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2-(6-Fluoro-5-hydroxy-2-methyl-1*H*-3-indenyl)acetic acid

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The bicylic system in the title compound, $C_{12}H_{11}FO_3$, is effectively planar, with the methylcarboxylic acid residue being located above and perpendicular to this portion of the molecule. The crystal structure is stabilized *via* a network of hydrogen-bonding and C—H$\cdots\pi$ interactions.

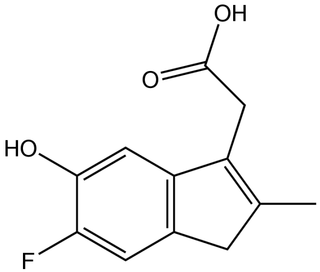
Key indicators

Single-crystal X-ray study
T = 173 K
Mean σ(C–C) = 0.003 Å
R factor = 0.047
wR factor = 0.141
Data-to-parameter ratio = 16.0

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Comment

The nine atoms comprising the bicyclic system in the title compound, (I) (Fig. 1), are coplanar, with the maximum deviation out of their least-squares plane of 0.012 (2) Å being found for the C5 atom. The distribution of bond distances (Table 1) suggests extensive delocalization of π-electron density over all non-H atoms comprising the bicyclic system. The respective C1—C2—C3—C31, C2—C3—C31—C32 and C3—C31—C32—O32*a* torsion angles of −177.75 (18), −99.4 (2) and 17.1 (3)° show that the methylcarboxylic acid residue lies to one side and approximately perpendicular to the five-membered ring. In the crystal structure, centrosymmetrically related molecules associate *via* the familiar carboxylic acid dimer motif (Fig. 1) so that O32—H$\cdots$O32$a^{\rm i}$ is 1.69 Å, O32$\cdots$O32$a^{\rm i}$ is 2.652 (2) Å and the angle at H is 175° [symmetry code: (i) $-x$, -y, 1-z]. The O32*a* atom also forms a weaker interaction with the hydroxyl–H atom so that O5—H$\cdots$O32$a^{\rm ii}$ is 2.11 Å, O5$\cdots$O32$a^{\rm ii}$ is 2.878 (3) Å and the angle at H is 143° [symmetry code: (ii) $\frac{1}{2}-x$, $-\frac{1}{2}+y$, $\frac{1}{2}-z$]. In addition, the hydroxyl–H atom also forms an intramolecular interaction with the fluoride so that H$\cdots$F6 is 2.39 Å with an angle at H of 103 Å.



(I)

The structure also features C—H...π interactions of note. Each of the ring methylene H atoms participates in a C—H...π contact so that this group bridges the six-membered ring of one molecule *via* H1B (the C—H1B...ring centroid distance is 2.70 Å with an angle at H of 160° for symmetry operation $\frac{1}{2}-x, \frac{1}{2}+y, \frac{1}{2}-z$) and the five-membered ring of another *via* H1*A* (2.75 Å, 151° for $-x, 1-y, -z$).

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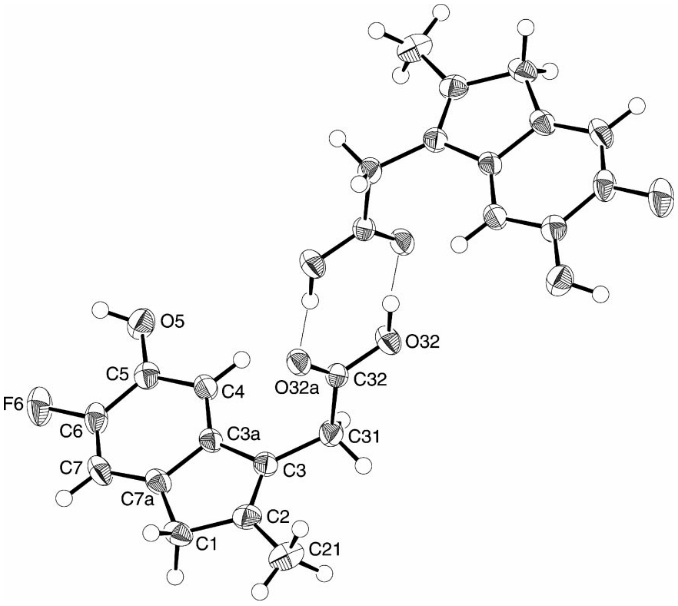

Figure 1
The molecular structure and crystallographic numbering scheme for (I). Displacement ellipsoids are shown at the 50% probability level (Johnson, 1976).

Experimental

The title compound, (I), was prepared from 4-(chloromethyl)-2-fluoro-1-methoxybenzene in an eight-step sequence. The key features of this synthesis were addition of a malonic ester enolate and a Friedel–Crafts ring closure, followed by addition of an acetate enolate and dehydration. Recrystallization from ethyl acetate/hexane (2:1) gave colourless crystals, m.p. 443–445 K (decomposition). ^{1}H NMR (CDCl$_3$, 300 MHz): δ 2.08, *s*, 3H; 3.26, *s*, 2H; 3.50, *s*, 2H; 6.88, *d*, J = 8 Hz, 1 H; 7.08, *d*, J = 10 Hz, 1H.

Crystal data

$C_{12}H_{11}FO_3$
$M_r = 222.22$
Monoclinic, $P2_1/n$
$a = 11.662$ (3) Å
$b = 7.579$ (2) Å
$c = 12.038$ (6) Å
$\beta = 106.89$ (3)°
$V = 1018.0$ (5) Å^3
$Z = 4$

$D_x = 1.450$ Mg m^{-3}
Mo Kα radiation
Cell parameters from 25 reflections
$\theta = 7.7$–12.0°
$\mu = 0.12$ mm^{-1}
$T = 173$ K
Block, pale yellow
0.40 × 0.32 × 0.24 mm

Data collection

Rigaku AFC-7*R* diffractometer
ω–2θ scans
5024 measured reflections
2337 independent reflections
1565 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.031$
$\theta_{\rm max} = 27.5$°

$h = -15 \rightarrow 15$
$k = 0 \rightarrow 9$
$l = 0 \rightarrow 15$
3 standard reflections every 400 reflections intensity decay: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.047$
$wR(F^2) = 0.141$
$S = 1.02$
2337 reflections
146 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0743P)^2 + 0.3569P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\rm max} < 0.001$
$\Delta\rho_{\rm max} = 0.38$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.32$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

F6—C6	1.368 (2)	C3a—C7a	1.403 (3)
O5—C5	1.361 (3)	C3a—C3	1.473 (3)
O32a—C32	1.223 (2)	C3—C31	1.500 (3)
O32—C32	1.316 (2)	C4—C5	1.394 (3)
C1—C7a	1.495 (3)	C5—C6	1.396 (3)
C1—C2	1.500 (3)	C6—C7	1.371 (3)
C2—C3	1.344 (3)	C7a—C7	1.385 (3)
C2—C21	1.498 (3)	C31—C32	1.501 (3)
C3a—C4	1.388 (3)		
C7a—C1—C2	103.33 (17)	C6—C5—C4	118.00 (19)
C3—C2—C21	127.96 (19)	F6—C6—C5	116.63 (19)
C3—C2—C1	110.43 (18)	F6—C6—C7	119.61 (18)
C21—C2—C1	121.6 (2)	C5—C6—C7	123.76 (18)
C4—C3a—C7a	121.20 (18)	C7—C7a—C3a	119.93 (19)
C4—C3a—C3	131.18 (17)	C7—C7a—C1	131.01 (18)
C7a—C3a—C3	107.61 (18)	C3a—C7a—C1	109.05 (17)
C2—C3—C3a	109.57 (17)	C6—C7—C7a	117.88 (18)
C2—C3—C31	127.18 (19)	C32—C31—C3	114.48 (16)
C3a—C3—C31	123.24 (18)	O32a—C32—O32	123.14 (17)
C3a—C4—C5	119.20 (18)	O32a—C32—C31	124.20 (18)
O5—C5—C6	122.11 (18)	O32—C32—C31	112.65 (17)
O5—C5—C4	119.88 (18)		

The C-bound H atoms were placed in geometrically calculated positions and included in the final refinement in the riding model approximation with an overall displacement parameter, $U_{\rm iso}$, with $1.25U_{\rm iso}$ for CH$_2$ and $1.5U_{\rm iso}$ for CH$_3$. The O—H atoms were located from a difference map and were assigned $1.25U_{\rm iso}$.

Data collection: *MSC/AFC Diffractometer Control Software* (Molecular Structure Corporation, 1996); cell refinement: *MSC/AFC Diffractometer Control Software*; data reduction: *TEXSAN for Windows* (Molecular Structure Corporation, 1997); program(s) used to solve structure: *SIR*97 (Altomare *et al.*, 1994); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); software used to prepare material for publication: *SHELXL*97.

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