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Crystal structure of 3-(hydroxymethyl)-chromone

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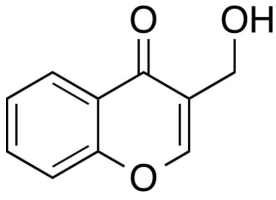
In the title compound, $C_{10}H_8O_3$ (systematic name 3-hydroxymethyl-4*H*-chromen-4-one), the fused-ring system is slightly puckered [dihedral angle between the rings = 3.84 (11)°]. The hydroxy O atom deviates from the heterocyclic ring by 1.422 (1) Å. In the crystal, inversion dimers linked by pairs of O—H···O hydrogen bonds generate $R_2^2(12)$ loops. The dimers are linked by aromatic π–π stacking [shortest centroid–centroid distance = 3.580 (3) Å], and C—H···O hydrogen bonds, generating a three-dimensional network.

Keywords: crystal structure; chromone; hydrogen bonding; π–π stacking.

CCDC reference: 1406927

1. Related literature

For the biological activities of related compounds, see: Sun *et al.* (2009); Helguera *et al.* (2013); Venkateswararao *et al.* (2014). For the synthesis of the title compound, see: Araya-Maturana *et al.* (2003).



2. Experimental

2.1. Crystal data

$C_{10}H_8O_3$
M_r = 176.17
Triclinic, $P\overline{1}$
a = 6.756 (4) Å
b = 7.988 (6) Å
c = 7.991 (6) Å
α = 94.48 (6)°
β = 108.27 (5)°
γ = 103.31 (5)°
V = 393.2 (5) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.11 mm^{-1}
T = 100 K
0.32 × 0.32 × 0.16 mm

2.2. Data collection

Rigaku AFC-7R diffractometer
2219 measured reflections
1805 independent reflections
1537 reflections with $F^2 > 2.0\sigma(F^2)$
R_{int} = 0.089
3 standard reflections every 150 reflections
intensity decay: 0.1%

2.3. Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.065
$wR(F^2)$ = 0.202
S = 1.06
1805 reflections
119 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.42 e Å^{-3}
$\Delta\rho_{min}$ = −0.49 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O3—H8···O2^{i}	0.84	1.94	2.757 (3)	165
C1—H1···O2^{ii}	0.95	2.58	3.283 (4)	131

Symmetry codes: (i) $-x+2, -y+1, -z+1$; (ii) $x-1, y, z$.

Data collection: *WinAFC Diffractometer Control Software* (Rigaku, 1999); cell refinement: *WinAFC Diffractometer Control Software*; data reduction: *WinAFC Diffractometer Control Software*; program(s) used to solve structure: *SIR2008* (Burla *et al.*, 2007); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *CrystalStructure* (Rigaku, 2010); software used to prepare material for publication: *CrystalStructure*.

Acknowledgements

The University of Shizuoka is acknowledged for instrumental support.

Supporting information for this paper is available from the IUCr electronic archives (Reference: HB7444).

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supporting information

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Crystal structure of 3-(hydroxymethyl)chromone

Yoshinobu Ishikawa

S1. Comment

Many derivatives of the title compound (3-hydroxymethylchromone) are reported as retinoic acid receptor binders (Sun *et al.* (2009)), human monoamine oxidase inhibitors (Helguera *et al.* (2013)) and anti-proliferative agents (Venkateswararao *et al.* (2014)).

The mean deviation of the least-square planes for the non-hydrogen atoms except hydroxy O3 atom is 0.0479 Å, and the largest deviation is 0.146 (2) Å for C10. These mean that these atoms are essentially coplanar (Fig.1). The dihedral angle of C3–C2–C10–O3 is 70.6 (2). In the crystal, the pyran rings are stacked [centroid–centroid distance between the pyran rings of the 4*H*-chromene units = 3.894 (3) Å], and C–H···O hydrogen bonds are formed to give dimers running along the *c* direction, as shown in Fig.2.

S2. Experimental

The title compound was synthesized from 3-formylchromone according to the literature method (Araya-Maturana *et al.* 2003). Colourless blocks were obtained by slow evaporation of an ethyl acetate solution of the title compound at room temperature.

S3. Refinement

All hydrogen atoms were placed in geometrical positions [C–H 0.95 Å and O–H 0.84 Å], and refined using a riding model with U_{iso}(H) = 1.2U_{eq} of the parent atoms.

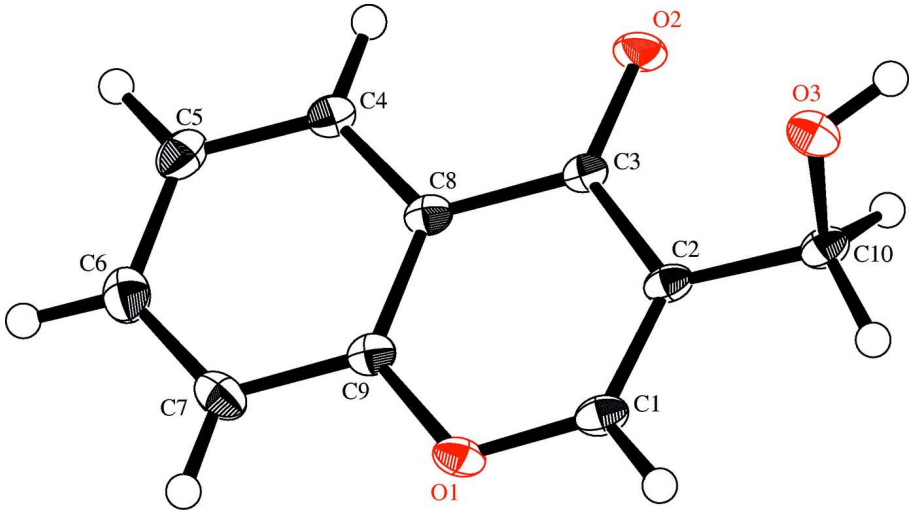

Figure 1

The molecular structure of the title compound, with displacement ellipsoids drawn at the 50% probability level.

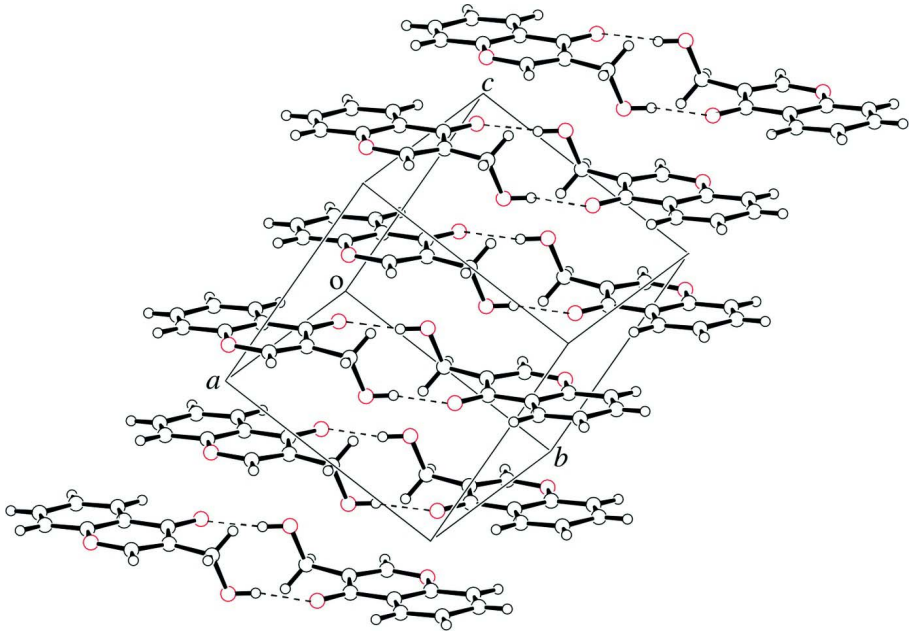

Figure 2

A view of the packing of the title compound. O—H···O hydrogen bonds are represented as dashed lines.

3-(Hydroxymethyl)-4*H*-chromen-4-one

Crystal data

$C_{10}H_8O_3$
M_r = 176.17
Triclinic, $P\overline{1}$
Hall symbol: -P 1
a = 6.756 (4) Å
b = 7.988 (6) Å
c = 7.991 (6) Å
α = 94.48 (6)°
β = 108.27 (5)°
γ = 103.31 (5)°
V = 393.2 (5) Å^3
Z = 2
$F(000)$ = 184.00
D_x = 1.488 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71069 Å
Cell parameters from 25 reflections
θ = 15.5–17.3°
μ = 0.11 mm^{-1}
T = 100 K
Block, colorless
0.32 × 0.32 × 0.16 mm

Data collection

Rigaku AFC-7R diffractometer
ω–2θ scans
2219 measured reflections
1805 independent reflections
1537 reflections with $F^2 > 2.0\sigma(F^2)$
R_{int} = 0.089
θ_{max} = 27.5°
h = −4→8
k = −10→10
l = −10→9
3 standard reflections every 150 reflections
intensity decay: 0.1%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.065$
$wR(F^2) = 0.202$
$S = 1.06$
1805 reflections
119 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
$w = 1/[\sigma^2(F_o^2) + (0.1399P)^2 + 0.1723P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.42$ e Å^{-3}
$\Delta\rho_{min} = -0.49$ e Å^{-3}

Special details

Refinement. Refinement was performed using all reflections. The weighted *R*-factor (*wR*) and goodness of fit (*S*) are based on F^2. *R*-factor (gt) are based on *F*. The threshold expression of $F^2 > 2.0\ \sigma(F^2)$ is used only for calculating *R*-factor (gt).

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
O1	0.72779 (19)	1.01991 (17)	0.70911 (18)	0.0179 (4)
O2	1.19522 (19)	0.80874 (17)	0.63213 (18)	0.0190 (4)
O3	0.7925 (2)	0.48525 (17)	0.56522 (17)	0.0195 (4)
C1	0.6785 (3)	0.8666 (3)	0.6006 (3)	0.0167 (4)
C2	0.8224 (3)	0.7871 (3)	0.5707 (3)	0.0146 (4)
C3	1.0527 (3)	0.8696 (3)	0.6575 (3)	0.0133 (4)
C4	1.3221 (3)	1.1214 (3)	0.8824 (3)	0.0168 (4)
C5	1.3681 (3)	1.2688 (3)	1.0053 (3)	0.0209 (5)
C6	1.1985 (3)	1.3325 (3)	1.0269 (3)	0.0218 (5)
C7	0.9868 (3)	1.2504 (3)	0.9255 (3)	0.0200 (5)
C8	1.1070 (3)	1.0325 (3)	0.7810 (3)	0.0147 (4)
C9	0.9416 (3)	1.0995 (3)	0.8033 (3)	0.0153 (4)
C10	0.7477 (3)	0.6132 (3)	0.4539 (3)	0.0165 (4)
H1	0.5294	0.8101	0.5403	0.0201*
H2	1.4369	1.0798	0.8665	0.0202*
H3	1.5139	1.3272	1.0751	0.0251*
H4	1.2306	1.4333	1.1123	0.0262*
H5	0.8729	1.2955	0.9382	0.0240*
H6A	0.5905	0.5855	0.3870	0.0198*
H7B	0.8247	0.6145	0.3669	0.0198*
H8	0.8200	0.4052	0.5093	0.0234*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0130 (6)	0.0202 (7)	0.0240 (7)	0.0070 (5)	0.0090 (5)	0.0043 (5)
O2	0.0112 (6)	0.0225 (7)	0.0252 (7)	0.0067 (5)	0.0075 (5)	0.0032 (6)
O3	0.0195 (7)	0.0188 (7)	0.0236 (7)	0.0069 (5)	0.0104 (6)	0.0052 (5)
C1	0.0110 (8)	0.0212 (9)	0.0193 (9)	0.0041 (7)	0.0061 (7)	0.0073 (7)
C2	0.0108 (8)	0.0202 (9)	0.0144 (8)	0.0046 (7)	0.0050 (6)	0.0067 (7)

C3	0.0115 (8)	0.0168 (9)	0.0129 (8)	0.0043 (6)	0.0052 (6)	0.0053 (6)
C4	0.0158 (8)	0.0179 (9)	0.0168 (8)	0.0054 (7)	0.0044 (7)	0.0050 (7)
C5	0.0205 (9)	0.0212 (10)	0.0174 (9)	0.0040 (7)	0.0021 (7)	0.0055 (7)
C6	0.0302 (10)	0.0184 (9)	0.0171 (9)	0.0071 (8)	0.0077 (8)	0.0042 (7)
C7	0.0264 (9)	0.0198 (9)	0.0212 (9)	0.0113 (8)	0.0136 (8)	0.0068 (7)
C8	0.0144 (8)	0.0179 (9)	0.0140 (8)	0.0055 (7)	0.0061 (7)	0.0071 (7)
C9	0.0149 (8)	0.0191 (9)	0.0144 (8)	0.0055 (7)	0.0068 (7)	0.0069 (7)
C10	0.0101 (8)	0.0204 (9)	0.0178 (9)	0.0026 (7)	0.0039 (6)	0.0038 (7)

Geometric parameters (Å, º)

O1—C1	1.352 (3)	C6—C7	1.375 (3)
O1—C9	1.371 (2)	C7—C9	1.400 (3)
O2—C3	1.236 (3)	C8—C9	1.396 (3)
O3—C10	1.429 (3)	O3—H8	0.840
C1—C2	1.346 (3)	C1—H1	0.950
C2—C3	1.455 (3)	C4—H2	0.950
C2—C10	1.495 (3)	C5—H3	0.950
C3—C8	1.468 (3)	C6—H4	0.950
C4—C5	1.381 (3)	C7—H5	0.950
C4—C8	1.404 (3)	C10—H6A	0.990
C5—C6	1.407 (4)	C10—H7B	0.990
C1—O1—C9	117.98 (17)	O3—C10—C2	108.17 (15)
O1—C1—C2	125.63 (15)	C10—O3—H8	109.472
C1—C2—C3	119.38 (17)	O1—C1—H1	117.186
C1—C2—C10	120.66 (15)	C2—C1—H1	117.188
C3—C2—C10	119.93 (18)	C5—C4—H2	119.761
O2—C3—C2	123.48 (17)	C8—C4—H2	119.765
O2—C3—C8	121.37 (15)	C4—C5—H3	120.061
C2—C3—C8	115.15 (18)	C6—C5—H3	120.064
C5—C4—C8	120.5 (2)	C5—C6—H4	119.658
C4—C5—C6	119.88 (16)	C7—C6—H4	119.653
C5—C6—C7	120.69 (19)	C6—C7—H5	120.506
C6—C7—C9	119.0 (3)	C9—C7—H5	120.503
C3—C8—C4	121.64 (19)	O3—C10—H6A	110.064
C3—C8—C9	119.77 (15)	O3—C10—H7B	110.063
C4—C8—C9	118.56 (17)	C2—C10—H6A	110.069
O1—C9—C7	116.70 (19)	C2—C10—H7B	110.064
O1—C9—C8	121.91 (17)	H6A—C10—H7B	108.407
C7—C9—C8	121.38 (16)		
C1—O1—C9—C7	−174.88 (15)	C5—C4—C8—C3	−176.20 (17)
C1—O1—C9—C8	4.2 (3)	C5—C4—C8—C9	1.9 (3)
C9—O1—C1—C2	−2.8 (3)	C8—C4—C5—C6	−1.2 (3)
C9—O1—C1—H1	177.2	C8—C4—C5—H3	178.8
H8—O3—C10—C2	−148.1	H2—C4—C5—C6	178.8
H8—O3—C10—H6A	91.6	H2—C4—C5—H3	−1.2

H8—O3—C10—H7B	−27.8	H2—C4—C8—C3	3.8
O1—C1—C2—C3	−1.0 (3)	H2—C4—C8—C9	−178.1
O1—C1—C2—C10	177.18 (16)	C4—C5—C6—C7	−0.7 (3)
H1—C1—C2—C3	179.0	C4—C5—C6—H4	179.3
H1—C1—C2—C10	−2.8	H3—C5—C6—C7	179.3
C1—C2—C3—O2	−177.19 (17)	H3—C5—C6—H4	−0.7
C1—C2—C3—C8	3.3 (3)	C5—C6—C7—C9	1.6 (3)
C1—C2—C10—O3	−107.56 (19)	C5—C6—C7—H5	−178.4
C1—C2—C10—H6A	12.7	H4—C6—C7—C9	−178.4
C1—C2—C10—H7B	132.2	H4—C6—C7—H5	1.6
C3—C2—C10—O3	70.6 (2)	C6—C7—C9—O1	178.25 (17)
C3—C2—C10—H6A	−169.1	C6—C7—C9—C8	−0.8 (3)
C3—C2—C10—H7B	−49.7	H5—C7—C9—O1	−1.8
C10—C2—C3—O2	4.6 (3)	H5—C7—C9—C8	179.2
C10—C2—C3—C8	−174.93 (15)	C3—C8—C9—O1	−1.8 (3)
O2—C3—C8—C4	−3.4 (3)	C3—C8—C9—C7	177.23 (16)
O2—C3—C8—C9	178.52 (16)	C4—C8—C9—O1	−179.96 (16)
C2—C3—C8—C4	176.19 (15)	C4—C8—C9—C7	−0.9 (3)
C2—C3—C8—C9	−1.9 (3)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O3—H8···O2^{i}	0.84	1.94	2.757 (3)	165
C1—H1···O2^{ii}	0.95	2.58	3.283 (4)	131

Symmetry codes: (i) −*x*+2, −*y*+1, −*z*+1; (ii) *x*−1, *y*, *z*.