Acta Crystallographica Section E
Structure Reports
Online

ISSN 1600-5368

Propyl gallate dihydrate

Nobuo Okabe* and Hasuyo Kyoyama

Faculty of Pharmaceutical Sciences, Kinki university, Kowakae 3-4-1, Higashiosaka, Osaka 577-8502, Japan

Correspondence e-mail:
okabe@phar.kindai.ac.jp

Received 13 December 2001
Accepted 30 January 2002
Online 8 February 2002

In the crystal structure of the title compound, $C_{10}H_{12}O_5 \cdot 2H_2O$, the molecule of *n*-propyl 3,4,5-trihydroxybenzoate adopts an essentially planar conformation with a fully extended *trans* zigzag propyl ester group. The gallate residue has two intramolecular hydrogen bonds between the hydroxyl groups. The crystal structure is stabilized by hydrogen-bonding interactions, utilizing all available sites, and hydrophobic interactions involving the propyl groups.

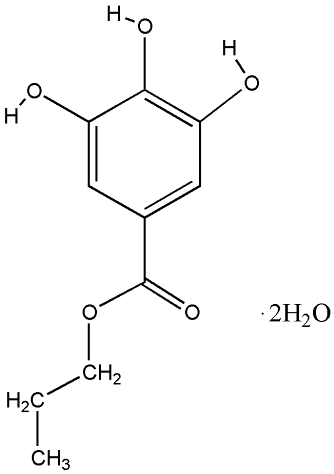
Key indicators

Single-crystal X-ray study
T = 296 K
Mean σ(C–C) = 0.004 Å
R factor = 0.046
wR factor = 0.175
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.

Comment

Gallic acid (3,4,5-trihydroxybenzoic acid) is a naturally occurring plant phenol having antitumor and antioxidative activity. It induces apoptosis in human myelogenenous leukemic cell lines (Sakaguchi *et al.*, 1999; Satoh & Sakagami, 1997) and it is suggested that the hydroxyl substituent plays an important role in its biological activity. We intend to study the structural characteristics of gallic acid derivatives with amphiphilic characteristics by systematically adding alkyl groups. The structure of the *n*-propyl ester of gallic acid (propyl gallate) as its dihydrate, (I), is reported here. The structure of the closely related gallic acid octyl ester (octyl gallate) with a long alkyl group has been determined (Jeffrey & Yeon, 1990).



(I)

The molecular structure of (I) is essentially planar, with a fully extended *trans*-zigzag propyl ester group, as shown in Fig. 1. In the molecule, all H atoms of the three hydroxy groups are oriented in the same direction, forming two intramolecular hydrogen bonds between a pair of hydroxyl groups at the 3- and 4-positions, and at the 4- and 5-positions. This intramol-

DOI: 10.1107/S1600536802002015

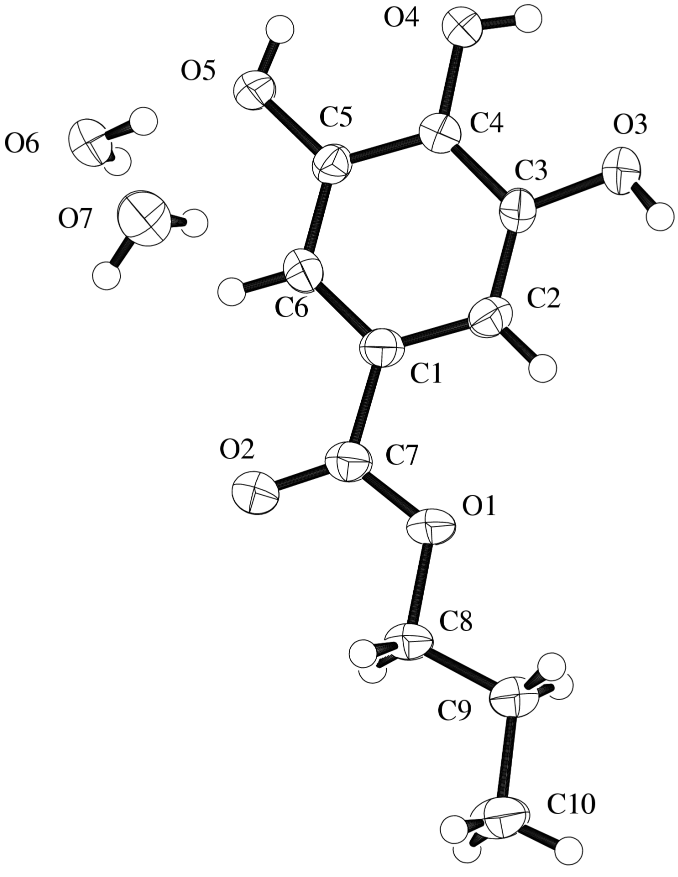


Figure 1
*ORTEP*II (Johnson, 1976) drawing of the title compound, showing the atomic numbering scheme. Ellipsoids for non-H atoms are shown at the 50% probability level.

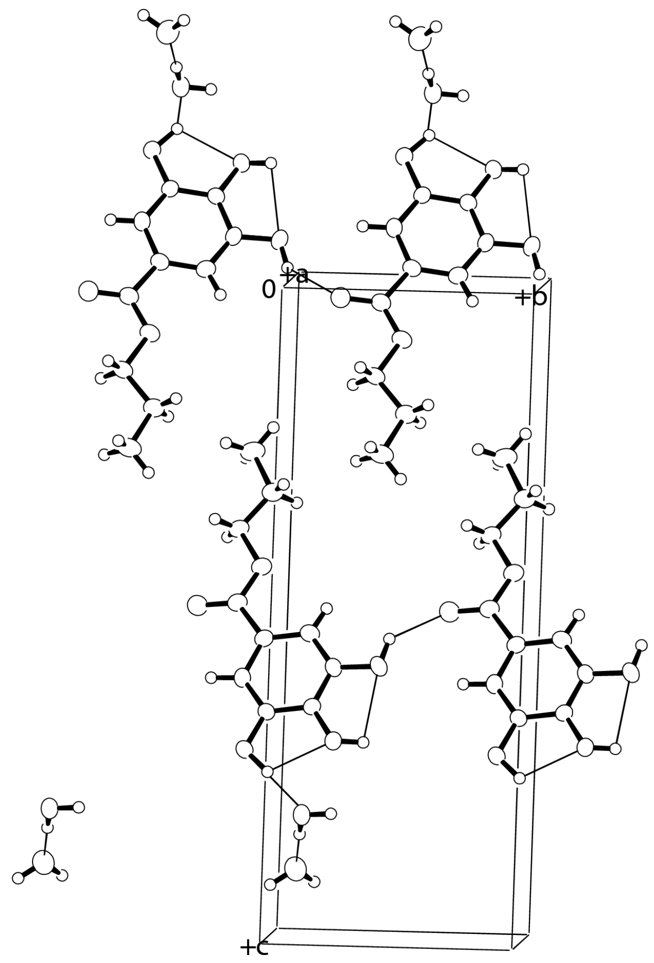


Figure 2
Crystal packing, viewed down the *c* axis, indicating the hydrogen bonds (fine lines) and interactions between the propyl groups.

ecular hydrogen-bonding scheme resembles that found in one polymorphic form of gallic acid monohydrate (Okabe *et al.*, 2001), but is different from that in another form (Jiang *et al.*, 2000). In the latter, one of the three H atoms of the hydroxy groups is oriented in the opposite direction to the others so that only one intramolecular hydrogen bond is present.

In the crystal packing, the methyl groups of the propyl ester regions interact with each other (Fig. 2). The interactions between the methyl groups of the short alkyl groups are different from the interdigitizing interaction with the long alkyl chains of octyl gallate (Jeffrey & Yeon, 1990). There are no stacking interactions between the phenyl rings. The benzene ring is shifted slightly towards the ester bond, as reflected by the C1$\cdots$C1^{i} separation [symmetry code: (i) $1-x$, $1-y$, $-z$] of 3.542 (5) Å and the separations O2$\cdots$C3^{i} of 3.580 (4) Å and O1$\cdots$C5^{i} of 3.492 (3) Å. All of the available hydrogen bonds are present as intra- and intermolecular interactions and are detailed in Table 2.

Experimental

Crystals were obtained by slow evaporation of an ethanol solution of the compound.

Crystal data

$C_{10}H_{12}O_5\cdot 2H_2O$
M_r = 248.23
Monoclinic, $P2_1/c$
a = 7.872 (2) Å
b = 7.560 (2) Å
c = 19.836 (2) Å
β = 101.03 (1)°
V = 1158.7 (4) Å^3
Z = 4

D_x = 1.423 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 25 reflections
θ = 13.7–14.7°
μ = 0.12 mm^{-1}
T = 296.2 K
Plate, colorless
0.30 × 0.30 × 0.20 mm

Data collection

Rigaku AFC-5*R* diffractometer
ω–2θ scans
Absorption correction: none
3072 measured reflections
2660 independent reflections
1259 reflections with $I > 2\sigma(I)$
$R_{\rm int}$ = 0.029

$\theta_{\rm max}$ = 27.5°
$h = 0 \rightarrow 10$
$k = 0 \rightarrow 9$
$l = -25 \rightarrow 25$
3 standard reflections
every 150 reflections
intensity decay: 0.8%

Refinement

Refinement on F^2
$R[(F^2) > 2\sigma(F^2)] = 0.046$
$wR(F^2) = 0.175$
S = 0.92
2660 reflections
170 parameters

H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.1P)^2]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\rm max} = 0.001$
$\Delta\rho_{\rm max}$ = 0.25 e Å^{-3}
$\Delta\rho_{\rm min}$ = −0.33 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

O1—C7	1.326 (3)	O4—C4	1.354 (3)
O1—C8	1.456 (4)	O5—C5	1.362 (3)
O2—C7	1.221 (4)	C1—C7	1.480 (4)
O3—C3	1.379 (3)		
C7—O1—C8	117.9 (2)	O5—C5—C4	120.9 (2)
C6—C1—C7	118.5 (2)	O5—C5—C6	119.0 (2)
O3—C3—C2	124.6 (2)	O1—C7—O2	123.3 (3)
O3—C3—C4	114.6 (2)	O1—C7—C1	112.6 (2)
O4—C4—C3	123.7 (2)	O2—C7—C1	124.1 (3)
O4—C4—C5	116.9 (2)		
O1—C7—C1—C6	178.2 (2)	O2—C7—C1—C2	178.6 (3)
O1—C8—C9—C10	178.2 (2)	C1—C7—O1—C8	−179.0 (2)
O2—C7—O1—C8	0.8 (4)		

Table 2
Hydrogen-bonding angles (°).

O4—H4···O3	113	O6—H14···O5	168 (4)
O5—H5···O4	113	O6—H15···O7	168 (4)
O3—H3···O2$^{\rm i}$	152	O7—H16···O2$^{\rm iii}$	166 (4)
O5—H5···O6$^{\rm ii}$	155	O7—H17···O2$^{\rm iv}$	167

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

The C-bound H atoms were placed in geometrically calculated positions and included in the final refinement in the riding-model approximation, with an overall $U_{\rm iso}$ value of $U_{\rm eq}$(C) for phenyl H atoms and 1.25 × $U_{\rm eq}$(C) for methylene H atoms. The H atoms bound to O atoms were located from a difference map but were included in idealized positions.

Data collection: *MSC/AFC Diffractometer Control Software* (Molecular Structure Corporation and Rigaku Corporation, 1999*a*); cell refinement: *MSC/AFC Diffractometer Control Software*; data reduction: *TEXSAN* (Molecular Structure Corporation and Rigaku Corporation, 1999*b*); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); software used to prepare material for publication: *TEXSAN*.

References

Jeffrey, G. A. & Yeon, Y. (1990). *Acta Cryst.* B**46**, 519–524.

Jiang, R.-W., Ming, D.-S., But, P. P. H. & Mak, T. C. W. (2000). *Acta Cryst.* C**56**, 594–595.

Johnson, C. K. (1976). *ORTEP*II. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.

Molecular Structure Corporation & Rigaku Corporation (1999*a*). *MSC/AFC Diffractometer Control Software*. MSC, 9009 New Trails Drive, The Woodlands, TX 77381-5209, USA, and Rigaku, 3-9-12 Akishima, Tokyo, Japan.

Molecular Structure Corporation & Rigaku Corporation (1999*b*). *TEXSAN*. Version 1.10. MSC, 9009 New Trails Drive, The Woodlands, TX 77381-5209, USA, and Rigaku, 3-9-12 Akishima, Tokyo, Japan.

Okabe, N., Kyoyama, H. & Suzuki, M. (2001). *Acta Cryst.* E**57**, o764–o766.

Satoh, K. & Sakagami, H. (1997). *Anticancer Res.* **17**, 2181–2184.

Sakaguchi, N., Inoue, M., Isuzugawa, K., Ogihara, Y. & Hosaka, K. (1999). *Biol. Pharm. Bull.* **22**, 471–475.

Sheldrick, G. M. (1997). *SHELXS*97 and *SHELXL*97. University of Göttingen, Germany.