

4-Chlorophenyl 4-chlorobenzoate

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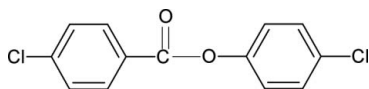
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Key indicators: single-crystal X-ray study; $T = 299$ K; mean $\sigma(\text{C}-\text{C}) = 0.004$ Å; R factor = 0.048; wR factor = 0.139; data-to-parameter ratio = 11.8.

The structure of the title compound (4CP4CBA), $\text{C}_{13}\text{H}_8\text{Cl}_2\text{O}_2$, resembles those of 4-methylphenyl 4-chlorobenzoate (4MP4CBA), 4-chlorophenyl 4-methylbenzoate (4CP4MBA) and 4-methylphenyl 4-methylbenzoate (4MP4MBA), with similar bond parameters. The dihedral angle between the two benzene rings in 4CP4CBA is 47.98 (7) $^\circ$, compared with 51.86 (4) $^\circ$ in 4MP4CBA, 63.89 (8) $^\circ$ in 4CP4MBA and 63.57 (5) $^\circ$ in 4MP4MBA. In the crystal structure, molecules are linked into helical chains running along the b axis by C—H—O hydrogen bonds.

Related literature

For related literature, see: Gowda *et al.* (2007); Gowda, Foro *et al.* (2008); Gowda, Svoboda *et al.* (2008); Nayak & Gowda (2008).



Experimental

Crystal data

$\text{C}_{13}\text{H}_8\text{Cl}_2\text{O}_2$
 $M_r = 267.09$
Monoclinic, $P2_1/n$
 $a = 15.370$ (2) Å
 $b = 3.9528$ (4) Å
 $c = 19.465$ (2) Å
 $\beta = 91.804$ (9) $^\circ$

$V = 1182.0$ (2) Å³
 $Z = 4$
Cu $K\alpha$ radiation
 $\mu = 4.83$ mm⁻¹
 $T = 299$ (2) K
 $0.45 \times 0.23 \times 0.18$ mm

Data collection

Enraf–Nonius CAD-4 diffractometer
Absorption correction: ψ scan (North *et al.*, 1968)
 $T_{\min} = 0.194$, $T_{\max} = 0.420$
4211 measured reflections

2109 independent reflections
1724 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.080$
3 standard reflections
frequency: 120 min
intensity decay: 1.0%

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.048$
 $wR(F^2) = 0.139$
 $S = 1.05$
2109 reflections

178 parameters
Only H-atom coordinates refined
 $\Delta\rho_{\max} = 0.39$ e Å⁻³
 $\Delta\rho_{\min} = -0.33$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, $^\circ$).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$\text{C5}-\text{H5}\cdots\text{O2}^i$	0.92 (3)	2.58 (3)	3.168 (3)	123 (2)

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

Data collection: *CAD-4-PC* (Enraf–Nonius, 1996); cell refinement: *CAD-4-PC*; data reduction: *REDU4* (Stoe & Cie, 1987); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *PLATON* (Spek, 2003); software used to prepare material for publication: *SHELXL97*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: CI2640).

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supplementary materials

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4-Chlorophenyl 4-chlorobenzoate

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Comment

As part of a study of the substituent effects on the solid state structures of aryl benzoates (Gowda *et al.*, 2007; Gowda, Foro *et al.*, 2008; Gowda, Svoboda *et al.*, 2008), the crystal structure of 4-chlorophenyl 4-chlorobenzoate (4CP4CBA) has been determined. The structure (Fig. 1) is similar to those of 4-methylphenyl 4-chlorobenzoate (4MP4CBA) (Gowda, Svoboda *et al.*, 2008), 4-chlorophenyl 4-methylbenzoate (4CP4MBA) (Gowda, Foro *et al.*, 2008) and 4-methylphenyl 4-methylbenzoate (4MP4MBA) (Gowda *et al.*, 2007). The bond parameters in 4CP4CBA are similar to those in afore mentioned 4MP4CBA, 4CP4MBA, 4MP4MBA and other aryl benzoates (Gowda *et al.*, 2007; Gowda, Foro *et al.*, 2008; Gowda, Svoboda *et al.*, 2008). The dihedral angle between the benzene and benzoyl rings in 4CP4CBA is $47.98(7)^\circ$, compared to the values of $51.86(4)^\circ$ in 4MP4CBA, $63.89(8)^\circ$ in 4CP4MBA and $63.57(5)^\circ$ in 4MP4MBA.

In the crystal structure, the molecules are linked into helical chains (Fig. 2) running along the *b* axis by C—H—O hydrogen bonds (Table 1).

Experimental

The title compound was prepared according to a literature method (Nayak & Gowda, 2008). The purity of the compound was checked by determining its melting point. It was characterized by recording its infrared and NMR spectra (Nayak & Gowda, 2008). Single crystals of the title compound used in X-ray diffraction studies were obtained by slow evaporation of its ethanol solution.

Refinement

H atoms were located in a difference map and their positional parameters were refined [C—H = 0.90 (3)–0.97 (3) Å] with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$.

Figures

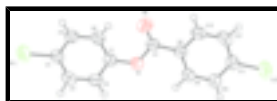


Fig. 1. Molecular structure of the title compound, showing the atom labelling scheme. Displacement ellipsoids are drawn at the 50% probability level. H atoms are represented as small spheres of arbitrary radius.

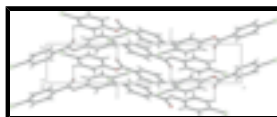


Fig. 2. Molecular packing of the title compound.

4-Chlorophenyl 4-chlorobenzoate

Crystal data

$C_{13}H_8Cl_2O_2$

$M_r = 267.09$

Monoclinic, $P2_1/n$

Hall symbol: -P 2yn

$a = 15.370$ (2) Å

$b = 3.9528$ (4) Å

$c = 19.465$ (2) Å

$\beta = 91.804$ (9)°

$V = 1182.0$ (2) Å³

$Z = 4$

$F_{000} = 544$

$D_x = 1.501$ Mg m⁻³

Cu $K\alpha$ radiation

$\lambda = 1.54180$ Å

Cell parameters from 25 reflections

$\theta = 11.5$ – 24.3 °

$\mu = 4.83$ mm⁻¹

$T = 299$ (2) K

Rod, colourless

$0.45 \times 0.23 \times 0.18$ mm

Data collection

Enraf–Nonius CAD-4
diffractometer

Radiation source: fine-focus sealed tube

Monochromator: graphite

$T = 299$ (2) K

$\omega/2\theta$ scans

Absorption correction: ψ scan
(North *et al.*, 1968)

$T_{\min} = 0.194$, $T_{\max} = 0.420$

4211 measured reflections

2109 independent reflections

1724 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.080$

$\theta_{\text{max}} = 67.0$ °

$\theta_{\text{min}} = 3.6$ °

$h = -18 \rightarrow 18$

$k = -4 \rightarrow 0$

$l = -23 \rightarrow 23$

3 standard reflections

every 120 min

intensity decay: 1.0%

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.048$

$wR(F^2) = 0.139$

$S = 1.05$

2109 reflections

178 parameters

Primary atom site location: structure-invariant direct
methods

Secondary atom site location: difference Fourier map

Hydrogen site location: difference Fourier map

Only H-atom coordinates refined

$$w = 1/[\sigma^2(F_o^2) + (0.0736P)^2 + 0.2805P]$$

where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\text{max}} = 0.021$

$\Delta\rho_{\text{max}} = 0.39$ e Å⁻³

$\Delta\rho_{\text{min}} = -0.33$ e Å⁻³

Extinction correction: none

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted R -factor wR and goodness 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 threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating R -factors(gt) *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 (\AA^2)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
C11	0.49228 (5)	0.7640 (2)	0.08027 (4)	0.0690 (3)
C12	0.72347 (5)	-0.0839 (2)	0.67678 (3)	0.0704 (3)
O1	0.59363 (10)	0.3353 (5)	0.36024 (9)	0.0529 (5)
O2	0.72448 (12)	0.5904 (7)	0.36130 (10)	0.0737 (7)
C1	0.57335 (13)	0.4453 (6)	0.29332 (12)	0.0444 (5)
C2	0.49523 (15)	0.6088 (7)	0.28247 (14)	0.0518 (6)
H2	0.4603 (19)	0.657 (8)	0.3193 (16)	0.062*
C3	0.46945 (15)	0.7062 (7)	0.21667 (15)	0.0546 (6)
H3	0.4132 (19)	0.820 (9)	0.2133 (15)	0.066*
C4	0.52292 (14)	0.6377 (6)	0.16294 (13)	0.0469 (5)
C5	0.60108 (15)	0.4709 (7)	0.17399 (13)	0.0492 (6)
H5	0.6337 (18)	0.438 (8)	0.1358 (16)	0.059*
C6	0.62658 (14)	0.3724 (7)	0.23892 (13)	0.0488 (6)
H6	0.6762 (19)	0.246 (8)	0.2482 (15)	0.059*
C7	0.67225 (14)	0.4158 (7)	0.38938 (12)	0.0482 (6)
C8	0.68345 (13)	0.2812 (6)	0.45961 (12)	0.0452 (5)
C9	0.61746 (15)	0.1152 (8)	0.49299 (14)	0.0530 (6)
H9	0.5619 (19)	0.089 (8)	0.4691 (16)	0.064*
C10	0.62957 (16)	0.0048 (8)	0.55961 (13)	0.0538 (6)
H10	0.584 (2)	-0.108 (8)	0.5807 (16)	0.065*
C11	0.70901 (15)	0.0570 (7)	0.59276 (13)	0.0494 (6)
C12	0.77573 (16)	0.2151 (8)	0.56050 (15)	0.0579 (7)
H12	0.826 (2)	0.225 (9)	0.5856 (17)	0.069*
C13	0.76306 (14)	0.3276 (8)	0.49402 (14)	0.0534 (6)
H13	0.8067 (19)	0.428 (9)	0.4718 (16)	0.064*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C11	0.0694 (4)	0.0793 (5)	0.0574 (4)	0.0037 (3)	-0.0138 (3)	0.0097 (4)
C12	0.0777 (5)	0.0819 (6)	0.0507 (4)	0.0114 (4)	-0.0142 (3)	0.0012 (3)
O1	0.0419 (8)	0.0685 (12)	0.0480 (9)	-0.0142 (8)	-0.0015 (6)	0.0071 (9)

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O2	0.0623 (11)	0.0998 (18)	0.0589 (11)	-0.0387 (11)	0.0032 (9)	0.0064 (11)
C1	0.0394 (10)	0.0469 (13)	0.0469 (12)	-0.0087 (9)	0.0002 (9)	-0.0007 (11)
C2	0.0404 (11)	0.0604 (16)	0.0553 (14)	-0.0009 (10)	0.0094 (10)	-0.0063 (12)
C3	0.0395 (11)	0.0581 (15)	0.0659 (16)	0.0061 (11)	-0.0023 (10)	-0.0024 (13)
C4	0.0447 (11)	0.0462 (13)	0.0492 (12)	-0.0044 (10)	-0.0046 (9)	0.0006 (11)
C5	0.0451 (11)	0.0537 (14)	0.0491 (13)	0.0034 (10)	0.0054 (10)	-0.0044 (12)
C6	0.0393 (11)	0.0553 (14)	0.0518 (13)	0.0056 (10)	0.0007 (9)	-0.0009 (12)
C7	0.0378 (10)	0.0605 (15)	0.0463 (12)	-0.0070 (10)	0.0038 (9)	-0.0042 (11)
C8	0.0367 (10)	0.0513 (13)	0.0477 (13)	-0.0032 (9)	0.0027 (9)	-0.0065 (11)
C9	0.0368 (10)	0.0715 (17)	0.0504 (13)	-0.0101 (11)	-0.0043 (9)	0.0033 (12)
C10	0.0428 (11)	0.0678 (16)	0.0507 (13)	-0.0070 (12)	0.0003 (10)	0.0049 (13)
C11	0.0514 (12)	0.0502 (14)	0.0460 (12)	0.0065 (10)	-0.0064 (10)	-0.0059 (11)
C12	0.0413 (11)	0.0657 (17)	0.0659 (16)	0.0002 (11)	-0.0123 (11)	-0.0106 (14)
C13	0.0356 (10)	0.0625 (16)	0.0622 (15)	-0.0079 (11)	0.0026 (10)	-0.0057 (13)

Geometric parameters (Å, °)

C11—C4	1.736 (3)	C5—H5	0.92 (3)
C12—C11	1.735 (3)	C6—H6	0.93 (3)
O1—C7	1.356 (3)	C7—C8	1.472 (3)
O1—C1	1.399 (3)	C8—C9	1.387 (3)
O2—C7	1.203 (3)	C8—C13	1.389 (3)
C1—C2	1.374 (3)	C9—C10	1.375 (4)
C1—C6	1.389 (3)	C9—H9	0.96 (3)
C2—C3	1.383 (4)	C10—C11	1.379 (3)
C2—H2	0.93 (3)	C10—H10	0.93 (3)
C3—C4	1.377 (4)	C11—C12	1.370 (4)
C3—H3	0.97 (3)	C12—C13	1.376 (4)
C4—C5	1.381 (3)	C12—H12	0.90 (3)
C5—C6	1.368 (4)	C13—H13	0.90 (3)
C7—O1—C1	119.07 (17)	O2—C7—C8	124.7 (2)
C2—C1—C6	120.9 (2)	O1—C7—C8	112.34 (19)
C2—C1—O1	117.2 (2)	C9—C8—C13	118.9 (2)
C6—C1—O1	121.7 (2)	C9—C8—C7	122.7 (2)
C1—C2—C3	119.8 (2)	C13—C8—C7	118.4 (2)
C1—C2—H2	120.1 (19)	C10—C9—C8	120.8 (2)
C3—C2—H2	120.1 (19)	C10—C9—H9	120.8 (18)
C4—C3—C2	119.2 (2)	C8—C9—H9	118.3 (18)
C4—C3—H3	126.1 (17)	C11—C10—C9	119.0 (2)
C2—C3—H3	114.7 (17)	C11—C10—H10	122 (2)
C3—C4—C5	120.8 (2)	C9—C10—H10	119 (2)
C3—C4—C11	119.77 (19)	C12—C11—C10	121.2 (2)
C5—C4—C11	119.41 (19)	C12—C11—C12	120.19 (19)
C6—C5—C4	120.1 (2)	C10—C11—C12	118.6 (2)
C6—C5—H5	124.0 (19)	C11—C12—C13	119.5 (2)
C4—C5—H5	115.8 (19)	C11—C12—H12	114 (2)
C5—C6—C1	119.1 (2)	C13—C12—H12	126 (2)
C5—C6—H6	123.0 (18)	C12—C13—C8	120.5 (2)
C1—C6—H6	117.8 (18)	C12—C13—H13	121 (2)

O2—C7—O1	122.9 (2)	C8—C13—H13	119 (2)
C7—O1—C1—C2	-129.5 (3)	O2—C7—C8—C9	172.1 (3)
C7—O1—C1—C6	55.0 (3)	O1—C7—C8—C9	-4.8 (4)
C6—C1—C2—C3	-0.8 (4)	O2—C7—C8—C13	-6.9 (4)
O1—C1—C2—C3	-176.3 (2)	O1—C7—C8—C13	176.2 (2)
C1—C2—C3—C4	-0.1 (4)	C13—C8—C9—C10	1.5 (4)
C2—C3—C4—C5	0.7 (4)	C7—C8—C9—C10	-177.5 (3)
C2—C3—C4—C11	-179.0 (2)	C8—C9—C10—C11	-0.9 (4)
C3—C4—C5—C6	-0.3 (4)	C9—C10—C11—C12	-0.2 (4)
C11—C4—C5—C6	179.4 (2)	C9—C10—C11—C12	-179.9 (2)
C4—C5—C6—C1	-0.6 (4)	C10—C11—C12—C13	0.7 (4)
C2—C1—C6—C5	1.1 (4)	C12—C11—C12—C13	-179.5 (2)
O1—C1—C6—C5	176.5 (2)	C11—C12—C13—C8	-0.1 (4)
C1—O1—C7—O2	3.2 (4)	C9—C8—C13—C12	-1.0 (4)
C1—O1—C7—C8	-179.9 (2)	C7—C8—C13—C12	178.0 (3)

Hydrogen-bond geometry (Å, °)

<i>D</i> —H... <i>A</i>	<i>D</i> —H	H... <i>A</i>	<i>D</i> ... <i>A</i>	<i>D</i> —H... <i>A</i>
C5—H5...O2 ⁱ	0.92 (3)	2.58 (3)	3.168 (3)	123 (2)

Symmetry codes: (i) $-x+3/2, y-1/2, -z+1/2$.

Fig. 1

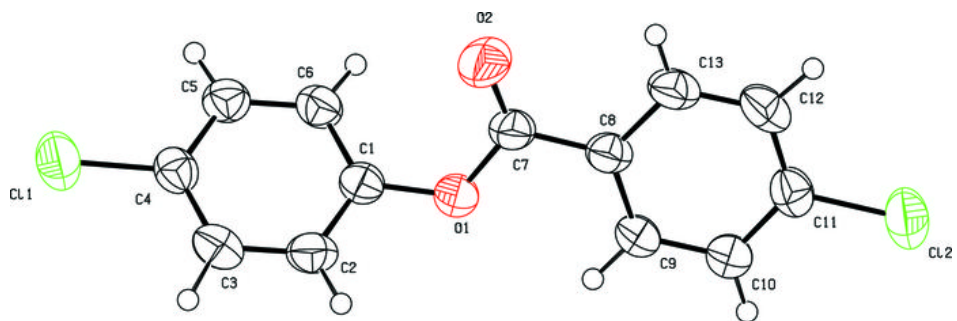


Fig. 2

