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5-Chloro-2-benzofuran-1,3-dione

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The asymmetric unit of the title compound, $C_8H_3ClO_3$, contains two molecules. The molecular packing is controlled by C—H···O hydrogen bonding.

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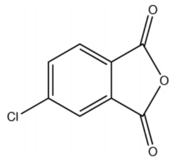
Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.003 Å
R factor = 0.036
wR factor = 0.104
Data-to-parameter ratio = 15.4

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

Comment

The title compound, (I), is a valuable intermediate for Cu phthalocyanine and anthraquinone dyes (Bansho *et al.*, 1960), quinophthalone pigments (Dietmar *et al.*, 1998) and polyether–polyimide polymers (Brunelle *et al.*, 1999). Compound (I) can be made from phthalic anhydride by chlorination, but it is very difficult to separate (I) from the by-product 4,5-dichlorophthalic anhydride by recrystallization. This paper reports the structure analysis of (I).



(I)

The asymmetric unit contains two molecules, which are very similar (Fig. 1). These two molecules are essentially planar, making a dihedral angle of 58.13 (5)°. Except for the C—Cl bond lengths, which are 0.01 Å longer than those observed in 4,5-dichlorophthalic anhydride (Ojala *et al.*, 1999), other bond lengths and angles are similar for the two compounds. The packing of the molecules in the crystal is governed by weak C—H···O hydrogen bonds (Fig. 2 and Table 2).

Experimental

The title compound was obtained by dehydration of 4-chlorophthalic acid, which was synthesized in our laboratory following the procedure described by Ayling (1929). Crystals suitable for X-ray analysis were obtained by slow evaporation of a saturated carbon tetrachloride solution at room temperature.

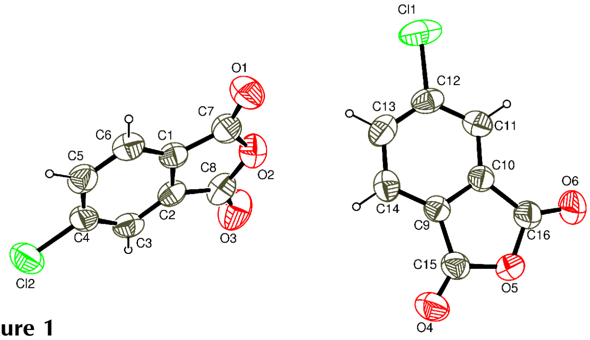


Figure 1
The asymmetric unit of (I), showing the atomic numbering. Displacement ellipsoids are drawn at the 50% probability level for non-H atoms.

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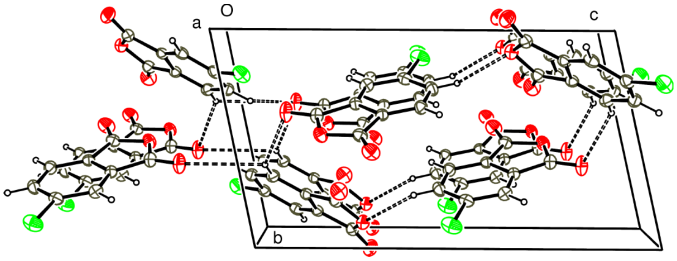

Figure 2
Packing diagram of (I), showing the hydrogen bonds as dashed lines.

Crystal data

$C_8H_3ClO_3$
$M_r = 182.55$
Triclinic, $P\bar{1}$
$a = 6.929\ (2)$ Å
$b = 7.8540\ (10)$ Å
$c = 14.262\ (2)$ Å
$\alpha = 77.69\ (1)^\circ$
$\beta = 88.09\ (1)^\circ$
$\gamma = 85.48\ (2)^\circ$
$V = 755.8\ (3)$ Å^3

$Z = 4$
$D_x = 1.604$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 4173 reflections
$\theta = 1.5$–27.2°
$\mu = 0.46$ mm^{-1}
$T = 293\ (2)$ K
Prism, colourless
0.4 × 0.2 × 0.1 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
$\omega/2\theta$ scans
Absorption correction: ψ scan (North *et al.*, 1968)
$T_{\rm min} = 0.837$, $T_{\rm max} = 0.955$
4173 measured reflections
3336 independent reflections
2184 reflections with $I > 2\sigma(I)$

$R_{\rm int} = 0.015$
$\theta_{\rm max} = 27.2^\circ$
$h = -1 \rightarrow 8$
$k = -10 \rightarrow 10$
$l = -18 \rightarrow 18$
3 standard reflections every 100 reflections intensity decay: 0.5%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.036$
$wR(F^2) = 0.104$
$S = 1.08$
3336 reflections
217 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0367P)^2 + 0.2624P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\rm max} = 0.001$
$\Delta\rho_{\rm max} = 0.25$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.24$ e Å^{-3}

Table 1
Selected geometric parameters (Å).

C1—C2	1.380 (3)	C9—C10	1.374 (3)
C1—C7	1.468 (3)	C9—C15	1.470 (3)
C2—C8	1.470 (3)	C10—C16	1.471 (3)
C4—Cl2	1.729 (2)	C12—Cl1	1.731 (2)
C7—O1	1.189 (3)	C15—O4	1.193 (3)
C7—O2	1.388 (3)	C15—O5	1.386 (3)
C8—O3	1.183 (3)	C16—O6	1.181 (3)
C8—O2	1.393 (3)	C16—O5	1.395 (3)

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

D—H$\cdots$$A$	D—H	H$\cdots$$A$	$D\cdots A$	D—H$\cdots$$A$
C3—H3$\cdots$O1$^{\rm i}$	0.93	2.80	3.292 (3)	114
C6—H6$\cdots$O6$^{\rm ii}$	0.93	3.03	3.466 (3)	111
C6—H6$\cdots$O6$^{\rm iii}$	0.93	2.89	3.690 (3)	145
C11—H11$\cdots$O2$^{\rm iv}$	0.93	2.94	3.675 (3)	137
C13—H13$\cdots$O1	0.93	2.63	3.522 (3)	161
C13—H13$\cdots$O2	0.93	2.81	3.453 (3)	127
C14—H14$\cdots$O4$^{\rm v}$	0.93	2.61	3.519 (3)	166

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

All H atoms were located in difference Fourier maps but were introduced in the refinement in calculated positions (C—H = 0.93 Å) and treated as riding, with $U_{\rm iso}({\rm H}) = 1.2U_{\rm eq}({\rm C})$.

Data collection: *CAD*-4 *EXPRESS* (Enraf–Nonius, 1994; cell refinement: *CAD*-4 *EXPRESS*; data reduction: *XCAD*4 (Harms & Wocadlo, 1995); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEP*-3 *for Windows* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

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