n-Propyl gallate–dichloromethane (3/0.5)
Akio Hitachi, Takashi Makino, Shohei Iwata and Jin Mizuguchi

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*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 $O-H \cdot \cdot \cdot O$ 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 electrophotography, 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), $3PG \cdot 0.5$chloroform (Iwata et al., 2005), the title compound, (II), $3PG \cdot 0.5$dichloromethane (this publication), and compound (III), $2PG \cdot$acetonitrile (Mizuguchi et al., 2005), which follows the present publication.

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 $O-H \cdot \cdot \cdot O$ 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 $O-H \cdot \cdot \cdot O$ hydrogen bonds. Similarly, short intermolecular $H \cdot \cdot \cdot H$ 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
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
3C10H12O5·0.5CH2Cl2
M_r = 679.07
Triclinic, P T
a = 9.9439 (12) Å
b = 12.8538 (16) Å
c = 13.8410 (17) Å
α = 65.311 (10)°
β = 80.168 (11)°
γ = 86.706 (11)°
V = 1557.0 (4) Å³

Data collection
Rigaku R-AXIS RAPID-F imaging-plate diffractometer
3794 independent reflections
14448 measured reflections
Rint = 0.034
h = −11 → 11
k = −15 → 15
l = −16 → 16

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

Table 2
Selected bond lengths (Å).

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

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
H-atom parameters not refined

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

References