organic compounds

6-Methyl-4-oxo-4*H*-chromene-3-carbaldehyde

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In the title compound, $C_{11}H_8O_3$, the benzopyran-4-one or chromone ring system is almost planar, with a maximum deviation of 0.045 (2) Å. The crystal structure is stablized by π–π interactions between the benzene and pyran rings of inversion-related molecules stacked along the *b* axis, with a centroid–centroid distance of 3.5463 (12) Å.

Related literature

For the biological activity of chromone, see: Patel *et al.* (2011); Khan *et al.* (2009, 2010); Gautam *et al.* (2010); Ishar *et al.* (2006); Hassan (1992); Nohara *et al.* (1974). For a related structure, see: Wang & Kong (2007).
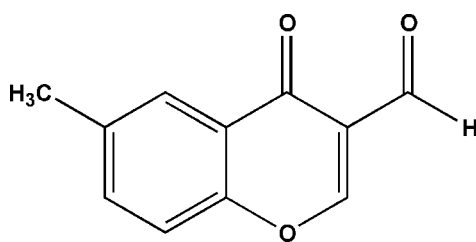
Experimental

Crystal data

$C_{11}H_8O_3$	Triclinic, $P\bar{1}$
$M_r = 188.17$	$a = 6.6945$ (7) Å
$b = 7.1079$ (7) Å	$Z = 2$
$c = 10.3032$ (11) Å	Mo $K\alpha$ radiation
$\alpha = 71.593$ (2)°	$\mu = 0.11$ mm^{-1}
$\beta = 84.962$ (2)°	$T = 273$ K
$\gamma = 69.843$ (2)°	$0.26 \times 0.23 \times 0.11$ mm
$V = 436.57$ (8) Å^3	

Data collection

Bruker SMART APEX CCD area-detector diffractometer	4974 measured reflections
Absorption correction: multi-scan (*SADABS*; Bruker, 2000)	1629 independent reflections
$T_{min} = 0.973$, $T_{max} = 0.989$	1300 reflections with $I > 2\sigma(I)$
	$R_{int} = 0.019$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.049$	128 parameters
$wR(F^2) = 0.152$	H-atom parameters constrained
$S = 1.07$	$\Delta\rho_{max} = 0.26$ e Å^{-3}
1629 reflections	$\Delta\rho_{min} = -0.19$ e Å^{-3}

Data collection: *SMART* (Bruker, 2000); cell refinement: *SAINT* (Bruker, 2000); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXTL*, *PARST* (Nardelli, 1995) and *PLATON* (Spek, 2009).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: RZ2798).

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supplementary materials

6-Methyl-4-oxo-4*H*-chromene-3-carbaldehyde

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Comment

Chromone is a heterocyclic compound containing a benzene ring fused with a pyran ring, so it is also called as benzo-pyran-4-one. Chromone moieties are associated with various physiological and biological properties such as antibacterial (Patel *et al.*, 2011), antioxidant (Gautam *et al.*, 2010; Hassan *et al.*, 1992), antianaphylactic (Nohara *et al.*, 1974), antiinflammatory (Khan *et al.*, 2010), anticancer (Ishar *et al.*, 2006), and thymidine phosphorylase inhibitor (Khan *et al.*, 2009) activities. The title compound is a chromone derivative synthesized as a part of our ongoing research to study different biological activities of this medicinally important class of organic compounds and establish their structure–activity relationship.

The structure of title compound (Fig. 1) is composed of a planar chromone moiety (O1/C1–C9) with maximum deviation of 0.045 (2) Å for atom C8. Bond lengths and angles are similar to those observed in a structurally related compound (Wang & Kong, 2007). In the crystal (Fig. 2), inversion-related molecules are linked along the *b* axis by significant π–π stacking interactions occurring between benzene and pyran rings of chromone moeities, with centroid-centroid distances of 3.5463 (12) Å.

Experimental

The title compound was synthesized by taking dry dimethylformamide (12.32 ml) into a three necked flask followed by slow addition of $POCl_3$ (49 mmol) with intensive stirring at 50°C. Heating and stirring was continued for 2 h at 45–55°C. A solution of 5-methyl-2-hydroxyacetophenone (10 mmol) in DMF was then slowly added under stirring at 50°C. The stirring was continued for additional 2 h at 55–60°C. After cooling, the mixture was kept over night at room temperature and diluted slowly by adding crushed ice (300 g) and stirred again for 6 h to obtain the crude product. Recrystallization from ethanol afforded crystals in 78.7% yield (1.48 g) which were found suitable for single-crystal X-ray diffraction studies. All chemicals were purchased by sigma Aldrich Germany.

Refinement

H atoms were positioned geometrically with C—H = 0.93–0.95 Å and constrained to ride on their parent atoms with $U_{iso}(H) = 1.5U_{eq}(CH_3)$ or $1.2U_{eq}(CH)$.

Computing details

Data collection: *SMART* (Bruker, 2000); cell refinement: *SAINT* (Bruker, 2000); data reduction: *SAINT* (Bruker, 2000); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXTL* (Sheldrick, 2008), *PARST* (Nardelli, 1995) and *PLATON* (Spek, 2009).
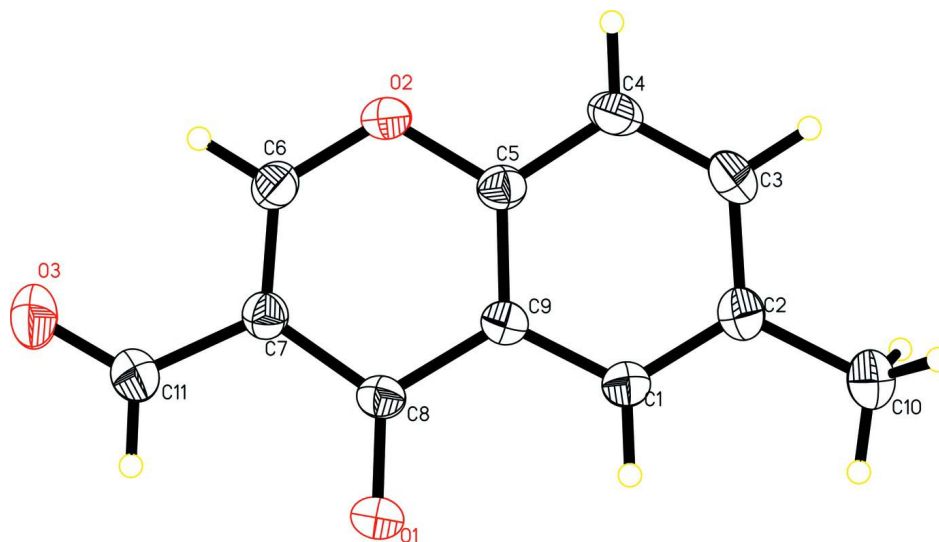
Figure 1

The molecular structure of the title compound with displacement ellipsoids drawn at the 30% probability level.
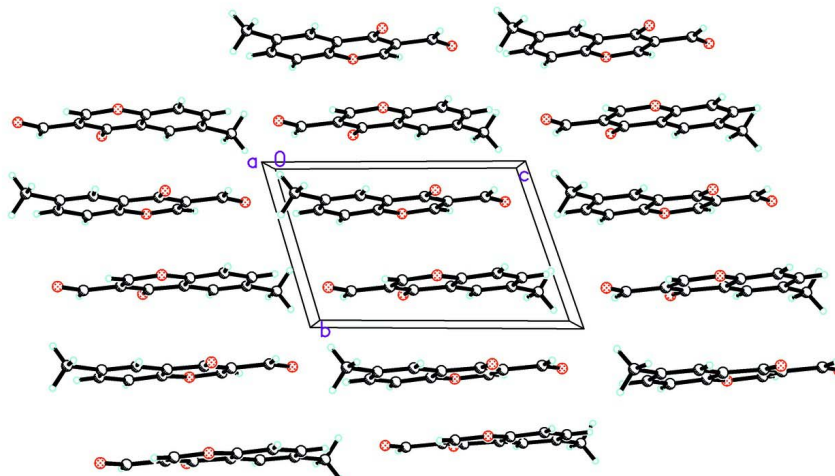
Figure 2

The crystal packing of the title compound viewed along the *a* axis.

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Crystal data

$C_{11}H_8O_3$	$\gamma = 69.843\ (2)°$
$M_r = 188.17$	$V = 436.57\ (8)$ Å^3
Triclinic, $P\overline{1}$	$Z = 2$
Hall symbol: -P 1	$F(000) = 196$
$a = 6.6945\ (7)$ Å	$D_x = 1.431$ Mg m^{-3}
$b = 7.1079\ (7)$ Å	Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
$c = 10.3032\ (11)$ Å	Cell parameters from 1636 reflections
$\alpha = 71.593\ (2)°$	$\theta = 3.2–28.1°$
$\beta = 84.962\ (2)°$	$\mu = 0.11$ mm^{-1}

$T = 273$ K

$0.26 \times 0.23 \times 0.11$ mm

Block, colorles

Data collection

Bruker SMART APEX CCD area-detector diffractometer	4974 measured reflections
Radiation source: fine-focus sealed tube	1629 independent reflections
Graphite monochromator	1300 reflections with $I > 2\sigma(I)$
ω scan	$R_{int} = 0.019$
Absorption correction: multi-scan (*SADABS*; Bruker, 2000)	$\theta_{max} = 25.5°$, $\theta_{min} = 2.1°$
	$h = -8 \rightarrow 8$
	$k = -8 \rightarrow 8$
$T_{min} = 0.973$, $T_{max} = 0.989$	$l = -12 \rightarrow 12$

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)] = 0.049$	$w = 1/[\sigma^2(F_o^2) + (0.0852P)^2 + 0.0886P]$
$wR(F^2) = 0.152$	where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.07$	$(\Delta/\sigma)_{max} < 0.001$
1629 reflections	$\Delta\rho_{max} = 0.26$ e Å^{-3}
128 parameters	$\Delta\rho_{min} = -0.19$ e Å^{-3}
0 restraints	Extinction correction: *SHELXTL* (Sheldrick, 2008), Fc*=kFc[1+0.001xFc$^2\lambda^3$/sin(2θ)]$^{-1/4}$
Primary atom site location: structure-invariant direct methods	Extinction coefficient: 0.013 (9)
Secondary atom site location: difference Fourier map	

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 $F^2 > \sigma(F^2)$ 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}
O1	0.2834 (2)	0.1857 (2)	0.62203 (13)	0.0573 (4)
O2	−0.30931 (18)	0.3149 (2)	0.47149 (12)	0.0476 (4)
O3	−0.2182 (3)	0.2434 (3)	0.87960 (15)	0.0749 (5)
C1	0.2333 (3)	0.2150 (3)	0.34035 (18)	0.0415 (4)
H1A	0.3724	0.1795	0.3703	0.050*
C2	0.1952 (3)	0.2394 (3)	0.20506 (19)	0.0455 (5)
C3	−0.0162 (3)	0.2971 (3)	0.16259 (19)	0.0497 (5)
H3A	−0.0452	0.3169	0.0717	0.060*
C4	−0.1816 (3)	0.3252 (3)	0.25048 (19)	0.0494 (5)
H4A	−0.3210	0.3642	0.2199	0.059*
C5	−0.1371 (3)	0.2942 (3)	0.38609 (18)	0.0397 (4)
C6	−0.2734 (3)	0.2855 (3)	0.60304 (19)	0.0443 (5)
H6A	−0.3894	0.2953	0.6604	0.053*

C7	−0.0835 (3)	0.2431 (3)	0.65872 (18)	0.0396 (4)
C8	0.1067 (3)	0.2197 (3)	0.57661 (18)	0.0386 (4)
C9	0.0686 (3)	0.2422 (2)	0.43295 (17)	0.0360 (4)
C10	0.3734 (4)	0.2037 (4)	0.1063 (2)	0.0654 (6)
H10A	0.5069	0.1645	0.1524	0.098*
H10B	0.3537	0.3308	0.0316	0.098*
H10C	0.3733	0.0931	0.0717	0.098*
C11	−0.0697 (3)	0.2176 (3)	0.8057 (2)	0.0548 (5)
H11A	0.0649	0.1784	0.8440	0.066*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0400 (8)	0.0876 (10)	0.0504 (8)	−0.0238 (7)	−0.0042 (6)	−0.0252 (7)
O2	0.0330 (7)	0.0611 (8)	0.0441 (7)	−0.0130 (6)	−0.0026 (5)	−0.0121 (6)
O3	0.0733 (11)	0.1117 (13)	0.0537 (9)	−0.0380 (10)	0.0201 (8)	−0.0413 (9)
C1	0.0385 (9)	0.0444 (10)	0.0434 (10)	−0.0157 (8)	0.0003 (7)	−0.0138 (8)
C2	0.0522 (11)	0.0442 (10)	0.0401 (10)	−0.0176 (8)	0.0025 (8)	−0.0117 (8)
C3	0.0609 (12)	0.0530 (11)	0.0348 (9)	−0.0209 (9)	−0.0073 (8)	−0.0087 (8)
C4	0.0436 (10)	0.0553 (11)	0.0459 (11)	−0.0163 (9)	−0.0118 (8)	−0.0077 (8)
C5	0.0373 (9)	0.0369 (9)	0.0423 (10)	−0.0120 (7)	−0.0017 (7)	−0.0085 (7)
C6	0.0389 (10)	0.0465 (10)	0.0440 (10)	−0.0114 (8)	0.0043 (7)	−0.0133 (8)
C7	0.0419 (10)	0.0387 (9)	0.0403 (10)	−0.0145 (7)	0.0017 (8)	−0.0141 (7)
C8	0.0369 (9)	0.0385 (9)	0.0427 (9)	−0.0131 (7)	−0.0033 (7)	−0.0137 (7)
C9	0.0372 (9)	0.0325 (8)	0.0384 (9)	−0.0124 (7)	−0.0024 (7)	−0.0094 (7)
C10	0.0634 (14)	0.0885 (16)	0.0457 (11)	−0.0245 (12)	0.0091 (10)	−0.0256 (11)
C11	0.0550 (12)	0.0696 (13)	0.0482 (11)	−0.0241 (10)	0.0037 (9)	−0.0262 (10)

Geometric parameters (Å, º)

O1—C8	1.227 (2)	C4—H4A	0.9300
O2—C6	1.335 (2)	C5—C9	1.385 (2)
O2—C5	1.383 (2)	C6—C7	1.339 (3)
O3—C11	1.196 (2)	C6—H6A	0.9300
C1—C2	1.385 (3)	C7—C8	1.455 (2)
C1—C9	1.396 (2)	C7—C11	1.475 (3)
C1—H1A	0.9300	C8—C9	1.473 (2)
C2—C3	1.399 (3)	C10—H10A	0.9600
C2—C10	1.505 (3)	C10—H10B	0.9600
C3—C4	1.368 (3)	C10—H10C	0.9600
C3—H3A	0.9300	C11—H11A	0.9300
C4—C5	1.387 (3)		
C6—O2—C5	117.99 (13)	C6—C7—C8	120.85 (16)
C2—C1—C9	121.75 (17)	C6—C7—C11	118.72 (16)
C2—C1—H1A	119.1	C8—C7—C11	120.43 (16)
C9—C1—H1A	119.1	O1—C8—C7	123.32 (16)
C1—C2—C3	117.68 (17)	O1—C8—C9	122.63 (16)
C1—C2—C10	121.76 (18)	C7—C8—C9	114.04 (15)
C3—C2—C10	120.56 (17)	C5—C9—C1	118.08 (16)

C4—C3—C2	122.15 (17)	C5—C9—C8	119.66 (16)
C4—C3—H3A	118.9	C1—C9—C8	122.26 (16)
C2—C3—H3A	118.9	C2—C10—H10A	109.5
C3—C4—C5	118.60 (17)	C2—C10—H10B	109.5
C3—C4—H4A	120.7	H10A—C10—H10B	109.5
C5—C4—H4A	120.7	C2—C10—H10C	109.5
O2—C5—C9	122.29 (16)	H10A—C10—H10C	109.5
O2—C5—C4	116.01 (15)	H10B—C10—H10C	109.5
C9—C5—C4	121.69 (16)	O3—C11—C7	125.06 (19)
O2—C6—C7	125.04 (16)	O3—C11—H11A	117.5
O2—C6—H6A	117.5	C7—C11—H11A	117.5
C7—C6—H6A	117.5		
C9—C1—C2—C3	1.3 (3)	C6—C7—C8—C9	−1.3 (2)
C9—C1—C2—C10	−178.14 (16)	C11—C7—C8—C9	177.95 (15)
C1—C2—C3—C4	−1.3 (3)	O2—C5—C9—C1	176.96 (14)
C10—C2—C3—C4	178.17 (18)	C4—C5—C9—C1	−1.9 (3)
C2—C3—C4—C5	−0.3 (3)	O2—C5—C9—C8	−3.7 (3)
C6—O2—C5—C9	0.8 (2)	C4—C5—C9—C8	177.44 (15)
C6—O2—C5—C4	179.73 (15)	C2—C1—C9—C5	0.3 (3)
C3—C4—C5—O2	−176.99 (15)	C2—C1—C9—C8	−179.10 (15)
C3—C4—C5—C9	2.0 (3)	O1—C8—C9—C5	−175.79 (16)
C5—O2—C6—C7	2.0 (3)	C7—C8—C9—C5	3.7 (2)
O2—C6—C7—C8	−1.6 (3)	O1—C8—C9—C1	3.6 (3)
O2—C6—C7—C11	179.14 (16)	C7—C8—C9—C1	−176.89 (14)
C6—C7—C8—O1	178.25 (17)	C6—C7—C11—O3	−4.4 (3)
C11—C7—C8—O1	−2.5 (3)	C8—C7—C11—O3	176.35 (19)