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3-Anilinothiocarbonyl-4-hydroxychromen-2-one

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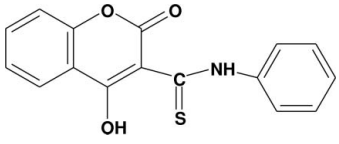
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.003 Å; R factor = 0.049; wR factor = 0.134; data-to-parameter ratio = 18.8.

The geometrical parameters of the title compound, $C_{16}H_{11}NO_3S$, are in the usual ranges. The two aromatic residues are not coplanar and are twisted by a dihedral angle of 66.63 (6)°. The crystal structure is stabilized by N—H···O and O—H···S interactions.

Related literature

For literature on coumarins, see: Campbell (1959); Murray *et al.* (1982); Wolska *et al.* (1990); Harvey (1999); Matern *et al.* (1999); Yang *et al.* (1992); Tsai *et al.* (2000).



Experimental

Crystal data

$C_{16}H_{11}NO_3S$
M_r = 297.33
Monoclinic, $P2_1/c$
a = 14.8059 (9) Å
b = 5.5245 (4) Å
c = 17.4438 (12) Å
β = 109.091 (7)°
V = 1348.34 (16) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.25 mm^{-1}
T = 293 K
0.30 × 0.24 × 0.18 mm

Data collection

Oxford Diffraction Xcalibur diffractometer
Absorption correction: none
11450 measured reflections
4408 independent reflections
2320 reflections with $I > 2\sigma(I)$
R_{int} = 0.036

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.049
$wR(F^2)$ = 0.134
S = 1.01
4408 reflections
235 parameters
All H-atom parameters refined
$\Delta\rho_{max}$ = 0.24 e Å^{-3}
$\Delta\rho_{min}$ = −0.23 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1A···O2	0.96 (2)	1.77 (2)	2.5923 (19)	141 (2)
O3—H3A···S1	1.05 (2)	1.81 (3)	2.8163 (15)	159 (2)

Data collection: *CrysAlisPro* (Oxford Diffraction, 2007); cell refinement: *CrysAlisPro*; data reduction: *CrysAlis RED* (Oxford Diffraction, 2007); program(s) used to solve structure: *SHELXS86* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: JH2075).

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supporting information

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3-Anilinothiocarbonyl-4-hydroxychromen-2-one

Rajni Kant, Lovely Sarmal, Sabeta Kohli, Kamni and Mehtab Parveen

S1. Comment

Coumarins belong to a group of compounds known as benzopyrones, all of which consist of a benzene ring joined to a pyrone moiety.

Coumarins are found to have a wide spectrum of biological activity, *e.g.* antithronbotic effect, vascodilating effect on vessel, tonic influence on capillary blood vessels, reduction of blood pressure, antispastic and photosensitizing effect (Wolska *et al.*, 1990).

Interestingly, coumarins exhibit inhibitory effect on DNA gyrase, which may be linked to anti-HIV (human immuno deficiency virus) activity (Matern *et al.*, 1999).

Coumarins are also found to exhibit anti-malarial activity (Yang *et al.*, 1992).

Recently Collinin, isolated from Zathoxylum Schinifolium, has been found to exhibit anti-HBV (hepatitis B virus) activity (Tsai *et al.*, 2000).

Owing to the general importance of these coumarin analogues we report herein the synthesis and crystal structure of a new coumarin 3-anilinothiocarbonyl-4-hydroxychromen-2-one, (I).

The geometrical parameters (*i.e.* bond distances and angles) of (I) are in the usual ranges. The two aromatic residues are not coplanar and are twisted by a dihedral angle of 66.63 (6)°. The crystal structure is stabilized by *X*—H···A interactions.

S2. Experimental

Scheme1: The mixture of 4-hydroxy coumarin, phenylisothiocyanate was taken in THF.The base Na_2CO_3 was also added to it. The reaction mixture was refluxed on water bath for three hours. Progress of the reaction was monitored by TLC. After completion the reaction mixture was poured into water and worked up with ether and then in ethyl acetate. The ether layer showed the presence of three compounds from which the title compound (I) was separated by column chromatography followed by crystallization from chloroform-methanol as white crystalline solid. Melting point: 421–423 K.

S3. Refinement

All H atoms were located from difference Fourier map and refined isotopically with distance restraints 0.86–1.05 Å.

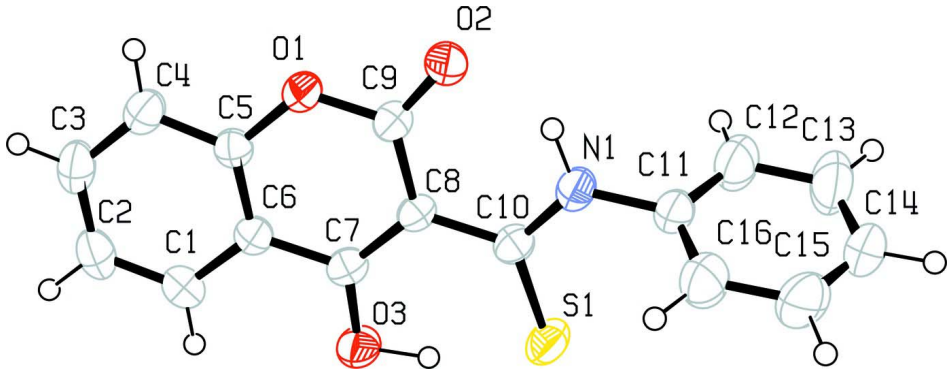

Figure 1

View of (I) (50% probability displacement ellipsoids)

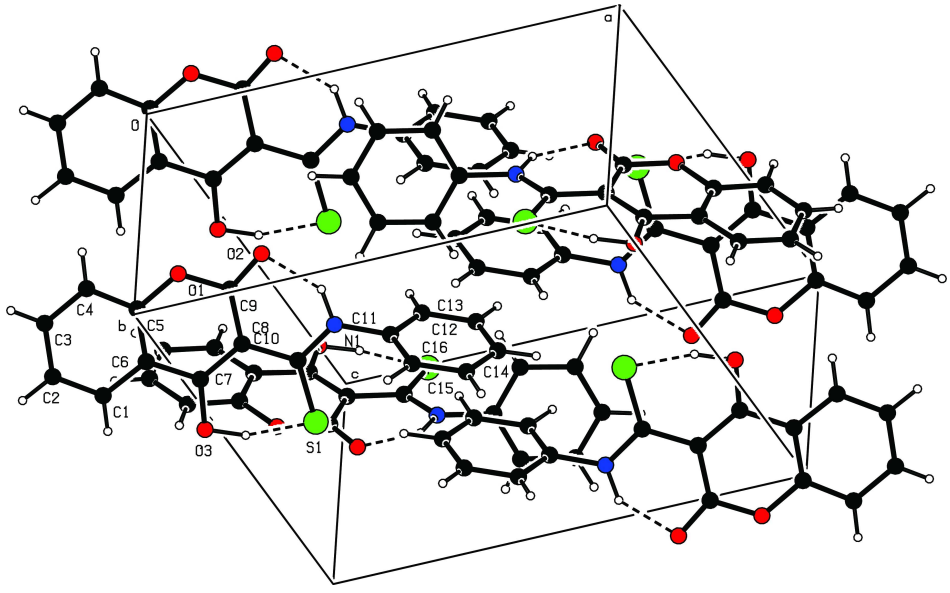

Figure 2

Depiction of *X*—H···A interactions in the title compound(I)

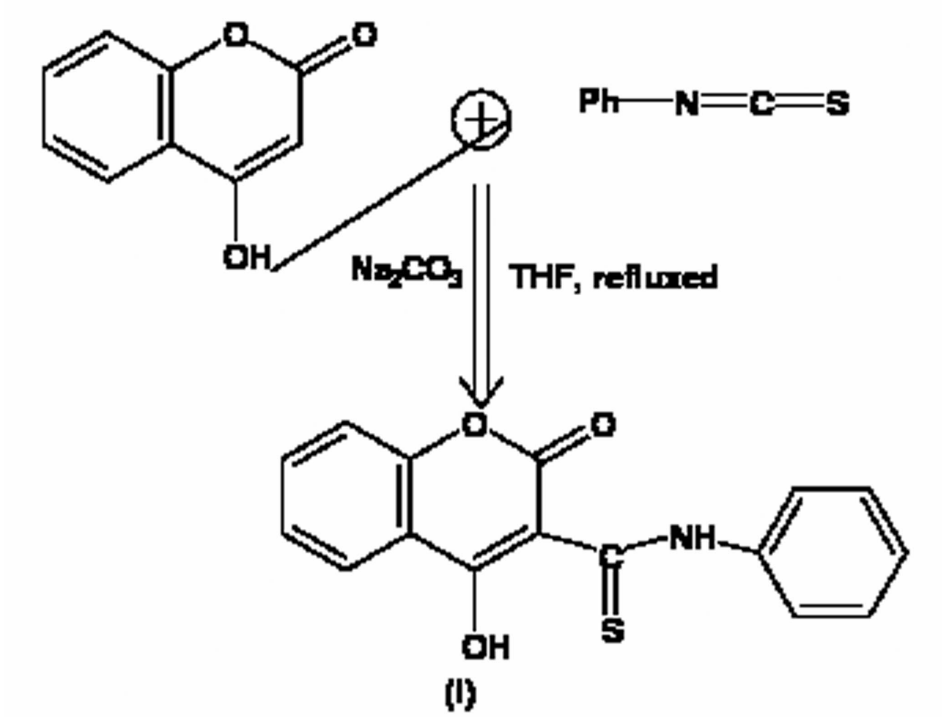

Figure 3

The synthesis of the title compound.

3-Anilinothiocarbonyl-4-hydroxychromen-2-one

Crystal data

$C_{16}H_{11}NO_3S$
$M_r = 297.33$
Monoclinic, $P2_1/c$
$a = 14.8059\ (9)$ Å
$b = 5.5245\ (4)$ Å
$c = 17.4438\ (12)$ Å
$\beta = 109.091\ (7)°$
$V = 1348.34\ (16)$ Å^3
$Z = 4$
$F(000) = 616$
$D_x = 1.465$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 2320 reflections
$\theta = 2.9–32.3°$
$\mu = 0.25$ mm^{-1}
$T = 293$ K
Rectangular, yellow
0.30 × 0.24 × 0.18 mm

Data collection

Oxford Diffraction Xcalibur diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
ω–2θ scans
11450 measured reflections
4408 independent reflections
2320 reflections with $I > 2\sigma(I)$
$R_{int} = 0.036$
$\theta_{max} = 32.3°$, $\theta_{min} = 2.9°$
$h = -20\rightarrow21$
$k = -8\rightarrow5$
$l = -25\rightarrow26$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.049$
$wR(F^2) = 0.134$
$S = 1.01$
4408 reflections
235 parameters
0 restraints

0 constraints
All H-atom parameters refined
$w = 1/[\sigma^2(F_o^2) + (0.0645P)^2]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$

$\Delta\rho_{max} = 0.24$ e Å^{-3}
$\Delta\rho_{min} = -0.23$ e Å^{-3}
Extinction correction: *SHELXL97* (Sheldrick, 2008), $Fc^* = kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.0079 (18)

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

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

	x	*y*	*z*	U_{iso}*/U_{eq}
H3	−0.2431 (14)	−0.089 (4)	−0.0463 (12)	0.058 (6)*
H1	−0.1035 (12)	0.344 (4)	0.1367 (11)	0.044 (5)*
H4	−0.1108 (14)	−0.346 (4)	−0.0190 (11)	0.053 (6)*
H2	−0.2420 (15)	0.255 (4)	0.0323 (13)	0.060 (6)*
H1A	0.2933 (15)	−0.272 (4)	0.2272 (13)	0.066 (7)*
H14	0.6547 (16)	−0.057 (4)	0.4609 (13)	0.074 (6)*
H12	0.4091 (17)	−0.402 (5)	0.3868 (13)	0.080 (8)*
H15	0.5906 (18)	0.239 (5)	0.3553 (16)	0.095 (9)*
H16	0.4290 (17)	0.200 (5)	0.2686 (16)	0.084 (8)*
H13	0.5615 (19)	−0.365 (5)	0.4674 (16)	0.099 (9)*
H3A	0.127 (2)	0.298 (5)	0.2783 (16)	0.102 (9)*
S1	0.24977 (3)	0.22219 (9)	0.33460 (3)	0.05005 (18)
C8	0.14300 (10)	−0.0442 (3)	0.20025 (9)	0.0319 (3)
O1	0.04858 (8)	−0.2950 (2)	0.08806 (7)	0.0400 (3)
O2	0.19660 (8)	−0.3959 (2)	0.14831 (7)	0.0497 (3)
C10	0.23535 (11)	0.0021 (3)	0.26441 (9)	0.0349 (4)
O3	0.06066 (9)	0.2954 (2)	0.23189 (8)	0.0470 (3)
C6	−0.02392 (11)	0.0583 (3)	0.12239 (9)	0.0329 (4)
C7	0.06394 (11)	0.1047 (3)	0.18839 (9)	0.0338 (4)
N1	0.30848 (10)	−0.1395 (3)	0.26561 (9)	0.0452 (4)
C5	−0.02859 (11)	−0.1432 (3)	0.07446 (9)	0.0333 (4)
C1	−0.10418 (12)	0.2061 (3)	0.10584 (12)	0.0423 (4)
C4	−0.10991 (12)	−0.2023 (4)	0.01022 (11)	0.0420 (4)
C9	0.13442 (12)	−0.2508 (3)	0.14694 (10)	0.0351 (4)
C11	0.40436 (12)	−0.1113 (4)	0.32054 (11)	0.0449 (4)
C2	−0.18535 (13)	0.1526 (4)	0.04245 (12)	0.0494 (5)
C3	−0.18805 (13)	−0.0511 (4)	−0.00491 (11)	0.0484 (5)
C16	0.45938 (15)	0.0776 (4)	0.31041 (15)	0.0619 (6)
C12	0.44115 (17)	−0.2784 (5)	0.37918 (17)	0.0725 (7)
C15	0.55294 (16)	0.0988 (5)	0.36243 (18)	0.0735 (7)
C14	0.58922 (15)	−0.0672 (5)	0.42249 (17)	0.0769 (8)
C13	0.53483 (19)	−0.2533 (5)	0.4308 (2)	0.0947 (11)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0422 (3)	0.0555 (3)	0.0476 (3)	−0.0063 (2)	0.00809 (19)	−0.0194 (2)
C8	0.0336 (8)	0.0328 (8)	0.0296 (8)	−0.0035 (7)	0.0105 (6)	−0.0054 (7)
O1	0.0368 (6)	0.0394 (7)	0.0388 (6)	0.0011 (5)	0.0053 (5)	−0.0093 (5)
O2	0.0435 (7)	0.0478 (8)	0.0498 (7)	0.0107 (6)	0.0044 (5)	−0.0145 (6)
C10	0.0351 (8)	0.0355 (9)	0.0351 (8)	−0.0044 (7)	0.0128 (6)	−0.0023 (7)
O3	0.0429 (7)	0.0470 (8)	0.0490 (7)	0.0045 (6)	0.0122 (6)	−0.0170 (6)
C6	0.0341 (8)	0.0331 (9)	0.0323 (8)	−0.0012 (7)	0.0119 (6)	0.0017 (7)
C7	0.0366 (8)	0.0337 (9)	0.0335 (8)	−0.0050 (7)	0.0149 (6)	−0.0015 (7)
N1	0.0335 (7)	0.0466 (9)	0.0490 (9)	−0.0002 (7)	0.0047 (6)	−0.0124 (8)
C5	0.0334 (8)	0.0325 (9)	0.0344 (8)	−0.0012 (7)	0.0113 (6)	0.0034 (7)
C1	0.0434 (10)	0.0391 (10)	0.0451 (10)	0.0058 (8)	0.0154 (8)	0.0049 (9)
C4	0.0413 (9)	0.0444 (11)	0.0373 (9)	−0.0086 (8)	0.0087 (7)	−0.0007 (8)
C9	0.0342 (8)	0.0365 (9)	0.0338 (8)	−0.0028 (7)	0.0099 (6)	−0.0032 (7)
C11	0.0334 (8)	0.0442 (11)	0.0521 (11)	−0.0010 (8)	0.0072 (8)	−0.0101 (9)
C2	0.0370 (9)	0.0545 (13)	0.0525 (11)	0.0086 (9)	0.0088 (8)	0.0123 (10)
C3	0.0347 (9)	0.0594 (13)	0.0439 (10)	−0.0065 (9)	0.0030 (8)	0.0094 (9)
C16	0.0458 (11)	0.0608 (14)	0.0740 (15)	−0.0072 (11)	0.0124 (10)	0.0046 (12)
C12	0.0509 (12)	0.0590 (15)	0.0860 (17)	−0.0148 (11)	−0.0073 (11)	0.0140 (13)
C15	0.0425 (11)	0.0662 (16)	0.110 (2)	−0.0167 (12)	0.0226 (12)	−0.0133 (15)
C14	0.0402 (11)	0.0640 (16)	0.103 (2)	−0.0036 (11)	−0.0091 (12)	−0.0183 (15)
C13	0.0616 (15)	0.0752 (18)	0.105 (2)	−0.0106 (14)	−0.0302 (15)	0.0220 (16)

Geometric parameters (Å, º)

S1—C10	1.6889 (17)	C1—H1	0.929 (19)
S1—H3A	1.81 (3)	C4—C3	1.381 (3)
C8—C7	1.390 (2)	C4—H4	0.94 (2)
C8—C9	1.452 (2)	C11—C12	1.353 (3)
C8—C10	1.479 (2)	C11—C16	1.370 (3)
O1—C9	1.3696 (19)	C2—C3	1.389 (3)
O1—C5	1.3737 (19)	C2—H2	0.98 (2)
O2—C9	1.215 (2)	C3—H3	0.919 (19)
C10—N1	1.330 (2)	C16—C15	1.391 (3)
O3—C7	1.308 (2)	C16—H16	0.99 (3)
O3—H3A	1.05 (3)	C12—C13	1.392 (3)
C6—C5	1.381 (2)	C12—H12	0.86 (3)
C6—C1	1.392 (2)	C15—C14	1.363 (4)
C6—C7	1.450 (2)	C15—H15	0.98 (3)
N1—C11	1.438 (2)	C14—C13	1.343 (4)
N1—H1A	0.97 (2)	C14—H14	0.98 (2)
C5—C4	1.389 (2)	C13—H13	0.88 (3)
C1—C2	1.373 (3)		
C10—S1—H3A	84.7 (8)	O2—C9—O1	114.13 (14)
C7—C8—C9	118.50 (14)	O2—C9—C8	126.81 (15)

C7—C8—C10	122.40 (14)	O1—C9—C8	119.06 (14)
C9—C8—C10	119.10 (14)	C12—C11—C16	120.52 (19)
C9—O1—C5	122.43 (13)	C12—C11—N1	119.68 (18)
N1—C10—C8	117.17 (15)	C16—C11—N1	119.71 (18)
N1—C10—S1	120.18 (12)	C1—C2—C3	120.06 (18)
C8—C10—S1	122.65 (12)	C1—C2—H2	119.6 (13)
C7—O3—H3A	104.8 (15)	C3—C2—H2	120.3 (12)
C5—C6—C1	118.51 (15)	C4—C3—C2	121.14 (17)
C5—C6—C7	118.51 (14)	C4—C3—H3	119.0 (13)
C1—C6—C7	122.98 (16)	C2—C3—H3	119.9 (13)
O3—C7—C8	125.40 (14)	C11—C16—C15	119.2 (2)
O3—C7—C6	114.13 (14)	C11—C16—H16	118.0 (14)
C8—C7—C6	120.45 (15)	C15—C16—H16	122.7 (15)
C10—N1—C11	124.58 (16)	C11—C12—C13	119.4 (2)
C10—N1—H1A	115.4 (13)	C11—C12—H12	123.2 (15)
C11—N1—H1A	120.0 (13)	C13—C12—H12	117.4 (15)
O1—C5—C6	120.90 (14)	C14—C15—C16	120.2 (2)
O1—C5—C4	116.65 (15)	C14—C15—H15	121.9 (15)
C6—C5—C4	122.44 (15)	C16—C15—H15	117.9 (15)
C2—C1—C6	120.24 (19)	C13—C14—C15	119.9 (2)
C2—C1—H1	118.9 (11)	C13—C14—H14	117.6 (13)
C6—C1—H1	120.8 (11)	C15—C14—H14	122.5 (13)
C3—C4—C5	117.60 (18)	C14—C13—C12	120.8 (3)
C3—C4—H4	123.0 (12)	C14—C13—H13	118.1 (18)
C5—C4—H4	119.3 (12)	C12—C13—H13	120.9 (18)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1*A*···O2	0.96 (2)	1.77 (2)	2.5923 (19)	141 (2)
O3—H3*A*···S1	1.05 (2)	1.81 (3)	2.8163 (15)	159 (2)