

## 2-(2-Chlorophenyl)-7-methyl-4H-chromen-4-one

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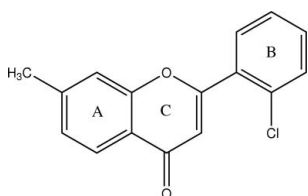
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Key indicators: single-crystal X-ray study;  $T = 298$  K; mean  $\sigma(\text{C}-\text{C}) = 0.005$  Å;  
 $R$  factor = 0.045;  $wR$  factor = 0.128; data-to-parameter ratio = 12.9.

The title compound,  $\text{C}_{16}\text{H}_{11}\text{ClO}_2$ , is a synthetic flavonoid. The fused-ring system is almost planar, with a mean deviation from the least-squares plane of  $0.0204$  (2) Å. The dihedral angle between the chromene group and the chlorophenyl ring is  $50.9$  (6)°, due to unfavourable steric interactions with the Cl atom. Aromatic  $\pi$ - $\pi$  stacking interactions between the fused benzene ( $\pi$ -rich) and pyran ( $\pi$ -deficient) rings are observed, with a centroid-centroid distance of  $3.578$  Å.

### Related literature

The  $\pi$ - $\pi$  stacking interaction observed in the title compound is a normal interaction found in flavonoids (see Jiang *et al.*, 2002; Etti *et al.*, 2005; Fun *et al.*, 2005; Stomberg *et al.*, 2002).



### Experimental

#### Crystal data

$\text{C}_{16}\text{H}_{11}\text{ClO}_2$   
 $M_r = 270.70$

Monoclinic,  $P2_1/n$   
 $a = 7.8394$  (10) Å

$b = 22.936$  (3) Å  
 $c = 7.8790$  (9) Å  
 $\beta = 116.462$  (2)°  
 $V = 1268.2$  (3) Å<sup>3</sup>  
 $Z = 4$

Mo  $K\alpha$  radiation  
 $\mu = 0.30$  mm<sup>-1</sup>  
 $T = 298$  (2) K  
 $0.49 \times 0.45 \times 0.28$  mm

#### Data collection

Bruker SMART-1000 CCD  
area-detector diffractometer  
Absorption correction: multi-scan  
(*SADABS*; Bruker, 2000)  
 $T_{\min} = 0.869$ ,  $T_{\max} = 0.922$

6274 measured reflections  
2231 independent reflections  
1362 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.035$

#### Refinement

$R[F^2 > 2\sigma(F^2)] = 0.045$   
 $wR(F^2) = 0.128$   
 $S = 1.01$   
2231 reflections

173 parameters  
H-atom parameters constrained  
 $\Delta\rho_{\max} = 0.20$  e Å<sup>-3</sup>  
 $\Delta\rho_{\min} = -0.22$  e Å<sup>-3</sup>

Data collection: *SMART* (Bruker, 2000); cell refinement: *SAINT* (Bruker, 2000); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 2000); software used to prepare material for publication: *SHELXTL* and *WinGX* (Farrugia, 1999).

The use of the Bruker X-ray Crystallographic Services at Liaocheng University and the valuable assistance from Professor Daqi Wang are gratefully acknowledged.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: FJ2043).

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**supplementary materials**

*Acta Cryst.* (2007). E63, o4340 [ doi:10.1107/S1600536807050155 ]

## 2-(2-Chlorophenyl)-7-methyl-4H-chromen-4-one

X.-B. Wang and L.-Y. Kong

### Comment

The title compound composed of a benzopyran ring system including ring A(C4—C9) and C(O1/C1—C4/C9), a phenyl ring B(C10—C15), a methyl group attached to ring A and a nitro group attached to ring B. The bond lengths and angles show normal values. The chromen including rings A and C is almost planar with mean deviation from their least square plane being 0.0204 (2) Å. A dihedral angle being 50.9 (6)° existed between the chromen part and ring B for the spatial conflict of the Cl atom.

It seems like that the  $\pi$ - $\pi$  stacking interaction is a normal interaction found in flavonoid (Jiang *et al.*, 2002; Etti, *et al.*, 2005; Fun, *et al.*, 2005; Stomberg, *et al.*, 2002). Fig. 2 shows the intermolecular stacking *via*  $\pi$ - $\pi$  stacking. In each molecule, ring A ( $\pi$ -rich) and C ( $\pi$ -deficient) was partial overlapped with ring C ( $\pi$ -deficient) and A ( $\pi$ -rich) from the other molecule at (2 - x, 1 - y, 2 - z), respectively, with intercentroid distances both of 3.578 Å (CgA and CgC represents the centroids of rings B and C) indicating that a strong  $\pi$ - $\pi$  stacking interaction exists in the title compound.

### Experimental

A mixture of 1-(2-hydroxy-5-methylphenyl)ethanone (3.0 g, 20 mmol) and oven-dried potassium carbonate (11.8 g, 90 mmol) in dry acetone was stirred with refluxing for 10 min. *o*-Chlorobenzoyl chloride (3.5 g, 20 mmol) was added to the reaction mixture, and the mixture was stirred for 8 h. The precipitate was acidified to pH 7.0~8.0 with 10% HOAc, and the solid was filtered to give 1-(2-hydroxy-5-methylphenyl)-3-(2-chlorophenyl)propane-1, 3-dione (A) 4.7 g, yield 82.3%. A (4.7 g) was mixed with HOAc (30 ml)-NaOAc (6 g) solution, and the mixture was stirred under refluxing for 6 h. The mixture was cooled to room temperature and water was added. The solid was filtered and washed with warm water, 5% NaHCO<sub>3</sub> solution and water, respectively, giving the title compound 3.8 g, yield 86.1%. ESI-MS:  $[M+H]^+ = 271$ . Prismatic crystals suitable for X-ray studies were grown from CH<sub>2</sub>Cl<sub>2</sub> by slow evaporation at room temperature.

### Refinement

The methyl H atoms were constrained to an ideal geometry with C—H = 0.96 Å and  $U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{C})$ , but were allowed to rotate freely about the C—C bonds. All remaining H atoms were placed in geometrically idealized positions (C—H = 0.93–0.97 Å) and constrained to ride on their parent atoms with  $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$ .

## Figures

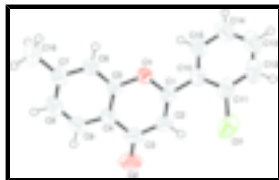


Fig. 1. A view of the asymmetric unit of the title compound, showing the atom labeling scheme. Displacement ellipsoids are drawn at the 50% probability level. H-atoms are shown as spheres of arbitrary radii.

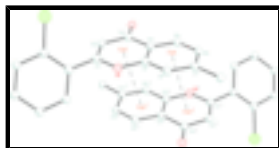


Fig. 2. Part of the crystal structure of (I), showing the  $\pi$ - $\pi$  stacking. Atoms marked with a (i) are at the symmetry positions  $(2-x, 1-y, 2-z)$ . CgA and CgC are the centroids of rings A and C.

## 2-(2-Chlorophenyl)-7-methyl-4H-chromen-4-one

### Crystal data

$C_{16}H_{11}ClO_2$

$M_r = 270.70$

Monoclinic,  $P2_1/n$

Hall symbol: -P 2yn

$a = 7.8394$  (10) Å

$b = 22.936$  (3) Å

$c = 7.8790$  (9) Å

$\beta = 116.462$  (2)°

$V = 1268.2$  (3) Å<sup>3</sup>

$Z = 4$

$F_{000} = 560$

$D_x = 1.418$  Mg m<sup>-3</sup>

Mo  $K\alpha$  radiation

$\lambda = 0.71073$  Å

Cell parameters from 1659 reflections

$\theta = 2.7$ – $23.0$ °

$\mu = 0.30$  mm<sup>-1</sup>

$T = 298$  (2) K

Prism, colourless

$0.49 \times 0.45 \times 0.28$  mm

### Data collection

Bruker SMART-1000 CCD area-detector diffractometer

Radiation source: fine-focus sealed tube

Monochromator: graphite

$T = 298$ (2) K

$\pi$  and  $\omega$  scans

Absorption correction: multi-scan (SADABS; Bruker, 2000)

$T_{\min} = 0.869$ ,  $T_{\max} = 0.922$

6274 measured reflections

2231 independent reflections

1362 reflections with  $I > 2\sigma(I)$

$R_{\text{int}} = 0.035$

$\theta_{\max} = 25.0$ °

$\theta_{\min} = 1.8$ °

$h = -9 \rightarrow 9$

$k = -27 \rightarrow 18$

$l = -9 \rightarrow 9$

### Refinement

Refinement on  $F^2$

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.045$

Secondary atom site location: difference Fourier map

Hydrogen site location: inferred from neighbouring sites

H-atom parameters constrained

$wR(F^2) = 0.128$	$w = 1/[\sigma^2(F_o^2) + (0.0477P)^2 + 0.646P]$
$S = 1.02$	where $P = (F_o^2 + 2F_c^2)/3$
2231 reflections	$(\Delta/\sigma)_{\max} < 0.001$
173 parameters	$\Delta\rho_{\max} = 0.20 \text{ e } \text{\AA}^{-3}$
Primary atom site location: structure-invariant direct methods	$\Delta\rho_{\min} = -0.22 \text{ e } \text{\AA}^{-3}$
	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  $F^2 > 2\sigma(F^2)$  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 ( $\text{\AA}^2$ )*

	$x$	$y$	$z$	$U_{\text{iso}}^*/U_{\text{eq}}$
C11	0.97339 (14)	0.22512 (4)	0.89359 (15)	0.0847 (4)
O1	0.8098 (3)	0.39353 (7)	0.9756 (3)	0.0480 (5)
O2	1.1790 (4)	0.42147 (11)	0.7482 (4)	0.0909 (8)
C1	0.9519 (4)	0.35489 (11)	1.0026 (4)	0.0460 (7)
C2	1.0726 (4)	0.36287 (13)	0.9272 (4)	0.0580 (8)
H2	1.1649	0.3346	0.9476	0.070*
C3	1.0661 (5)	0.41337 (14)	0.8157 (4)	0.0607 (9)
C4	0.9143 (4)	0.45480 (12)	0.7903 (4)	0.0489 (7)
C5	0.7900 (4)	0.44250 (11)	0.8673 (4)	0.0445 (7)
C6	0.6394 (4)	0.47863 (12)	0.8413 (4)	0.0498 (7)
H6	0.5569	0.4685	0.8923	0.060*
C7	0.6113 (4)	0.52959 (12)	0.7399 (4)	0.0547 (8)
C8	0.7362 (5)	0.54276 (14)	0.6632 (4)	0.0632 (9)
H8	0.7188	0.5770	0.5943	0.076*
C9	0.8835 (5)	0.50654 (14)	0.6869 (4)	0.0623 (9)
H9	0.9642	0.5164	0.6336	0.075*
C10	0.9574 (4)	0.30831 (12)	1.1331 (4)	0.0498 (7)
C11	0.9700 (4)	0.24929 (13)	1.1007 (5)	0.0593 (8)
C12	0.9784 (4)	0.20779 (15)	1.2319 (7)	0.0752 (11)
H12	0.9865	0.1684	1.2081	0.090*
C13	0.9748 (5)	0.22476 (18)	1.3959 (6)	0.0776 (11)
H13	0.9807	0.1968	1.4839	0.093*
C14	0.9626 (5)	0.28284 (17)	1.4327 (5)	0.0749 (10)
H14	0.9620	0.2941	1.5458	0.090*
C15	0.9513 (4)	0.32395 (15)	1.3025 (5)	0.0617 (8)

## supplementary materials

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H15	0.9393	0.3630	1.3267	0.074*
C16	0.4510 (5)	0.56963 (14)	0.7152 (5)	0.0734 (10)
H16A	0.3497	0.5475	0.7208	0.110*
H16B	0.4049	0.5888	0.5944	0.110*
H16C	0.4957	0.5983	0.8144	0.110*

### Atomic displacement parameters ( $\text{\AA}^2$ )

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
C11	0.0859 (7)	0.0583 (5)	0.1092 (8)	0.0015 (4)	0.0427 (6)	-0.0188 (5)
O1	0.0508 (12)	0.0408 (11)	0.0590 (12)	0.0009 (9)	0.0304 (10)	0.0048 (9)
O2	0.109 (2)	0.0878 (18)	0.122 (2)	-0.0074 (14)	0.0931 (19)	-0.0010 (15)
C1	0.0444 (17)	0.0404 (15)	0.0534 (17)	-0.0043 (12)	0.0219 (15)	-0.0046 (13)
C2	0.0584 (19)	0.0502 (18)	0.079 (2)	-0.0035 (14)	0.0433 (19)	-0.0064 (16)
C3	0.074 (2)	0.0588 (19)	0.065 (2)	-0.0183 (17)	0.0460 (19)	-0.0149 (16)
C4	0.0592 (18)	0.0465 (17)	0.0469 (17)	-0.0145 (14)	0.0290 (15)	-0.0096 (14)
C5	0.0544 (18)	0.0373 (15)	0.0414 (16)	-0.0103 (13)	0.0210 (14)	-0.0032 (12)
C6	0.0577 (19)	0.0441 (17)	0.0513 (17)	-0.0028 (14)	0.0276 (15)	0.0010 (14)
C7	0.065 (2)	0.0419 (17)	0.0474 (17)	-0.0053 (14)	0.0160 (16)	-0.0036 (14)
C8	0.086 (3)	0.0483 (18)	0.0516 (19)	-0.0162 (18)	0.0271 (19)	0.0019 (15)
C9	0.084 (2)	0.058 (2)	0.0543 (19)	-0.0199 (18)	0.0392 (19)	-0.0043 (16)
C10	0.0362 (16)	0.0457 (17)	0.065 (2)	-0.0019 (12)	0.0204 (15)	0.0047 (15)
C11	0.0372 (17)	0.0475 (18)	0.084 (2)	0.0019 (13)	0.0190 (17)	0.0054 (17)
C12	0.045 (2)	0.053 (2)	0.113 (3)	0.0057 (15)	0.022 (2)	0.020 (2)
C13	0.051 (2)	0.079 (3)	0.097 (3)	0.0005 (18)	0.028 (2)	0.039 (2)
C14	0.061 (2)	0.089 (3)	0.078 (3)	-0.0028 (19)	0.034 (2)	0.023 (2)
C15	0.0525 (19)	0.062 (2)	0.074 (2)	0.0002 (15)	0.0304 (18)	0.0128 (18)
C16	0.080 (3)	0.054 (2)	0.070 (2)	0.0079 (17)	0.018 (2)	0.0111 (17)

### Geometric parameters ( $\text{\AA}$ , $^\circ$ )

C11—C11	1.735 (4)	C8—C9	1.366 (4)
O1—C1	1.364 (3)	C8—H8	0.9300
O1—C5	1.377 (3)	C9—H9	0.9300
O2—C3	1.233 (3)	C10—C11	1.389 (4)
C1—C2	1.336 (4)	C10—C15	1.404 (4)
C1—C10	1.470 (4)	C11—C12	1.385 (5)
C2—C3	1.441 (4)	C12—C13	1.362 (5)
C2—H2	0.9300	C12—H12	0.9300
C3—C4	1.466 (4)	C13—C14	1.375 (5)
C4—C5	1.387 (4)	C13—H13	0.9300
C4—C9	1.398 (4)	C14—C15	1.366 (4)
C5—C6	1.381 (4)	C14—H14	0.9300
C6—C7	1.377 (4)	C15—H15	0.9300
C6—H6	0.9300	C16—H16A	0.9600
C7—C8	1.393 (4)	C16—H16B	0.9600
C7—C16	1.498 (4)	C16—H16C	0.9600
C1—O1—C5	119.1 (2)	C8—C9—H9	119.6

C2—C1—O1	122.2 (3)	C4—C9—H9	119.6
C2—C1—C10	127.2 (3)	C11—C10—C15	117.4 (3)
O1—C1—C10	110.4 (2)	C11—C10—C1	124.2 (3)
C1—C2—C3	122.8 (3)	C15—C10—C1	118.5 (3)
C1—C2—H2	118.6	C12—C11—C10	121.1 (3)
C3—C2—H2	118.6	C12—C11—C11	117.8 (3)
O2—C3—C2	123.2 (3)	C10—C11—C11	121.1 (3)
O2—C3—C4	122.6 (3)	C13—C12—C11	119.8 (3)
C2—C3—C4	114.2 (2)	C13—C12—H12	120.1
C5—C4—C9	117.1 (3)	C11—C12—H12	120.1
C5—C4—C3	119.6 (3)	C12—C13—C14	120.6 (3)
C9—C4—C3	123.3 (3)	C12—C13—H13	119.7
O1—C5—C6	115.8 (2)	C14—C13—H13	119.7
O1—C5—C4	122.0 (3)	C15—C14—C13	119.9 (4)
C6—C5—C4	122.3 (3)	C15—C14—H14	120.1
C7—C6—C5	120.0 (3)	C13—C14—H14	120.1
C7—C6—H6	120.0	C14—C15—C10	121.3 (3)
C5—C6—H6	120.0	C14—C15—H15	119.4
C6—C7—C8	118.3 (3)	C10—C15—H15	119.4
C6—C7—C16	120.3 (3)	C7—C16—H16A	109.5
C8—C7—C16	121.4 (3)	C7—C16—H16B	109.5
C9—C8—C7	121.6 (3)	H16A—C16—H16B	109.5
C9—C8—H8	119.2	C7—C16—H16C	109.5
C7—C8—H8	119.2	H16A—C16—H16C	109.5
C8—C9—C4	120.8 (3)	H16B—C16—H16C	109.5
C5—O1—C1—C2	-0.1 (4)	C6—C7—C8—C9	-0.1 (5)
C5—O1—C1—C10	175.8 (2)	C16—C7—C8—C9	179.5 (3)
O1—C1—C2—C3	1.6 (5)	C7—C8—C9—C4	-0.2 (5)
C10—C1—C2—C3	-173.5 (3)	C5—C4—C9—C8	-0.3 (4)
C1—C2—C3—O2	178.8 (3)	C3—C4—C9—C8	178.1 (3)
C1—C2—C3—C4	-0.7 (4)	C2—C1—C10—C11	-51.4 (4)
O2—C3—C4—C5	178.9 (3)	O1—C1—C10—C11	133.0 (3)
C2—C3—C4—C5	-1.6 (4)	C2—C1—C10—C15	128.0 (3)
O2—C3—C4—C9	0.5 (5)	O1—C1—C10—C15	-47.6 (3)
C2—C3—C4—C9	180.0 (3)	C15—C10—C11—C12	-0.7 (4)
C1—O1—C5—C6	178.0 (2)	C1—C10—C11—C12	178.7 (3)
C1—O1—C5—C4	-2.3 (4)	C15—C10—C11—C11	178.6 (2)
C9—C4—C5—O1	-178.3 (2)	C1—C10—C11—C11	-2.0 (4)
C3—C4—C5—O1	3.2 (4)	C10—C11—C12—C13	-0.2 (5)
C9—C4—C5—C6	1.3 (4)	C11—C11—C12—C13	-179.5 (3)
C3—C4—C5—C6	-177.2 (3)	C11—C12—C13—C14	0.1 (5)
O1—C5—C6—C7	178.0 (2)	C12—C13—C14—C15	0.9 (5)
C4—C5—C6—C7	-1.6 (4)	C13—C14—C15—C10	-1.8 (5)
C5—C6—C7—C8	1.0 (4)	C11—C10—C15—C14	1.7 (4)
C5—C6—C7—C16	-178.6 (3)	C1—C10—C15—C14	-177.7 (3)

Fig. 1

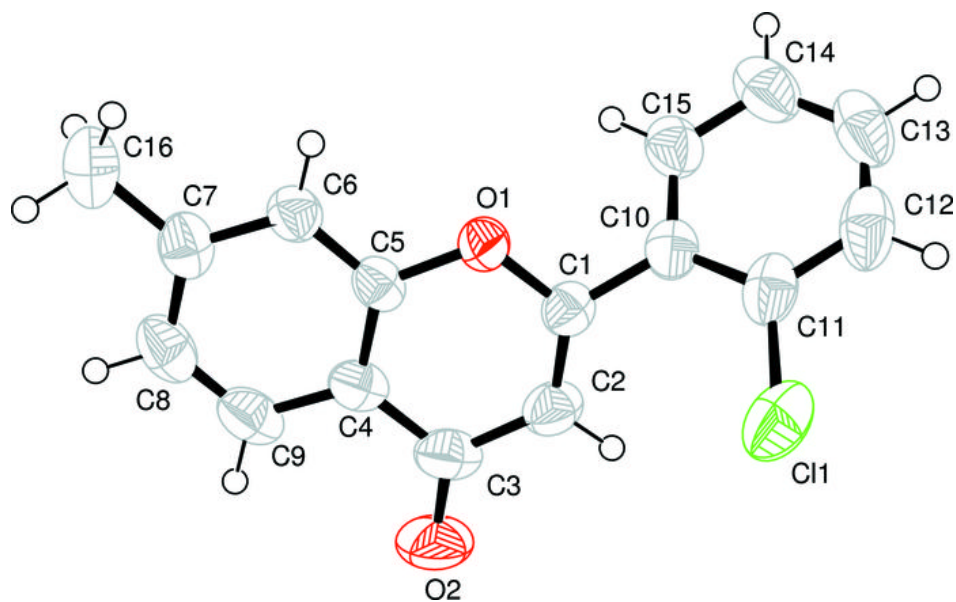




Fig. 2

