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(7,8-Dimethyl-2-oxo-2*H*-chromen-4-yl)methyl piperidine-1-carbodithioate

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Keywords: crystal structure; coumarin; piperidine; hydrogen bonding.

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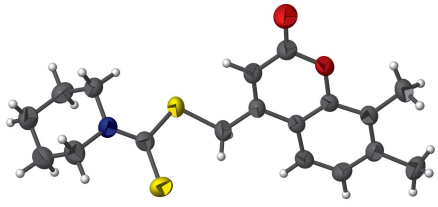
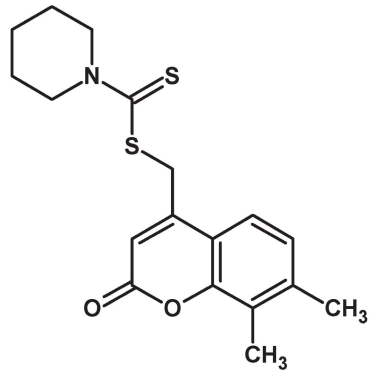
Structural data: full structural data are available from iucrdata.iucr.org

In the title compound, $C_{18}H_{21}NO_2S_2$, the 2*H*-chromene ring systems is nearly planar, with a maximum deviation of 0.023 (2) Å. The coumarin unit makes a dihedral angle of 60.54 (8)° with the piperidine ring, which adopts a chair conformation. The carbodithioate group is present in a synperiplanar conformation with respect to the piperidine ring, as indicated by the torsion angle of −4.7 (2)°. A short intramolecular C—H···S conatct generates an *S*(5) ring. No directional interactions beyond van der Waals contacts could be identified in the crystal.

3D view



Chemical scheme



Structure description

As partof our onging structural studies of coumarin derivatives (Kumar *et al.*, 2012; Anitha *et al.*, 2016), we now describe the structure of the title compound.

The asymmetric unit is shown in Fig. 1. The 2*H*-chromene ring systems is nearly planar, with a maximum deviation of 0.0233 (20) Å for atom C10. The coumarin unit makes a dihedral angle of 60.54 (8)° with the mean plane of the piperidine ring. The carbodithioate group is present in a synperiplanar conformation with respect to the piperidine ring, as indicated by the torsion angle value of −4.7 (2)°. A short intramolecular C—H···S contact (Table 1) generates an S(5) ring. No directional interactions beyond van der Waals' contacts could be identified in the crystal.

Synthesis and crystallization

The title compound was synthesized according to the reported method (Kumar *et al.*, 2012). The compound was recrystallized from an ethanol–chloroform solvent mixture (*v*/*v* = 2/1) to yield colourless needles. Yield = 80%. m.p. 439–441, IR (KBr, cm^{-1}): 999,

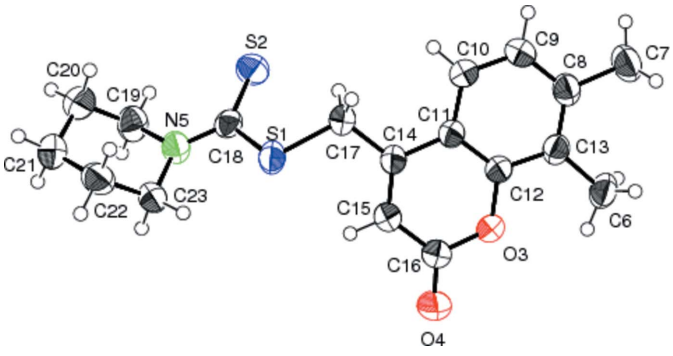


Figure 1
Displacement ellipsoid plot of the title compound.

1220, 1424, 1479, 1721. GCMS: *m*/*e*: 347. 1H NMR (400 MHz, $CDCl_3$, δ, p.p.m): *d* 7.03 (*s*, 1H, Ar—H), 6.92 (*s*, 1H, Ar—H), 6.53 (*s*, 1H, Ar—H), 4.80 (*s*, 2H, CH_2), 4.29 (*s*, 2H, CH_2), 3.90 (*s*, 2H, CH_2), 2.79 (*s*, 3H, CH_3), 2.38 (*s*, 3H, CH_3), 1.73 (*m*, 6H, CH_3). Mol. formula: $C_{18}H_{21}NO_2S_2$. Elemental analysis: C, 62.21; H, 6.09; N, 4.03 (calculated); C, 62.25; H, 6.04; N, 4.07 (found).

Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2.

Acknowledgements

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C17—H17*A*···S2	0.97	2.45	3.134 (2)	127

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{18}H_{21}NO_2S_2$
M_r	347.48
Crystal system, space group	Monoclinic, $P2_1/c$
Temperature (K)	296
a, *b*, *c* (Å)	11.6767 (3), 16.2058 (3), 9.1417 (2)
β (°)	99.486 (2)
V (Å^3)	1706.23 (7)
Z	4
Radiation type	Mo $K\alpha$
μ (mm^{-1})	0.32
Crystal size (mm)	0.24 × 0.20 × 0.12
Data collection	
Diffractometer	Bruker SMART CCD area-detector
Absorption correction	Multi-scan (*SADABS*; Bruker, 2009)
T_{min}, T_{max}	0.770, 1.000
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	19606, 5070, 3111
R_{int}	0.036
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.711
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, *S*	0.049, 0.140, 1.06
No. of reflections	5070
No. of parameters	208
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.29, −0.18

Computer programs: *SMART* (Bruker, 2009), *SAINT* (Bruker, 2009), *SHELXS97* (Sheldrick, 2008), *SHELXL2014* (Sheldrick, 2015), *ORTEP-3 for Windows* (Farrugia, 2012).

full crystallographic data

IUCrData (2016). **1**, x160171 [https://doi.org/10.1107/S2414314616001711]

(7,8-Dimethyl-2-oxo-2*H*-chromen-4-yl)methyl piperidine-1-carbodithioate

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Crystal data

$C_{18}H_{21}NO_2S_2$
M_r = 347.48
Monoclinic, $P2_1/c$
a = 11.6767 (3) Å
b = 16.2058 (3) Å
c = 9.1417 (2) Å
β = 99.486 (2)°
V = 1706.23 (7) Å^3
Z = 4
$F(000)$ = 736
D_x = 1.353 Mg m^{-3}
Melting point: 439 K
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 5070 reflections
θ = 1.8–30.3°
μ = 0.32 mm^{-1}
T = 296 K
Plate, colourless
0.24 × 0.20 × 0.12 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
ω and φ scans
Absorption correction: multi-scan (SADABS; Bruker, 2009)
T_{min} = 0.770, T_{max} = 1.000
19606 measured reflections
5070 independent reflections
3111 reflections with $I > 2\sigma(I)$
R_{int} = 0.036
θ_{max} = 30.3°, θ_{min} = 1.8°
h = −14→16
k = −22→22
l = −11→12

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.049
$wR(F^2)$ = 0.140
S = 1.06
5070 reflections
208 parameters
0 restraints
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0649P)^2 + 0.1225P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.29 e Å^{-3}
$\Delta\rho_{min}$ = −0.18 e Å^{-3}

Special details

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

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
S1	0.72757 (4)	0.85043 (3)	0.04750 (6)	0.05596 (17)
S2	0.97470 (5)	0.86854 (3)	0.20673 (7)	0.06158 (18)
O3	0.52322 (11)	1.12401 (7)	0.20252 (15)	0.0522 (3)
O4	0.43238 (14)	1.02016 (9)	0.2869 (2)	0.0849 (5)
N5	0.85029 (13)	0.73033 (9)	0.18090 (18)	0.0505 (4)
C6	0.52787 (18)	1.29307 (12)	0.1852 (3)	0.0624 (5)
H6A	0.4757	1.2581	0.2278	0.094*
H6B	0.5689	1.3282	0.2607	0.094*
H6C	0.4844	1.3263	0.1085	0.094*
C7	0.7133 (2)	1.36691 (12)	0.0386 (3)	0.0676 (6)
H7A	0.7770	1.3786	−0.0125	0.101*
H7B	0.6433	1.3901	−0.0157	0.101*
H7C	0.7285	1.3907	0.1361	0.101*
C8	0.69952 (16)	1.27483 (11)	0.0510 (2)	0.0502 (4)
C9	0.77554 (17)	1.22298 (12)	−0.0077 (2)	0.0560 (5)
H9	0.8318	1.2462	−0.0562	0.067*
C10	0.77018 (16)	1.13909 (11)	0.0036 (2)	0.0500 (4)
H10	0.8223	1.1062	−0.0368	0.060*
C11	0.68595 (14)	1.10240 (10)	0.07637 (19)	0.0419 (4)
C12	0.60991 (14)	1.15510 (10)	0.13132 (19)	0.0419 (4)
C13	0.61317 (15)	1.24084 (11)	0.1205 (2)	0.0456 (4)
C14	0.67517 (14)	1.01447 (10)	0.0980 (2)	0.0438 (4)
C15	0.59226 (16)	0.98717 (11)	0.1716 (2)	0.0511 (4)
H15	0.5870	0.9308	0.1884	0.061*
C16	0.51136 (17)	1.04126 (12)	0.2253 (2)	0.0547 (5)
C17	0.75634 (18)	0.95856 (11)	0.0327 (2)	0.0565 (5)
H17A	0.8351	0.9695	0.0817	0.068*
H17B	0.7522	0.9723	−0.0713	0.068*
C18	0.85766 (15)	0.81038 (11)	0.15314 (18)	0.0440 (4)
C19	0.95023 (19)	0.68230 (13)	0.2544 (2)	0.0607 (5)
H19A	0.9315	0.6568	0.3435	0.073*
H19B	1.0163	0.7185	0.2827	0.073*
C20	0.98043 (19)	0.61655 (13)	0.1501 (3)	0.0673 (6)
H20A	1.0057	0.6425	0.0653	0.081*
H20B	1.0441	0.5834	0.2005	0.081*
C21	0.8778 (2)	0.56170 (13)	0.0977 (3)	0.0731 (7)
H21A	0.8587	0.5304	0.1808	0.088*
H21B	0.8976	0.5230	0.0249	0.088*
C22	0.77386 (19)	0.61218 (12)	0.0296 (2)	0.0609 (5)
H22A	0.7893	0.6376	−0.0612	0.073*
H22B	0.7070	0.5763	0.0047	0.073*
C23	0.74742 (16)	0.67820 (11)	0.1354 (2)	0.0533 (5)
H23A	0.6834	0.7120	0.0876	0.064*
H23B	0.7245	0.6527	0.2221	0.064*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0489 (3)	0.0383 (3)	0.0793 (4)	0.00565 (19)	0.0064 (2)	−0.0011 (2)
S2	0.0517 (3)	0.0575 (3)	0.0752 (4)	−0.0109 (2)	0.0093 (3)	−0.0097 (2)
O3	0.0466 (7)	0.0446 (7)	0.0696 (9)	0.0051 (5)	0.0225 (6)	0.0014 (6)
O4	0.0832 (11)	0.0629 (10)	0.1247 (14)	−0.0001 (8)	0.0649 (11)	0.0065 (9)
N5	0.0483 (9)	0.0424 (8)	0.0579 (10)	0.0004 (6)	0.0000 (7)	−0.0011 (7)
C6	0.0590 (13)	0.0456 (11)	0.0829 (15)	0.0110 (9)	0.0125 (11)	−0.0083 (10)
C7	0.0735 (15)	0.0421 (11)	0.0856 (16)	−0.0061 (10)	0.0086 (13)	0.0034 (10)
C8	0.0484 (10)	0.0398 (10)	0.0593 (12)	−0.0009 (8)	0.0001 (9)	0.0008 (8)
C9	0.0506 (11)	0.0480 (11)	0.0719 (13)	−0.0061 (8)	0.0174 (10)	0.0064 (9)
C10	0.0431 (10)	0.0471 (10)	0.0623 (12)	0.0029 (8)	0.0163 (9)	0.0026 (8)
C11	0.0381 (9)	0.0385 (9)	0.0487 (10)	0.0030 (7)	0.0062 (7)	0.0021 (7)
C12	0.0367 (9)	0.0405 (9)	0.0483 (10)	0.0012 (7)	0.0064 (7)	0.0017 (7)
C13	0.0414 (9)	0.0410 (9)	0.0524 (10)	0.0033 (7)	0.0015 (8)	−0.0032 (7)
C14	0.0428 (9)	0.0365 (9)	0.0519 (10)	0.0040 (7)	0.0073 (8)	0.0021 (7)
C15	0.0521 (11)	0.0401 (9)	0.0638 (12)	0.0014 (8)	0.0181 (9)	0.0054 (8)
C16	0.0519 (11)	0.0471 (10)	0.0688 (13)	−0.0009 (8)	0.0212 (10)	0.0031 (9)
C17	0.0575 (12)	0.0383 (10)	0.0786 (14)	0.0069 (8)	0.0260 (10)	0.0031 (9)
C18	0.0459 (10)	0.0450 (10)	0.0428 (10)	0.0024 (7)	0.0126 (8)	−0.0070 (7)
C19	0.0600 (12)	0.0545 (12)	0.0633 (13)	0.0082 (9)	−0.0025 (10)	0.0071 (10)
C20	0.0577 (13)	0.0602 (13)	0.0856 (16)	0.0086 (10)	0.0169 (11)	0.0068 (11)
C21	0.0715 (15)	0.0473 (12)	0.1060 (19)	0.0015 (10)	0.0307 (14)	−0.0117 (11)
C22	0.0647 (13)	0.0533 (12)	0.0672 (13)	−0.0136 (10)	0.0182 (11)	−0.0062 (10)
C23	0.0498 (11)	0.0409 (9)	0.0695 (13)	−0.0025 (8)	0.0104 (10)	0.0029 (9)

Geometric parameters (Å, º)

S1—C18	1.7825 (18)	C11—C12	1.385 (2)
S1—C17	1.7937 (18)	C11—C14	1.447 (2)
S2—C18	1.6660 (18)	C12—C13	1.394 (2)
O3—C16	1.368 (2)	C14—C15	1.342 (2)
O3—C12	1.385 (2)	C14—C17	1.504 (2)
O4—C16	1.206 (2)	C15—C16	1.434 (2)
N5—C18	1.327 (2)	C15—H15	0.9300
N5—C19	1.470 (2)	C17—H17A	0.9700
N5—C23	1.471 (2)	C17—H17B	0.9700
C6—C13	1.501 (2)	C19—C20	1.510 (3)
C6—H6A	0.9600	C19—H19A	0.9700
C6—H6B	0.9600	C19—H19B	0.9700
C6—H6C	0.9600	C20—C21	1.506 (3)
C7—C8	1.507 (3)	C20—H20A	0.9700
C7—H7A	0.9600	C20—H20B	0.9700
C7—H7B	0.9600	C21—C22	1.510 (3)
C7—H7C	0.9600	C21—H21A	0.9700
C8—C13	1.391 (3)	C21—H21B	0.9700
C8—C9	1.392 (3)	C22—C23	1.508 (3)

C9—C10	1.366 (3)	C22—H22A	0.9700
C9—H9	0.9300	C22—H22B	0.9700
C10—C11	1.407 (2)	C23—H23A	0.9700
C10—H10	0.9300	C23—H23B	0.9700

C18—S1—C17	104.07 (9)	O4—C16—C15	125.74 (18)
C16—O3—C12	121.90 (14)	O3—C16—C15	117.27 (15)
C18—N5—C19	122.23 (16)	C14—C17—S1	114.87 (13)
C18—N5—C23	125.64 (15)	C14—C17—H17A	108.5
C19—N5—C23	112.02 (15)	S1—C17—H17A	108.5
C13—C6—H6A	109.5	C14—C17—H17B	108.5
C13—C6—H6B	109.5	S1—C17—H17B	108.5
H6A—C6—H6B	109.5	H17A—C17—H17B	107.5
C13—C6—H6C	109.5	N5—C18—S2	124.93 (14)
H6A—C6—H6C	109.5	N5—C18—S1	112.53 (13)
H6B—C6—H6C	109.5	S2—C18—S1	122.54 (11)
C8—C7—H7A	109.5	N5—C19—C20	109.61 (17)
C8—C7—H7B	109.5	N5—C19—H19A	109.7
H7A—C7—H7B	109.5	C20—C19—H19A	109.7
C8—C7—H7C	109.5	N5—C19—H19B	109.7
H7A—C7—H7C	109.5	C20—C19—H19B	109.7
H7B—C7—H7C	109.5	H19A—C19—H19B	108.2
C13—C8—C9	119.55 (16)	C21—C20—C19	111.18 (18)
C13—C8—C7	121.40 (17)	C21—C20—H20A	109.4
C9—C8—C7	119.05 (18)	C19—C20—H20A	109.4
C10—C9—C8	122.10 (17)	C21—C20—H20B	109.4
C10—C9—H9	118.9	C19—C20—H20B	109.4
C8—C9—H9	118.9	H20A—C20—H20B	108.0
C9—C10—C11	120.07 (17)	C20—C21—C22	110.78 (17)
C9—C10—H10	120.0	C20—C21—H21A	109.5
C11—C10—H10	120.0	C22—C21—H21A	109.5
C12—C11—C10	116.80 (15)	C20—C21—H21B	109.5
C12—C11—C14	118.84 (15)	C22—C21—H21B	109.5
C10—C11—C14	124.35 (15)	H21A—C21—H21B	108.1
C11—C12—O3	120.53 (15)	C23—C22—C21	110.82 (18)
C11—C12—C13	124.22 (16)	C23—C22—H22A	109.5
O3—C12—C13	115.24 (14)	C21—C22—H22A	109.5
C8—C13—C12	117.23 (16)	C23—C22—H22B	109.5
C8—C13—C6	122.32 (17)	C21—C22—H22B	109.5
C12—C13—C6	120.44 (16)	H22A—C22—H22B	108.1
C15—C14—C11	118.65 (15)	N5—C23—C22	110.17 (15)
C15—C14—C17	123.66 (16)	N5—C23—H23A	109.6
C11—C14—C17	117.67 (15)	C22—C23—H23A	109.6
C14—C15—C16	122.75 (17)	N5—C23—H23B	109.6
C14—C15—H15	118.6	C22—C23—H23B	109.6
C16—C15—H15	118.6	H23A—C23—H23B	108.1
O4—C16—O3	116.98 (17)		

C13—C8—C9—C10	−1.6 (3)	C11—C14—C15—C16	2.2 (3)
C7—C8—C9—C10	178.0 (2)	C17—C14—C15—C16	−176.22 (19)
C8—C9—C10—C11	0.0 (3)	C12—O3—C16—O4	−179.19 (18)
C9—C10—C11—C12	1.2 (3)	C12—O3—C16—C15	−0.4 (3)
C9—C10—C11—C14	−178.15 (19)	C14—C15—C16—O4	176.9 (2)
C10—C11—C12—O3	179.04 (16)	C14—C15—C16—O3	−1.8 (3)
C14—C11—C12—O3	−1.6 (3)	C15—C14—C17—S1	4.6 (3)
C10—C11—C12—C13	−0.9 (3)	C11—C14—C17—S1	−173.78 (14)
C14—C11—C12—C13	178.49 (17)	C18—S1—C17—C14	−119.89 (15)
C16—O3—C12—C11	2.0 (3)	C19—N5—C18—S2	−4.7 (2)
C16—O3—C12—C13	−178.05 (17)	C23—N5—C18—S2	179.53 (14)
C9—C8—C13—C12	1.9 (3)	C19—N5—C18—S1	174.31 (14)
C7—C8—C13—C12	−177.70 (18)	C23—N5—C18—S1	−1.5 (2)
C9—C8—C13—C6	−179.57 (18)	C17—S1—C18—N5	177.30 (13)
C7—C8—C13—C6	0.9 (3)	C17—S1—C18—S2	−3.67 (13)
C11—C12—C13—C8	−0.6 (3)	C18—N5—C19—C20	−116.9 (2)
O3—C12—C13—C8	179.44 (16)	C23—N5—C19—C20	59.4 (2)
C11—C12—C13—C6	−179.25 (18)	N5—C19—C20—C21	−56.5 (2)
O3—C12—C13—C6	0.8 (3)	C19—C20—C21—C22	54.5 (3)
C12—C11—C14—C15	−0.5 (3)	C20—C21—C22—C23	−54.1 (3)
C10—C11—C14—C15	178.87 (18)	C18—N5—C23—C22	116.7 (2)
C12—C11—C14—C17	178.01 (17)	C19—N5—C23—C22	−59.4 (2)
C10—C11—C14—C17	−2.7 (3)	C21—C22—C23—N5	55.9 (2)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C17—H17*A*···S2	0.97	2.45	3.134 (2)	127