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3-Cyano-4-[2-(4-methylthiophenyl)ethenyl]-2*H*-1-benzopyran-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 = 0.030
wR factor = 0.086
Data-to-parameter ratio = 8.0

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

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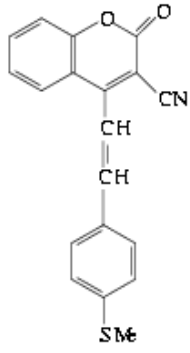
In the title compound, 4-[2-(4-methylthiophenyl)ethenyl]-2-oxo-2*H*-1-benzopyran-3-carbonitrile, $C_{19}H_{13}NO_2S$, the benzopyran and phenyl rings are individually planar, but the phenyl ring is twisted 56.7 (1)° out of the benzopyran ring plane. The configuration about the ethenyl double bond is *E*.

Comment

The structure determination of the title compound, (I), was taken up as part of our studies on coumarin derivatives which possess a variety of medicinal and biological properties (Parrish *et al.*, 1974; Evans *et al.*, 1981; Fujiwara *et al.*, 1978; Song & Gordon, 1970; Kawase *et al.*, 2001). It is of physiological interest that there is an apparent close chemical similarity between coumarin and vitamin K (Kralt & Claassen, 1972).



(I)

The molecular geometry of (I) is similar to that of 3-cyano-6-methyl-4-[2-(4-methoxyphenyl)ethenyl]-2*H*-1-benzopyran-2-one (Vijayalakshmi *et al.*, 2001). The bond lengths and angles in the coumarin moiety agree well with the reported values (Jha *et al.*, 2000; Chinnakali *et al.*, 1998, 1999; Vijayalakshmi *et al.*, 2000, 2001). The dihedral angle between the phenyl and planar benzopyran rings is 56.7 (1)°. The widening of the bond angle C11—C12—C18 to 126.8 (2)° is due to the close approach of the H11 and H17 atoms (2.256 Å). A similar feature is observed in the structures of cinnamanilides (Renganayaki *et al.*, 1999, 2000; Subramanian *et al.*, 1999) and dienethioamide (Nesterov *et al.*, 2000). Also, the slight increase in C4—C11—C12 to 122.2 (2)° is due to steric repulsion between H12 and C21 (H12···C21 2.629 Å). The Csp^2—S [1.755 (2) Å] and Csp^3—S [1.777 (4) Å] distances show partial double-bond character (Malhotra *et al.*, 1997; Azim *et al.*, 1997; Kumar *et al.*, 1999; Allen *et al.*, 1987). A C—H···N intermolecular short contact is observed

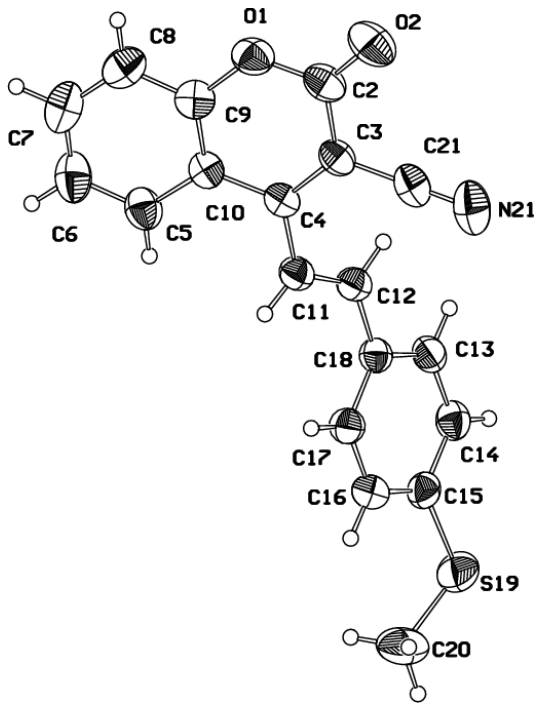


Figure 1
The molecular structure of (I) showing 50% probability displacement ellipsoids.

[C16···N21^{i} 3.337 (4) Å and H16···N21^{i} 2.56 Å; symmetry code: (i) x, $y+1$, z].

Experimental

A mixture of 3-cyano-4-methyl-2*H*-1-benzopyran-2-one (0.01 mol) and 4-methylthiobenzaldehyde (0.01 mol) was dissolved in chloroform (75 ml) and a few drops of piperidine (8–10 drops) was added as catalyst. The mixture was heated on a hotplate with stirring for 15–16 h. After evaporation of the solvent, the solid residue was recrystallized from dimethylformamide to give yellow crystals (m.p. 482 K; yield 64%).

Crystal data

$C_{19}H_{13}NO_2S$
M_r = 319.36
Orthorhombic, $P2_12_12_1$
a = 7.783 (3) Å
b = 7.8653 (10) Å
c = 25.610 (3) Å
V = 1567.7 (7) Å^3
Z = 4
D_x = 1.353 Mg m^{-3}
Cu $K\alpha$ radiation
Cell parameters from 25 reflections
θ = 2–25°
μ = 1.91 mm^{-1}
T = 293 (2) K
Needle, yellow
0.15 × 0.10 × 0.09 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
ω–2θ scans
Absorption correction: ψ scan (North *et al.*, 1968)
T_{min} = 0.784, T_{max} = 0.891
1676 measured reflections
1676 independent reflections
1606 reflections with $I > 2\sigma(I)$
θ_{max} = 67.9°
$h = 0 \rightarrow 9$
$k = 0 \rightarrow 9$
$l = 0 \rightarrow 30$
3 standard reflections
every 100 reflections
intensity decay: negligible

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.030
$wR(F^2)$ = 0.086
S = 1.06
1676 reflections
210 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0501P)^2 + 0.3363P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.18 e Å^{-3}
$\Delta\rho_{min}$ = −0.21 e Å^{-3}
Extinction correction: *SHELXL*97
Extinction coefficient: 0.0037 (4)

Table 1
Selected torsion angles (°).

C3—C4—C11—C12	−45.5 (3)	C16—C15—S19—C20	4.9 (3)
C10—C4—C11—C12	135.5 (2)	C14—C15—S19—C20	−175.5 (2)

All H atoms were fixed using geometrical considerations. The absolute configuration is indeterminate for the title compound.

Data collection: *CAD*-4 *Software* (Enraf–Nonius, 1989); cell refinement: *MolEN* (Fair, 1990); data reduction: *MolEN*; program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ZORTEP* (Zsolnai, 1997); software used to prepare material for publication: *SHELXL*97.

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