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Crystal structure of (*Z*)-3-(3-(4-hydroxyphenyl)-2-(phenylimino)-2,3-dihydrothiazol-4-yl)-2*H*-chromen-2-one, $C_{24}H_{16}N_2O_3S$

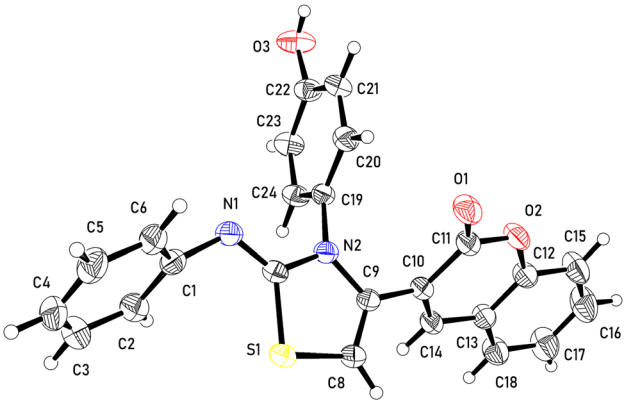

Figure 1: Oretep representation of $C_{24}H_{16}N_2O_3S$ showing 50 % probability ellipsoids.

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Abstract

$C_{24}H_{16}N_2O_3S$, triclinic, $P\bar{1}$ (no. 2), a = 6.7738(3) Å, b = 11.6072(6) Å, c = 13.6060(9) Å, α = 69.197(6)°, β = 87.025(5)°, γ = 76.990(4)°, V = 973.90(10) Å^3, Z = 2, $R_{gt}(F)$ = 0.0482, $wR_{ref}(F^2)$ = 1191, T = 293(2) K.

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The molecular structure is shown in the figure. Table 1 contains crystallographic data and Table 2 contains the list of the atoms including atomic coordinates and displacement parameters.

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Table 1: Data collection and handling.

Crystal:	Needle
Size:	0.51 × 0.18 × 0.09 mm
Wavelength:	Mo *K*α radiation (0.71073 Å)
μ:	0.20 mm^{-1}
Diffractometer, scan mode:	SuperNova, ω
θ_{max}, completeness:	29.8°, >99 %
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	9085, 4643, 0.027
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\sigma(I_{obs})$, 3456
$N(param)_{refined}$:	272
Programs:	CrysAlis^{Pro} [1], SHELX [2], WinGX/ORTEP [3], CHEMDRAW [4]

1 Source of material

A mixture of 4-hydroxyaniline (0.55 g, 5 mmol) and phenyl isothiocyanate (0.68 g, 5 mmol) in EtOH (15 mL) was refluxed for 15 min. 3-(2-Bromoacetyl)-2*H*-chromen-2-one (1.33 g, 5 mmol) was added, and the mixture was refluxed for 4 h. The mixture was left overnight, and the solid formed was filtered, dried, and recrystallized from DMF to give the title heterocycle in 82 % yield, mp 179–181 °C. **IR** (KBr; cm^{-1}): 3121, 1610, 1517. **^{1}H NMR** (δ): 6.56 (s, 1H, thiazolyl), 6.67 (d, 8.6 Hz, 2H, Ar), 6.87 (d, 7.6 Hz, 2H, Ar), 6.98 (t, 7.2 Hz, 1H, Ar), 7.10 (d, 8.6 Hz, 2H, Ar), 7.26–7.36 (m, 4H, Ar), 7.59 (t, 7.2 Hz, 1H, Ar), 7.66 (d, 7.6 Hz, 1H, Ar), 8.15 (s, 1H, Ar), 9.58 (s, exch., 1H, OH). **^{13}C NMR** (δ): 100.7, 115.7, 116.7, 118.8, 119.8, 121.5, 123.4, 125.5, 128.9, 129.4, 130.1, 130.5, 133.2, 134.6, 145.0, 152.0, 153.8, 157.3, 158.4, 159.3. **Anal. calcd.** for $C_{24}H_{16}N_2O_3S$ (412.46): C, 69.89; H, 3.91; N, 6.79; found: C, 69.93; H, 4.01; N, 6.88 %.

2 Experimental details

The hydrogen atoms were located in the difference Fourier map and refined with idealized geometry using a riding model. The O–H bond distance was set at 0.82 Å with free rotation about the C–O bond and displacement parameter 1.5 times U_{iso}(O). The C–H distances were set to 0.93 Å and their U(iso) to 1.2 times the U_{iso}(C). Crystal data, data collection and structure refinement details are summarized in Table 1.

Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2).

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
C1	0.3230 (3)	0.09965 (18)	0.71824 (15)	0.0383 (4)
C2	0.1327 (3)	0.0800 (2)	0.75093 (17)	0.0472 (5)
H2	0.021607	0.118125	0.704308	0.057*
C3	0.1077 (4)	0.0035 (2)	0.85323 (19)	0.0597 (6)
H3	−0.020624	−0.009146	0.874940	0.072*
C4	0.2710 (4)	−0.0540 (2)	0.92320 (18)	0.0599 (6)
H4	0.253591	−0.105995	0.991549	0.072*
C5	0.4589 (4)	−0.0337 (2)	0.89088 (17)	0.0545 (6)
H5	0.569540	−0.071982	0.937827	0.065*
C6	0.4864 (3)	0.0429 (2)	0.78950 (16)	0.0453 (5)
H6	0.614734	0.056533	0.768821	0.054*
C7	0.3107 (3)	0.15544 (17)	0.53638 (15)	0.0341 (4)
C8	0.2202 (3)	0.07071 (19)	0.40507 (16)	0.0410 (5)
H8	0.181907	0.025773	0.367486	0.049*
C9	0.2674 (3)	0.18276 (18)	0.36010 (15)	0.0352 (4)
C10	0.2666 (3)	0.25009 (18)	0.24588 (15)	0.0372 (4)
C11	0.4554 (3)	0.2818 (2)	0.19874 (16)	0.0417 (5)
C12	0.2898 (3)	0.3519 (2)	0.02815 (16)	0.0452 (5)
C13	0.1133 (3)	0.31867 (19)	0.07210 (15)	0.0419 (5)
C14	0.1051 (3)	0.26890 (19)	0.18428 (15)	0.0399 (4)
H14	−0.014458	0.249166	0.215191	0.048*
C15	0.3129 (4)	0.3981 (3)	−0.07901 (18)	0.0638 (7)
H15	0.432265	0.421168	−0.107204	0.077*
C16	0.1541 (4)	0.4092 (3)	−0.14293 (19)	0.0754 (8)
H16	0.167762	0.438865	−0.215479	0.090*
C17	−0.0245 (4)	0.3776 (3)	−0.10238 (19)	0.0680 (7)
H17	−0.130147	0.386257	−0.147271	0.082*
C18	−0.0465 (3)	0.3334 (2)	0.00444 (17)	0.0538 (6)
H18	−0.167949	0.313113	0.031971	0.065*
C19	0.3226 (3)	0.36330 (17)	0.40678 (14)	0.0342 (4)
C20	0.5033 (3)	0.40177 (19)	0.39629 (16)	0.0418 (5)
H20	0.625552	0.343916	0.401233	0.050*
C21	0.5014 (3)	0.52668 (19)	0.37840 (17)	0.0447 (5)
H21	0.622755	0.553395	0.370296	0.054*
C22	0.3191 (3)	0.61256 (18)	0.37248 (15)	0.0416 (5)
C23	0.1393 (3)	0.57410 (19)	0.37911 (17)	0.0451 (5)
H23	0.016550	0.632289	0.371565	0.054*
C24	0.1414 (3)	0.44943 (18)	0.39693 (15)	0.0402 (5)
H24	0.019877	0.423325	0.402319	0.048*
N1	0.3543 (2)	0.18229 (15)	0.61556 (12)	0.0395 (4)
N2	0.3184 (2)	0.23212 (14)	0.43302 (12)	0.0349 (4)
O1	0.6119 (2)	0.26286 (16)	0.24484 (12)	0.0547 (4)
O2	0.4518 (2)	0.33949 (14)	0.09103 (11)	0.0494 (4)
O3	0.3099 (2)	0.73376 (13)	0.36354 (15)	0.0613 (5)
H3A	0.424333	0.742938	0.370313	0.092*
S1	0.23759 (8)	0.01755 (5)	0.54105 (4)	0.04174 (15)

3 Comment

Thiazoles display ample biological activity, and many natural products contain such ring systems [5–7]. Coumarin is a naturally occurring heterocycle, and has various applications [8, 9]. The design, synthesis, and structure elucidation of heterocycles containing thiazole and coumarin moieties is therefore of general interest. The X-ray crystal structures of other related heterocycles have been reported [10–12].

The crystal structure of $C_{24}H_{16}N_2O_3S$ is triclinic, with an asymmetric unit consisting of one molecule (Figure 1) which comprises five planar groups, namely: aniline (**A**: C1–C6, N1), thiazole (**B**: C7–C9, N2, S1), benzopyranone (**C**: C10–C18, O1, O2) and phenol (**D**: C19–C24, O1). The planes through neighbouring groups in the molecules are twisted in relation to each other, with angles **A**/**B** = 63.88(7)°, **B**/**C** = 54.59(6)°, **C**/**D** = 82.09(5)°, **B**/**D** = 86.91(7)°.

In the crystal structure, two O–H···N hydrogen bonds (O3···N1 = 2.733(2) Å, O3–H3A···N1 = 161.5°) link pairs of molecules related by inversion symmetry, with the aniline group accepting a contact from the phenol group. These molecular pairs are also involved in $\pi\cdots\pi$ contacts between neighbouring like groups, specifically symmetry-related benzopyranone pairs and thiazole pairs. The centroid-to-centroid distances are 4.069 Å for the benzopyranone groups and 3.734 Å for the thiazole groups.

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Conflict of interest statement: The authors declare no conflicts of interest regarding this article.

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