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## Key indicators

Single-crystal X-ray study

T = 100 K

Mean  $\sigma(\text{C}-\text{C}) = 0.003 \text{ \AA}$ 

R factor = 0.058

wR factor = 0.116

Data-to-parameter ratio = 13.6

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

## 6-Acetyl-5-hydroxy-2,2-dimethyl-2H-chromene

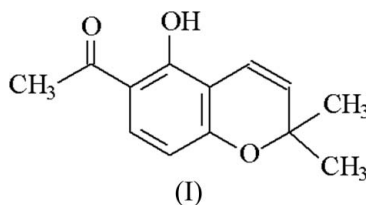
The title compound,  $\text{C}_{13}\text{H}_{14}\text{O}_3$ , is a natural product isolated from *Brickellia cavanillesii* and is of interest with respect to its biological activity. The structure displays an intramolecular  $\text{O}-\text{H}\cdots\text{O}$  hydrogen bond and the crystal structure is stabilized by intermolecular  $\text{C}-\text{H}\cdots\pi$  and  $\text{C}-\text{H}\cdots\text{O}$  interactions. The pyran ring exists in the half-chair conformation.

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## Comment

The title chromene, (I), reported as an intermediate in the synthesis of isoencecalin (Ahluwalia & Arora, 1981), was later isolated from *Blepharispermum subsessile* (Kulkarni *et al.*, 1987) and showed antifungal activity against *Candida albicans* and *Cryptococcus neoformans* and antifeedant activity against *Spilarctia obliqua* (Agarwal *et al.*, 2000). In our ongoing studies of natural products with biological activity we isolated the chromene (I) from *Brickellia cavanillesii*, a plant used in traditional Mexican medicine to alleviate some gastrointestinal problems.



In the crystal structure of (I) (Fig. 1), the pyran ring exists in the half-chair form, with a  $\text{C}14-\text{C}2-\text{C}3-\text{C}4$  torsion angle of  $90.5(3)^\circ$  (Table 1). An intramolecular  $\text{O}2-\text{H}2\cdots\text{O}3$  hydrogen bond [ $\text{H}2\cdots\text{O}3 = 1.84 \text{ \AA}$ ,  $\text{O}2\cdots\text{O}3 = 2.558(2) \text{ \AA}$  and  $\text{O}2-\text{H}2\cdots\text{O}3 = 146^\circ$ ] is also present.

The crystal structure is stabilized by weak intermolecular hydrogen bonds  $\text{C}8-\text{H}8\cdots\text{O}2^i$  [ $\text{H}8\cdots\text{O}2^i = 2.59 \text{ \AA}$ ,  $\text{C}8\cdots\text{O}2^i = 3.480(3) \text{ \AA}$  and  $\text{C}8-\text{H}8\cdots\text{O}2^i = 160^\circ$ ; symmetry code: (i)  $-\frac{1}{2} + x, \frac{3}{2} - y, -z$ ] (Desiraju, 1996) and shows edge-to-face  $\text{C}-\text{H}\cdots\pi$  interactions. The distance between the benzene and pyran ring centers in these interactions is  $5.1 \text{ \AA}$  and the shortest  $\text{C}\cdots\text{C}$  distance is  $3.738 \text{ \AA}$  (Hunter, 1994; Adams, *et al.*, 1996) (Fig. 2).

## Experimental

The title chromene was isolated from *Brickellia cavanillesii*. The methanolic extract was subjected to vacuum liquid chromatography ( $11 \times 3 \text{ cm}$ ) over silica gel. Elution with benzene-hexane (1:1) afforded chromene (I) (55 mg). Recrystallization from hexane- $\text{CH}_2\text{Cl}_2$  (75:25) by slow evaporation gave yellow plates (m.p.  $376.6 \text{ K}$ ).

## Crystal data

$C_{13}H_{14}O_3$   
 $M_r = 218.24$   
 Orthorhombic, *Pbca*  
 $a = 10.269 (2) \text{ \AA}$   
 $b = 11.194 (2) \text{ \AA}$   
 $c = 19.006 (4) \text{ \AA}$   
 $V = 2184.8 (7) \text{ \AA}^3$

$Z = 8$   
 $D_x = 1.327 \text{ Mg m}^{-3}$   
 Mo  $K\alpha$  radiation  
 $\mu = 0.09 \text{ mm}^{-1}$   
 $T = 100 (2) \text{ K}$   
 Block, yellow  
 $0.22 \times 0.20 \times 0.10 \text{ mm}$

## Data collection

Bruker SMART CCD area-detector  
 diffractometer  
 $\varphi$  and  $\omega$  scans  
 Absorption correction: none  
 10526 measured reflections

2038 independent reflections  
 1867 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.045$   
 $\theta_{\text{max}} = 25.5^\circ$

## Refinement

Refinement on  $F^2$   
 $R[F^2 > 2\sigma(F^2)] = 0.058$   
 $wR(F^2) = 0.116$   
 $S = 1.23$   
 2038 reflections  
 150 parameters  
 H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0256P)^2 + 1.8648P]$   
 where  $P = (F_o^2 + 2F_c^2)/3$   
 $(\Delta/\sigma)_{\text{max}} < 0.001$   
 $\Delta\rho_{\text{max}} = 0.27 \text{ e \AA}^{-3}$   
 $\Delta\rho_{\text{min}} = -0.17 \text{ e \AA}^{-3}$

Table 1

Selected geometric parameters ( $\text{\AA}$ ,  $^\circ$ ).

C2—O1	1.473 (3)	C11—O3	1.241 (3)
C2—C3	1.503 (3)	O2—H2	0.8200
C3—C4	1.325 (3)		
O1—C2—C3—C4	−28.5 (3)	C13—C2—C3—C4	−143.8 (2)
C14—C2—C3—C4	90.5 (3)	C4—C5—C10—O1	−1.0 (3)

The hydroxy H atom was located in a difference Fourier map; it was then refined as riding with O—H = 0.82  $\text{\AA}$ ; the isotropic displacement parameter was refined freely. All the remaining H atoms were treated as riding, with methyl C—H = 0.96  $\text{\AA}$  and aromatic C—H = 0.93  $\text{\AA}$ , and with  $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$  for aromatic and  $1.5U_{\text{eq}}(\text{C})$  for methyl H atoms.

Data collection: *SMART* (Bruker, 2000); cell refinement: *SAINT-Plus* (Bruker, 2000); data reduction: *SAINT-Plus*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 2000); software used to prepare material for publication: *SHELXTL* and *pubCIF* (Westrip, 2006).

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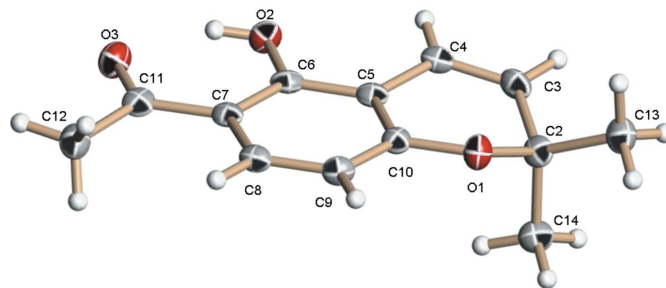


Figure 1

The molecular structure of (I), showing the atom-labeling scheme. Displacement ellipsoids are drawn at the 50% probability level and H atoms are shown as small spheres of arbitrary radii.

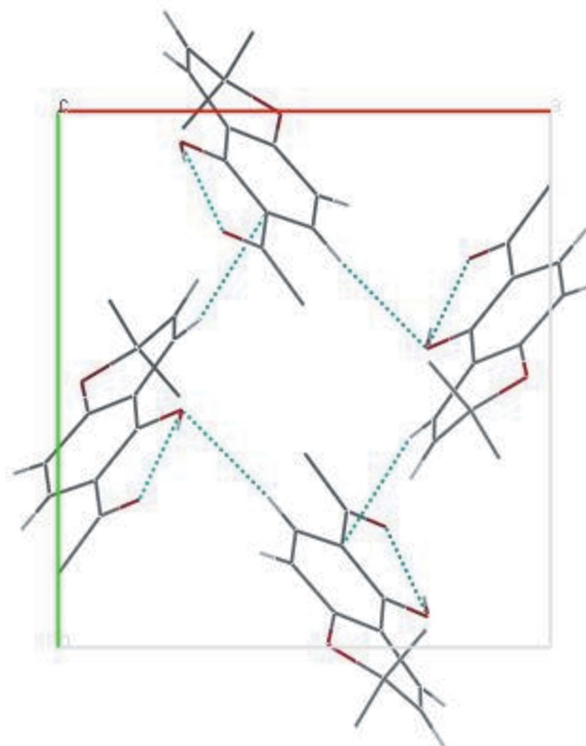


Figure 2

Crystal packing of (I), showing the intermolecular C7—H7...O2 hydrogen bonds and edge-to-face C—H... $\pi$  interactions; these interactions are represented by dashed lines and H atoms not involved in hydrogen bonding have been omitted for clarity.

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