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N-[4-(2-Oxo-2*H*-chromen-3-yl)-3-phenyl-3*H*-thiazol-2-ylidene]anilinium bromide methanol solvate

T. Seethalakshmi,[a] V. Rajeswar Rao,[b] P. Vijaya Kumar[b] and P. Kaliannan[a]*

[a]School of Physics, Bharathidasan University, Tiruchirappalli 620 024, India, and [b]Department of Chemistry, National Institute of Technology, Warangal 506 004, Andhra Pradesh, India

Correspondence e-mail: kal_44in@yahoo.co.in

Key indicators

Single-crystal X-ray study
T = 160 K
Mean σ(C–C) = 0.003 Å
R factor = 0.038
wR factor = 0.092
Data-to-parameter ratio = 21.9

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

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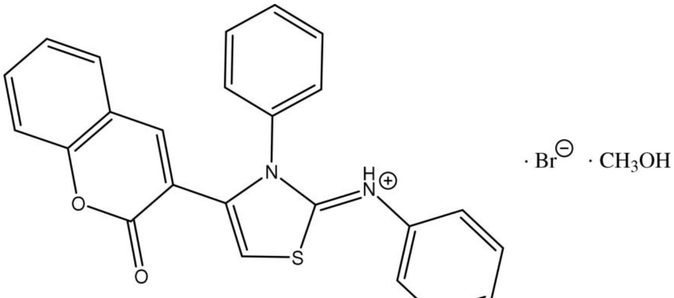
In the title structure, $C_{24}H_{17}N_2O_2S^+\cdot Br^-\cdot CH_3OH$, the cations, anions and solvent molecules form centrosymmetric clusters *via* a combination of intermolecular N—H···O, O—H···Br and C—H···Br interactions.

Comment

It is well known that heterocycles containing a 1,3-thiazole ring form the central structural group of a number of biologically active natural products (Crews *et al.*, 1988) and of pharmacologically active compounds (Metzger, 1979). We report here the crystal structure of the title compound, (I), containing a 1,3-thiazole ring.



(I)

The asymmetric unit of (I) is shown in Fig. 1. The cation has normal geometric parameters (Allen *et al.*, 1987); the C2—S1 [1.728 (2) Å] and C5—S1 [1.733 (2) Å] bond lengths are intermediate between typical C—S single- and double-bond distances, indicating significant electron delocalization. The C2—S1—C5 [89.81 (8)°] bond angle in (I) is almost the same as the corresponding value in *N*-[4-(*N*,*N*-diethylamino)benzylidene]-4-phenyl-5-(1*H*-1,2,4-triazol-1-yl)thiazol-2-amine [88.17(10°); Zhou *et al.*, 2005]. The value of the N3—C2—N21—C21 torsion angle [178.4 (2)°] indicates that the ammonium benzylidene ring has an *E* configuration with respect to the phenyl ring (C31–C36) at position N3 of the thiazole ring. All the rings are individually planar. However, atom O42 deviates by 0.185 (2) Å from the mean plane of the coumarin ring (O41/C42–C50). The thiazole ring makes dihedral angles of 50.4 (1), 76.1 (1) and 75.7 (1)° with the C21–C26, C31–C36 and O41/C42–C50 rings, respectively.

In the crystal structure of (I), the methanol solvent atom O52 acts as donor for an intermolecular O—H···Br hydrogen bond with a symmetry-related Br^- anion, and also acts as acceptor for an intermolecular N—H···O hydrogen bond from a symmetry-related cation. Atom C32 participates in a weak intermolecular C—H···Br interaction with a symmetry-

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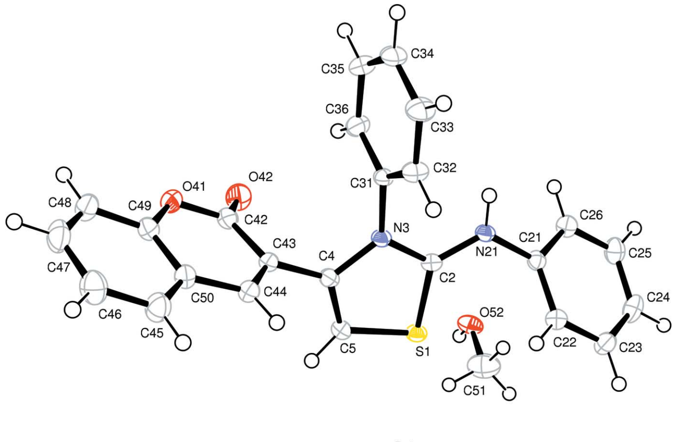

Figure 1
A view of the asymmetric unit of (I), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level and H atoms are represented by spheres of arbitrary radii.

related Br^- anion. The combination of the N—H···O, O—H···Br and C—H···Br interactions connects the cations, anions and solvent molecules into centrosymmtric clusters (Fig. 2 and Table 1). The shortest intermolecular S1···Br contact is *ca* 3.20 Å.

Experimental

3-(2-Bromoacetyl)coumarin (0.01 mol) and diphenylthiourea (0.01 mol) were dissolved in anhydrous ethanol (20 ml) and the solution was heated to reflux over a period of 30 min. Upon cooling to room temperature (298 K), the solid which separated was filtered off to give the title compound, (I), which was recrystallized from methanol.

Crystal data

$C_{24}H_{17}N_2O_2S^+ \cdot Br^- \cdot CH_4O$
$M_r = 509.41$
Triclinic, $P\bar{1}$
$a = 10.5909$ (1) Å
$b = 10.9374$ (1) Å
$c = 11.0418$ (2) Å
$\alpha = 82.2188$ (8)°
$\beta = 62.9030$ (6)°
$\gamma = 79.2876$ (8)°
$V = 1116.93$ (2) Å^3
$Z = 2$
$D_x = 1.515$ Mg m^{-3}
Mo $K\alpha$ radiation
$\mu = 1.96$ mm^{-1}
$T = 160$ (2) K
Prism, colourless
0.28 × 0.25 × 0.20 mm

Data collection

Nonius KappaCCD area-detector diffractometer
φ and ω scans with κ offsets
Absorption correction: multi-scan (*SORTAV*; Blessing, 1995)
$T_{\min} = 0.565$, $T_{\max} = 0.678$
31111 measured reflections
6538 independent reflections
5532 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.057$
$\theta_{\max} = 30.0°$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.038$
$wR(F^2) = 0.092$
$S = 1.11$
6538 reflections
299 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0463P)^2 + 0.3319P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.001$
$\Delta\rho_{\max} = 0.56$ e Å^{-3}
$\Delta\rho_{\min} = -0.57$ e Å^{-3}
Extinction correction: *SHELXL97* (Sheldrick, 1997)
Extinction coefficient: 0.089 (3)

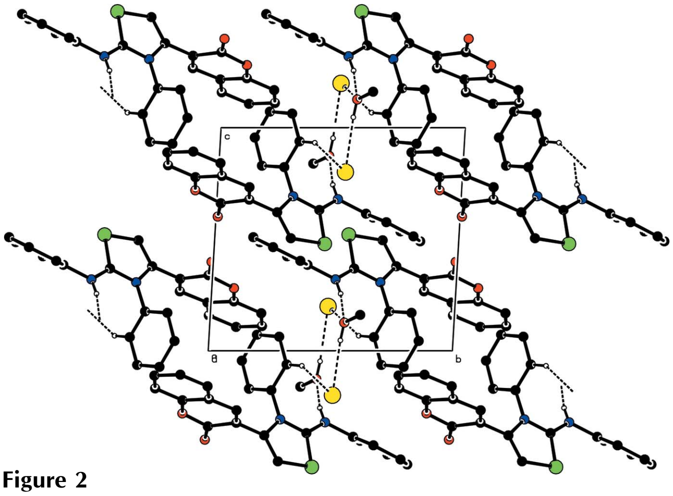

Figure 2
Part of the crystal structure of (I), viewed along the *a* axis. Intermolecular N—H···O, O—H···Br and C—H···Br hydrogen bonds are indicated by dashed lines. All H atoms, except those involved in hydrogen bonding, have been omitted for clarity.

Table 1
Hydrogen-bond geometry (Å, °).

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N21—H21···O52$^{\rm i}$	0.84 (2)	1.92 (2)	2.734 (2)	165 (2)
O52—H52···Br1$^{\rm ii}$	0.80 (3)	2.46 (3)	3.2412 (15)	164 (3)
C32—H32···Br1$^{\rm iii}$	0.95	2.75	3.6296 (18)	153

Symmetry codes: (i) $-x, -y+1, -z+1$; (ii) $-x+1, -y+1, -z$; (iii) $-x+1, -y+1, -z+1$.

H atoms bonded to O and N atoms were located in a difference Fourier map and refined isotropically. Methyl H atoms were constrained to an ideal geometry (C—H = 0.98 Å), with $U_{\rm iso}$(H) = $1.5U_{\rm eq}$(C), but were allowed to rotate about the C—O bond. All remaining H atoms were placed in geometrically idealized positions (C—H = 0.95 Å) and were constrained to ride on their parent atoms, with $U_{\rm iso}$(H) = $1.2U_{\rm eq}$(C).

Data collection: *COLLECT* (Nonius, 2000); cell refinement: *DENZO-SMN* (Otwinowski & Minor, 1997); data reduction: *DENZO-SMN* and *SCALEPACK* (Otwinowski & Minor, 1997); structure solution: *SIR92* (Altomare *et al.*, 1994); structure refinement: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3* (Version 1.07; Farrugia, 1997); software used to prepare material for publication: *SHELXL97* and *PLATON* (Spek, 2003).

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