Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

n-Propyl gallate–acetonitrile (2/1)

Jin Mizuguchi,[a]* Akio Hitachi,[b] Shohei Iwata[a] and Takashi Makino[a]

[a]Department of Applied Physics, Graduate School of Engineering, Yokohama National University, Tokiwadai 79-5, Hodogaya-ku, Yokohama 240-8501, Japan, and [b]Sony Corporation, Media Format Department, 6-7-35 Kitashinagawa, Shinagawa-ku, Tokyo 141-0001, Japan

Correspondence e-mail: mizu-j@ynu.ac.jp

Received 20 June 2005
Accepted 11 July 2005
Online 16 July 2005

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.

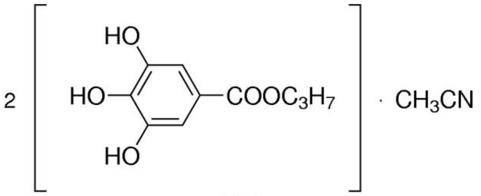
Key indicators

Single-crystal X-ray study
T = 93 K
Mean σ(C–C) = 0.002 Å
R factor = 0.047
wR factor = 0.134
Data-to-parameter ratio = 11.6

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

Comment

PG is widely used as a charge-control agent (CCA) of the negative type for toners in electrophotograpy, 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·0.5chloroform (Iwata *et al.*, 2005), compound (II), 3PG·0.5dichloromethane (Hitachi *et al.*, 2005), and the title compound, (III), 2PG·acetonitrile (this publication).



(III)

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

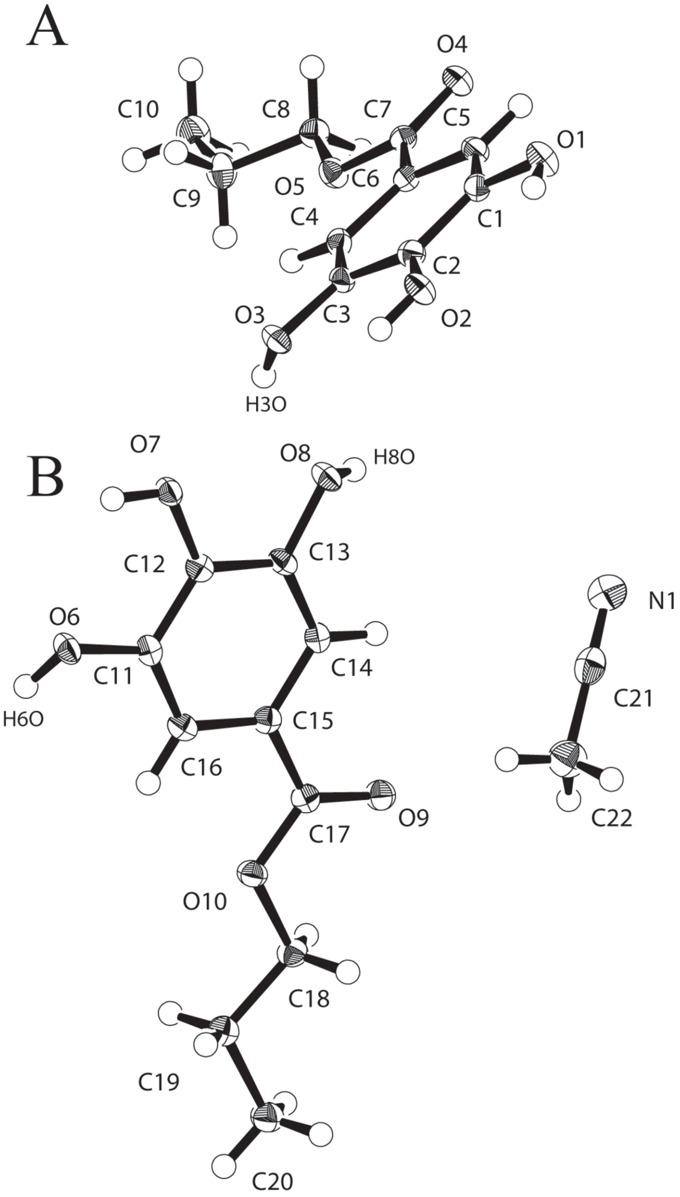


Figure 1
A view of the molecular conformation of (III), showing 50% probability displacement ellipsoids.

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.

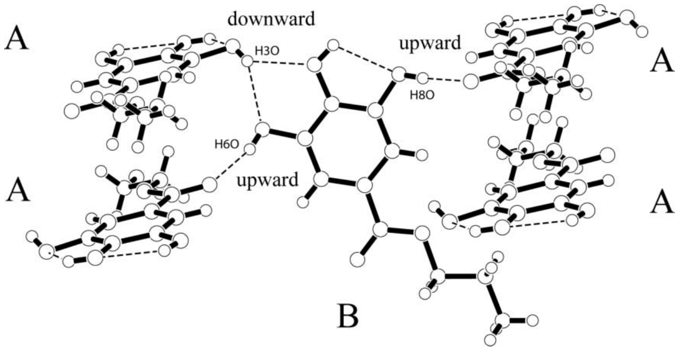


Figure 2
The hydrogen-bond network (dashed lines) of PG in (III). H atoms that participate in the formation of O—H···O intermolecular hydrogen bonds deviate from the molecular plane. These are designated by 'downward' or 'upward' bending.

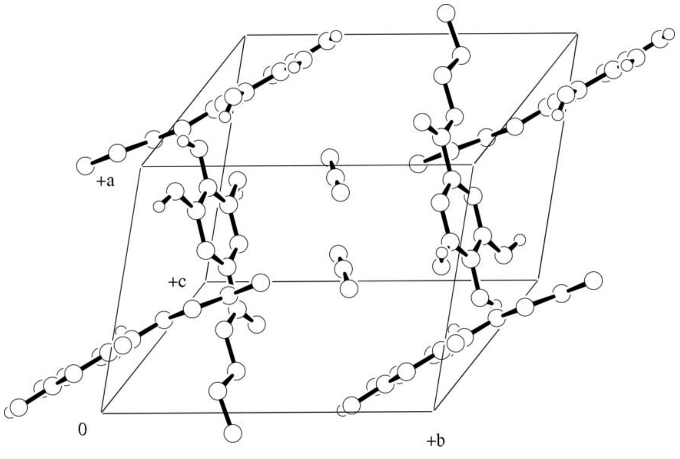


Figure 3
The crystal structure of (III). H atoms bonded to C atoms have been omitted for clarity.

Crystal data

$2C_{10}H_{12}O_5 \cdot C_2H_3N$
$M_r = 465.45$
Triclinic, $P\overline{1}$
$a = 8.5884$ (11) Å
$b = 11.3727$ (13) Å
$c = 12.5530$ (14) Å
$\alpha = 67.648$ (9)°
$\beta = 74.069$ (10)°
$\gamma = 81.488$ (11)°
$V = 1089.1$ (2) Å^3
$Z = 2$
$D_x = 1.419$ Mg m^{-3}
Cu $K\alpha$ radiation
Cell parameters from 18945 reflections
$\theta = 3.9$–68.2°
$\mu = 0.96$ mm^{-1}
$T = 93.1$ K
Block, colourless
0.10 × 0.10 × 0.10 mm

Data collection

Rigaku R-AXIS RAPID-F imaging-plate diffractometer
ω scans
Absorption correction: multi-scan (*ABSCOR*; Higashi, 1995)
$T_{\min} = 0.898$, $T_{\max} = 0.909$
9830 measured reflections
3588 independent reflections
3257 reflections with $F^2 > 2\sigma(F^2)$
$R_{\rm int} = 0.045$
$\theta_{\max} = 68.2°$
$h = -10 \rightarrow 10$
$k = -13 \rightarrow 13$
$l = -14 \rightarrow 15$

o2594 Mizuguchi *et al.* • $2C_{10}H_{12}O_5 \cdot C_2H_3N$ *Acta Cryst.* (2005). E**61**, o2593–o2595

Refinement

Refinement on F
$R[F^2 > 2\sigma(F^2)] = 0.047$
$wR(F^2) = 0.134$
$S = 1.89$
3588 reflections
310 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + \{0.05[\max(F_o^2,0) + 2F_c^2]/3\}^2]$
$(\Delta/\sigma)_{\max} = 0.002$
$\Delta\rho_{\max} = 0.49$ e Å^{-3}
$\Delta\rho_{\min} = -0.43$ e Å^{-3}

Table 1
Selected bond lengths (Å).

O1—C1	1.365 (2)	O6—C11	1.367 (2)
O2—C2	1.3508 (17)	O7—C12	1.359 (2)
O3—C3	1.375 (2)	O8—C13	1.382 (2)

Table 2
Hydrogen-bond geometry (Å, °).

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1O···O2	0.82 (2)	2.20 (2)	2.677 (2)	117 (2)
O1—H1O···N1^{i}	0.82 (2)	2.39 (2)	3.1675 (19)	158 (2)
O2—H2O···O3	0.83 (2)	2.31 (2)	2.735 (2)	112 (2)
O2—H2O···O9^{i}	0.83 (2)	1.92 (2)	2.6888 (17)	153 (2)
O3—H3O···O7	0.75 (2)	2.16 (2)	2.761 (2)	137 (2)
O3—H3O···O8	0.75 (2)	2.23 (2)	2.8606 (18)	142 (2)
O6—H6O···O4^{ii}	0.87 (2)	1.86 (2)	2.725 (2)	172 (2)
O7—H7O···O3^{iii}	0.88 (2)	1.91 (2)	2.7346 (17)	156 (2)
O7—H7O···O6	0.88 (2)	2.28 (2)	2.698 (2)	109 (2)
O8—H8O···O4^{iv}	0.84 (2)	2.04 (2)	2.8528 (15)	164 (2)

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

H atoms attached to O atoms were found in difference density maps and their coordinates were refined, with $U_{\rm iso}$(H) = 0.027 Å^2. All other H atoms were positioned geometrically and included in a riding-model approximation, with C—H = 0.95 Å, and with $U_{\rm iso}$(H) = $1.2U_{\rm eq}$(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: *CrystalStructure* (Rigaku/MSC, 2005).

References

Burnett, M. N. & Johnson, C. K. (1996). *ORTEPIII*. Report ORNL-6895. Oak Ridge National Laboratory, Tennessee, USA.

Higashi, T. (1995). *ABSCOR*. Rigaku Corporation, Tokyo, Japan.

Hitachi, A., Iwata, S., Makino, T. & Mizuguchi, J. (2005). *Acta Cryst.* E**61**, o2590–o2592.

Iwata, S., Hitachi, A., Makino, T. & Mizuguchi, J. (2005). *Acta Cryst.* E**61**, o2587–o2589.

Molecular Structure Corporation (2001). *TEXSAN*. Version 1.11. MSC, 9009 New Trails Drive, The Woodlands, TX 77381-5209, USA.

Rigaku (1998). *PROCESS-AUTO*. Rigaku Corporation, Tokyo, Japan.

Rigaku/MSC (2005). *CrystalStructure*. Version 3.7.0. Rigaku/MSC, 9009 New Trails Drive, The Woodlands, TX 77381-5209, USA.

Sheldrick, G. M. (1985). *SHELXS86*. University of Göttingen, Germany.

Acta Cryst. (2005). E**61**, o2593–o2595 Mizuguchi *et al.* • $2C_{10}H_{12}O_5{\cdot}C_2H_3N$ **o2595**