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3-[3-(4-Chlorophenyl)-4-oxothiazolidin-2-yl]-6-methyl-4*H*-benzopyran-4-one

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.003 Å
R factor = 0.044
wR factor = 0.134
Data-to-parameter ratio = 16.5

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

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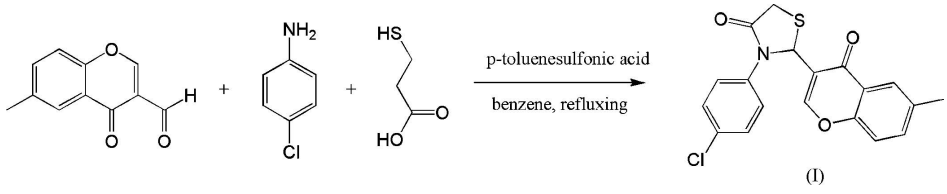
The molecular structure of the title compound, $C_{19}H_{14}ClNO_3S$, contains a 4-oxothiazolidine ring in a near planar geometry.

Comment

3-Substituted chromones are important because of their widespread occurrence in nature and their interesting biological activities. Meanwhile, thiazolidinones have been reported to possess a wide range of biological activities, including antifungal, antibacterial, antihistaminic, antimicrobial and anti-inflammatory activities. We are interested in heterocyclic compounds with a combination of thiazolidinonyl and chromonyl groups, in the hope of discovering novel lead structures for the development of antifungal and antimicrobial agents. The title compound, (I), is readily synthesized by condensing 6-methyl-4-oxo-4*H*-chromene-3-carbaldehyde with *p*-chloroaniline and mercaptoacetic acid (Fitton *et al.*, 1984).



The crystal structure (Fig. 1) shows that the benzene ring of the 4-chlorophenyl substituent is not coplanar with the thiazolidine ring, and the thiazolidine ring is also not coplanar

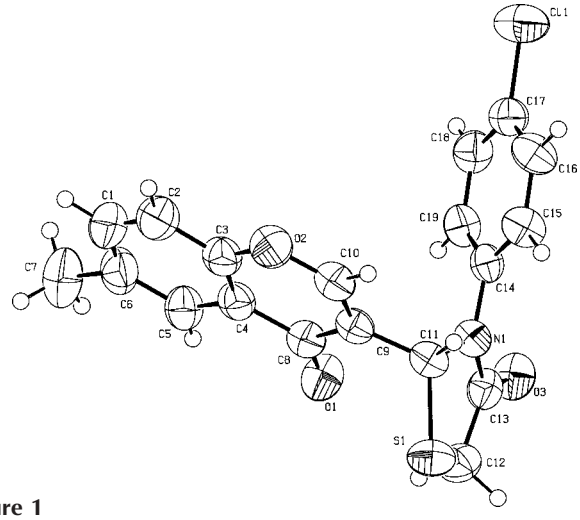


Figure 1
View of the molecule of (I), showing the atom-labeling scheme and 50% probability displacement ellipsoids.

with the pyran ring [the dihedral angles between the thiazolidine ring and pyran ring is 85.69 (4)°] so they cannot form a conjugated system in the solid state (Table 1).

Experimental

A mixture of 6-methyl-4-oxo-4*H*-chromene-3-carbaldehyde (5 mmol), 4-chlorobenzenamine (5 mmol) and *p*-toluenesulfonic acid (10 mg, 0.058 mmol) was refluxed and stirred in benzene (40 ml) for 1 h, and then mercaptoacetic acid (25 mmol) was added. The reactant was refluxed for another 8 h. The solvent was removed *in vacuo*. The resulting residue was washed with ethanol to afford the crude product, which was then recrystallized from ethanol to give colorless crystals. ^{1}H NMR (400 MHz, $CDCl_3$): δ 2.45 (*s*, 3H), 3.77 (*d*, J = 15.6 Hz, 1H), 4.11 (*dd*, J = 15.6 Hz, J = 1.4 Hz, 1H), 6.08 (*s*, 1H), 7.25–7.32 (*m*, 5H), 7.47 (*dd*, J = 8.6 Hz, J = 1.8 Hz, 1H), 7.74 (*s*, 1H), 7.99 (*s*, 1H); MS (EI, *m*/*z*): 373 (M^+ + 1), 372 (M^+); elemental analysis calculated for $C_{19}H_{14}ClNO_3S$: C 61.37, H 3.79, N 3.77%; found: C 61.46, H 3.78, N 3.71%.

Crystal data

$C_{19}H_{14}ClNO_3S$
M_r = 371.82
Monoclinic, $P2_1/n$
a = 11.1830 (9) Å
b = 13.9851 (10) Å
c = 12.3419 (9) Å
β = 116.445 (1)°
V = 1728.2 (2) Å^3
Z = 4
D_x = 1.429 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 3801 reflections
θ = 2.4–27.7°
μ = 0.36 mm^{-1}
T = 293 (2) K
Block, colorless
0.40 × 0.38 × 0.34 mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1997)
T_{min} = 0.870, T_{max} = 0.888
9946 measured reflections
3747 independent reflections
2974 reflections with $I > 2\sigma(I)$
R_{int} = 0.018
θ_{max} = 27.0°
$h = -13 \rightarrow 14$
$k = -17 \rightarrow 13$
$l = -14 \rightarrow 15$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.044
$wR(F^2)$ = 0.134
S = 1.05
3747 reflections
227 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0722P)^2 + 0.3678P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.31 e Å^{-3}
$\Delta\rho_{min}$ = −0.20 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

C3—O2	1.382 (3)	C11—N1	1.473 (2)
C8—O1	1.225 (2)	C11—S1	1.8197 (18)
C8—C9	1.463 (3)	C12—S1	1.803 (2)
C9—C10	1.336 (3)	C13—O3	1.219 (2)
C9—C11	1.505 (3)	C13—N1	1.354 (2)
C10—O2	1.344 (3)	C14—N1	1.425 (2)
N1—C11—C9	111.39 (14)	C13—N1—C14	124.09 (15)
N1—C11—S1	104.79 (12)	C13—N1—C11	118.00 (15)
C9—C11—S1	115.60 (13)	C14—N1—C11	116.83 (13)
C13—C12—S1	107.61 (13)	C10—O2—C3	117.83 (15)
N1—C13—C12	112.55 (17)	C12—S1—C11	93.10 (9)

All H atoms were initially located in a difference Fourier map. The methyl H atoms were then constrained to an ideal geometry, with C—H distances of 0.96 Å and U_{iso}(H) = 1.5U_{eq}(C), but each group was allowed to rotate freely about its C—C bond. All other H atoms were placed in geometrically idealized positions and constrained to ride on their parent atoms, with C—H distances in the range 0.93–0.98 Å and with U_{iso}(H) = 1.2U_{eq}(C).

Data collection: *SMART* (Bruker, 2003); cell refinement: *SAINT* (Bruker, 2003); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997*a*); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997*a*); molecular graphics: *SHELXTL* (Sheldrick, 1997*b*); software used to prepare material for publication: *SHELXTL*.

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