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2*H*-Chromene-2-thione

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.013 Å
R factor = 0.054
wR factor = 0.185
Data-to-parameter ratio = 8.1

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

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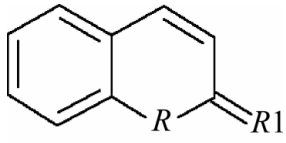
The crystal structure of the title compound, C_9H_6OS, also known as 2-thiocoumarin, has been determined. The molecule is essentially planar and crystals may display second harmonic generation (SHG) effects.

Comment

Coumarin has been identified as an important compound in the area of photochemistry, especially as a building block in the crystal lattice for photo-cycloaddition reactions (Vishnumurthy *et al.*, 2001). Coumarin derivatives are known to generate quality dyes, and are prominent in natural products chemistry (Dean, 1963; Murray *et al.*, 1982). They have been found to possess a wide variety of uses in the perfume industry, as flavour enhancers, sunscreens, laser dyes (Khalfan *et al.*, 1987), and in the pharmaceutical industry (Hooper *et al.*, 1982; Morris & Russell, 1971). Theoretical calculations using semi-empirical methods demonstrate that coumarin and its sulfur derivatives possess well defined residual dipole moments and the title compound, 2*H*-chromene-2-thione, (II), crystallizes in a non-centrosymmetric space group. These features make coumarin and its derivatives excellent candidates for organic SHG materials. We have synthesized a series of coumarins in this context (see Scheme below). Here we report the structure of (II). The molecular structure of (II) is illustrated in Fig. 1. Fig. 2 shows the packing of the molecules in the crystal lattice. Since the data were restricted to a unique octant of reflections only, the Flack (1983) parameter does not give a reliable indication of the absolute configuration.



(I) $R = R1 = O$
(II) $R = O$, $R1 = S$
(III) $R = S$, $R1 = O$
(IV) $R = R1 = S$

Experimental

The title compound was synthesized in a single step (Scheibye *et al.*, 1979). Compound (I) was converted to compound (II) with an equimolar amount of methoxyphenylthionophosphine sulfide (Lawesson's Reagent). Crystals were grown by slow evaporation at low temperature from ethanol as yellow needles (m.p. 371 K).

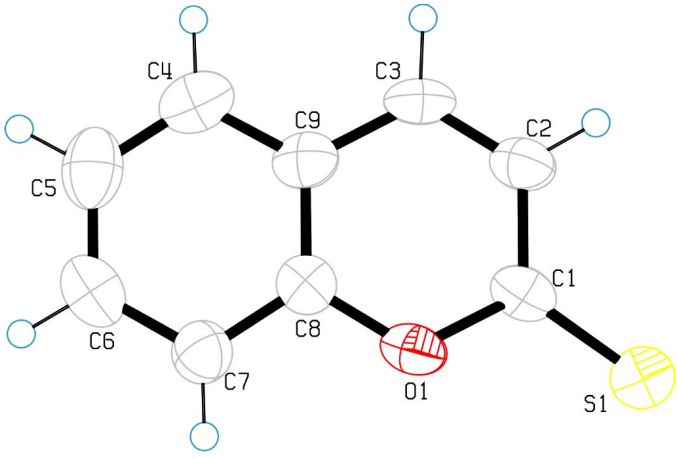


Figure 1
The molecular structure of (II), showing the atomic numbering. Displacement ellipsoids are drawn at the 50% probability level. H atoms are shown as spheres of arbitrary radii.

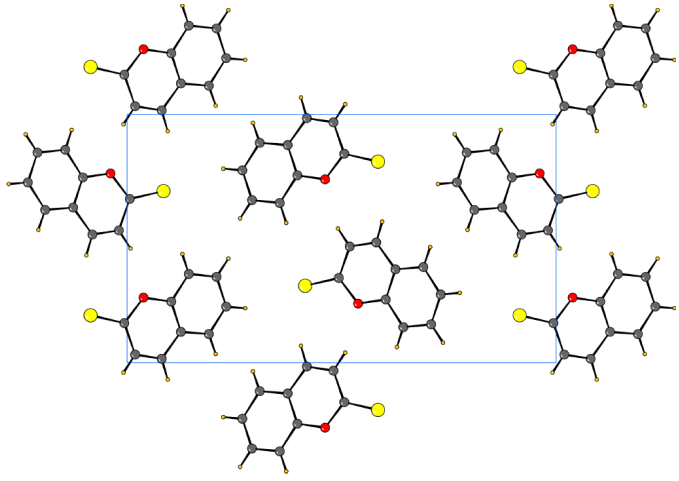


Figure 2
Packing diagram of the title compound, viewed down the *a* axis.

Crystal data

C_9H_6OS	D_m not measured
M_r = 162.21	Mo Kα radiation
Orthorhombic, $P2_12_12_1$	Cell parameters from 25 reflections
a = 4.126 (1) Å	θ = 6.1–13.3°
b = 17.779 (3) Å	μ = 0.36 mm^{-1}
c = 10.268 (3) Å	T = 293 (2) K
V = 753.2 (3) Å^3	Needle, yellow
Z = 4	0.38 × 0.05 × 0.04 mm
D_x = 1.431 Mg m^{-3}	

Data collection

Enraf–Nonius Turbo-CAD-4 diffractometer	θ_{max} = 25.0°
Non-profiled $\omega/2\theta$ scans	$h = 0 \rightarrow 4$
Absorption correction: none	$k = 0 \rightarrow 21$
815 measured reflections	$l = 0 \rightarrow 12$
815 independent reflections	3 standard reflections
375 reflections with $I > 2\sigma(I)$	frequency: 60 min
	intensity decay: 3%

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.1169P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)]$ = 0.054	$(\Delta/\sigma)_{max} < 0.001$
$wR(F^2)$ = 0.186	$\Delta\rho_{max}$ = 0.20 e Å^{-3}
S = 0.84	$\Delta\rho_{min}$ = −0.26 e Å^{-3}
815 reflections	Absolute structure: Flack (1983)
100 parameters	Flack parameter = 0.1 (5)
H-atom parameters constrained	

Data collection: *CAD-4 EXPRESS* (Enraf–Nonius, 1994); cell refinement: *CAD-4 EXPRESS*; data reduction: *XCAD*4 (Harms & Wocadlo, 1995); program(s) used to solve structure: *SIR*92 (Altomare *et al.*, 1993); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEP*3 for Windows (Farrugia, 1997); software used to prepare material for publication: *PLATON* (Spek, 1990).

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