n-Propyl gallate–acetonitrile (2/1)
Jin Mizuguchi, Akio Hitachi, Shohei Iwata and Takashi Makino
\( n \)-Propyl gallate–acetonitrile (2/1)

\( n \)-Propyl gallate (PG) is a charge-control agent used for toners in electrophotography, and shows pseudo-polymorphism. In the asymmetric unit of the title compound, \( 2C_{10}H_{12}O_5\cdot CH_3CN \), there are two PG molecules and one solvent molecule. There are intra- and intermolecular O—H—O hydrogen bonds, forming a three-dimensional hydrogen-bond network.

Comment

PG is widely used as a charge-control agent (CCA) of the negative type for toners in electrophotography, as well as the colour former of leuco dyes. The acidic nature of PG plays an important role in these applications. The background of the present study has been set out in our previous paper (Iwata et al., 2005). Recently, we have isolated three kinds of solvated PG crystals, namely compound (I), 3PG\( \cdot 0.5\)chloroform (Iwata et al., 2005), compound (II), 3PG\( \cdot 0.5\)dichloromethane (Hitachi et al., 2005), and the title compound, (III), 2PG-acetonitrile (this publication).

![ORTEPIII plot](https://example.com/ortep.png)

Fig. 1 shows an ORTEPIII plot (Burnett & Johnson, 1996) of (III). There are two independent PG molecules (A and B) and one solvent molecule in the asymmetric unit. In molecule A, all OH groups are bent in the opposite direction to the alkyl chain. This is in contrast with the situation in (I) and (II), where all OH groups are bent in the same direction as the alkyl chain. In molecule B, however, two OH groups (O6—H6 and O7—H7) are bent in the opposite direction to the alkyl chain, while one OH group (O8—H8) is bent in the same direction as the alkyl chain. Furthermore, atom H3O in molecule A and atoms H6O and H8O in molecule B deviate considerably from the PG molecular planes, due to the formation of intermolecular O—H—O hydrogen bonds, as described below.

There are intra- and intermolecular O—H—O hydrogen bonds (Table 2), as found in compounds (I) and (II). However, compound (III) is characterized by a three-dimensional hydrogen-bond network, as shown in Fig. 2. The deviation of atom H3O in molecule A from the molecular plane, as well as of atoms H6O and H8O in molecule B, is clearly recognized as

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**Acta Cryst.** (2005). **E61**, o2593–o2595

Received 20 June 2005

Accepted 11 July 2005

Online 16 July 2005

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designated by ‘downward’ or ‘upward’ bending, and this is caused by the formation of intermolecular O—H···O hydrogen bonds. In addition, the present three-dimensional hydrogen-bond network contrasts strikingly with the two-dimensional one in (I) and (II). Here again, the acid strength of PG required for applications as CCAs and colour formers is considerably weakened by the formation of intra- and intermolecular O—H···O hydrogen bonds. Fig. 3 shows the crystal structure of (III).

**Experimental**

PG was purchased from Kanto Chemicals Ltd. and used without further purification. Single crystals of (III) were grown from an acetonitrile solution by slow evaporation. After a week, a number of colourless transparent crystals were obtained in the form of blocks.

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**Figure 1**  
A view of the molecular conformation of (III), showing 50% probability displacement ellipsoids.

**Figure 2**  
The hydrogen-bond network (dashed lines) of PG in (III). H atoms that participate in the formation of O—H···O intermolecular hydrogen bonds deviate from the molecular plane. These are designated by ‘downward’ or ‘upward’ bending.

**Figure 3**  
The crystal structure of (III). H atoms bonded to C atoms have been omitted for clarity.

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**Crystal data**

$2\mathrm{C}_{10}\mathrm{H}_{12}\mathrm{O}_{5}\mathrm{C}_{2}\mathrm{H}_{3}\mathrm{~N}$

$M_r = 465.45$

Triclinic, $P\overline{1}$

$a = 8.5884$ (11) Å

$b = 11.3727$ (13) Å

$c = 12.5530$ (14) Å

$\alpha = 67.648$ (9)$^\circ$

$\beta = 74.069$ (10)$^\circ$

$\gamma = 81.488$ (11)$^\circ$

$V = 1089.1$ (2) Å$^3$

$Z = 2$

$D_{r} = 1.419$ Mg m$^{-3}$

Cu Kα radiation

Cell parameters from 18945 reflections

$\theta = 3.9\text{–}68.2^\circ$

$\mu = 0.96$ mm$^{-1}$

$T = 93.1$ K

Block, colourless

$0.10 \times 0.10 \times 0.10$ mm

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**Data collection**

Rigaku R-AXIS RAPID-F imaging-plate diffractometer

$\omega$ scans

Absorption correction: multi-scan

(ABSCOR; Higashi, 1995)

$\omega = 0.898$, $\omega_{max} = 0.909$

9830 measured reflections

$R_{int} = 0.045$

3588 independent reflections

3257 reflections with $F^2 > 2\sigma(F^2)$

$\theta_{max} = 68.2^\circ$

$h = -10 \rightarrow 10$

$k = -13 \rightarrow 13$

$l = -14 \rightarrow 15$
Refinement

Refinement on $F^2$

$R[F^2 > 2\sigma(F^2)] = 0.047$

$wR^2(F^2) = 0.134$

$S = 1.89$

3588 reflections

310 parameters

H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.05\max(F_o^2,F_c^2))^2]$

$(\Delta \sigma)_{max} = 0.002$

$\Delta \rho_{max} = 0.49 \text{ e} \ 	ext{Å}^{-3}$

$\Delta \rho_{min} = -0.43 \text{ e} \ 	ext{Å}^{-3}$

Table 1

Selected bond lengths (Å).

<table>
<thead>
<tr>
<th>Bond</th>
<th>Length (Å)</th>
</tr>
</thead>
<tbody>
<tr>
<td>O1–C1</td>
<td>1.365 (2)</td>
</tr>
<tr>
<td>O2–C2</td>
<td>1.3508 (17)</td>
</tr>
<tr>
<td>O3–C3</td>
<td>1.375 (2)</td>
</tr>
<tr>
<td>O6–C11</td>
<td>1.367 (2)</td>
</tr>
<tr>
<td>O7–C12</td>
<td>1.359 (2)</td>
</tr>
<tr>
<td>O8–C13</td>
<td>1.382 (2)</td>
</tr>
</tbody>
</table>

Table 2

Hydrogen-bond geometry (Å, °).

<table>
<thead>
<tr>
<th>H···A</th>
<th>D–H</th>
<th>H···A</th>
<th>D–A</th>
<th>D···A</th>
</tr>
</thead>
<tbody>
<tr>
<td>O1–H1O···O2</td>
<td>0.82 (2)</td>
<td>2.20 (2)</td>
<td>2.677 (2)</td>
<td>117 (2)</td>
</tr>
<tr>
<td>O1–H1O···N1i</td>
<td>0.82 (2)</td>
<td>2.39 (2)</td>
<td>3.1675 (19)</td>
<td>158 (2)</td>
</tr>
<tr>
<td>O2–H2O···O3</td>
<td>0.83 (2)</td>
<td>2.31 (2)</td>
<td>2.735 (2)</td>
<td>112 (2)</td>
</tr>
<tr>
<td>O2–H2O···O9ii</td>
<td>0.87 (2)</td>
<td>1.86 (2)</td>
<td>2.7346 (17)</td>
<td>153 (2)</td>
</tr>
<tr>
<td>O3–H3O···O7</td>
<td>0.75 (2)</td>
<td>2.16 (2)</td>
<td>2.761 (2)</td>
<td>137 (2)</td>
</tr>
<tr>
<td>O3–H3O···O8</td>
<td>0.75 (2)</td>
<td>2.23 (2)</td>
<td>2.8060 (18)</td>
<td>142 (2)</td>
</tr>
<tr>
<td>O6–H6O···O4iv</td>
<td>0.88 (2)</td>
<td>2.28 (2)</td>
<td>2.698 (2)</td>
<td>109 (2)</td>
</tr>
<tr>
<td>O7–H7O···O3iii</td>
<td>0.88 (2)</td>
<td>2.04 (2)</td>
<td>2.8528 (15)</td>
<td>164 (2)</td>
</tr>
</tbody>
</table>

Symmetry codes: (i) x+1, y, z; (ii) x, y, z+1; (iii) x+2, y, z+1; (iv) x+1, y, z.

H atoms attached to O atoms were found in difference density maps and their coordinates were refined, with $U_{eq}(H) = 0.027 \text{ Å}^2$. All other H atoms were positioned geometrically and included in a riding-model approximation, with C–H = 0.95 Å, and with $U_{eq}(H) = 1.2U_{eq}(C)$.

Data collection: PROCESS-AUTO (Rigaku, 1998); cell refinement: PROCESS-AUTO; data reduction: TEXSAN (Molecular Structure Corporation, 2001); program(s) used to solve structure: SHELXS86 (Sheldrick, 1985); program(s) used to refine structure: TEXSAN; molecular graphics: ORTEPIII (Burnett & Johnson, 1996); software used to prepare material for publication: Crystal-Structure (Rigaku/MSC, 2005).

References


