quantum yield is much higher (Schimitschek, Trias, Hammond & Atkins, 1974). The decrease in the fluorescence quantum yield is a result of free rotation around the N—C(sp^2) bond in the excited state and the stabilization of the non-emissive new conformer in the solvent media (Jones, Jackson & Halpern, 1980; Jones, Jackson, Choi & Bergmark, 1985; Loboda, Sokolova, Fofonova & Khochkina, 1989). The structural study, performed in order to understand the ground-state conformation of the diethylamino group, shows that any rotation about the N—C(sp^2) bond is facilitated since the methyl C atoms lie on either sides of the coumarin moiety. 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; Chinnakali, Sivakumar, Natarajan & Mathews, 1992).

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Structure of 3-*tert*-Butyl-1-[(4-chlorophenyl)sulfonyl]-1-methylurea

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Abstract. $C_{12}H_{17}ClN_2O_3S$, $M_r = 304.8$, triclinic, $P\bar{1}$, $a = 13.075(2)$, $b = 13.284(3)$, $c = 9.924(2)$ Å, $\alpha = 103.68(3)$, $\beta = 95.28(3)$, $\gamma = 66.34(4)^\circ$, $V = 1534$ Å^3, $Z = 4$, $D_m = 1.33$, $D_x = 1.3197\ \mathrm{g\,cm^{-3}}$, $\lambda(\mathrm{Cu}\ K\alpha) = 1.54178$ Å, $\mu = 35.4\ \mathrm{cm^{-1}}$, $F(000) = 640$, $T = 296$ K, $R = 0.061$, $wR = 0.075$ for 4694 reflections $[I > 2\sigma(I)]$. There are two molecules in the asymmetric unit. In both molecules, the N atom attached to the *tert*-butyl group forms an intramolecular hydrogen bond with one of the sulfonyl O atoms, forming a six-membered ring. In the absence of intermolecular hydrogen bonds, the structure is stabilized by non-bonded interactions.

Experimental. Crystallization from petroleum ether, colourless thick-plate-shaped single crystal, 0.22 × 0.37 × 0.59 mm. The unit-cell parameters were refined from least-squares analysis of θ values for 29 reflections from $13.1 < \theta < 39.3^\circ$. Intensities for 5822 unique reflections having $3 < \theta < 70^\circ$, $-15 \le h \le 15$, $-16 \le k \le 15$, $0 \le l \le 12$, measured on a Siemens AED diffractometer, using ω–2θ scan mode, variable scan speed from 0.3 to $1.2^\circ\ \mathrm{min}^{-1}$. During data collection, one standard reflection was monitored every 100 measurements with no decrease in intensity. The data were corrected for Lorentz and polarization effects. Absorption corrections were applied following Walker & Stuart (1983) after isotropic refinement using *ABSORB* (Ugozzoli, 1987). The

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

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

	Molecule *A*				Molecule *B*			
	x	y	z	$U_{\rm eq}$(Å^2)	x	y	z	$U_{\rm eq}$(Å^2)
Cl	0.8250 (1)	0.0333 (1)	−1.2170 (1)	0.1069 (6)	0.8697 (1)	0.6880 (1)	0.6404 (1)	0.0872 (4)
S	0.7600 (1)	0.3226 (1)	−0.6162 (1)	0.0706 (3)	0.8548 (1)	0.7458 (1)	0.0347 (1)	0.0559 (2)
N(1)	0.7840 (2)	0.2273 (2)	−0.5223 (3)	0.067 (1)	0.7251 (2)	0.7738 (2)	−0.0181 (2)	0.055 (1)
N(3)	0.6011 (2)	0.2474 (2)	−0.4917 (3)	0.065 (1)	0.7385 (2)	0.5874 (2)	−0.0849 (2)	0.057 (1)
O(1)	0.6462 (2)	0.3982 (2)	−0.5964 (3)	0.079 (1)	0.8748 (2)	0.8443 (2)	0.0384 (2)	0.075 (1)
O(2)	0.8464 (2)	0.3640 (2)	−0.5834 (3)	0.101 (2)	0.9245 (1)	0.6404 (2)	−0.0503 (2)	0.065 (1)
O(3)	0.7422 (2)	0.0752 (2)	−0.5333 (3)	0.087 (1)	0.5728 (2)	0.7306 (2)	−0.0227 (3)	0.082 (1)
C(1)	0.7782 (2)	0.2447 (2)	−0.7875 (3)	0.056 (1)	0.8593 (2)	0.7270 (2)	0.2048 (3)	0.048 (1)
C(2)	0.8799 (2)	0.2035 (3)	−0.8536 (4)	0.067 (1)	0.8946 (2)	0.6193 (2)	0.2301 (2)	0.055 (1)
C(3)	0.8953 (2)	0.1363 (3)	−0.9853 (3)	0.069 (1)	0.8970 (2)	0.6075 (2)	0.3638 (3)	0.064 (1)
C(4)	0.8060 (3)	0.1158 (3)	−1.0507 (3)	0.067 (1)	0.8642 (2)	0.7038 (2)	0.4709 (3)	0.061 (1)
C(5)	0.7031 (3)	0.1582 (3)	−0.9883 (4)	0.065 (1)	0.8294 (3)	0.8087 (2)	0.4458 (3)	0.066 (1)
C(6)	0.6888 (2)	0.2226 (2)	−0.8558 (3)	0.060 (1)	0.8262 (2)	0.8221 (2)	0.3121 (3)	0.061 (1)
C(7)	0.7067 (2)	0.1762 (2)	−0.5193 (3)	0.059 (1)	0.6721 (2)	0.6952 (2)	−0.0400 (3)	0.052 (1)
C(9)	0.9013 (3)	0.1516 (4)	−0.5130 (6)	0.111 (2)	0.6456 (3)	0.8934 (3)	0.0157 (5)	0.095 (2)
C(10)	0.5054 (3)	0.2174 (2)	−0.4851 (4)	0.069 (1)	0.7049 (3)	0.4913 (2)	−0.1066 (3)	0.065 (1)
C(11)	0.4058 (3)	0.3282 (4)	−0.4544 (7)	0.125 (3)	0.6144 (3)	0.4985 (3)	−0.2150 (5)	0.094 (2)
C(12)	0.5195 (4)	0.1557 (5)	−0.3704 (6)	0.122 (3)	0.8105 (3)	0.3864 (3)	−0.1587 (5)	0.100 (2)
C(13)	0.4879 (5)	0.1484 (4)	−0.6228 (5)	0.125 (3)	0.6647 (5)	0.4891 (4)	0.0305 (5)	0.113 (3)

Table 2. *Selected bond lengths* (Å), *bond angles* (°), *torsion angles* (°) *and hydrogen-bonding geometry*

	Molecule *A*	Molecule *B*		Molecule *A*	Molecule *B*
S—O(1)	1.422 (2)	1.425 (3)	N(1)—C(9)	1.467 (4)	1.484 (4)
S—O(2)	1.425 (3)	1.436 (2)	C(7)—O(3)	1.208 (3)	1.204 (3)
S—N(1)	1.660 (3)	1.646 (2)	C(7)—N(3)	1.332 (3)	1.332 (3)
S—C(1)	1750 (3)	1.756 (3)	N(3)—C(10)	1.468 (5)	1.474 (5)
N(1)—C(7)	1.431 (4)	1.436 (4)	Cl—C(4)	1.729 (3)	1.735 (3)
O(1)—S—C(1)	108.7 (2)	108.8 (1)	S—N(1)—C(9)	115.5 (3)	117.0 (2)
O(2)—S—C(1)	108.7 (2)	107.8 (1)	C(7)—N(1)—C(9)	113.8 (3)	113.6 (3)
O(1)—S—O(2)	120.1 (2)	119.3 (1)	N(1)—C(7)—O(3)	118.6 (3)	118.9 (3)
O(1)—S—N(1)	107.6 (2)	106.1 (2)	N(1)—C(7)—N(3)	115.5 (2)	115.7 (2)
O(2)—S—N(1)	106.5 (2)	107.8 (1)	C(7)—N(3)—C(10)	126.6 (3)	125.6 (3)
C(1)—S—N(1)	104.0 (1)	106.3 (1)	N(3)—C(7)—O(3)	125.7 (3)	125.3 (3)
S—N(1)—C(7)	121.5 (2)	124.2 (2)			

	Molecule *A*	Molecule *B*
C(6)—C(1)—S—O(1)	−29.8 (3)	30.7 (3)
C(6)—C(1)—S—O(2)	−162.1 (2)	161.5 (2)
C(6)—C(1)—S—N(1)	84.6 (3)	−83.1 (2)
C(1)—S—N(1)—C(7)	−71.5 (3)	−66.2 (3)
C(1)—S—N(1)—C(9)	73.5 (3)	86.7 (3)
S—N(1)—C(7)—N(3)	−51.1 (4)	−33.1 (4)
S—N(1)—C(7)—O(3)	133.2 (3)	149.9 (3)

D—H···*A*	*D*···A (Å)
N3*A*—H1N3*A*···O1*A*	2.736 (5)
N3*B*—H1N3*B*···O2*B*	2.763 (4)

absorption coefficients $A_{p,s}$ calculated in the polar angles of the incident and diffracted beam range from 1.171 to 0.85 while the spherical absorption coefficients A_θ range from 1.22 to 0.97.

Direct methods using *SHELXS*86 (Sheldrick, 1986) revealed the positions of all non-H atoms. The positional and anisotropic displacement parameters of all non-H atoms were refined by full-matrix least squares on F using *SHELX*76 (Sheldrick, 1976) with 4694 reflections having $I > 2\sigma(I)$. The H atoms, located in a difference Fourier map, were refined isotropically. Final $R = 0.061$, $wR = 0.075$, $S = 4.22$ for observed reflections and $w = 1/[\sigma^2(F) + 0.008565F^2]$, $(\Delta/\sigma)_{\max} < 1.0$. Final difference map showed maximum and minimum peaks of $\Delta\rho = +0.59$ and -0.63 e Å^{-3}. All the atomic scattering factors are from *SHELX*76.

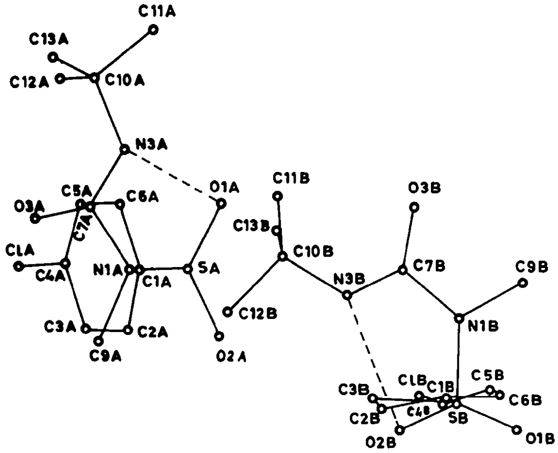

Fig. 1. Molecular diagram projected along the c axis.

Atomic parameters are listed in Table 1.* Selected bond lengths, angles, torsion angles and hydrogen-bonding geometry are given in Table 2. A molecular diagram with atomic numbering scheme is shown in Fig. 1.

Related literature. The structural and conformational features of the present compound are similar to those observed in several sulfonamide structures (Kálmán, Czugler & Argay, 1981; Alléaume, Gulko, Herbstein, Kapon & Marsh, 1976). The compound was supplied by Herbert T. Nagasawa, Medical Research Laboratories, VA Medical Center and Department of

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* Lists of structure factors, anisotropic thermal parameters, H-atom parameters and equations of least-squares planes have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 55009 (33 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: AS0517]

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Structure of Corydaline

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Abstract. 5,8,13,13a-Tetrahydro-2,3,9,10-tetramethoxy-1-methyl-6*H*-dibenzo[*a*,*g*]quinolizine, $C_{22}H_{27}NO_4$, $M_r = 369.46$, monoclinic, $P2_1$, $a = 8.732\,(6)$, $b = 7.639\,(5)$, $c = 14.966\,(10)$ Å, $\beta = 95.85\,(2)°$, $V = 993\,(1)$ Å^3, $Z = 2$, $D_x = 1.235$ g cm,$^{-3}$, λ(Mo $K\alpha$) = 0.7107 Å, $\mu = 0.79$ cm^{-1}, $F(000) = 396$, $T = 293\,(2)$ K, $R = 0.054$ for 1625 reflections with $I > 3.5\sigma(I)$. Ring *B* assumes an almost perfect half-chair conformation while ring *C* has a transitional form between sofa and half-chair. The O(1)—C(18), O(2)—C(19) and O(3)—C(21) bonds are in the planes of their respective phenyl rings, *D* and *A*, as indicated by the corresponding torsion angles, while the O(4)—C(22) bond is perpendicular to the plane of phenyl ring *A*. The mean value of the three C—N—C angles is 111.5 (4)° indicating sp^3 hybridization of the N atom. The C(20)-methyl group is in an axial position. Molecules are held together by van der Waals interactions.

Experimental. In a previous work the crystal and molecular structure of bulbocapnine was solved by Ribár, Mészáros, Gašić, Kanyó & Engel (1991), isolated from the aerial parts of the wild-growing medicinal plants *Corydalis cava* and *C. solida* (L). In a later investigation the minor alkaloid corydaline was isolated from the roots of both species. The isolation of crude alkaloids was carried out according to Preininger, Vesely, Gašić, Šimanek & Dolejš (1975) and Gašić, Popović & Dragutinović (1985). Corydaline was isolated by column chromatography as well as by preparative thin-layer chromatography as described by Gašić, Kanyó, Loukis & Bačić (1991).

Data were collected on a crystal of irregular shape (approximate dimensions 0.33 × 0.33 × 0.33 mm) mounted on a CAD-4 diffractometer (Berne) equipped with a graphite monochromator. Cell constants were refined by least-squares fit for 20 reflections in the range $2.7 < \theta < 9.9°$. Data were collected by ω–2θ scan in the range $\theta < 25°$ with h −10 to 10, k 0 to 9, l 0 to 18. Of 2253 measured reflections, 1625 with $I > 3.5\sigma(I)$ were taken as observed. Only one standard reflection ($\bar{1}2\bar{1}$) was measured every 200 min to control intensity decay and orientation; no intensity variation was recorded. Data were corrected for Lorentz and polarization effects. The structure was solved with *SHELXS*86 (Sheldrick, 1986) with all non-H atoms obtained from an *E* map. Full-matrix least-squares refinement with *SHELX*76 (Sheldrick, 1976) minimized $\sum w(\Delta F)^2$ for 270 parameters, with $w = [\sigma^2(F_o) + (15.98 \times$