Acta Cryst E STRUCTURE REPORTS

OPEN ACCESS

ISSN 1600-5368

Crystal structure of 1-(8-methoxy-2*H*-chromen-3-yl)ethanone

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Received 16 July 2014; accepted 21 July 2014

Edited by E. R. T. Tiekink, University of Malaya, Malaysia

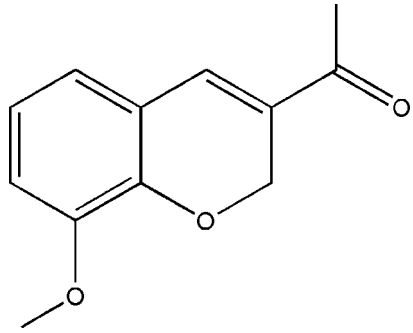
In the structure of the title compound, $C_{12}H_{12}O_3$, the dihydropyran ring is fused with the benzene ring. The dihydropyran ring is in a half-chair conformation, with the ring O and methylene C atoms positioned 1.367 (3) and 1.504 (4) Å, respectively, on either side of the mean plane formed by the other four atoms. The methoxy group is coplanar with the benzene ring to which it is connected [$C_b-Cb-O_m-C_m$ torsion angle = −0.2 (4)°; b = benzene and m = methoxy], and similarly the aldehyde is coplanar with respect to the double bond of the dihydropyran ring [$C_{dh}-C_{dh}-C_a-O_a$ = −178.1 (3)°; dh = dihydropyran and a = aldehyde]. In the crystal, molecules are linked by weak methyl–methoxy C—H···O hydrogen bonds into supramolecular chains along the *a*-axis direction.

Keywords: crystal structure; hydrogen bonding; dihydropyran ring; chromenes.

CCDC reference: 1015076

1. Related literature

For the synthesis and biological properties of chromene derivatives, see: Choi *et al.* (2014); Mun *et al.* (2012); Yoon *et al.* (2012). For the chromene group in natural products, see: Starks *et al.* (2014); Escandón-Rivera *et al.* (2012). For related structures, see: Yan & Zhang (2013): Yusufzai *et al.* (2012).



2. Experimental

2.1. Crystal data

$C_{12}H_{12}O_3$
M_r = 204.22
Orthorhombic, $P2_12_12_1$
a = 5.1000 (4) Å
b = 12.7455 (9) Å
c = 15.130 (1) Å
V = 983.48 (12) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.10 mm^{-1}
T = 173 K
0.26 × 0.20 × 0.04 mm

2.2. Data collection

Bruker SMART CCD area-detector diffractometer
7256 measured reflections
2439 independent reflections
1943 reflections with $I > 2\sigma(I)$
R_{int} = 0.029

2.3. Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.041
$wR(F^2)$ = 0.148
S = 1.15
2439 reflections
138 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.30 e Å^{-3}
$\Delta\rho_{min}$ = −0.36 e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
C11—H11A···O2^{i}	0.98	2.56	3.429 (4)	148

Symmetry code: (i) $x+\frac{1}{2}, -y+\frac{1}{2}, -z+1$.

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*.

Acknowledgements

The author acknowledges financial support from Dongduk Women's University.

Supporting information for this paper is available from the IUCr electronic archives (Reference: TK5330).

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supporting information

Acta Cryst. (2014). E**70**, o936–o937 [doi:10.1107/S1600536814016808]

Crystal structure of 1-(8-methoxy-2*H*-chromen-3-yl)ethanone

Dongsoo Koh

S1. Experimental

S1.1. Synthesis and crystallization

To a solution of 2-hydroxy-3-methoxy-benzaldehyde (750 mg, 5 mmol) in 1,4-dioxane (15 ml) was added excess amount of methyl vinyl ketone (0.7 mL, 8 mmol) and potassium carbonate (700 mg, 5 mmol) at room temperature. The reaction mixture was refluxed for 12 h and TLC showed no evidence for the starting material. After cooling to room temperature, the mixture was poured into iced water (40 ml) and extracted with methylene chloride (3 x 20 ml) and the combined organic layers were dried under $MgSO_4$. Filtration, evaporation of filtrate gave residue which was purified by flash chromatography to give the titled compound (22%). Recrystallization from its ethanol solution gave crystals (M.pt: 375–376 K).

S1.2. Refinement

The H atoms were placed in calculated positions and refined as riding with C—H = 0.95 Å, and with U_{iso}(H) = 1.2U_{eq}(C).

S2. Results and discussion

Chromenes have been shown to be potential pharmaceuticals which show anti-inflammatory (Choi *et al.*, 2014) and anti-cancer (Mun *et al.*, 2012) activities. Especially, the 2*H*-chromene skeleton is a core structure of oxygen heterocycles in many natural products having versatile biological activities (Starks *et al.*, 2014; Escandón-Rivera *et al.*, 2012). In continuation of our research interest to develop novel chromene derivatives (Yoon *et al.*, 2012), the title compound was synthesized and its crystal structure was determined (Fig. 1). In the chromene compound, the dihydropyran ring is fused with the benzene ring and is in a half-chair conformation with atoms C1 and O1 positioned 1.367 (3) and 1.504 (4) Å, respectively, on either side of the mean plane formed by the other four atoms (C2/C3/C4/C5). In the crystal, weak C—H—-O hydrogen bonds link molecules along [100] (Fig. 2). Examples of structures of chromene compounds have been published (Yan *et al.*, 2013; Yusufzai *et al.*, 2012).

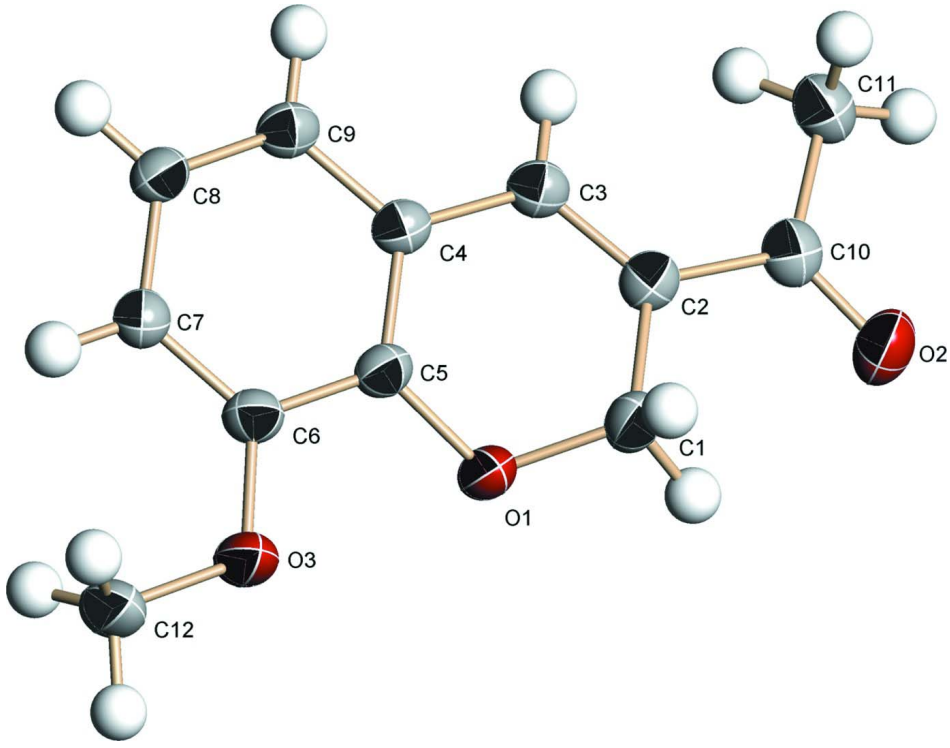

Figure 1

The molecular structure of the title compound, showing the atom labelling scheme and displacement ellipsoids drawn at the 50% probability level.

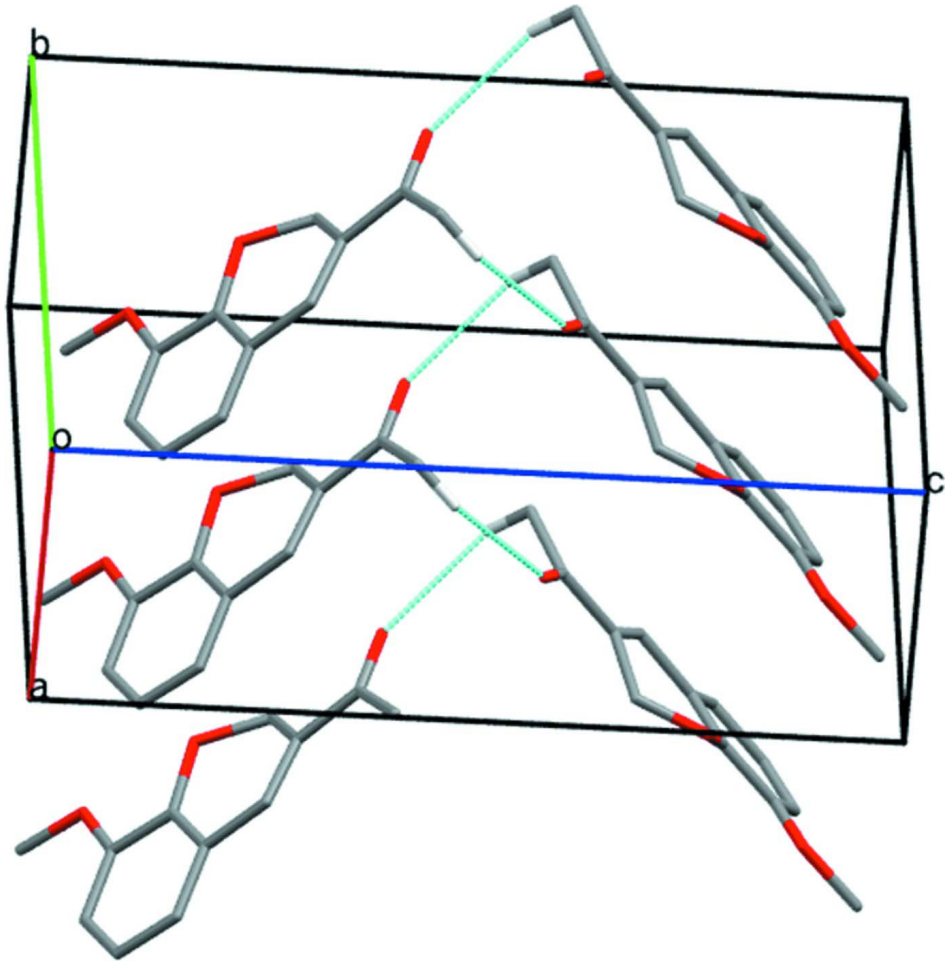

Figure 2

Part of the crystal structure with weak intermolecular C—H···O hydrogen bonds shown as dashed lines.

1-(8-Methoxy-2*H*-chromen-3-yl)ethanone

Crystal data

$C_{12}H_{12}O_3$
$M_r = 204.22$
Orthorhombic, $P2_12_12_1$
Hall symbol: P 2ac 2ab
$a = 5.1000$ (4) Å
$b = 12.7455$ (9) Å
$c = 15.130$ (1) Å
$V = 983.48$ (12) Å^3
$Z = 4$
$F(000) = 432$
$D_x = 1.379$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 4995 reflections
$\theta = 2.7$–$28.3°$
$\mu = 0.10$ mm^{-1}
$T = 173$ K
Block, white
0.26 × 0.20 × 0.04 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
φ and ω scans
7256 measured reflections
2439 independent reflections
1943 reflections with $I > 2\sigma(I)$
$R_{int} = 0.029$
$\theta_{max} = 28.3°$, $\theta_{min} = 2.1°$

$h = -4 \rightarrow 6$
$k = -16 \rightarrow 16$
$l = -20 \rightarrow 19$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.041$
$wR(F^2) = 0.148$
$S = 1.15$
2439 reflections
138 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0443P)^2 + 0.8069P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.30$ e Å^{-3}
$\Delta\rho_{min} = -0.36$ e Å^{-3}

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 (Å²)

	x	y	z	U_{iso}*/U_{eq}
O1	0.2139 (4)	0.13132 (13)	0.78457 (12)	0.0369 (5)
C1	0.0935 (6)	0.1378 (2)	0.69905 (18)	0.0382 (6)
H1A	−0.0596	0.0900	0.6976	0.046*
H1B	0.2202	0.1130	0.6541	0.046*
C2	0.0045 (5)	0.2465 (2)	0.67470 (17)	0.0317 (5)
C3	0.1318 (5)	0.32861 (19)	0.70942 (16)	0.0315 (5)
H3	0.0841	0.3977	0.6921	0.038*
C4	0.3416 (5)	0.31384 (19)	0.77314 (16)	0.0290 (5)
C5	0.3744 (5)	0.21278 (18)	0.80743 (16)	0.0304 (5)
C6	0.5662 (5)	0.19364 (19)	0.87130 (17)	0.0323 (6)
C7	0.7236 (6)	0.27498 (19)	0.90030 (17)	0.0327 (5)
H7	0.8543	0.2621	0.9437	0.039*
C8	0.6916 (6)	0.3759 (2)	0.86613 (17)	0.0335 (6)
H8	0.8013	0.4314	0.8860	0.040*
C9	0.5011 (5)	0.39520 (19)	0.80357 (17)	0.0320 (5)
H9	0.4783	0.4642	0.7811	0.038*
C10	−0.2116 (5)	0.2531 (2)	0.61005 (18)	0.0357 (6)
O2	−0.3175 (4)	0.17219 (16)	0.58488 (14)	0.0471 (5)
C11	−0.2923 (6)	0.3576 (2)	0.57362 (18)	0.0414 (7)
H11A	−0.1666	0.3797	0.5283	0.062*
H11B	−0.2956	0.4095	0.6214	0.062*
H11C	−0.4673	0.3519	0.5473	0.062*

O3	0.5790 (4)	0.09230 (15)	0.90143 (14)	0.0430 (5)
C12	0.7723 (6)	0.0714 (2)	0.9676 (2)	0.0435 (7)
H12A	0.9471	0.0861	0.9437	0.065*
H12B	0.7620	−0.0025	0.9853	0.065*
H12C	0.7403	0.1162	1.0191	0.065*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0406 (10)	0.0278 (9)	0.0424 (10)	−0.0065 (8)	−0.0104 (9)	0.0029 (7)
C1	0.0443 (15)	0.0297 (12)	0.0405 (13)	−0.0008 (12)	−0.0109 (13)	−0.0034 (11)
C2	0.0296 (12)	0.0307 (12)	0.0350 (12)	0.0021 (10)	0.0010 (10)	−0.0026 (10)
C3	0.0318 (13)	0.0285 (12)	0.0341 (12)	0.0045 (10)	0.0022 (11)	−0.0001 (10)
C4	0.0286 (12)	0.0268 (11)	0.0316 (12)	0.0013 (10)	0.0034 (10)	−0.0018 (9)
C5	0.0336 (13)	0.0257 (11)	0.0318 (12)	−0.0022 (10)	0.0013 (11)	−0.0016 (9)
C6	0.0347 (13)	0.0272 (12)	0.0349 (12)	0.0009 (10)	−0.0002 (11)	0.0018 (10)
C7	0.0335 (13)	0.0334 (13)	0.0311 (12)	−0.0017 (10)	−0.0015 (11)	−0.0003 (10)
C8	0.0379 (14)	0.0285 (12)	0.0342 (12)	−0.0044 (11)	0.0000 (11)	−0.0013 (10)
C9	0.0348 (13)	0.0261 (11)	0.0351 (12)	0.0023 (10)	0.0034 (11)	−0.0016 (10)
C10	0.0340 (13)	0.0400 (14)	0.0330 (12)	0.0047 (12)	0.0014 (11)	−0.0041 (11)
O2	0.0446 (12)	0.0441 (12)	0.0527 (12)	−0.0028 (10)	−0.0110 (10)	−0.0106 (9)
C11	0.0437 (16)	0.0451 (15)	0.0354 (13)	0.0064 (14)	−0.0037 (12)	0.0011 (11)
O3	0.0498 (12)	0.0285 (9)	0.0508 (11)	−0.0036 (9)	−0.0161 (10)	0.0082 (8)
C12	0.0464 (17)	0.0345 (14)	0.0497 (16)	0.0006 (13)	−0.0131 (14)	0.0096 (12)

Geometric parameters (Å, º)

O1—C5	1.366 (3)	C7—C8	1.396 (3)
O1—C1	1.435 (3)	C7—H7	0.9500
C1—C2	1.504 (4)	C8—C9	1.378 (4)
C1—H1A	0.9900	C8—H8	0.9500
C1—H1B	0.9900	C9—H9	0.9500
C2—C3	1.339 (4)	C10—O2	1.224 (3)
C2—C10	1.476 (4)	C10—C11	1.499 (4)
C3—C4	1.452 (4)	C11—H11A	0.9800
C3—H3	0.9500	C11—H11B	0.9800
C4—C9	1.396 (3)	C11—H11C	0.9800
C4—C5	1.399 (3)	O3—C12	1.430 (3)
C5—C6	1.397 (3)	C12—H12A	0.9800
C6—O3	1.371 (3)	C12—H12B	0.9800
C6—C7	1.383 (4)	C12—H12C	0.9800
C5—O1—C1	116.2 (2)	C8—C7—H7	119.8
O1—C1—C2	113.8 (2)	C9—C8—C7	120.1 (2)
O1—C1—H1A	108.8	C9—C8—H8	120.0
C2—C1—H1A	108.8	C7—C8—H8	120.0
O1—C1—H1B	108.8	C8—C9—C4	120.3 (2)
C2—C1—H1B	108.8	C8—C9—H9	119.8

H1A—C1—H1B	107.7	C4—C9—H9	119.8
C3—C2—C10	125.3 (2)	O2—C10—C2	119.2 (2)
C3—C2—C1	118.5 (2)	O2—C10—C11	120.8 (2)
C10—C2—C1	116.1 (2)	C2—C10—C11	119.9 (2)
C2—C3—C4	121.1 (2)	C10—C11—H11A	109.5
C2—C3—H3	119.5	C10—C11—H11B	109.5
C4—C3—H3	119.5	H11A—C11—H11B	109.5
C9—C4—C5	119.5 (2)	C10—C11—H11C	109.5
C9—C4—C3	123.5 (2)	H11A—C11—H11C	109.5
C5—C4—C3	117.0 (2)	H11B—C11—H11C	109.5
O1—C5—C6	117.5 (2)	C6—O3—C12	116.2 (2)
O1—C5—C4	122.3 (2)	O3—C12—H12A	109.5
C6—C5—C4	120.1 (2)	O3—C12—H12B	109.5
O3—C6—C7	125.0 (2)	H12A—C12—H12B	109.5
O3—C6—C5	115.3 (2)	O3—C12—H12C	109.5
C7—C6—C5	119.7 (2)	H12A—C12—H12C	109.5
C6—C7—C8	120.4 (2)	H12B—C12—H12C	109.5
C6—C7—H7	119.8		

C5—O1—C1—C2	−39.4 (3)	O1—C5—C6—C7	−176.4 (2)
O1—C1—C2—C3	28.1 (4)	C4—C5—C6—C7	−0.1 (4)
O1—C1—C2—C10	−154.7 (2)	O3—C6—C7—C8	−179.0 (3)
C10—C2—C3—C4	179.7 (2)	C5—C6—C7—C8	0.0 (4)
C1—C2—C3—C4	−3.5 (4)	C6—C7—C8—C9	0.5 (4)
C2—C3—C4—C9	172.2 (2)	C7—C8—C9—C4	−0.9 (4)
C2—C3—C4—C5	−10.5 (4)	C5—C4—C9—C8	0.8 (4)
C1—O1—C5—C6	−156.5 (2)	C3—C4—C9—C8	178.1 (2)
C1—O1—C5—C4	27.3 (3)	C3—C2—C10—O2	−178.1 (3)
C9—C4—C5—O1	175.8 (2)	C1—C2—C10—O2	5.0 (4)
C3—C4—C5—O1	−1.7 (4)	C3—C2—C10—C11	4.2 (4)
C9—C4—C5—C6	−0.3 (4)	C1—C2—C10—C11	−172.7 (2)
C3—C4—C5—C6	−177.7 (2)	C7—C6—O3—C12	−0.2 (4)
O1—C5—C6—O3	2.8 (3)	C5—C6—O3—C12	−179.3 (2)
C4—C5—C6—O3	179.0 (2)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C11—H11*A*···O2^{i}	0.98	2.56	3.429 (4)	148

Symmetry code: (i) *x*+1/2, −*y*+1/2, −*z*+1.