

## Luteolin

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## Key indicators

Single-crystal X-ray study

$T = 120\text{ K}$

Mean  $\sigma(\text{C}-\text{C}) = 0.010\text{ \AA}$

Disorder in solvent or counterion

$R$  factor = 0.093

w $R$  factor = 0.190

Data-to-parameter ratio = 7.2

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

Molecules of the tetrahydroxyflavone luteolin,  $\text{C}_{15}\text{H}_{10}\text{O}_6$ , are essentially planar and contain an intramolecular hydrogen bond. The crystal structure is stabilized by  $\text{O}-\text{H}\cdots\text{O}$ ,  $\text{C}-\text{H}\cdots\text{O}$  and  $\text{C}-\text{H}\cdots\pi$  interactions, and contains disordered water molecules, probably corresponding to a hemihydrate.

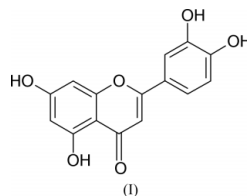
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## Comment

There is an extensive body of literature on the biological activity of luteolin (5,7,3',4'-tetrahydroxyflavone), (I), including a dedicated web site ([www.luteolin.com](http://www.luteolin.com)). Many herbs containing luteolin have been used as traditional medicines to treat a wide variety of symptoms, and anti-inflammatory and anti-oxidant activity are particularly important features of this flavonoid. A recent example of its structure elucidation by spectroscopy has been published (Owen *et al.*, 2003).

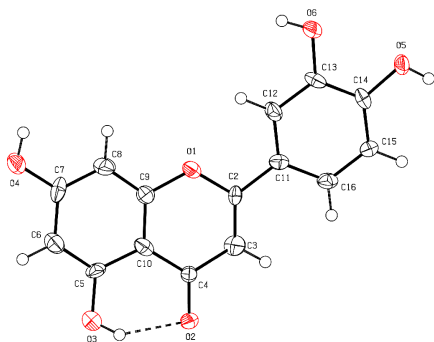


The atomic arrangement in (I) is shown in Fig. 1. The molecule is essentially planar, with a dihedral angle between the endocyclic atoms of the pyrone ring system and the substituted phenyl ring of only  $2.1(3)^\circ$ . The largest torsion angle [ $\text{O}6-\text{C}13-\text{C}14-\text{O}5 = 4.7(9)^\circ$ ] involves hydroxy O atoms associated with intermolecular hydrogen bonding.

Within the crystal structure, hydrogen bonding is extensive (Table 2). An intramolecular hydrogen bond is present between the carbonyl and hydroxy groups at C4 and C5, respectively. The  $\text{H}12\cdots\text{O}1$  separation is also short at  $2.31\text{ \AA}$ , but the  $\text{C}12-\text{H}12\cdots\text{O}1$  angle is only  $101^\circ$ . The hydroxy group at C7 is believed to be linked with the solvent through hydrogen bonding, but the quality of the data cannot confirm this association. Apart from the classical hydrogen bonding, a  $\text{C}-\text{H}\cdots\text{O}$  interaction and a  $\text{C}-\text{H}\cdots\pi$  (Table 3) contact are also present. Crystal structures of related hydroxyflavones include cirsimaritin (Chou *et al.*, 2002), pachypodol (Smith *et al.*, 2001) and 5-hydroxy-3,7,4'-trimethoxyflavone (Gajhede *et al.*, 1989).

## Experimental

The seeds of *Daucus carota* L. (family Apiaceae), commonly known as 'Wild Carrot', were purchased from B&T World Seeds, France. A voucher specimen (PH700 003) has been deposited in the herbarium of the Plant and Soil Science Department, University of Aberdeen,



**Figure 1**  
The molecular structure of (I). Displacement ellipsoids are drawn at the 50% probability level.

Scotland (ABD). Ground seeds were Soxhlet-extracted, successively, with *n*-hexane, dichloromethane and methanol (1.1 l each). The methanol extract was subjected to Sep-Pak (C<sub>18</sub>, 10 g cartridge) fractionation using 30, 40, 60, 80 and 100% aqueous MeOH (200 ml each). The preparative HPLC analysis (A Luna C<sub>18</sub> preparative column 10  $\mu$ , 250  $\times$  21.2 mm; mobile phase: 30–80% MeOH in water gradient over 50 min, flow rate = 20 ml min<sup>-1</sup>) of the Sep-Pak 60% MeOH fraction yielded luteolin (19 mg, retention time = 26 min), the structure of which was preliminarily determined by UV, MS, <sup>1</sup>H and <sup>13</sup>C NMR data analyses.

#### Crystal data

C <sub>15</sub> H <sub>10</sub> O <sub>6</sub>	$D_x = 1.463 \text{ Mg m}^{-3}$
$M_r = 286.23$	Mo $K\alpha$ radiation
Monoclinic, C2	Cell parameters from 8254 reflections
$a = 35.327 (5) \text{ \AA}$	$\theta = 2.9\text{--}27.5^\circ$
$b = 5.618 (1) \text{ \AA}$	$\mu = 0.12 \text{ mm}^{-1}$
$c = 6.682 (1) \text{ \AA}$	$T = 120 (2) \text{ K}$
$\beta = 101.554 (5)^\circ$	Plate, light brown
$V = 1299.3 (4) \text{ \AA}^3$	$0.40 \times 0.10 \times 0.01 \text{ mm}$
$Z = 4$	

#### Data collection

Nonius KappaCCD area-detector diffractometer	1471 independent reflections
$\varphi$ and $\omega$ scans	1262 reflections with $I > 2\sigma(I)$
Absorption correction: multi-scan (SORTAV; Blessing, 1995, 1997)	$R_{\text{int}} = 0.087$
$T_{\text{min}} = 0.911$ , $T_{\text{max}} = 1.000$	$\theta_{\text{max}} = 27.6^\circ$
8254 measured reflections	$h = -45 \rightarrow 45$
	$k = -7 \rightarrow 7$
	$l = -8 \rightarrow 8$

#### Refinement

Refinement on $F^2$	$w = 1/[\sigma^2(F_o^2) + (0.0452P)^2 + 6.6344P]$
$R[F^2 > 2\sigma(F^2)] = 0.093$	where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.190$	$(\Delta/\sigma)_{\text{max}} = 0$
$S = 1.18$	$\Delta\rho_{\text{max}} = 0.35 \text{ e \AA}^{-3}$
1471 reflections	$\Delta\rho_{\text{min}} = -0.34 \text{ e \AA}^{-3}$
204 parameters	
Only H-atom $U$ 's refined	

**Table 1**  
Selected geometric parameters ( $\text{\AA}$ ,  $^\circ$ ).

O2—C4	1.260 (8)	O5—C14	1.367 (7)
O3—C5	1.354 (8)	O6—C13	1.375 (8)
O4—C7	1.349 (8)	C2—C11	1.457 (9)
C3—C2—C11	127.4 (6)	O1—C2—C11	111.1 (6)
O6—C13—C14—O5	4.7 (9)		

**Table 2**  
Hydrogen-bonding geometry ( $\text{\AA}$ ,  $^\circ$ ).

$D\text{—}H\cdots A$	$D\text{—}H$	$H\cdots A$	$D\cdots A$	$D\text{—}H\cdots A$
O3—H3 $\cdots$ O2	0.84	2.00	2.614 (7)	130
O5—H5 $\cdots$ O5 <sup>i</sup>	0.84	2.26	2.955 (7)	141
O5—H5 $\cdots$ O6 <sup>i</sup>	0.84	2.19	2.862 (7)	137
O6—H6 $\cdots$ O2 <sup>ii</sup>	0.84	1.84	2.658 (7)	165
C3—H3A $\cdots$ O6 <sup>iii</sup>	0.95	2.46	3.159 (9)	130

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

**Table 3**  
C—H $\cdots\pi$  interactions ( $\text{\AA}$ ,  $^\circ$ ).

C—H	$CgI$	Symmetry code	$H\cdots CgI$	C—H $\cdots CgI$	C $\cdots CgI$
C15—H15	1	$\frac{1}{2} - x, \frac{1}{2} + y, 1 - z$	2.87	130	3.553 (7)

Data were collected on a weakly diffracting thin plate over 46 h. H atoms were placed in calculated positions (O—H = 0.84  $\text{\AA}$  and C—H = 0.95  $\text{\AA}$ ) and allowed to ride on their parent atoms with freely refined  $U_{\text{iso}}(\text{H})$ . The structure contains disordered solvent, which was treated with the *SQUEEZE* procedure in *PLATON* (Spek, 2002). There are two solvent cavities per unit cell, at positions (0, 0.261, 0) and (0.5, 0.761, 0), each with a volume of 35  $\text{\AA}^3$  and each containing approximately 13 electrons. At least two diffuse peaks could be seen in each cavity in a difference map, but the small volume indicates the presence of a single water molecule (10 electrons) disordered in each cavity.

In the absence of elements heavier than oxygen, Friedel pairs were merged prior to refinement. The absolute polarity of the crystal was assigned arbitrarily (for a planar molecule, absolute configuration is meaningless).

The high value of  $R$  relates to the difficulty in obtaining suitable data from a small, thin plate of diffracting material containing diffuse solvent.

Data collection: *DENZO* (Otwinowski & Minor, 1997) and *COLLECT* (Hooft, 1998); cell refinement: *DENZO* and *COLLECT*; data reduction: *DENZO* and *COLLECT*; program(s) used to solve structure: *SIR97* (Altomare *et al.*, 1999); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *PLATON* (Spek, 2002); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

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