organic compounds

3-Butyl-1H-isochromene-1-thione

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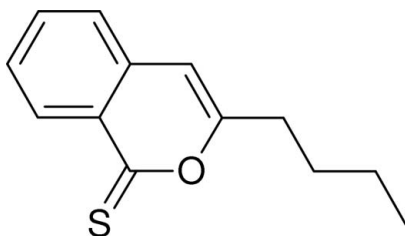Correspondence e-mail: ssctng@sscu.iisc.ernet.in

In the title compound, $C_{13}H_{14}OS$, a derivative of thio-isocoumarin, the packing is stabilized by weak $C-H \cdots S$, $C-H \cdots \pi$ and $\pi-\pi$ [separation 3.7481 (12) Å] interactions.

Related literature

For a related structure, see: Hathwar *et al.* (2007). For related literature, see: Saeed *et al.* (2006, 2007). For background, see: Barry (1964); Napolitano (1997).

Experimental

Crystal data

$C_{13}H_{14}OS$	$\gamma = 62.924\ (1)°$
$M_r = 218.31$	$V = 573.75\ (7)$ Å^3
Triclinic, $P\bar{1}$	$Z = 2$
$a = 7.8699\ (6)$ Å	Mo $K\alpha$ radiation
$b = 8.5840\ (6)$ Å	$\mu = 0.25$ mm^{-1}
$c = 9.8083\ (7)$ Å	$T = 290\ (2)$ K
$\alpha = 82.032\ (1)°$	$0.20 \times 0.11 \times 0.05$ mm
$\beta = 76.713\ (1)°$	

Data collection

Bruker SMART CCD area-detector diffractometer	6031 measured reflections
	2249 independent reflections
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)	1777 reflections with $I > 2\sigma(I)$
$T_{min} = 0.938$, $T_{max} = 0.988$	$R_{int} = 0.014$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.041$	192 parameters
$wR(F^2) = 0.107$	All H-atom parameters refined
$S = 1.05$	$\Delta\rho_{max} = 0.17$ e Å^{-3}
2249 reflections	$\Delta\rho_{min} = -0.21$ e Å^{-3}

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

Cg is the centroid of the C2–C7 ring.

$D-H \cdots A$	$D-H$	$H \cdots A$	$D \cdots A$	$D-H \cdots A$
$C3-H3 \cdots S1$	0.92 (2)	2.77 (2)	3.1621 (18)	106.7 (16)
$C10-H10B \cdots Cg^i$	0.98 (2)	2.98 (2)	3.813 (3)	144.1 (13)

Symmetry code: (i) $-x, -y + 1, -z$.

Data collection: *SMART* (Bruker, 2004); cell refinement: *SAINT* (Bruker, 2004); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Bruker, 2000); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3* (Farrugia, 1997) and *CAMERON* (Watkin *et al.*, 1993); software used to prepare material for publication: *PLATON* (Spek, 2003).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HB2499).

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supplementary materials

3-Butyl-1*H*-isochromene-1-thione

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Comment

Thioisocoumarin compounds are widely used in medicinal chemistry and are commonly found in higher plants, marine organisms and in a variety of fungi, lichens and bacteria (Barry, 1964; Napolitano,1997).

In the thioisocoumarin derivative title compound, (I), (Fig. 1), the dihedral angle between the isocoumarin moiety and the *n*-butyl side chain is 1.90°. The *n*-butyl group adopts an all *trans* configuration.

The crystal structure is stabilized by an acute C—H···S intramolecular interaction, an intermolecular C—H···π bond (Table 1) along with an aromatic π···π interaction with a separation of 3.7481 (12) Å (symmetry code: $1 - x$, $1 - y$, $-z$) between the centroids of the aromatic rings.

It is noteworthy that in the related 3-butyl-*1*H isochromene-1-one (ref, date), the molecules are held together only *via* C—H···O interactions.

Experimental

The title compound was synthesized from a mixture of 3-butyl isocoumarin (1 mmol) and Lawesson's reagent (1.2 mmol). The mixture was placed in a glass tube fitted with a tightened rubber septum and was refluxed at 393 K in the presence of toluene solvent under a nitrogen atmosphere. The completion of the reaction was monitored by TLC using a hexane:ethylacetate (9:1 *v/v*) mixture. After the completion of reaction,the mixture was dissolved in dichloromethane and adsorbed on silica gel·The compound was purified by column chromatography using a mixture of hexane/ethyl acetate (9:1 *v/v*). Yellow crystals of (I) were recrystalized from ether.

Refinement

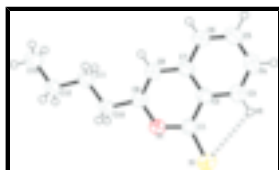All the H atoms were located and refined isotropically resulting in C—H bond lengths of 0.89 (2)–0.97 (2) Å.

Figures

Fig. 1. View of the molecular structure of (I) showing 50% displacement ellipsoids (H atoms are shown as spheres of arbitrary radius).
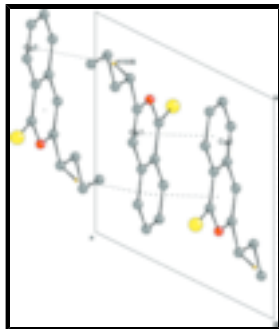
supplementary materials

Fig. 2. Packing diagram of (I) viewed down the *c* axis. The dotted lines indicate intermolecular C—H⋯π and π⋯π stacking interactions.

3-Butyl-1*H*-isochromene-1-thione

Crystal data

$C_{13}H_{14}OS$	$Z = 2$
$M_r = 218.31$	$F_{000} = 232$
Triclinic, $P\overline{1}$	$D_x = 1.264$ Mg m^{-3}
Hall symbol: -P 1	Mo $K\alpha$ radiation $\lambda = 0.71073$ Å
$a = 7.8699$ (6) Å	Cell parameters from 2281 reflections
$b = 8.5840$ (6) Å	$\theta = 2.7–25.3°$
$c = 9.8083$ (7) Å	$\mu = 0.25$ mm^{-1}
$\alpha = 82.032$ (1)°	$T = 290$ (2) K
$\beta = 76.713$ (1)°	Plate, yellow
$\gamma = 62.924$ (1)°	$0.20 \times 0.11 \times 0.05$ mm
$V = 573.75$ (7) Å^3	

Data collection

Bruker SMART CCD area-detector diffractometer	2249 independent reflections
Radiation source: fine-focus sealed tube	1777 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{int} = 0.014$
$T = 290(2)$ K	$\theta_{max} = 26.0°$
ω scans	$\theta_{min} = 2.1°$
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	$h = -9 \rightarrow 9$
$T_{min} = 0.938$, $T_{max} = 0.988$	$k = -10 \rightarrow 10$
6031 measured reflections	$l = -12 \rightarrow 12$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: difference Fourier map
$R[F^2 > 2\sigma(F^2)] = 0.041$	All H-atom parameters refined
$wR(F^2) = 0.107$	$w = 1/[\sigma^2(F_o^2) + (0.0594P)^2 + 0.0575P]$ where $P = (F_o^2 + 2F_c^2)/3$

$S = 1.05$	$(\Delta/\sigma)_{max} < 0.001$
2249 reflections	$\Delta\rho_{max} = 0.17$ e Å^{-3}
192 parameters	$\Delta\rho_{min} = -0.21$ e Å^{-3}
Primary atom site location: structure-invariant direct methods	Extinction correction: none

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.

Refinement. Refinement of F^2 against ALL reflections. The weighted R-factor wR and goodness of fit S are based on F^2, conventional R-factors R are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > 2\mathrm{sigma}(F^2)$ is used only for calculating R-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. R-factors based on F^2 are statistically about twice as large as those based on F, and R– factors based on ALL data will be even larger.

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

	x	y	z	$U_{iso}*/U_{eq}$
S1	0.43615 (8)	0.25638 (6)	−0.32536 (5)	0.0693 (2)
O1	0.30654 (16)	0.27779 (13)	−0.06157 (11)	0.0519 (3)
C1	0.3570 (2)	0.3626 (2)	−0.17997 (17)	0.0487 (4)
C2	0.3340 (2)	0.53771 (19)	−0.16539 (16)	0.0473 (4)
C3	0.3829 (3)	0.6351 (2)	−0.28165 (19)	0.0558 (4)
C4	0.3587 (3)	0.8002 (2)	−0.2656 (2)	0.0618 (5)
C5	0.2869 (3)	0.8736 (2)	−0.1350 (2)	0.0632 (5)
C6	0.2392 (3)	0.7808 (2)	−0.0203 (2)	0.0582 (4)
C7	0.2605 (2)	0.6114 (2)	−0.03317 (17)	0.0490 (4)
C8	0.2109 (3)	0.5103 (2)	0.08443 (19)	0.0522 (4)
C9	0.2350 (2)	0.3500 (2)	0.06889 (17)	0.0485 (4)
C10	0.1948 (3)	0.2248 (2)	0.17760 (18)	0.0530 (4)
C11	0.1120 (3)	0.2967 (2)	0.32359 (18)	0.0515 (4)
C12	0.0685 (3)	0.1694 (2)	0.43241 (19)	0.0588 (5)
C13	−0.0232 (4)	0.2464 (3)	0.5754 (2)	0.0713 (6)
H3	0.429 (3)	0.585 (3)	−0.368 (2)	0.066 (5)*
H4	0.395 (3)	0.857 (3)	−0.341 (2)	0.067 (6)*
H5	0.274 (3)	0.990 (3)	−0.127 (2)	0.074 (6)*
H6	0.196 (3)	0.824 (3)	0.065 (2)	0.068 (6)*
H8	0.165 (3)	0.557 (2)	0.170 (2)	0.063 (5)*
H10A	0.316 (3)	0.124 (2)	0.1765 (17)	0.055 (5)*
H10B	0.106 (3)	0.193 (2)	0.1454 (19)	0.066 (5)*
H11A	0.201 (2)	0.328 (2)	0.3513 (17)	0.052 (4)*
H11B	−0.010 (3)	0.403 (3)	0.3205 (19)	0.063 (5)*
H12A	0.189 (3)	0.066 (3)	0.438 (2)	0.069 (5)*
H12B	−0.015 (3)	0.136 (3)	0.397 (2)	0.081 (6)*
H13A	0.054 (3)	0.288 (3)	0.611 (2)	0.095 (7)*

H13B	−0.139 (4)	0.348 (3)	0.566 (3)	0.099 (8)*
H13C	−0.053 (3)	0.171 (3)	0.645 (3)	0.100 (8)*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0912 (4)	0.0545 (3)	0.0547 (3)	−0.0277 (3)	−0.0019 (2)	−0.0138 (2)
O1	0.0646 (7)	0.0413 (6)	0.0495 (6)	−0.0236 (5)	−0.0082 (5)	−0.0040 (5)
C1	0.0479 (9)	0.0433 (8)	0.0517 (9)	−0.0163 (7)	−0.0117 (7)	−0.0012 (7)
C2	0.0477 (8)	0.0418 (8)	0.0514 (9)	−0.0176 (7)	−0.0136 (7)	0.0007 (7)
C3	0.0620 (10)	0.0541 (10)	0.0501 (10)	−0.0251 (8)	−0.0116 (8)	0.0024 (8)
C4	0.0695 (12)	0.0543 (10)	0.0646 (12)	−0.0320 (9)	−0.0169 (9)	0.0135 (9)
C5	0.0739 (12)	0.0456 (9)	0.0746 (13)	−0.0297 (9)	−0.0166 (10)	0.0003 (9)
C6	0.0725 (12)	0.0439 (9)	0.0599 (11)	−0.0270 (8)	−0.0103 (9)	−0.0055 (8)
C7	0.0521 (9)	0.0416 (8)	0.0529 (9)	−0.0192 (7)	−0.0124 (7)	−0.0016 (7)
C8	0.0639 (10)	0.0459 (9)	0.0478 (9)	−0.0257 (8)	−0.0079 (8)	−0.0040 (7)
C9	0.0529 (9)	0.0439 (8)	0.0484 (9)	−0.0205 (7)	−0.0096 (7)	−0.0037 (7)
C10	0.0611 (11)	0.0424 (9)	0.0574 (10)	−0.0253 (8)	−0.0100 (8)	−0.0007 (7)
C11	0.0562 (10)	0.0446 (9)	0.0566 (10)	−0.0257 (8)	−0.0084 (8)	−0.0017 (7)
C12	0.0722 (12)	0.0466 (10)	0.0577 (11)	−0.0296 (9)	−0.0046 (9)	−0.0027 (8)
C13	0.0934 (16)	0.0656 (13)	0.0614 (12)	−0.0470 (13)	0.0032 (11)	−0.0091 (10)

Geometric parameters (Å, °)

S1—C1	1.6396 (16)	C8—C9	1.326 (2)
O1—C1	1.361 (2)	C8—H8	0.904 (19)
O1—C9	1.3817 (19)	C9—C10	1.493 (2)
C1—C2	1.453 (2)	C10—C11	1.518 (2)
C2—C7	1.402 (2)	C10—H10A	0.950 (17)
C2—C3	1.405 (2)	C10—H10B	0.98 (2)
C3—C4	1.368 (3)	C11—C12	1.516 (2)
C3—H3	0.92 (2)	C11—H11A	0.954 (18)
C4—C5	1.387 (3)	C11—H11B	0.985 (19)
C4—H4	0.89 (2)	C12—C13	1.510 (3)
C5—C6	1.368 (3)	C12—H12A	0.97 (2)
C5—H5	0.97 (2)	C12—H12B	0.97 (2)
C6—C7	1.407 (2)	C13—H13A	0.97 (2)
C6—H6	0.89 (2)	C13—H13B	0.95 (3)
C7—C8	1.436 (2)	C13—H13C	0.94 (3)
C1—O1—C9	123.44 (12)	C8—C9—C10	128.69 (15)
O1—C1—C2	117.11 (14)	O1—C9—C10	110.82 (13)
O1—C1—S1	116.27 (11)	C9—C10—C11	113.79 (14)
C2—C1—S1	126.61 (13)	C9—C10—H10A	105.7 (10)
C7—C2—C3	119.31 (15)	C11—C10—H10A	110.4 (10)
C7—C2—C1	119.49 (14)	C9—C10—H10B	106.5 (11)
C3—C2—C1	121.20 (15)	C11—C10—H10B	111.4 (11)
C4—C3—C2	120.19 (18)	H10A—C10—H10B	108.7 (15)
C4—C3—H3	121.6 (13)	C12—C11—C10	113.22 (14)

C2—C3—H3	118.2 (13)	C12—C11—H11A	109.4 (10)
C3—C4—C5	120.79 (18)	C10—C11—H11A	109.4 (10)
C3—C4—H4	117.8 (13)	C12—C11—H11B	107.9 (11)
C5—C4—H4	121.3 (13)	C10—C11—H11B	108.4 (11)
C6—C5—C4	120.01 (17)	H11A—C11—H11B	108.4 (15)
C6—C5—H5	121.2 (12)	C13—C12—C11	112.48 (16)
C4—C5—H5	118.8 (12)	C13—C12—H12A	109.5 (11)
C5—C6—C7	120.73 (18)	C11—C12—H12A	108.4 (12)
C5—C6—H6	121.3 (14)	C13—C12—H12B	112.2 (12)
C7—C6—H6	118.0 (14)	C11—C12—H12B	106.3 (13)
C2—C7—C6	118.97 (15)	H12A—C12—H12B	107.7 (17)
C2—C7—C8	118.61 (14)	C12—C13—H13A	113.6 (14)
C6—C7—C8	122.42 (16)	C12—C13—H13B	107.5 (15)
C9—C8—C7	120.86 (16)	H13A—C13—H13B	104 (2)
C9—C8—H8	120.5 (12)	C12—C13—H13C	114.5 (15)
C7—C8—H8	118.6 (12)	H13A—C13—H13C	108 (2)
C8—C9—O1	120.49 (15)	H13B—C13—H13C	109 (2)
C9—O1—C1—C2	0.4 (2)	C1—C2—C7—C8	0.2 (2)
C9—O1—C1—S1	−179.15 (11)	C5—C6—C7—C2	0.8 (3)
O1—C1—C2—C7	−0.6 (2)	C5—C6—C7—C8	−179.58 (17)
S1—C1—C2—C7	178.82 (12)	C2—C7—C8—C9	0.5 (2)
O1—C1—C2—C3	179.74 (14)	C6—C7—C8—C9	−179.20 (16)
S1—C1—C2—C3	−0.8 (2)	C7—C8—C9—O1	−0.8 (3)
C7—C2—C3—C4	−0.1 (3)	C7—C8—C9—C10	179.12 (16)
C1—C2—C3—C4	179.51 (16)	C1—O1—C9—C8	0.3 (2)
C2—C3—C4—C5	0.4 (3)	C1—O1—C9—C10	−179.55 (14)
C3—C4—C5—C6	−0.1 (3)	C8—C9—C10—C11	1.2 (3)
C4—C5—C6—C7	−0.5 (3)	O1—C9—C10—C11	−178.92 (14)
C3—C2—C7—C6	−0.4 (2)	C9—C10—C11—C12	178.94 (16)
C1—C2—C7—C6	179.91 (15)	C10—C11—C12—C13	−177.06 (19)
C3—C2—C7—C8	179.89 (14)		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C3—H3···S1	0.92 (2)	2.77 (2)	3.1621 (18)	106.7 (16)
C10—H10B···Cg[i]	0.98 (2)	2.98 (2)	3.813 (3)	144.1 (13)

Symmetry codes: (i) −*x*, −*y*+1, −*z*.

Fig. 1

Fig. 2