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10-(1,3-Benzothiazol-2-yl)-2,3,6,7-tetrahydro-1*H*,5*H*-11*H*-[1]benzopyrano[6,7,8-*ij*]-quinoliz-11-one

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Key indicators

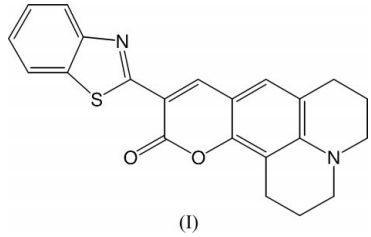
Single-crystal X-ray study
T = 296 K
Mean σ(C–C) = 0.004 Å
Disorder in main residue
R factor = 0.045
wR factor = 0.143
Data-to-parameter ratio = 15.0

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

The title compound, $C_{22}H_{18}N_2O_2S$, is also known as coumarin 545. The 2-benzothiazolyl and quinolizine groups are each nearly planar and are twisted slightly at 6.4 (1)° to each other. The molecules are stacked in layers oblique to (110) and (111).

Comment

The title compound, (I) (Fig. 1), also known as coumarin 545 (Exiton Inc.), is an efficient laser dye compound, exhibiting lasing and fluorescent properties in the 541–564 nm region. It produces a lasing maximum, λ_{max}, at 547 and 555 nm in ethanol and ethanol–water (1:1), respectively (Fletcher *et al.*, 1983). A variety of quinolizine-related derivatives, all exhibiting a structurally rigid amino group, show a high quantum yield of fluorescence in polar solvents and have been characterized in this regard [LD 490 (Gridunova *et al.*, 1992), Coumarin 314 (or 504) (Honda *et al.*, 1996*a*,*b*; Yip *et al.*, 1995), Coumarin 338 (or 519) (Honda *et al.*, 1996*c*)]. The (2-benzothiazolyl) group is structurally related to its counterpart in Coumarin 6 [or 520; Jasinski & Paight (1995)]. Structural analysis of this compound was carried out to provide evidence of the ground-state conformation of the parent-fused 2,3,6,7-tetrahydro-1*H*,5*H*,11*H*-[1]benzopyrano[6,7,8-*i*,*j*]quinoliz-11-one group connected to a 2-benzothiazolyl ring through the C2′—C3 bond. Examination of the rigid nitrogen centers in the two connected moieties provides additional insight regarding these centers and lasing efficiency.



(I)

Bond lengths and angles for the 2-benzothiazolyl and quinolizine groups of (I) closely resemble those reported for the related derivatives mentioned earlier, within experimental error. Atoms C13 and C17 are disordered. Relatively short C12—C13*A* [1.451 (6) Å], C12—C13*B* [1.447 (7) Å], C16—C17A [1.447 (5) Å], and C16—C17*B* [1.445 (6) Å] bonds have been observed in similar structurally related compounds (Yagi *et al.*, 2000; Baraznenok *et al.*, 2000; Dunlop *et al.*, 1979). The mean deviations from the least-squares planes for the 2-benzothiazolyl (S1′/C2′/N3′/C9′/C4′/C5′/C6′/C7′/C8′; plane 1), coumarin (C2/C3/C4/C10/C5/C6/C7/C8/C9/O1; plane 2) and quinolizine (C2/C3/C4/C10/C5/C6/C12/C13/C14/N15/C16/C17/C18/C7/C8/C9/O1; plane 3) groups are 0.008 (9), 0.021 (1) and 0.092 (2) Å, respectively. The dihedral angles between the

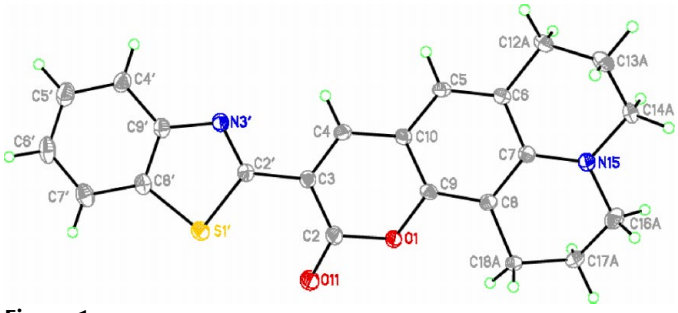


Figure 1
*ORTEP*II (Johnson, 1976) drawing of (I), showing 30% probability displacement ellipsoids and the atomic numbering scheme.

least-squares planes of these ring systems are 4.4 (1)° (between planes 1 and 2), 6.4 (1)° (between planes 1 and 3) and 2.2 (6)° (between planes 2 and 3). The deviations of the N atom from planes 1 and 3 are 0.006 (7) and 0.12 (6) Å, respectively. The distance between atoms N3′ and N15 is 8.42 (3) Å.

A packing diagram of the molecule (Fig. 2), viewed down the *a* axis, shows that the molecules are stacked in layers oblique to the (111) and (110) planes, with a closest contact interlayer spacing of 3.544 (5) Å.

Experimental

Commercial coumarin 545 (Exiton Inc., Ohio, USA) was crystallized by slow evaporation from acetonitrile.

Crystal data

$C_{22}H_{18}N_2O_2S$
M_r = 374.44
Monoclinic, $P2_1/n$
a = 12.2528 (18) Å
b = 11.702 (2) Å
c = 13.263 (2) Å
β = 110.915 (10)°
V = 1776.3 (5) Å^3
Z = 4
D_x = 1.400 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 20 reflections
θ = 18.8–22.4°
μ = 0.20 mm^{-1}
T = 296 (2) K
Prism, red
0.60 × 0.50 × 0.50 mm

Data collection

Rigaku AFC-6*S* diffractometer
$2\theta/\omega$ scans
Absorption correction: ψ scan (North *et al.*, 1968)
T_{min} = 0.888, T_{max} = 0.903
4241 measured reflections
4057 independent reflections
2088 reflections with $I > 2\sigma(I)$
R_{int} = 0.032
θ_{max} = 27.5°
$h = 0 \rightarrow 15$
$k = 0 \rightarrow 15$
$l = -17 \rightarrow 16$
3 standard reflections
every 150 reflections
intensity decay: 0.5%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.045
$wR(F^2)$ = 0.143
S = 1.01
4057 reflections
270 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0504P)^2 + 1.155P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.30 e Å^{-3}
$\Delta\rho_{min}$ = −0.25 e Å^{-3}

H atoms on C2, C4, C5, C4′, C5′, C6′ and C7′ were included in calculated positions and refined as riding atoms. The disordered atoms C13 and C17, together with the H atoms on C12, C14, C16 and C18, were split into two components with appropriate H-atom constraints.

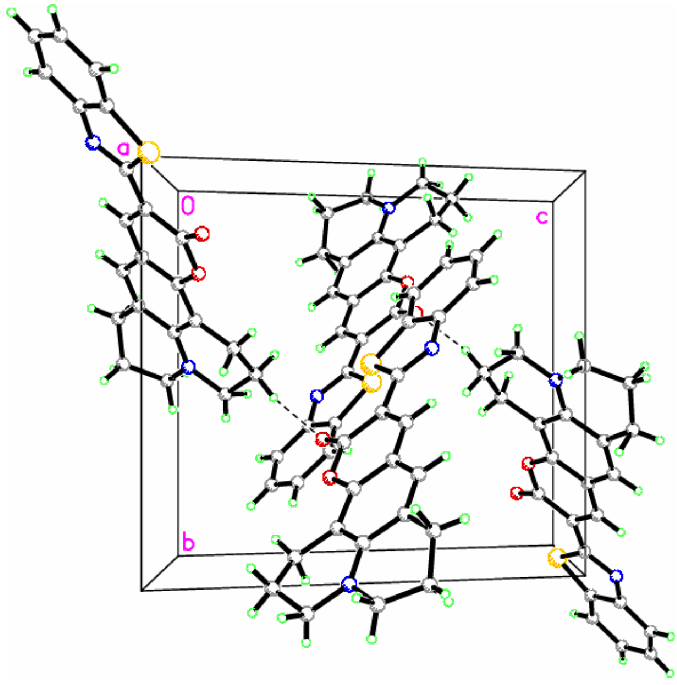


Figure 2
*ORTEP*II (Johnson, 1976) packing diagram of (I), viewed down the *a* axis. The minor disorder component of the quinolizine group is not shown.

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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