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Malabaricone C monohydrate

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Key indicators

Single-crystal X-ray study
T = 299 K
Mean σ(C–C) = 0.003 Å
R factor = 0.048
wR factor = 0.137
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.

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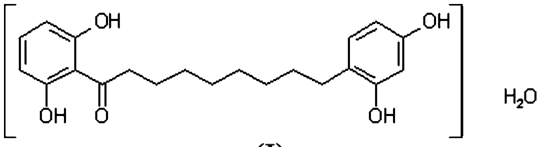
The crystal structure of the title compound, 1-(2,6-dihydroxyphenyl)-9-(3,4-dihydroxyphenyl)nonan-1-one monohydrate, also known as malabaricone C, $C_{21}H_{26}O_5 \cdot H_2O$, is stabilized by both intra- and intermolecular O—H$\cdots$O hydrogen bonds.

Comment

The fruit rind of *Myristica malabarica*, popularly known as Rampatri in Mumbai (India), is used as an exotic spice in various Indian cuisines, as well as in phytomedicine (Forrest & Heacock, 1972, and references therein). It is credited with hepatoprotective, anticarcinogenic and antithrombotic properties and is found as a constituent in many ayurvedic preparations such as Pasupasi. Previous phytochemical investigations of *M. malabarica* fruit rinds revealed the presence of four novel diarylnonanoids named as malabaricone A–D (Purushothaman *et al.*, 1977). In addition, a lignan malabericanol A and an isoflavone were also isolated from heart wood of the plant (Purushothaman *et al.*, 1974; Talukdar *et al.*, 2000). During our studies of the antioxidant activity of methanol extracts of *M. malabarica* fruit rinds, we have isolated malabaricone C as a major product. The compound was assayed against breast and colon cancer cells, and the result was quite promising (Patro *et al.*, 2005).



(I)

A view of the title compound, (I), is shown in Fig. 1 and a packing diagram depicting the hydrogen bonds is shown in Fig. 2. Details of hydrogen bonding are given in Table 1.

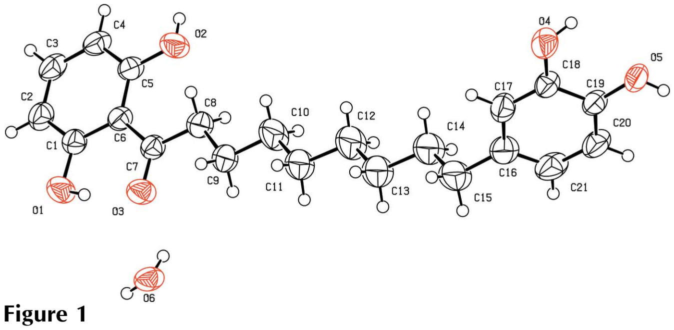


Figure 1
Molecular structure of (I), showing the atom labeling and displacement ellipsoids drawn at the 50% probability level.

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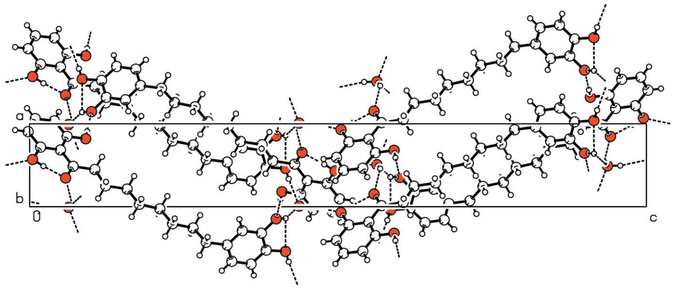

Figure 2
Molecular packing of (I) with hydrogen bonding shown as dashed lines.

Experimental

The title compound was isolated as a major product from a methanol extract of *M. malabarica* by column chromatography over silica gel with gradient elution by changing the polarity of the solvent system using ethyl acetate in petroleum ether followed by purification by preparative thin-layer chromatography. Crystals suitable for X-ray data collection were obtained by recrystallization from aqueous methanol at room temperature by slow evaporation.

Crystal data

$C_{21}H_{26}O_5\cdot H_2O$
$M_r = 376.43$
Orthorhombic, $P2_12_12_1$
$a = 5.4549$ (6) Å
$b = 9.176$ (1) Å
$c = 40.718$ (3) Å
$V = 2038.1$ (3) Å^3
$Z = 4$
$D_x = 1.227$ Mg m^{-3}
Cu $K\alpha$ radiation
$\mu = 0.73$ mm^{-1}
$T = 299$ (2) K
Long needle, dark yellow
0.55 × 0.25 × 0.20 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
$\omega/2\theta$ scans
Absorption correction: none
3822 measured reflections
3566 independent reflections
3323 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.032$
$\theta_{\rm max} = 67.0°$
3 standard reflections
frequency: 120 min
intensity decay: 1%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.048$
$wR(F^2) = 0.137$
$S = 1.04$
3566 reflections
263 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.1014P)^2 + 0.1804P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\rm max} = 0.007$
$\Delta\rho_{\rm max} = 0.20$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.21$ e Å^{-3}
Extinction correction: *SHELXL97*
Extinction coefficient: 0.0035 (6)
Absolute structure: Flack (1983), 1404 Friedel pairs
Flack parameter: −0.1 (2)

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

$D-\mathrm{H}\cdots A$	$D-\mathrm{H}$	$\mathrm{H}\cdots A$	$D\cdots A$	$D-\mathrm{H}\cdots A$
$O1-H1O\cdots O3$	0.86 (2)	1.67 (2)	2.4938 (19)	160 (3)
$O2-H2O\cdots O4^{i}$	0.84 (2)	1.92 (2)	2.748 (2)	173 (3)
$O4-H4O\cdots O6^{ii}$	0.87 (2)	1.98 (2)	2.788 (2)	153 (3)
$O4-H4O\cdots O5$	0.87 (2)	2.34 (3)	2.675 (2)	103 (2)
$O5-H5O\cdots O6^{iii}$	0.80 (2)	1.95 (2)	2.724 (2)	164 (3)
$O6-H61O\cdots O1^{iv}$	0.85 (2)	1.96 (2)	2.8006 (19)	169 (3)
$O6-H62O\cdots O3$	0.82 (2)	2.12 (2)	2.788 (2)	138 (2)

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

The O-bound H atoms were located in a difference map and their coordinates were refined. The H atoms of the water molecule were refined with geometry restraints (Nardelli, 1999), *viz*. O—H distances were restrained to 0.85 (2) Å and the H···H distance was restrained to 1.36 (2) Å. The C-bound H atoms were positioned with idealized geometry using a riding model, with C—H = 0.93 Å (aromatic) and 0.97 Å (methylene groups). For all H atoms, $U_{\rm iso}(\mathrm{H}) = 1.2U_{\rm eq}(\mathrm{C,O})$.

Data collection: *CAD-4-PC* (Enraf–Nonius, 1996); cell refinement: *CAD-4-PC*; data reduction: *REDU4* (Stoe & Cie, 1987); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *PLATON* (Spek, 2003); software used to prepare material for publication: *SHELXL97*.

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