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n-Propyl gallate–dichloromethane (3/0.5)

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Key indicators

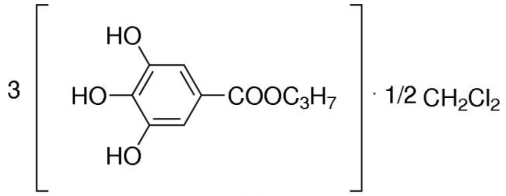
Single-crystal X-ray study
T = 93 K
Mean σ(C–C) = 0.003 Å
Disorder in solvent or counterion
R factor = 0.045
wR factor = 0.134
Data-to-parameter ratio = 11.0

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

n-Propyl gallate (PG) is a charge-control agent used for toners in electrophotography, and shows pseudo-polymorphism. The title compound, $3C_{10}H_{12}O_5 \cdot 0.5CH_2Cl_2$, is isostructural with the corresponding chloroform solvate. The asymmetric unit is composed of three PG molecules and a half-molecule of dichloromethane, the solvent being disordered over an inversion centre. There are intra- and intermolecular O—H···O hydrogen bonds, forming a two-dimensional hydrogen-bond network in the (421) plane.

Comment

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



(II)

Compound (II) was found to be entirely isostructural with (I). The asymmetric unit is composed of three independent PG molecules and half of a solvent molecule, as shown in Fig. 1. The solvent molecule is disordered around an inversion centre, just as in (I).

The nature of the intra- and intermolecular O—H···O hydrogen-bond network and the molecular arrangement are exactly the same as found in (I) (Fig. 2). All molecules lie in the (421) plane, forming a sheet-like layered structure. 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. Similarly, short intermolecular H···H contacts of less than 2.4 Å are also found, as shown in Fig. 2, between atoms H12 and H12* [2.07 Å; symmetry codes (x, y, z) and ($-x$, $-y$, $2-z$), respectively], H22 and H22* [2.10 Å; symmetry codes ($1-x$, $1-y$, $1-z$) and ($1+x$, y, z), respectively], and H2

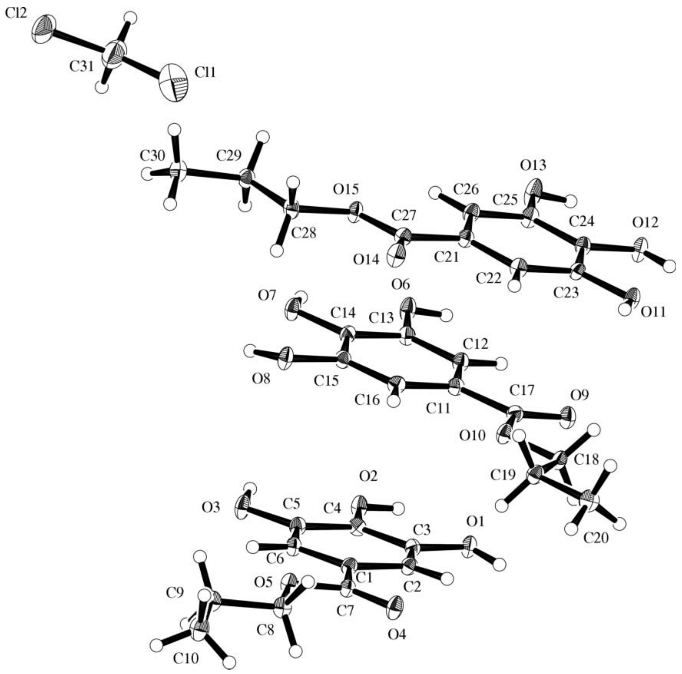


Figure 1
The asymmetric unit of (II), showing 30% probability displacement ellipsoids. The solvent molecule is disordered around an inversion centre (occupancy 50%).

and H2* [2.14 Å; symmetry codes ($-x$, $1-y$, $1-z$) and (x, $1+y$, z), respectively]. This results from two intermolecular O—H···O hydrogen bonds which exist on each side of the H···H contact.

Experimental

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

Crystal data

$3C_{10}H_{12}O_5\cdot 0.5CH_2Cl_2$
$M_r = 679.07$
Triclinic, $P\overline{1}$
$a = 9.9439$ (12) Å
$b = 12.8538$ (16) Å
$c = 13.8410$ (17) Å
$\alpha = 63.311$ (10)°
$\beta = 80.168$ (11)°
$\gamma = 86.706$ (11)°
$V = 1557.0$ (4) Å^3

$Z = 2$
$D_x = 1.448$ Mg m^{-3}
Cu $K\alpha$ radiation
Cell parameters from 13196 reflections
$\theta = 3.6$–68.2°
$\mu = 1.74$ mm^{-1}
$T = 93.1$ K
Block, colourless
0.40 × 0.40 × 0.40 mm

Data collection

Rigaku R-AXIS RAPID-F imaging-plate diffractometer
ω scans
Absorption correction: multi-scan (*ABSCOR*; Higashi, 1995)
$T_{\min} = 0.468$, $T_{\max} = 0.498$
14448 measured reflections

5149 independent reflections
3794 reflections with $F^2 > 2\sigma(F^2)$
$R_{\rm int} = 0.034$
$\theta_{\max} = 68.2°$
$h = -11 \rightarrow 11$
$k = -15 \rightarrow 15$
$l = -16 \rightarrow 16$

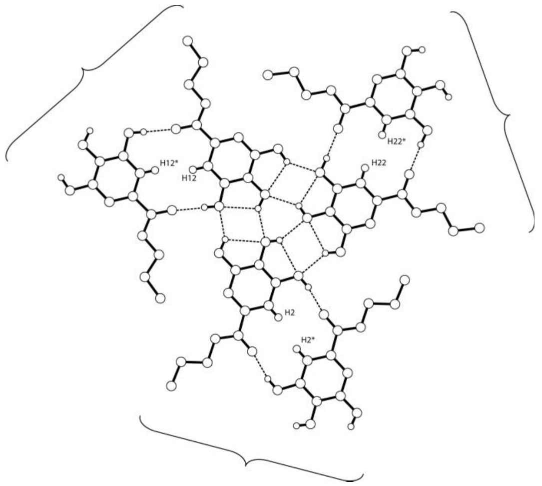


Figure 2
The six molecules involved in the intra- and intermolecular O—H···O hydrogen bonds. The hydrogen bonds are designated by dotted lines.

Refinement

Refinement on F
$R[F^2 > 2\sigma(F^2)] = 0.045$
$wR(F^2) = 0.134$
$S = 1.05$
5149 reflections
470 parameters

H-atom parameters not refined
$w = 1/[\sigma^2(F_o^2) + \{0.05[\max(F_o^2,0) + 2F_c^2]/3\}^2]$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.52$ e Å^{-3}
$\Delta\rho_{\min} = -0.51$ e Å^{-3}

Table 1
Selected bond lengths (Å).

O1—C3	1.369 (2)	O8—C15	1.366 (2)
O2—C4	1.369 (2)	O11—C23	1.369 (2)
O3—C5	1.362 (2)	O12—C24	1.373 (2)
O6—C13	1.373 (2)	O13—C25	1.361 (2)
O7—C14	1.377 (2)		

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

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1O···O4$^{\rm i}$	0.84	1.90	2.7260 (18)	170
O2—H2O···O1	0.85	2.27	2.7187 (19)	113
O2—H2O···O12$^{\rm ii}$	0.85	2.07	2.801 (2)	144
O3—H3O···O2	0.91	2.34	2.749 (2)	107
O3—H3O···O6$^{\rm iii}$	0.91	1.95	2.7672 (18)	149
O6—H6O···O9$^{\rm ii}$	0.86	1.87	2.724 (2)	171
O7—H7O···O2$^{\rm iii}$	0.89	2.03	2.7879 (18)	143
O7—H7O···O6	0.89	2.28	2.732 (2)	111
O8—H8O···O7	0.95	2.32	2.7542 (19)	107
O8—H8O···O11$^{\rm iv}$	0.95	1.99	2.7866 (19)	140
O11—H11O···O14$^{\rm v}$	0.87	1.87	2.736 (2)	171
O12—H12O···O7$^{\rm vi}$	0.85	2.03	2.766 (2)	144
O12—H12O···O11	0.85	2.28	2.7198 (19)	112
O13—H13O···O1$^{\rm ii}$	0.89	1.97	2.770 (2)	149
O13—H13O···O12	0.89	2.37	2.753 (2)	106

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

H atoms attached to O atoms were found in difference density maps and fixed during the least-squares refinement, with $U_{\rm iso}({\rm H})$ =

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$1.2U_{eq}$(O). All other H atoms were positioned geometrically and included in a riding-model approximation, with C—H = 0.95 Å, and with U_{iso}(H) = $1.2U_{eq}$(C). The dichloromethane molecule is disordered over two possible sites around an inversion centre, each with occupancy 0.5.

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).

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