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2-Chloro-6-fluorophenyl 4-chlorobenzoate

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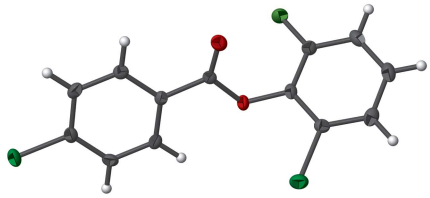
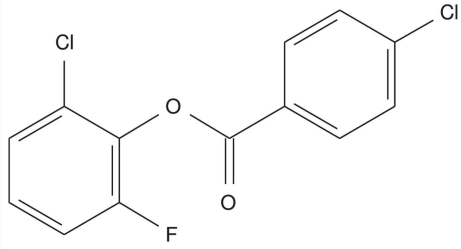
In the title compound, $C_{13}H_7Cl_2FO_2$, the dihedral angle between the aromatic rings is 49.96 (12)° and the fluorine atom is *syn* to the C=O group. In the crystal, the molecules are linked into [010] chains by C—H···O hydrogen bonds and weak C—H···Cl interactions link these chains into sheets parallel to the (101) plane.

3D view



Chemical scheme



Structure description

Phenyl benzoate derivatives are widely used as intermediates in the synthesis of bioactive compounds (Prashanth *et al.*, 2014; Khanum *et al.*, 2004), which exhibit a broad range of biological activities. As part of our studies in this area, we obtained the title compound, namely 2-chloro-6-fluorophenyl 4-chlorobenzoate, (I), the structure of which we report here.

The molecular structure of (I) (Fig. 1) closely resembles that of 2,6-dichlorophenyl 4-chlorobenzoate (Abdoh *et al.*, 2012), (II), with similar bond lengths and angles. The dihedral angle between the planes of the aromatic rings is 49.96 (12)° [*cf.* 82.1 (2)° in (II)]. The ester grouping in (I) is twisted at an angle of 66.3 (3)° out of the 2-chloro-6-fluorophenyl plane, while it forms a dihedral angle of 10.5 (4)° with the plane of the 2-chloro-6-fluorophenyl ring. In the crystal, C—H···O hydrogen bonds (Table 1) link the molecules into chains extending along [010] (Fig. 2) and weak C—H···Cl interactions (Table 1) further link these chains into sheets parallel to (101).

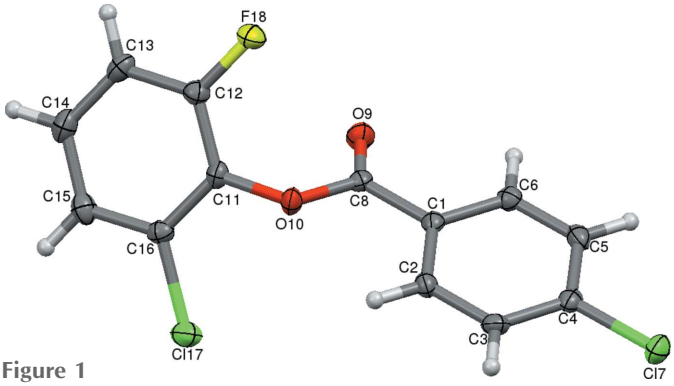


Figure 1
The molecular structure of (I), showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level.

Synthesis and crystallization

2-Chloro-6-fluorophenol (0.20 mol) was dissolved in dichloromethane (DCM) and triethylamine (0.45 mol) was added. The reaction mixture was cooled to 273 K. A solution of chlorobenzoyl chloride (0.21 mol) in DCM was added slowly to the reaction mixture and the resulting solution stirred for 3 h. The completion of the reaction was monitored by thin-layer chromatography (TLC) using a 4:1 *n*-hexane–ethyl acetate solvent mixture. The reaction mass was diluted with DCM (100 ml) and washed successively with 10% sodium hydroxide solution (3 × 40 ml) and water (3 × 30 ml). The organic layer was dried over sodium sulfate and the solid obtained after evaporation of the solvent was recrystallized from ethanol, giving white crystals in good yield (95%; m.p. 325.1–326.5 K).

Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2. H atoms were positioned geometrically (C—H = 0.93–0.96 Å) and refined as riding, with U_{iso}(H) = 1.2–1.5U_{eq}(C).

Acknowledgements

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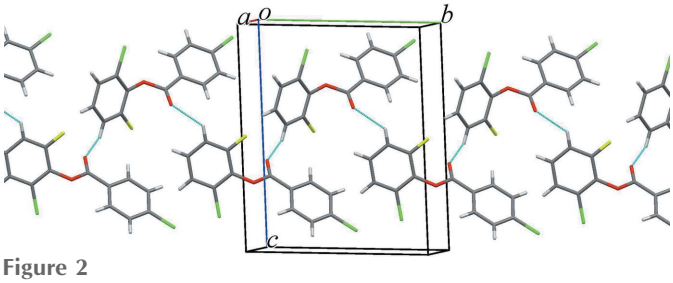

Figure 2
A portion of the crystal packing, viewed approximately down the *a* axis, and showing the hydrogen-bonded (dashed lines) chain of molecules running in the [010] direction.

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C13—H13···O9^{i}	0.93	2.54	3.142 (3)	123
C2—H2···Cl7^{ii}	0.93	2.94	3.581 (3)	127

Symmetry codes: (i) $-x, y-\frac{1}{2}, -z+1$; (ii) $-x+1, y-\frac{1}{2}, -z$.

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{13}H_7Cl_2FO_2$
M_r	285.09
Crystal system, space group	Monoclinic, $P2_1$
Temperature (K)	296
a, *b*, *c* (Å)	3.8882 (3), 11.2750 (7), 13.5058 (8)
β (°)	95.520 (3)
V (Å^3)	589.34 (7)
Z	2
Radiation type	Cu $K\alpha$
μ (mm^{-1})	5.01
Crystal size (mm)	0.30 × 0.28 × 0.23
Data collection	
Diffractometer	Bruker X8 Proteum
Absorption correction	Multi-scan (*SADABS*; Bruker, 2013)
T_{min}, T_{max}	0.278, 0.316
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	4587, 1724, 1702
R_{int}	0.036
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.583
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.031, 0.080, 1.06
No. of reflections	1724
No. of parameters	163
No. of restraints	1
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.27, −0.21
Absolute structure	Flack (1983), 877 Friedel pairs
Absolute structure parameter	0.051 (17)

Computer programs: *APEX2* (Bruker, 2013), *SAINT* (Bruker, 2013), *SHELXS97* (Sheldrick, 2008), *SHELXL97* (Sheldrick, 2008), *Mercury* (Macrae *et al.*, 2008).

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full crystallographic data

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Crystal data

$C_{13}H_7Cl_2FO_2$
M_r = 285.09
Monoclinic, $P2_1$
Hall symbol: P 2yb
a = 3.8882 (3) Å
b = 11.2750 (7) Å
c = 13.5058 (8) Å
β = 95.520 (3)°
V = 589.34 (7) Å^3
Z = 2
$F(000)$ = 288
D_x = 1.607 Mg m^{-3}
Cu $K\alpha$ radiation, λ = 1.54178 Å
Cell parameters from 1724 reflections
θ = 3.3–64.1°
μ = 5.01 mm^{-1}
T = 296 K
Prism, white
0.30 × 0.28 × 0.23 mm

Data collection

Bruker X8 Proteum diffractometer
Radiation source: Bruker MicroStar microfocus rotating anode
Helios multilayer optics monochromator
Detector resolution: 18.4 pixels mm^{-1}
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2013)
T_{min} = 0.278, T_{max} = 0.316
4587 measured reflections
1724 independent reflections
1702 reflections with $I > 2\sigma(I)$
R_{int} = 0.036
θ_{max} = 64.1°, θ_{min} = 3.3°
h = −4→3
k = −12→12
l = −15→15

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.031
$wR(F^2)$ = 0.080
S = 1.06
1724 reflections
163 parameters
1 restraint
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.052P)^2 + 0.0506P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.27 e Å^{-3}
$\Delta\rho_{min}$ = −0.21 e Å^{-3}
Absolute structure: Flack (1983), 877 Friedel pairs
Absolute structure parameter: 0.051 (17)

Special details

Experimental. ^{1}H NMR(400 MHz, DMSO-d_6 δ p.p.m.)7.39–8.17 (m, 7H, Ar—H).
IR (KBr) (ν_{max}/cm^{-1}):1750 (ester, C=O).
Mass spectra of the compound showed molecular ion peaks at m/z = 285 [M^+], 287 (M^{+2}) and 289 (M^{+4}).
Anal. Calcd. for $C_{13}H_7O_2Cl_2F$: C, 54.77; H, 2.47; Cl, 24.87; F, 6.66. Found: C, 54.57; H, 2.33; Cl, 24.64; F, 6.42%.

Geometry. Bond distances, angles *etc*. have been calculated using the rounded fractional coordinates. All su's are estimated from the variances of the (full) variance-covariance matrix. The cell e.s.d.'s are taken into account in the estimation of distances, angles and torsion angles

Refinement. Refinement on F^2 for ALL reflections except those flagged by the user for potential systematic errors. Weighted *R*-factors *wR* and all goodnesses of fit *S* are based on F^2, conventional *R*-factors *R* are based on *F*, with *F* set to zero for negative F^2. The observed criterion of $F^2 > \sigma(F^2)$ is used only for calculating -*R*-factor-obs *etc*. and is not relevant to the choice of reflections for refinement. *R*-factors based on F^2 are statistically about twice as large as those based on *F*, and *R*-factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
Cl7	0.60980 (16)	0.92140 (6)	0.06763 (5)	0.0271 (2)
Cl17	−0.06501 (14)	0.25211 (6)	0.13366 (4)	0.0255 (2)
F18	0.5317 (4)	0.34204 (15)	0.47450 (11)	0.0248 (5)
O9	0.0700 (4)	0.51053 (19)	0.37803 (14)	0.0225 (6)
O10	0.3621 (4)	0.39889 (16)	0.27475 (13)	0.0185 (5)
C1	0.3494 (6)	0.6054 (2)	0.2487 (2)	0.0161 (7)
C2	0.4895 (6)	0.5885 (2)	0.1588 (2)	0.0185 (7)
C3	0.5675 (6)	0.6864 (2)	0.10226 (19)	0.0203 (7)
C4	0.5093 (6)	0.7989 (2)	0.1372 (2)	0.0192 (7)
C5	0.3728 (6)	0.8170 (2)	0.2276 (2)	0.0205 (8)
C6	0.2917 (6)	0.7197 (2)	0.28295 (19)	0.0181 (7)
C8	0.2427 (6)	0.5038 (2)	0.30924 (18)	0.0159 (7)
C11	0.2481 (6)	0.2947 (2)	0.3165 (2)	0.0170 (7)
C12	0.3379 (6)	0.2650 (3)	0.41579 (19)	0.0190 (7)
C13	0.2396 (7)	0.1580 (3)	0.4542 (2)	0.0222 (8)
C14	0.0485 (7)	0.0793 (3)	0.3933 (2)	0.0243 (8)
C15	−0.0440 (6)	0.1067 (3)	0.2939 (2)	0.0228 (8)
C16	0.0548 (6)	0.2149 (2)	0.25638 (19)	0.0181 (7)
H2	0.53080	0.51220	0.13650	0.0220*
H3	0.65790	0.67600	0.04160	0.0240*
H5	0.33680	0.89340	0.25040	0.0250*
H6	0.19840	0.73040	0.34320	0.0220*
H13	0.30190	0.13930	0.52060	0.0270*
H14	−0.01920	0.00730	0.41890	0.0290*
H15	−0.17100	0.05300	0.25290	0.0270*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl7	0.0352 (3)	0.0206 (3)	0.0248 (4)	−0.0058 (3)	−0.0003 (2)	0.0072 (3)
Cl17	0.0294 (3)	0.0285 (4)	0.0180 (3)	0.0007 (3)	−0.0007 (2)	−0.0016 (3)
F18	0.0282 (8)	0.0255 (8)	0.0193 (8)	−0.0051 (6)	−0.0047 (6)	−0.0002 (7)

O9	0.0261 (9)	0.0216 (10)	0.0206 (10)	0.0014 (8)	0.0069 (7)	0.0008 (9)
O10	0.0233 (8)	0.0129 (9)	0.0200 (9)	−0.0004 (7)	0.0056 (7)	0.0014 (8)
C1	0.0159 (11)	0.0147 (13)	0.0167 (12)	−0.0001 (9)	−0.0033 (9)	0.0012 (10)
C2	0.0198 (11)	0.0149 (14)	0.0202 (13)	−0.0004 (10)	−0.0004 (10)	−0.0013 (12)
C3	0.0230 (11)	0.0247 (15)	0.0129 (12)	−0.0007 (11)	−0.0002 (10)	0.0004 (11)
C4	0.0185 (10)	0.0183 (14)	0.0196 (14)	−0.0032 (10)	−0.0035 (9)	0.0027 (11)
C5	0.0211 (12)	0.0167 (15)	0.0225 (14)	0.0003 (10)	−0.0037 (10)	−0.0038 (11)
C6	0.0180 (10)	0.0189 (14)	0.0172 (13)	0.0024 (10)	0.0005 (9)	−0.0005 (11)
C8	0.0166 (11)	0.0165 (13)	0.0141 (13)	0.0011 (10)	−0.0013 (9)	0.0011 (11)
C11	0.0174 (10)	0.0140 (12)	0.0201 (13)	0.0003 (10)	0.0043 (10)	0.0019 (10)
C12	0.0181 (10)	0.0201 (14)	0.0186 (12)	−0.0005 (10)	0.0006 (9)	−0.0004 (11)
C13	0.0225 (12)	0.0237 (14)	0.0210 (14)	0.0019 (11)	0.0048 (10)	0.0068 (12)
C14	0.0255 (12)	0.0186 (15)	0.0299 (15)	0.0018 (11)	0.0078 (11)	0.0058 (13)
C15	0.0185 (11)	0.0195 (14)	0.0304 (16)	−0.0026 (11)	0.0026 (10)	−0.0028 (12)
C16	0.0178 (11)	0.0205 (14)	0.0159 (12)	0.0028 (10)	0.0019 (9)	−0.0001 (11)

Geometric parameters (Å, º)

Cl7—C4	1.736 (2)	C11—C12	1.394 (4)
Cl17—C16	1.730 (3)	C11—C16	1.384 (3)
F18—C12	1.355 (3)	C12—C13	1.382 (5)
O9—C8	1.200 (3)	C13—C14	1.378 (4)
O10—C8	1.369 (3)	C14—C15	1.391 (4)
O10—C11	1.394 (3)	C15—C16	1.389 (4)
C1—C2	1.391 (4)	C2—H2	0.9300
C1—C6	1.395 (3)	C3—H3	0.9300
C1—C8	1.489 (3)	C5—H5	0.9300
C2—C3	1.392 (3)	C6—H6	0.9300
C3—C4	1.380 (3)	C13—H13	0.9300
C4—C5	1.392 (4)	C14—H14	0.9300
C5—C6	1.381 (3)	C15—H15	0.9300
C8—O10—C11	117.34 (18)	C12—C13—C14	119.4 (3)
C2—C1—C6	120.3 (2)	C13—C14—C15	120.5 (3)
C2—C1—C8	121.8 (2)	C14—C15—C16	119.5 (3)
C6—C1—C8	117.8 (2)	Cl17—C16—C11	119.04 (18)
C1—C2—C3	119.6 (2)	Cl17—C16—C15	120.27 (19)
C2—C3—C4	119.3 (2)	C11—C16—C15	120.7 (2)
Cl7—C4—C3	119.6 (2)	C1—C2—H2	120.00
Cl7—C4—C5	118.86 (18)	C3—C2—H2	120.00
C3—C4—C5	121.6 (2)	C2—C3—H3	120.00
C4—C5—C6	119.0 (2)	C4—C3—H3	120.00
C1—C6—C5	120.1 (2)	C4—C5—H5	121.00
O9—C8—O10	123.5 (2)	C6—C5—H5	120.00
O9—C8—C1	125.6 (2)	C1—C6—H6	120.00
O10—C8—C1	110.9 (2)	C5—C6—H6	120.00
O10—C11—C12	122.1 (2)	C12—C13—H13	120.00
O10—C11—C16	119.1 (2)	C14—C13—H13	120.00

C12—C11—C16	118.7 (2)	C13—C14—H14	120.00
F18—C12—C11	118.9 (3)	C15—C14—H14	120.00
F18—C12—C13	119.9 (2)	C14—C15—H15	120.00
C11—C12—C13	121.2 (3)	C16—C15—H15	120.00
C11—O10—C8—C1	−172.5 (2)	Cl7—C4—C5—C6	179.81 (19)
C8—O10—C11—C12	−66.3 (3)	C4—C5—C6—C1	0.6 (4)
C8—O10—C11—C16	117.2 (2)	O10—C11—C12—F18	2.5 (4)
C11—O10—C8—O9	6.2 (3)	O10—C11—C12—C13	−176.0 (2)
C8—C1—C6—C5	−177.4 (2)	C16—C11—C12—F18	179.0 (2)
C2—C1—C8—O10	11.7 (3)	C16—C11—C12—C13	0.4 (4)
C6—C1—C8—O9	10.5 (4)	O10—C11—C16—Cl17	−4.6 (3)
C2—C1—C8—O9	−166.9 (2)	O10—C11—C16—C15	175.8 (2)
C6—C1—C2—C3	−0.9 (4)	C12—C11—C16—Cl17	178.78 (19)
C8—C1—C2—C3	176.5 (2)	C12—C11—C16—C15	−0.8 (4)
C2—C1—C6—C5	0.1 (4)	F18—C12—C13—C14	−178.7 (2)
C6—C1—C8—O10	−170.8 (2)	C11—C12—C13—C14	−0.2 (4)
C1—C2—C3—C4	1.0 (4)	C12—C13—C14—C15	0.3 (4)
C2—C3—C4—Cl7	179.39 (19)	C13—C14—C15—C16	−0.7 (4)
C2—C3—C4—C5	−0.3 (4)	C14—C15—C16—Cl17	−178.7 (2)
C3—C4—C5—C6	−0.5 (4)	C14—C15—C16—C11	0.9 (4)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C13—H13···O9^{i}	0.93	2.54	3.142 (3)	123
C2—H2···Cl7^{ii}	0.93	2.94	3.581 (3)	127

Symmetry codes: (i) −*x*, *y*−1/2, −*z*+1; (ii) −*x*+1, *y*−1/2, −*z*.