Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

(2*S**,3*S**,4*R**,5*R**)-3,4,5-Trihydroxy-6-(hydroxymethyl)-3,4,5,6-tetrahydro-2*H*-pyran-2-yl benzoate

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Received 7 October 2005
Accepted 23 December 2005
Online 7 January 2006

Key indicators

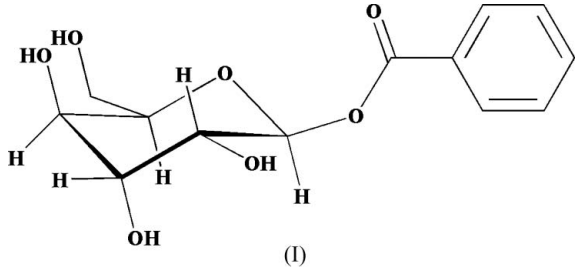
Single-crystal X-ray study
T = 173 K
Mean σ(C–C) = 0.001 Å
Disorder in main residue
R factor = 0.032
wR factor = 0.092
Data-to-parameter ratio = 15.5

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

The title compound, $C_{13}H_{16}O_7$, was extracted from fresh cranberries (*Vaccinium macrocarpon*). In the crystal structure, molecules are linked into a two-dimensional framework *via* O—H···O hydrogen bonds.

Comment

Cranberries (*Vaccinium macrocarpo*n), a native fruit of North America, have attracted public attention due to potential health benefits. Recently, cranberries have been found to be rich in phenolics (Sun *et al.*, 2002), to prevent bacterial adhesion in urinary tract infections of *E. coli* and stomach ulcers (Howell *et al.*, 1998; Foo *et al.*, 2000; Burger *et al.*, 2000), to exhibit *in vitro* anticancer activity (Sun *et al.*, 2002; Bomser *et al.*, 1996) and to protect against lipoprotein oxidation (Chu & Liu, 2005; Wilson *et al.*, 1998). The attractive bright red appearance and distinctive flavor of cranberries is recognized as a concentrated source of dietary flavonoids (Sun *et al.*, 2002), including anthocyanins (Zapsalis & Francis, 1965), proanthocyanidins (Foo & Porter, 1980; Hale *et al.*, 1986) and flavonol glycosides (Puski & Francis, 1967), as well as various phenolic acids (Schmid, 1977).



(I)

In our continuing efforts to seek bioactive components from fruits, vegetables and other natural products, bioactivity-guided fractionation of cranberries was used to determine the identity of the bioactive compounds from cranberries, which inhibit tumor cell growth and may play a role in cancer prevention and therapy (He *et al.*, 2005). The title compound, (I), was isolated from the active fraction of cranberry extracts. This compound may be responsible for the antimicrobial activity of cranberries.

A view of the title molecule is shown in Fig. 1. In the crystal structure, intermolecular O—H···O hydrogen bonds connect molecules into a two-dimensional framework (Table 1).

Experimental

Fresh cranberries of Stevens cultivar (7.0 kg) were homogenized five times with chilled 80% acetone (1:2 *w*/*v*) using a chilled Waring

blender (Sun *et al.*, 2002). The samples were then homogenized for an additional 3 min using a Polytron homogenizer. The homogenates were filtered and the filtrate was evaporated under vacuum at 318 K until approximately 90% of the filtrate had been evaporated. The residue was then recovered with 4000 ml water and extracted with the same volume of ethyl acetate three times, and then water-saturated *n*-butanol three times. The butanol fraction (204.0 g) was subjected to Diaion HP-20 column chromatography (55 × 550 mm) and eluted with 3000 ml water, 30% methanol (*v*/*v*), 50% methanol (*v*/*v*) and methanol. The 30% methanol elution (16.50 g) was further isolated by a silica-gel column (230–400 mesh, 75 × 235 mm) and eluted with dichloromethane–methanol with a gradually increasing proportion of methanol. The title compound (I) (605.2 mg) was obtained from dichloromethane–methanol elution (10:1, *v*/*v*) as colorless crystals. Spectroscopic analysis: ^{1}H NMR (400 MHz, DMSO-d_6, δ,p.p.m.): 8.04 (2H, *dt*, *J* = 8.7 and 1.5 Hz), 7.70 (H, *td*, *J* = 7.5 and 1.5 Hz), 7.56 (2H, *td*, *J* = 7.5 and 1.5 Hz), 5.61 (H, *d*, *J* = 7.5 Hz), 5.45 (H, *d*, *J* = 5.1 Hz), 5.20 (H, *d*, *J* = 4.8 Hz), 5.08 (H, *d*, *J* = 5.4 Hz), 4.62 (H, *t*, *J* = 6.0 Hz), 3.69 (H, *ddd*, *J* = 12.0, 5.4 and 1.5 Hz), 3.49 (H, *p*, *J* = 6.0 Hz), 3.37–3.16 (4H, *m*); ^{13}C NMR (400 MHz, DMSO-d_6, δ, p.p.m.): 164.8 (C), 133.9 (C), 129 (2CH), 129.2 (CH), 128.9 (2CH), 95.1 (CH), 78.0 (CH), 76.4 (CH), 72.6 (CH), 69.5 (CH), and 60.0 (CH_2).

Crystal data

$C_{13}H_{16}O_7$
M_r = 284.26
Orthorhombic, $P2_12_12_1$
a = 5.8022 (2) Å
b = 7.6865 (3) Å
c = 28.3221 (12) Å
V = 1263.13 (8) Å^3
Z = 4
D_x = 1.495 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 8636 reflections
θ = 2.7–37.4°
μ = 0.12 mm^{-1}
T = 173 (2) K
Block, colorless
0.70 × 0.60 × 0.50 mm

Data collection

Bruker X8 APEX-II diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.919, T_{max} = 0.941
15473 measured reflections
3768 independent reflections
3448 reflections with $I > 2\sigma(I)$
R_{int} = 0.024
θ_{max} = 37.8°
h = −10 → 6
k = −13 → 7
l = −48 → 39

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.032
$wR(F^2)$ = 0.092
S = 1.06
3768 reflections
243 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0678P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.41 e Å^{-3}
$\Delta\rho_{min}$ = −0.21 e Å^{-3}

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1*O*···O7^{i}	0.97 (3)	1.81 (3)	2.7607 (10)	166 (2)
O3—H3*O*···O2^{ii}	0.82 (2)	1.88 (2)	2.6774 (10)	166.1 (16)
O4—H4*A*···O1^{iii}	0.84	2.19	3.0121 (12)	167
O4*A*—H4*AA*···O7^{iii}	0.84	2.19	2.953 (11)	151

Symmetry codes: (i) $x+1, y, z$; (ii) $-x+1, y+\frac{1}{2}, -z+\frac{3}{2}$; (iii) $x, y+1, z$.

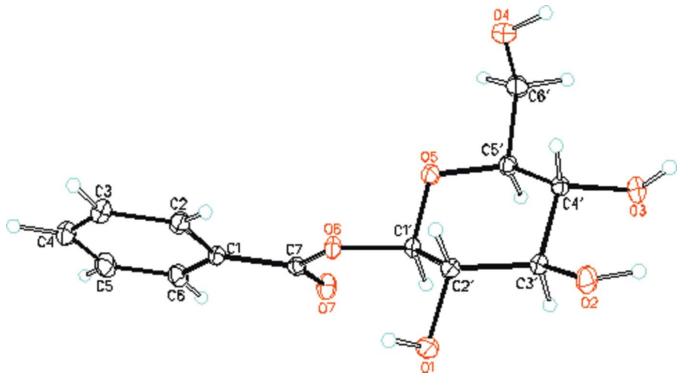


Figure 1
Molecular structure of (I), showing 40% probability displacement ellipsoids and H atoms as spheres of arbitrary radii. Only the major disorder component is shown.

In the absence of significant anomalous dispersion effects, Friedel pairs were merged before the refinement and the assignment of the absolute stereochemistry is arbitrary. The methylhydroxy group containing atom O4 is disordered over two sites with refined occupancies of 0.889 (3) and 0.111 (3) for the major and minor components, respectively. The disorder corresponds to a rotation about the C5′—C6′ bond. The disordered H atoms were included in calculated positions, with C—H = 0.99 Å and U_{iso}(H) = 1.2U_{eq}(C), and O—H = 0.84 Å and U_{iso}(H) = 1.5U_{eq}(O). All other H atoms were refined isotropically. The C—H distances are in the range 0.936 (15)–1.026 (15) Å.

Data collection: *APEX2* (Bruker, 2004); cell refinement: *SAINT-Plus* (Bruker, 2003); data reduction: *SAINT-Plus*; program(s) used to solve structure: *SHELXTL* (Bruker, 1999); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

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