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2-(4,6-Dimethyl-1-benzofuran-3-yl)acetic acid

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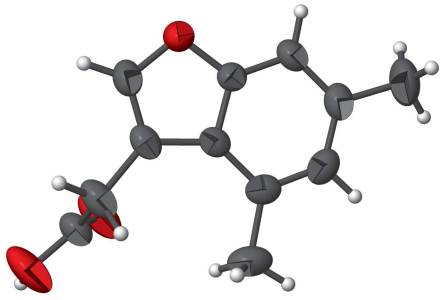
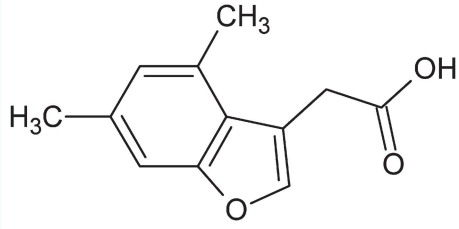
Structural data: full structural data are available from iucrdata.iucr.org

In the title compound, $C_{12}H_{12}O_3$, the dihedral angle between the planes of the carboxylic acid group and the benzofuran ring system (r.m.s. deviation = 0.012 Å) is 76.53 (10)°. In the crystal, carboxylic acid inversion dimers linked by pairs of O—H···O hydrogen bonds generate $R_2^2(8)$ loops. C—H···O interactions link the dimers into (101) sheets.

3D view



Chemical scheme



Structure description

Carboxylic acids, such as arylalkanoic acids, exhibit anti-inflammatory, analgesic and antipyretic properties and have been in widespread clinical use for a number of years (Basanagouda *et al.*, 2015). As part of our studies in this area, we now report the crystal structure of the title compound. All the bond lengths and angles are close to those observed for similar structures (Gowda *et al.*, 2015; Ramprasad *et al.*, 2016).

The X-ray structure of the title molecule (Fig. 1) reveals its non-planar nature; the plane of the acetic acid group makes a dihedral angle of 76.53 (10)° with that of the benzofuran ring system. The C9—C12 bond length [1.512 (3) Å] reflects the sp^2(C1)—sp^3(C12) hybridization of these atoms; this is also reflected in the C7—C11 bond length [1.497 (3) Å].

In the crystal, molecules are linked into carboxylic acid inversion dimers by pairs of O—H···O hydrogen bonds. The dimers are linked into (101) sheets by a very weak C—H···O hydrogen bond (Fig. 2 and Table 1).

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

D—H···A	D—H	H···A	D···A	D—H···A
C4—H4···O3^{i}	0.93	2.58	3.328 (2)	137
O2—H2···O1^{ii}	0.82	1.83	2.645 (2)	171

Symmetry codes: (i) $-x+\frac{3}{2}, y-\frac{1}{2}, -z+\frac{1}{2}$; (ii) $-x+1, -y, -z$.

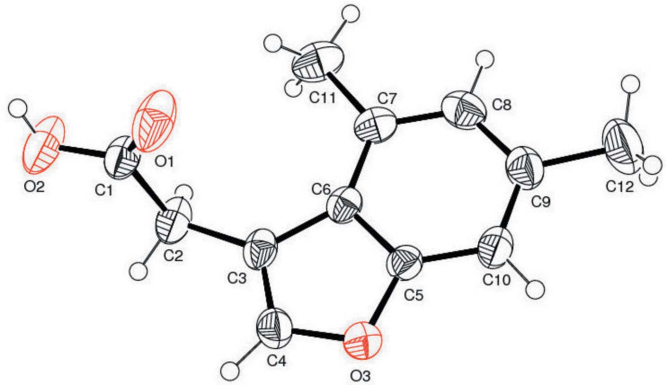


Figure 1
The molecular structure of the title compound, showing 40% probability displacement ellipsoids.

Synthesis and crystallization

4-Bromomethyl-5,7-dimethylcoumarin (10 m*M*) was refluxed in 1 *M* NaOH (100 ml) for 2 h (the completion of the reaction was monitored by thin-layer chromatography). The reaction mixture was cooled, neutralized with 1 *M* HCl and the obtained product was filtered off and dried. Colourless blocks were obtained by recrystallization from an ethanol and ethyl acetate solvent mixture by slow evaporation (m.p. 442–443 K) (Basanagouda *et al.*, 2015).

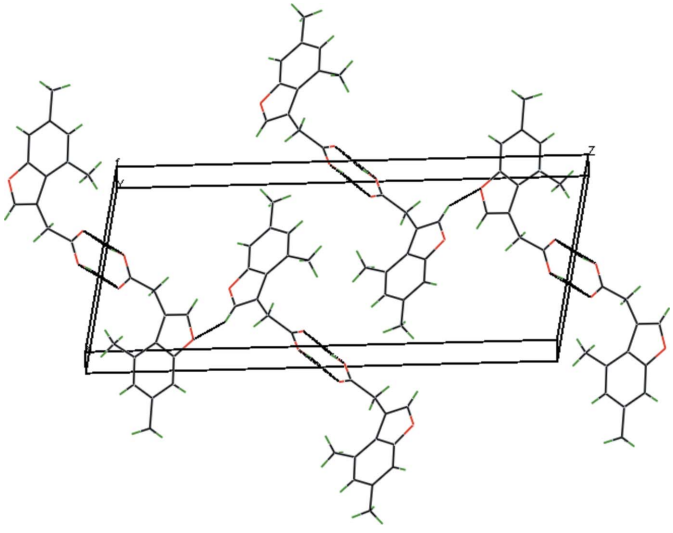


Figure 2
The crystal packing diagram of the title compound. The dotted lines indicate intermolecular hydrogen bonds. H atoms not involved in these interactions have been omitted for clarity.

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{12}H_{12}O_3$
M_r	204.22
Crystal system, space group	Monoclinic, $P2_1/n$
Temperature (K)	293
a, b, c (Å)	9.5048 (4), 4.8237 (2), 23.3395 (11)
β (°)	100.829 (2)
V (Å^3)	1051.02 (8)
Z	4
Radiation type	Mo $K\alpha$
μ (mm^{-1})	0.09
Crystal size (mm)	0.25 × 0.20 × 0.20
Data collection	
Diffractometer	Bruker Kappa APEXII CCD
Absorption correction	Multi-scan (*SADABS*; Bruker, 2004)
T_{min}, T_{max}	0.969, 0.992
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	15929, 2056, 1581
R_{int}	0.027
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.616
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.043, 0.129, 1.06
No. of reflections	2056
No. of parameters	139
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.27, −0.30

Computer programs: *APEX2* and *SAINT* (Bruker, 2004), *SIR92* (Altomare *et al.*, 1994), *SHELXL2014* (Sheldrick, 2015), *ORTEP-3 for Windows* (Farrugia, 2012) and *Mercury* (Macrae *et al.*, 2008).

Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2.

Acknowledgements

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full crystallographic data

IUCrData (2016). **1**, x161032 [https://doi.org/10.1107/S2414314616010324]

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Crystal data

$C_{12}H_{12}O_3$
M_r = 204.22
Monoclinic, $P2_1/n$
a = 9.5048 (4) Å
b = 4.8237 (2) Å
c = 23.3395 (11) Å
β = 100.829 (2)°
V = 1051.02 (8) Å^3
Z = 4
$F(000)$ = 432
D_x = 1.291 Mg m^{-3}
Melting point = 433–432 K
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 4624 reflections
θ = 2.2–25.6°
μ = 0.09 mm^{-1}
T = 293 K
Block, colourless
0.25 × 0.20 × 0.20 mm

Data collection

Bruker axs kappa apex2 CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
ω and φ scan
Absorption correction: multi-scan (SADABS; Bruker, 2004)
T_{min} = 0.969, T_{max} = 0.992
15929 measured reflections
2056 independent reflections
1581 reflections with $I > 2\sigma(I)$
R_{int} = 0.027
θ_{max} = 26.0°, θ_{min} = 2.2°
h = −11→11
k = −5→5
l = −28→28

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.043
$wR(F^2)$ = 0.129
S = 1.06
2056 reflections
139 parameters
0 restraints
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0551P)^2 + 0.4386P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.27 e Å^{-3}
$\Delta\rho_{min}$ = −0.30 e Å^{-3}
Extinction correction: SHELXL2014 (Sheldrick, 2015), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.021 (3)

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

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

	x	*y*	*z*	U_{iso}*/U_{eq}
C1	0.6178 (2)	−0.1293 (4)	0.07038 (8)	0.0483 (5)
C2	0.7035 (2)	−0.2388 (4)	0.12645 (9)	0.0544 (5)
H2A	0.6384	−0.2841	0.1525	0.065*
H2B	0.7501	−0.4093	0.1182	0.065*
C3	0.81502 (18)	−0.0435 (4)	0.15717 (8)	0.0431 (4)
C4	0.79779 (19)	0.1147 (4)	0.20245 (8)	0.0486 (5)
H4	0.7150	0.1145	0.2183	0.058*
C5	1.01240 (18)	0.2144 (4)	0.18824 (7)	0.0391 (4)
C6	0.95676 (17)	0.0184 (3)	0.14658 (7)	0.0384 (4)
C7	1.0429 (2)	−0.0704 (4)	0.10711 (8)	0.0458 (5)
C8	1.1781 (2)	0.0431 (4)	0.11406 (9)	0.0540 (5)
H8	1.2370	−0.0135	0.0886	0.065*
C9	1.23245 (19)	0.2375 (4)	0.15682 (9)	0.0519 (5)
C10	1.14744 (19)	0.3273 (4)	0.19488 (8)	0.0480 (5)
H10	1.1798	0.4580	0.2237	0.058*
C11	0.9921 (3)	−0.2776 (5)	0.06001 (9)	0.0667 (6)
H11A	0.9718	−0.4502	0.0773	0.100*
H11B	1.0651	−0.3056	0.0372	0.100*
H11C	0.9067	−0.2099	0.0353	0.100*
C12	1.3832 (2)	0.3480 (6)	0.16180 (13)	0.0832 (8)
H12A	1.4150	0.3199	0.1256	0.125*
H12B	1.4459	0.2517	0.1924	0.125*
H12C	1.3843	0.5425	0.1706	0.125*
O1	0.64980 (17)	0.0843 (4)	0.04769 (7)	0.0808 (6)
O2	0.50928 (17)	−0.2762 (4)	0.04947 (7)	0.0828 (6)
H2	0.4680	−0.2081	0.0187	0.124*
O3	0.91475 (13)	0.2772 (3)	0.22303 (5)	0.0484 (4)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0459 (10)	0.0473 (11)	0.0473 (10)	−0.0099 (8)	−0.0028 (8)	0.0048 (8)
C2	0.0515 (11)	0.0519 (11)	0.0527 (11)	−0.0142 (9)	−0.0087 (9)	0.0131 (9)
C3	0.0398 (9)	0.0435 (10)	0.0413 (9)	−0.0045 (7)	−0.0040 (7)	0.0106 (8)
C4	0.0377 (9)	0.0612 (12)	0.0459 (10)	−0.0028 (8)	0.0053 (8)	0.0064 (9)
C5	0.0388 (9)	0.0427 (9)	0.0348 (8)	0.0014 (7)	0.0038 (7)	−0.0003 (7)
C6	0.0412 (9)	0.0356 (9)	0.0358 (8)	0.0002 (7)	0.0003 (7)	0.0050 (7)
C7	0.0576 (11)	0.0396 (10)	0.0392 (9)	0.0058 (8)	0.0068 (8)	0.0018 (8)
C8	0.0531 (11)	0.0596 (12)	0.0529 (11)	0.0104 (10)	0.0192 (9)	0.0045 (10)
C9	0.0405 (10)	0.0592 (12)	0.0555 (11)	−0.0007 (9)	0.0074 (8)	0.0084 (10)
C10	0.0423 (10)	0.0511 (11)	0.0471 (10)	−0.0067 (8)	−0.0005 (8)	−0.0018 (8)
C11	0.0917 (17)	0.0557 (13)	0.0519 (12)	0.0043 (12)	0.0110 (11)	−0.0118 (10)
C12	0.0465 (12)	0.108 (2)	0.0973 (19)	−0.0135 (13)	0.0180 (12)	0.0074 (16)
O1	0.0777 (11)	0.0734 (11)	0.0753 (11)	−0.0296 (9)	−0.0270 (8)	0.0324 (9)
O2	0.0786 (11)	0.0869 (12)	0.0666 (10)	−0.0386 (9)	−0.0284 (8)	0.0240 (9)

O3	0.0433 (7)	0.0587 (8)	0.0435 (7)	−0.0017 (6)	0.0089 (5)	−0.0080 (6)

Geometric parameters (Å, º)

C1—O1	1.223 (2)	C7—C8	1.378 (3)
C1—O2	1.271 (2)	C7—C11	1.497 (3)
C1—C2	1.501 (3)	C8—C9	1.396 (3)
C2—C3	1.497 (2)	C8—H8	0.9300
C2—H2A	0.9700	C9—C10	1.378 (3)
C2—H2B	0.9700	C9—C12	1.512 (3)
C3—C4	1.338 (3)	C10—H10	0.9300
C3—C6	1.446 (2)	C11—H11A	0.9600
C4—O3	1.371 (2)	C11—H11B	0.9600
C4—H4	0.9300	C11—H11C	0.9600
C5—C10	1.376 (2)	C12—H12A	0.9600
C5—O3	1.377 (2)	C12—H12B	0.9600
C5—C6	1.388 (2)	C12—H12C	0.9600
C6—C7	1.409 (2)	O2—H2	0.8200
O1—C1—O2	123.60 (18)	C7—C8—C9	124.24 (18)
O1—C1—C2	122.38 (16)	C7—C8—H8	117.9
O2—C1—C2	114.02 (16)	C9—C8—H8	117.9
C3—C2—C1	114.56 (15)	C10—C9—C8	119.28 (17)
C3—C2—H2A	108.6	C10—C9—C12	120.1 (2)
C1—C2—H2A	108.6	C8—C9—C12	120.6 (2)
C3—C2—H2B	108.6	C5—C10—C9	116.77 (18)
C1—C2—H2B	108.6	C5—C10—H10	121.6
H2A—C2—H2B	107.6	C9—C10—H10	121.6
C4—C3—C6	105.86 (15)	C7—C11—H11A	109.5
C4—C3—C2	123.83 (17)	C7—C11—H11B	109.5
C6—C3—C2	130.31 (18)	H11A—C11—H11B	109.5
C3—C4—O3	113.03 (16)	C7—C11—H11C	109.5
C3—C4—H4	123.5	H11A—C11—H11C	109.5
O3—C4—H4	123.5	H11B—C11—H11C	109.5
C10—C5—O3	124.36 (16)	C9—C12—H12A	109.5
C10—C5—C6	124.98 (17)	C9—C12—H12B	109.5
O3—C5—C6	110.65 (15)	H12A—C12—H12B	109.5
C5—C6—C7	118.25 (16)	C9—C12—H12C	109.5
C5—C6—C3	105.41 (15)	H12A—C12—H12C	109.5
C7—C6—C3	136.34 (17)	H12B—C12—H12C	109.5
C8—C7—C6	116.47 (17)	C1—O2—H2	109.5
C8—C7—C11	121.12 (18)	C4—O3—C5	105.04 (14)
C6—C7—C11	122.41 (18)		
O1—C1—C2—C3	9.8 (3)	C3—C6—C7—C8	−178.26 (18)
O2—C1—C2—C3	−169.37 (19)	C5—C6—C7—C11	−179.04 (16)
C1—C2—C3—C4	99.9 (2)	C3—C6—C7—C11	1.5 (3)
C1—C2—C3—C6	−80.0 (3)	C6—C7—C8—C9	−0.4 (3)

C6—C3—C4—O3	0.2 (2)	C11—C7—C8—C9	179.83 (19)
C2—C3—C4—O3	−179.76 (15)	C7—C8—C9—C10	−0.6 (3)
C10—C5—C6—C7	−1.2 (3)	C7—C8—C9—C12	179.1 (2)
O3—C5—C6—C7	−179.97 (15)	O3—C5—C10—C9	178.86 (16)
C10—C5—C6—C3	178.44 (17)	C6—C5—C10—C9	0.2 (3)
O3—C5—C6—C3	−0.37 (19)	C8—C9—C10—C5	0.6 (3)
C4—C3—C6—C5	0.13 (19)	C12—C9—C10—C5	−179.02 (19)
C2—C3—C6—C5	−179.96 (17)	C3—C4—O3—C5	−0.4 (2)
C4—C3—C6—C7	179.6 (2)	C10—C5—O3—C4	−178.36 (17)
C2—C3—C6—C7	−0.5 (3)	C6—C5—O3—C4	0.46 (19)
C5—C6—C7—C8	1.2 (2)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C4—H4···O3^{i}	0.93	2.58	3.328 (2)	137
O2—H2···O1^{ii}	0.82	1.83	2.645 (2)	171

Symmetry codes: (i) −*x*+3/2, *y*−1/2, −*z*+1/2; (ii) −*x*+1, −*y*, −*z*.