{\AA}$ ,  $Z = 2$ ,  $D_m = 1.59$ ,  $D_x = 1.60$   $\text{g cm}^{-3}$ ,  $b = 6.883$  (4),  $c = 14.165$  (4)  $\text{\AA}$ ,  $\alpha = 51.90$  (3),  $\beta = 6.883$  (4),  $\gamma = 72.67$  (4) $^\circ$ ,  $V = 13.0$   $\text{cm}^{-3}$ , filtered Cu  $K\alpha$  radiation,  $\lambda = 1.5418$   $\text{\AA}$ ,  $\mu = 13.0$   $\text{cm}^{-1}$ ,  $F(000) = 232$ ,  $T = 296$  K,  $R = 0.05$ ,  $wR = 0.05$ .

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bond angles in  $Bu_2Sn(O_2CC_6H_4NO_2-p)_2$  [81.7 (3), 170.0 (2) and 137.1 (6) $^\circ$  respectively] are also similar to those in the related  $R_2Sn(O_2CR')_2$  structures [*viz.* 79.5 (1)–81.8 (1), 168.0–171.1 (1) and 130.6 (2)–140.7 (1) $^\circ$  respectively]. The nitro groups in  $Bu_2Sn(O_2CC_6H_4NO_2-p)$  are not involved in coordination to Sn, the shortest Sn—O(nitro) being 4.532 (8)  $\text{\AA}$ .

Unlike the  $R_2Sn(O_2CR')_2$  compounds mentioned above, the *o*-carboxylatopyridine complex,  $Me_2Sn(O_2CC_5H_4N-o)_2$ , has been reported to be polymeric with the Sn atom seven coordinate (Lockhart & Davidson, 1987), while the formate,  $Me_2Sn(O_2CH)_2$  (with six-coordinate Sn), is a sheet polymer with linear  $Me_2Sn$  moieties nearly symmetrically bridged by formate anions (Mistry, Rettig, Trotter & Aubke, 1990).

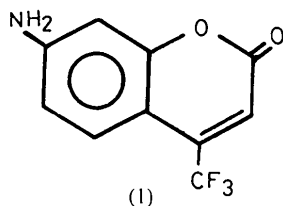
## References

- CHANDRASEKHAR, V., DAY, R. O., HOLMES, J. M. & HOLMES, R. R. (1988). *Inorg. Chem.* **27**, 958–964.
- KLEBANOV, K. S. & SHOLOGON, T. M. (1979). *Zh. Obshch. Khim.* **49**, 812–816.
- LOCKHART, T. P., CALABRESE, J. C. & DAVIDSON, F. (1987). *Organometallics*, **6**, 2479–2483.
- LOCKHART, T. P. & DAVIDSON, F. (1987). *Organometallics*, **6**, 2471–2478.
- MISTRY, F., RETTIG, S. J., TROTTER, J. & AUBKE, F. (1990). *Acta Cryst.* **C46**, 2091–2093.
- NG, S. W., KUMAR DAS, V. G., SKELTON, B. W. & WHITE, A. H. (1989). *J. Organomet. Chem.* **377**, 221–225.
- NG, S. W., KUMAR DAS, V. G., YIP, W.-H., WANG, R.-J. & MAK, T. C. W. (1990). *J. Organomet. Chem.* **393**, 201–204.
- SANDHU, G. K., SHARMA, N. & TIEKINK, E. R. T. (1989). *J. Organomet. Chem.* **371**, C1–C3.
- SANDHU, G. K., SHARMA, N. & TIEKINK, E. R. T. (1991). *J. Organomet. Chem.* **403**, 119–231.
- SHELDRIK, G. M. (1976). *SHELX76*. Program for crystal structure determination. Univ. of Cambridge, England.
- TIEKINK, E. R. T. (1991). *Appl. Organomet. Chem.* In the press.
- VOLLANO, J. F., DAY, R. O., RAU, D. N., CHANDRASEKHAR, V. & HOLMES, R. R. (1984). *Inorg. Chem.* **23**, 3153–3160.
- WILLIAMS, D. H. & FLEMING, I. (1989). *Spectroscopic Methods in Organic Chemistry*, 4th ed. Maidenhead, England: McGraw-Hill.

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0.076 for 1763 observed reflections. The benzene and pyrone rings are planar with maximum deviations of 0.016 Å at C(3) and -0.015 Å at C(10). The amino group is coplanar with the benzene and pyrone rings. The molecular packing involves van der Waals contacts only.

**Introduction.** Coumarin compounds have been found to be very useful in dye laser studies (Masilamani, 1979). Under laser excitation some coumarin dyes in weakly polar solvents give dual fluorescence. This is attributed to twisting of the amino groups attached to C(7), and is called twisted intramolecular charge transfer (TICT) (Masilamani, Chandrasekar, Sivaram, Sivasankar & Natarajan, 1986). Since structural information about the molecule may explain its laser characteristics, the X-ray crystal structure determination of the title compound (1), also called coumarin 490, has been performed.



**Experimental.** The compound was purchased from Exciton Chemicals, Inc., USA. A small needle-shaped yellow crystal of dimensions 0.55 × 0.15 × 0.10 mm, grown from a mixture of chloroform and ethanol by slow evaporation, was mounted on an Enraf-Nonius CAD-4 diffractometer. Accurate cell parameters were obtained with use of Ni-filtered Cu K $\alpha$  radiation from a least-squares refinement of the setting angles of 25 carefully centred reflections in the range 35 < 2 $\theta$  < 45°. The density of the crystal was determined by flotation in KI solution. The intensity data were collected by the  $\omega$ -2 $\theta$  scan technique to a maximum 2 $\theta$  of ~150°. Range of  $hkl$ :  $h$  - 6 → 6,  $k$  - 8 → 8,  $l$  0 → 17. Two standard reflections were checked for every 100 intensity measurements and their variation was less than 2%. Out of the 1984 unique reflections measured, 1763 reflections with  $I > 2\sigma(I)$  were considered observed and 149 reflections unobserved.  $R_{\text{int}} = 0.01$ . The data were corrected for direct-beam polarization and Lorentz effects, but not for absorption.

The structure was solved by direct methods by use of the program *SHELXS86* (Sheldrick, 1986) and refinement was carried out by the full-matrix least-squares method using *SHELX76* (Sheldrick, 1976). All of the H atoms were located in the difference Fourier map and refined isotropically. In the final stage of refinement, weights were introduced resulting in  $R = 0.05$ ,  $wR = 0.076$  [ $w = 1/(\sigma^2|F_o| +$

Table 1. Fractional coordinates and equivalent isotropic thermal parameters for non-H atoms, with e.s.d.'s in parentheses

$$B_{\text{eq}} = (8\pi^2/3) \sum_i \sum_j U_{ij} a_i^* a_j^* a_i a_j$$

	x	y	z	$B_{\text{eq}}$ (Å <sup>2</sup> )
O(1)	0.4605 (2)	0.1710 (2)	0.6146 (1)	3.84 (4)
C(2)	0.5375 (3)	0.3067 (2)	0.6596 (1)	3.92 (5)
O(2)	0.6893 (3)	0.3927 (2)	0.6148 (1)	5.02 (4)
C(3)	0.4319 (4)	0.3359 (3)	0.7555 (1)	4.32 (5)
C(4)	0.2713 (3)	0.2272 (3)	0.7996 (1)	3.98 (5)
C(5)	0.0287 (4)	-0.0413 (3)	0.7899 (1)	4.16 (4)
C(6)	-0.0315 (4)	-0.1730 (3)	0.7363 (1)	4.23 (5)
C(7)	0.0726 (3)	0.1917 (2)	0.6416 (1)	3.74 (5)
N(7)	0.0146 (4)	-0.3220 (3)	0.5881 (1)	5.00 (5)
C(8)	0.2412 (3)	-0.0743 (2)	0.6040 (1)	3.71 (5)
C(9)	0.2978 (3)	0.0576 (2)	0.6585 (1)	3.30 (4)
C(10)	0.1977 (3)	0.0800 (2)	0.7527 (1)	3.54 (4)
C(11)	0.1677 (4)	0.2651 (4)	0.8997 (1)	5.63 (7)
F(12)	-0.0993 (3)	0.3354 (3)	0.9053 (1)	7.99 (6)
F(13)	0.2360 (4)	0.0946 (3)	0.9593 (1)	8.38 (7)
F(14)	0.2606 (3)	0.4003 (3)	0.9336 (1)	8.78 (7)

Table 2. Bond lengths (Å) and angles (°) involving non-H atoms, with e.s.d.'s in parentheses

O(1)—C(2)	1.361 (2)	C(6)—C(7)	1.413 (2)
O(1)—C(9)	1.377 (2)	C(7)—N(7)	1.356 (3)
C(2)—O(2)	1.216 (2)	C(7)—C(8)	1.394 (2)
C(2)—C(3)	1.440 (2)	C(8)—C(9)	1.370 (2)
C(3)—C(4)	1.348 (3)	C(9)—C(10)	1.404 (2)
C(4)—C(10)	1.433 (3)	C(11)—F(12)	1.324 (3)
C(4)—C(11)	1.503 (2)	C(11)—F(13)	1.328 (3)
C(5)—C(6)	1.370 (3)	C(11)—F(14)	1.321 (4)
C(5)—C(10)	1.413 (3)		
C(2)—O(1)—C(9)	122.6 (1)	C(7)—C(8)—C(9)	119.7 (2)
O(1)—C(2)—C(3)	117.3 (2)	C(8)—C(9)—C(10)	123.4 (2)
O(1)—C(2)—O(2)	116.8 (2)	O(1)—C(9)—C(8)	115.6 (2)
C(2)—C(3)—C(4)	121.0 (2)	O(1)—C(9)—C(10)	121.1 (2)
O(2)—C(2)—C(3)	125.9 (2)	C(5)—C(10)—C(9)	116.2 (2)
C(3)—C(4)—C(11)	118.3 (2)	C(4)—C(10)—C(9)	116.6 (2)
C(3)—C(4)—C(10)	121.4 (1)	C(4)—C(10)—C(5)	127.1 (1)
C(10)—C(4)—C(11)	120.3 (2)	C(4)—C(11)—F(14)	112.8 (2)
C(6)—C(5)—C(10)	121.2 (1)	C(4)—C(11)—F(13)	111.7 (2)
C(5)—C(6)—C(7)	121.1 (2)	C(4)—C(11)—F(12)	111.6 (2)
C(6)—C(7)—C(8)	118.4 (2)	F(13)—C(11)—F(14)	107.0 (2)
C(6)—C(7)—N(7)	121.5 (2)	F(12)—C(11)—F(14)	106.7 (2)
N(7)—C(7)—C(8)	120.1 (1)	F(12)—C(11)—F(13)	106.7 (2)

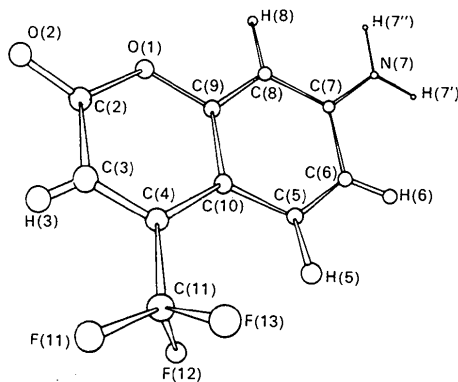


Fig. 1. Molecular diagram and atomic numbering scheme.

$0.006655|F_o|^2$ ], max. shift to e.s.d. = 0.09. A final difference Fourier map was featureless with  $\Delta\rho$  within  $\pm 0.25 \text{ e } \text{Å}^{-3}$ . The atomic scattering factors used in the calculations for all non-H atoms were taken from *International Tables for X-ray Crystallography* (1974, Vol. IV), while those for the H atoms were taken from Stewart, Davidson & Simpson (1965). Computer programs: *PARST* (Nardelli, 1983) for geometrical calculation; *MOLDRW* (Ugliengo, Borzani & Viterbo, 1988) for molecular illustrations. All calculations were performed on a VAX 11/780 computer.

**Discussion.** The final positional parameters and equivalent isotropic thermal parameters for the non-H atoms are given in Table 1.\* Fig. 1 shows the molecular diagram and atomic numbering scheme. Bond lengths and angles for non-H atoms are listed in Table 2. Bond lengths and angles in the benzene and pyrone rings are close to the mean values observed for the same distances and angles in other coumarin derivatives. Calculation of the least-squares plane shows that the benzene and pyrone rings are planar with maximum deviations of 0.016 Å at C(3) and  $-0.015 \text{ Å}$  at C(10). The  $\text{NH}_2$  group is coplanar with the benzene and pyrone rings. The deviation of atom N(7) is  $-0.02 \text{ Å}$ . The C—N distance of  $1.356 \text{ Å}$  is normal. A similar result has been observed for 7-diethylamino-4-methylcoumarin

\* Lists of structure factors, anisotropic thermal parameters, bond lengths and angles involving H atoms, torsion angles, least-squares-planes calculations and H-atom parameters, and a packing diagram have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54440 (23 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

(Messagar & Delugeard, 1974). The carbonyl O atom O(2) lies in the plane of the benzene and pyrone rings [the deviation of O(2) is  $-0.04 \text{ Å}$ ].

Coumarins are well known photosensitizing agents in photodynamic action. Song & Gordon (1970) concluded from both spectroscopic results and theoretical calculations that this property might be assigned either to carbonyl stretching or to C=C stretching in the pyrone moiety. Because of steric effects, the O(2)—C(2)—O(1) angle is generally smaller than the O(2)—C(2)—C(3) angle in coumarin and its derivatives. It is also found that the angles C(8)—C(9)—O(1) and C(4)—C(10)—C(5) at the junctions of the two rings are smaller and greater respectively than  $120^\circ$ . This causes the approach of O(1) to C(8) and the separation of C(4) from C(5). All of these common features found in coumarin derivatives are also present in the title compound. There are no short contacts. The structure is stabilized by van der Waals interactions.

#### References

- MASILAMANI, V. (1979). *A Study of Coumarin Dye Lasers*. PhD Thesis, IIT, Madras, India.  
 MASILAMANI, V., CHANDRASEKAR, V., SIVARAM, B. M., SIVASANKAR, B. & NATARAJAN, S. (1986). *Opt. Commun.* **59**, 203.  
 MESSAGAR, J. C. & DELUGEARD, Y. (1974). *Cryst. Struct. Commun.* **3**, 391–396.  
 NARDELLI, M. (1983). *Comput. Chem.* **7**, 95–98.  
 SHELDRIK, G. M. (1976). *SHELX76*. Program for crystal structure determination. Univ. of Cambridge, England.  
 SHELDRIK, G. M. (1986). *SHELXS86*. Program for the solution of crystal structures. Univ. of Göttingen, Germany.  
 SONG, P. S. & GORDON, W. H. (1970). *J. Phys. Chem.* **74**, 4234–4240.  
 STEWART, R. F., DAVIDSON, E. R. & SIMPSON, W. T. (1965). *J. Chem. Phys.* **42**, 3175–3187.  
 UGLIENGO, P., BORZANI, G. & VITERBO, D. (1988). *J. Appl. Cryst.* **21**, 75.

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## Structure of 2,2-Dimethyl-3-(2-methyl-1,3-dithian-2-yl)-3-phenylaziridine

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**Abstract.**  $\text{C}_{15}\text{H}_{21}\text{NS}_2$ ,  $M_r = 279.45$ , triclinic,  $P\bar{1}$ ,  $a = 741.7(6) \text{ Å}^3$ ,  $Z = 2$ ,  $D_m = 1.25(2)$ ,  $D_x = 7.472(3)$ ,  $b = 10.535(2)$ ,  $c = 10.857(5) \text{ Å}$ ,  $\alpha = 1.254(1) \text{ Mg m}^{-3}$ ,  $\lambda(\text{Cu K}\alpha) = 1.5418 \text{ Å}$ ,  $\mu = 99.99(3)$ ,  $\beta = 90.97(4)$ ,  $\gamma = 117.53(2)^\circ$ ,  $V = 3.01 \text{ mm}^{-1}$ ,  $F(000) = 300$ , room temperature,  $R =$