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Methyl 2-benzyloxy-6-(2-methyl-1,3-dioxolan-2-yl)benzoate monohydrate

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Key indicators

Single-crystal X-ray study
T = 292 K
Mean σ(C–C) = 0.002 Å
R factor = 0.045
wR factor = 0.155
Data-to-parameter ratio = 15.6

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

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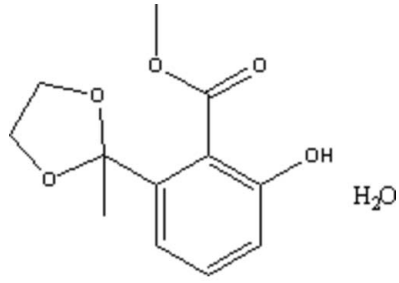
The crystal structure of the title compound, $C_{12}H_{14}O_5\cdot H_2O$, displays a supramolecular structure *via* O—H···O hydrogen bonds. No π–π stacking is observed.

Comment

The title compound, (I) (Fig. 1), is a key intermediate in the synthesis of pesticides such as the herbicide KIH-6127 (Tamaru & Saito, 1996). In this paper, we report the results of an X-ray diffraction study of this compound, as its monohydrate. The five-membered heterocycle adopts an envelope conformation, with C10 as the flap atom, and is twisted around the C5—C10 bond to lie approximately perpendicular to the benzene ring.



(I)

In the crystal structure, strong hydrogen-bonding interactions are observed (Fig. 2 and Table 1). Each ester molecule forms three hydrogen bonds with three solvent water molecules. No π–π stacking is observed.

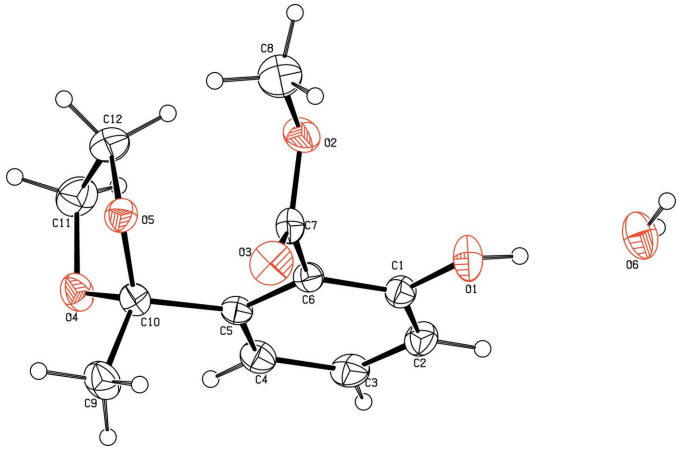


Figure 1
View of the asymmetric unit of (I), showing the atom-labeling scheme. Displacement ellipsoids are drawn at the 50% probability level and H atoms are represented by circles of arbitrary size.

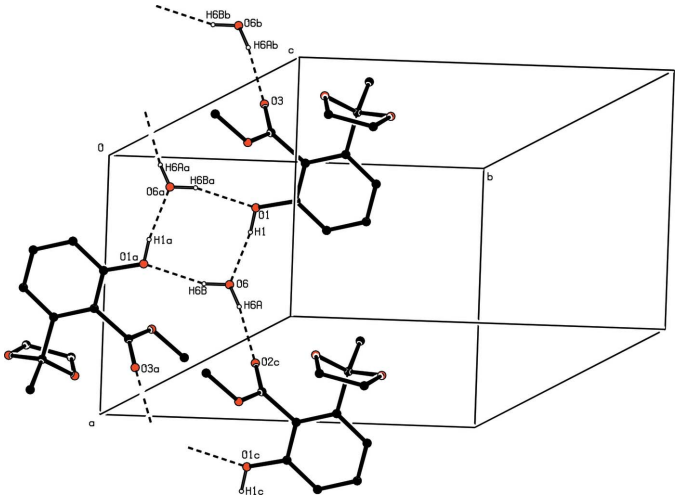


Figure 2
Hydrogen bonding in the crystal structure of (I). Hydrogen bonds are shown as dashed lines. [Symmetry codes: (*a*) $1-x, -y, 1-z$; (*b*) $-1+x, y, z$; (*c*) $1+x, y, z$.] H atoms not involved in hydrogen bonding have been omitted for clarity.

Experimental

The title compound was synthesized according to the literature procedure of Tamaru & Saito (1996). Crystals appropriate for data collection were obtained by slow evaporation of an isopropyl ether solution at 298 K.

Crystal data

$C_{12}H_{14}O_5\cdot H_2O$
$M_r = 256.25$
Monoclinic, $P2_1/n$
$a = 8.3939$ (10) Å
$b = 15.8376$ (19) Å
$c = 9.9983$ (12) Å
$\beta = 109.821$ (2)°
$V = 1250.4$ (3) Å^3
$Z = 4$
$D_x = 1.361$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 2626 reflections
$\theta = 2.5$–26.9°
$\mu = 0.11$ mm^{-1}
$T = 292$ (2) K
Block, colorless
0.30 × 0.20 × 0.20 mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: none
7256 measured reflections
2718 independent reflections
2197 reflections with $I > 2\sigma(I)$
$R_{int} = 0.022$
$\theta_{max} = 27.0°$
$h = -10 \rightarrow 10$
$k = -17 \rightarrow 20$
$l = -9 \rightarrow 12$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.045$
$wR(F^2) = 0.156$
$S = 1.14$
2718 reflections
174 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0916P)^2 + 0.0507P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.35$ e Å^{-3}
$\Delta\rho_{min} = -0.27$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

C1—O1	1.365 (2)	C7—O2	1.326 (2)
C5—C10	1.532 (2)	C9—C10	1.510 (2)
C6—C7	1.500 (2)	O4—C10	1.4158 (19)
C7—O3	1.203 (2)	C11—O4	1.420 (2)
C5—C6—C7—O3	80.5 (2)	C4—C5—C10—O4	−32.18 (19)
C6—C5—C10—O5	34.01 (19)	C4—C5—C10—C9	89.18 (18)

Table 2
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
O6—H6A···O3^{i}	0.82 (2)	1.97 (1)	2.799 (2)	170 (3)
O6—H6B···O1^{ii}	0.82 (1)	2.08 (1)	2.877 (2)	162 (2)
O1—H1···O6	0.82 (1)	1.85 (1)	2.666 (2)	171 (3)

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

H atoms bound to C atoms were positioned geometrically and treated as riding, with C—H = 0.93–0.97 Å. U_{iso}(H) values were set equal to xU_{eq}(carrier atom), where $x = 1.5$ for CH_3 and $x = 1.2$ for CH_2 and CH. Atoms H1, H6*A* and H6*B* were located in a difference Fourier map and refined with distance restraints of O—H = 0.82 (1) Å and with $U_{iso}(H) = 1.5U_{eq}(O)$.

Data collection: *SMART* (Bruker, 1997); cell refinement: *SAINT* (Bruker, 1999); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 2001); software used to prepare material for publication: *SHELXTL*.

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