Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

3,5-Di-*tert*-butyl-2-hydroxybenzoic acid

Jin Mizuguchi

Department of Applied Physics, Graduate School of Engineering, Yokohama National University, 79-5 Tokiwadai, Hodogaya-ku, 240-8501 Yokohama, Japan

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

Key indicators

Single-crystal X-ray study
T = 93 K
Mean σ(C–C) = 0.003 Å
R factor = 0.043
wR factor = 0.104
Data-to-parameter ratio = 14.1

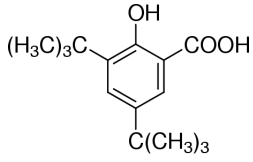
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Received 27 August 2003
Accepted 18 September 2003
Online 24 September 2003

The title compound, $C_{15}H_{22}O_3$, is a charge-control agent used in electrophotography. There are two independent molecules *A* and *B* in the asymmetric unit. The molecules form centrosymmetric dimers *A*–*A* and *B*–*B* *via* pairs of O—H···O hydrogen bonds between the carboxyl groups.

Comment

3,5-Di-tert-butyl-2-hydroxybenzoic acid (TBS), (I), is an industrially important charge-control agent (CCA) of the negative type used in electrophotography, either embedded in toners or mixed with toners (Suganami *et al.*, 2002). Furthermore, the charge-control power is greatly enhanced when TBS is combined with a variety of metals such as Zn, Al, Cr and Fe to yield metal complexes. An attempt has, therefore, been made in the present investigation to study the correlation between the structure and the charge-control effect.



(I)

There are two independent molecules *A* and *B* (Fig. 1 and Table 1). The conformation of both molecules is quite similar, and there exists an intramolecular hydrogen bond between the phenol OH group and the O atom of the carboxylic acid (Table 2). The molecules are dimerized in the crystal structure, as shown in Fig. 2, by O—H···O intermolecular hydrogen bonds between the carboxyl groups. There are two types of centrosymmetric dimers, *A*–*A* and *B*–*B*, in the unit cell.

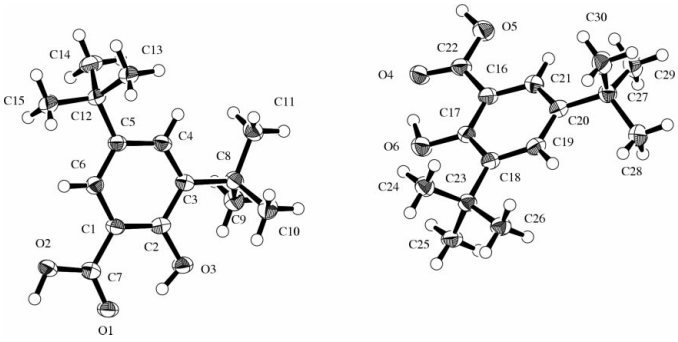


Figure 1
Two independent molecules (left: *A*; right: *B*) of (I), showing 50% displacement ellipsoids for non-H atoms.

Although the present hydrogen-bond formation is typical of carboxylic acids, there is a small step of *ca* 0.89 Å between the two hydrogen-bonded molecular planes for both *A*–*A* and *B*–*B* dimers (Fig. 3).

Experimental

The title compound was obtained from the API Corporation. Single crystals were grown by slow evaporation of an ethanol solution.

Crystal data

$C_{15}H_{22}O_3$
M_r = 250.34
Triclinic, $P\overline{1}$
a = 9.597 (2) Å
b = 9.828 (2) Å
c = 17.783 (3) Å
α = 74.16 (1)°
β = 78.85 (1)°
γ = 65.11 (1)°
V = 1457.9 (5) Å^3
Z = 4
D_x = 1.140 Mg m^{-3}
Cu Kα radiation
Cell parameters from 1379 reflections
θ = 6.9–65.2°
μ = 0.63 mm^{-1}
T = 93.2 K
Block, colorless
0.30 × 0.10 × 0.10 mm

Data collection

Rigaku R-AXIS RAPID Imaging Plate diffractometer
ω scans
Absorption correction: multi-scan (*ABSCOR*; Higashi, 1995)
T_{min} = 0.688, T_{max} = 0.939
13032 measured reflections
4803 independent reflections
3053 reflections with $F^2 > 2\sigma(F^2)$
R_{int} = 0.037
θ_{max} = 68.3°
$h = -11 \rightarrow 11$
$k = -11 \rightarrow 11$
$l = -21 \rightarrow 20$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.043
$wR(F^2)$ = 0.104
S = 0.95
4766 reflections
337 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.05(\mathrm{Max}(F_o^2,0) + 2F_c^2)/3)^2]$
$(\Delta/\sigma)_{max}$ = 0.050
$\Delta\rho_{max}$ = 0.33 e Å^{-3}
$\Delta\rho_{min}$ = −0.20 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

O1—C7	1.243 (2)	C1—C7	1.464 (3)
O2—C7	1.324 (2)	C3—C8	1.541 (3)
O3—C2	1.353 (2)	C5—C12	1.535 (3)
O4—C22	1.248 (2)	C16—C22	1.466 (3)
O5—C22	1.325 (2)	C18—C23	1.547 (3)
O6—C17	1.364 (2)	C20—C27	1.538 (3)
O1—C7—O2	120.4 (2)	O4—C22—O5	121.2 (2)
O1—C7—C1	123.7 (2)	O4—C22—C16	122.9 (2)
O2—C7—C1	115.9 (2)	O5—C22—C16	115.9 (2)

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

D—H···A	D—H	H···A	D···A	D—H···A
O3—H3···O1	0.93 (2)	1.76 (2)	2.613 (2)	151 (2)
O6—H7···O4	1.07 (2)	1.56 (2)	2.579 (2)	158 (2)
O2—H4···O1^{i}	0.97 (2)	1.69 (2)	2.659 (2)	177 (2)
O5—H8···O4^{ii}	0.92 (2)	1.74 (2)	2.661 (2)	179 (2)

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

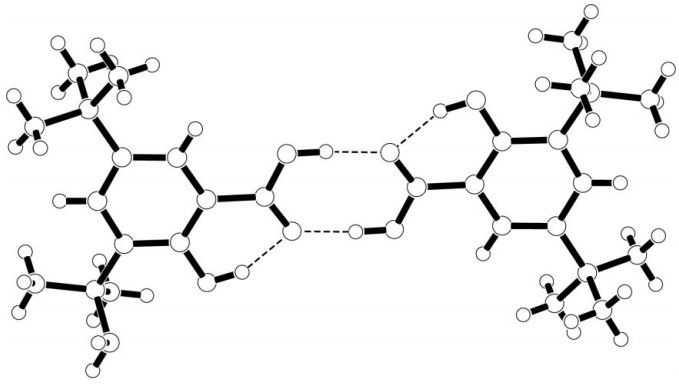


Figure 2
Top view of the hydrogen-bonded dimer *A*–*A*.

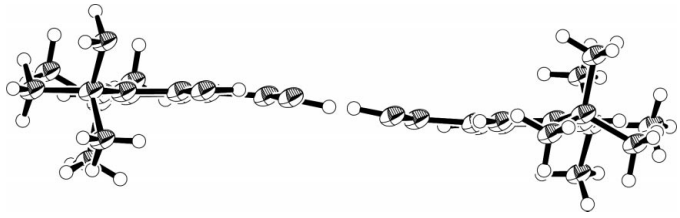


Figure 3
Side view of the hydrogen-bonded dimer *A*–*A*.

The H atoms of the carboxyl and hydroxyl groups were found in difference density maps and their coordinates were refined, with U_{iso} = 0.0541 Å^2. All other H atoms were positioned by calculation and not refined [C—H = 0.95 Å and U_{iso} =1.2U_{eq}(C)]. Reflections with $|F_o^2 - F_c^2|/|\sigma(F_o^2)|$ greater than 5% have not been used for the refinement.

Data collection: *PROCESS-AUTO* (Rigaku, 1998); cell refinement: *PROCESS-AUTO*; data reduction: *TEXSAN* (Molecular Structure Corporation, 2001); program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1985); program(s) used to refine structure: *TEXSAN*; molecular graphics: *ORTEP*III (Burnett & Johnson, 1996); software used to prepare material for publication: *TEXSAN*.

The author expresses his sincere thanks to Mr I. Suzuki for experimental assistance.

References

Burnett, M. N. & Johnson, C. K. (1996). *ORTEP*III. Report ORNL-6895. Oak Ridge National Laboratory. Tennessee, USA.
Higashi, T. (1995). *ABSCOR*. Rigaku Corporation, Tokyo, Japan.
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.
Sheldrick, G. M. (1985). *SHELXS*86. University of Göttingen, Germany.
Suganami, K., Takeuchi, M. & Oguchi, T. (2002). IS and T NIP18 International Conference on Digital Printing Technologies. Abstracts, pp. 302–305.