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4-Nitrophenyl 2-chlorobenzoate

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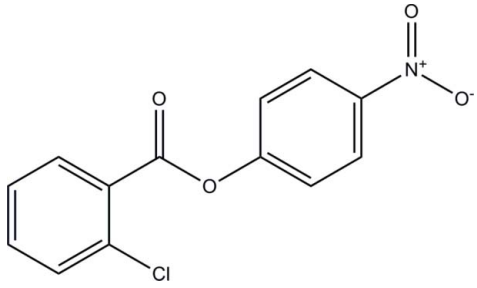
Key indicators: single-crystal X-ray study; T = 173 K; mean σ(C–C) = 0.002 Å; R factor = 0.044; wR factor = 0.105; data-to-parameter ratio = 18.9.

The aromatic rings in the title compound, $C_{13}H_8ClNO_4$, enclose a dihedral angle of 39.53 (3)°. The nitro group is almost coplanar with the ring to which it is attached [dihedral angle = 4.31 (1)°]. In the crystal, molecules are connected by C—H···O hydrogen bonds into chains running along [001].

Related literature

For the use of 4-nitrophenyl-2-chlorobenzoate as a starting material for the synthesis of pain-relieving and anti-inflammatory drugs, see: Selvakumar *et al.* (2002); Jefford & Zaslona (1985). For a similar hydrogen-bonding pattern in a related structure, see: Akhter *et al.* (2012).



Experimental

Crystal data

$C_{13}H_8ClNO_4$
M_r = 277.65
Orthorhombic, *Pbca*
a = 11.4790 (4) Å
b = 14.0461 (5) Å
c = 14.3702 (7) Å
V = 2316.98 (16) Å^3
Z = 8
Mo $K\alpha$ radiation
μ = 0.34 mm^{-1}
T = 173 K
0.36 × 0.15 × 0.15 mm

Data collection

Stoe IPDS II two-circle diffractometer
Absorption correction: multi-scan (*X-AREA*; Stoe & Cie, 2001)
T_{min} = 0.888, T_{max} = 0.951
54293 measured reflections
3251 independent reflections
3071 reflections with $I > 2\sigma(I)$
R_{int} = 0.057

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.044
$wR(F^2)$ = 0.105
S = 1.14
3251 reflections
172 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.32 e Å^{-3}
$\Delta\rho_{min}$ = −0.41 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
C4—H4···O1^{i}	0.95	2.48	3.3368 (19)	150

Symmetry code: (i) $x, y, z-1$.

Data collection: *X-AREA* (Stoe & Cie, 2001); cell refinement: *X-AREA*; data reduction: *X-AREA*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *XP* in *SHELXTL-Plus* (Sheldrick, 2008); 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: FF2092).

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supplementary materials

Acta Cryst. (2013). E**69**, o96 [doi:10.1107/S1600536812050362]

4-Nitrophenyl 2-chlorobenzoate

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Comment

The aromatic esters, like 4-Nitrophenyl-2-chlorobenzoate, can be used as starting materials for the synthesis of several pain-relieving and anti-inflammatory drugs (Selvakumar *et al.*, 2002; Jefford & Zaslona, 1985).

The two aromatic rings in the title compound enclose a dihedral angle of 39.53 (3)°. The nitro group is almost coplanar with the phenyl ring to which it is attached [dihedral angle 4.31 (1)°]. In the crystal, the molecules are connected by C—H···O bonds to chains running along [0 0 1].

Experimental

2-Chlorobenzoyl chloride was added drop wise to the solution of 4-nitrophenol (in a mixture of dry tetrahydrofuran (THF) and triethyl amine). The reaction mixture was stirred at room temperature for two hours and then was poured into the cold water. The oily product settled down after allowing the solution to stand for two hours, the supernatant liquid was decanted. The product was extracted from the solution by extraction with ethyl acetate, washed with 10% $NaHCO_3$ solution to remove any traces of reactants and recrystallized from methanol. Yield 78%, m.p. 416–418 K.

Refinement

All H atoms were initially located by difference Fourier synthesis. Subsequently all H atoms were refined using a riding model with C—H = 0.95 Å and with U_{iso}(H) = 1.2U_{eq}(C).

Computing details

Data collection: *X-AREA* (Stoe & Cie, 2001); cell refinement: *X-AREA* (Stoe & Cie, 2001); data reduction: *X-AREA* (Stoe & Cie, 2001); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *XP* in *SHELXTL-Plus* (Sheldrick, 2008); software used to prepare material for publication: *SHELXL97* (Sheldrick, 2008).

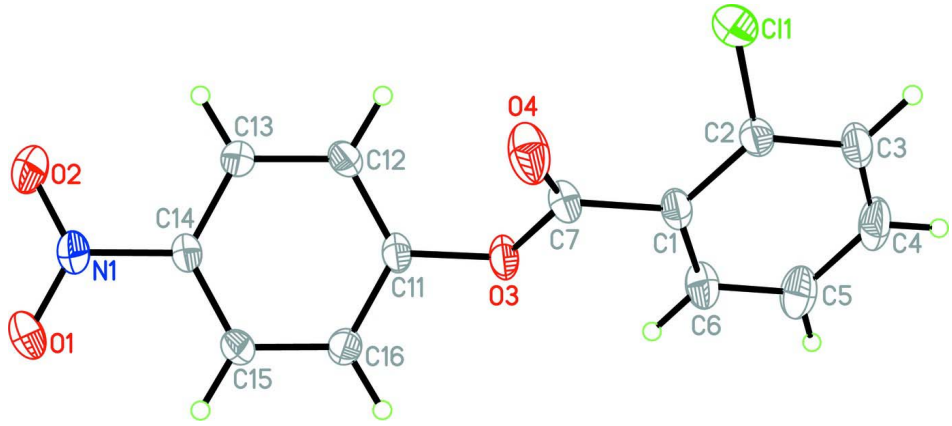

Figure 1

Perspective view of the title compound with the atom labeling scheme. Displacement ellipsoids are drawn at the 50% probability level.

4-Nitrophenyl 2-chlorobenzoate

Crystal data

$C_{13}H_8ClNO_4$
$M_r = 277.65$
Orthorhombic, *Pbca*
Hall symbol: -P 2ac 2ab
$a = 11.4790\ (4)$ Å
$b = 14.0461\ (5)$ Å
$c = 14.3702\ (7)$ Å
$V = 2316.98\ (16)$ Å^3
$Z = 8$
$F(000) = 1136$
$D_x = 1.592$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 57790 reflections
$\theta = 3.2–30.1°$
$\mu = 0.34$ mm^{-1}
$T = 173$ K
Rod, colourless
0.36 × 0.15 × 0.15 mm

Data collection

Stoe IPDS II two-circle diffractometer
Radiation source: Genix 3D IμS microfocus X-ray source
Genix 3D multilayer optics monochromator
ω scans
Absorption correction: multi-scan (*X-AREA*; Stoe & Cie, 2001)
$T_{min} = 0.888$, $T_{max} = 0.951$
54293 measured reflections
3251 independent reflections
3071 reflections with $I > 2\sigma(I)$
$R_{int} = 0.057$
$\theta_{max} = 29.7°$, $\theta_{min} = 3.2°$
$h = -15\rightarrow15$
$k = -19\rightarrow18$
$l = -19\rightarrow19$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.044$
$wR(F^2) = 0.105$
$S = 1.14$
3251 reflections
172 parameters
0 restraints
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
$w = 1/[\sigma^2(F_o^2) + (0.0411P)^2 + 1.2306P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.32$ e Å^{-3}
$\Delta\rho_{min} = -0.41$ e Å^{-3}

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 (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
Cl1	0.66372 (3)	0.20873 (4)	0.41090 (3)	0.04536 (14)
N1	0.85406 (11)	0.06742 (9)	1.00169 (8)	0.0282 (2)
O1	0.93046 (12)	0.09364 (13)	1.05429 (8)	0.0557 (4)
O2	0.76471 (11)	0.02876 (9)	1.02808 (8)	0.0417 (3)
O3	0.92469 (8)	0.11781 (8)	0.62155 (6)	0.0280 (2)
O4	0.76541 (11)	0.20800 (9)	0.59665 (7)	0.0403 (3)
C1	0.88309 (12)	0.15657 (9)	0.46702 (8)	0.0236 (2)
C2	0.80645 (13)	0.17488 (10)	0.39336 (9)	0.0272 (3)
C3	0.84243 (15)	0.16241 (11)	0.30152 (10)	0.0352 (3)
H3	0.7894	0.1738	0.2520	0.042*
C4	0.95451 (17)	0.13369 (11)	0.28236 (10)	0.0372 (4)
H4	0.9786	0.1251	0.2197	0.045*
C5	1.03234 (16)	0.11733 (11)	0.35436 (10)	0.0357 (3)
H5	1.1102	0.0989	0.3411	0.043*
C6	0.99620 (14)	0.12797 (10)	0.44555 (9)	0.0297 (3)
H6	1.0496	0.1155	0.4946	0.036*
C7	0.84707 (12)	0.16577 (10)	0.56623 (9)	0.0246 (3)
C11	0.90125 (11)	0.10896 (9)	0.71610 (8)	0.0219 (2)
C12	0.79705 (12)	0.07089 (9)	0.74716 (9)	0.0242 (3)
H12	0.7375	0.0541	0.7043	0.029*
C13	0.78119 (11)	0.05777 (9)	0.84200 (9)	0.0232 (2)
H13	0.7102	0.0325	0.8655	0.028*
C14	0.87097 (11)	0.08221 (9)	0.90168 (8)	0.0214 (2)
C15	0.97586 (11)	0.11932 (10)	0.87098 (9)	0.0233 (2)
H15	1.0360	0.1350	0.9137	0.028*
C16	0.99071 (11)	0.13298 (9)	0.77635 (9)	0.0235 (2)
H16	1.0615	0.1586	0.7530	0.028*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.02762 (19)	0.0729 (3)	0.0356 (2)	−0.00627 (18)	−0.00863 (14)	0.01702 (19)
N1	0.0323 (6)	0.0349 (6)	0.0174 (5)	−0.0006 (5)	0.0003 (4)	0.0019 (4)
O1	0.0494 (7)	0.0997 (12)	0.0180 (5)	−0.0213 (8)	−0.0083 (5)	0.0016 (6)
O2	0.0450 (7)	0.0552 (7)	0.0251 (5)	−0.0142 (6)	0.0084 (5)	0.0051 (5)
O3	0.0270 (5)	0.0425 (6)	0.0144 (4)	0.0076 (4)	0.0011 (3)	0.0034 (4)
O4	0.0442 (7)	0.0520 (7)	0.0246 (5)	0.0231 (5)	0.0002 (4)	−0.0003 (5)

C1	0.0312 (6)	0.0239 (6)	0.0155 (5)	−0.0014 (5)	−0.0013 (5)	0.0006 (4)
C2	0.0313 (7)	0.0293 (6)	0.0209 (6)	−0.0097 (5)	−0.0052 (5)	0.0051 (5)
C3	0.0509 (9)	0.0372 (8)	0.0174 (6)	−0.0186 (7)	−0.0075 (6)	0.0062 (5)
C4	0.0610 (10)	0.0341 (8)	0.0166 (6)	−0.0105 (7)	0.0064 (6)	−0.0017 (5)
C5	0.0484 (9)	0.0335 (7)	0.0251 (7)	0.0047 (7)	0.0102 (6)	−0.0009 (5)
C6	0.0362 (7)	0.0332 (7)	0.0196 (6)	0.0064 (6)	0.0015 (5)	−0.0004 (5)
C7	0.0293 (6)	0.0278 (6)	0.0167 (5)	0.0027 (5)	−0.0027 (5)	0.0002 (5)
C11	0.0243 (6)	0.0269 (6)	0.0146 (5)	0.0042 (5)	0.0005 (4)	0.0009 (4)
C12	0.0235 (6)	0.0304 (6)	0.0187 (5)	−0.0008 (5)	−0.0031 (4)	−0.0040 (5)
C13	0.0230 (6)	0.0263 (6)	0.0204 (5)	−0.0025 (5)	0.0011 (4)	−0.0008 (5)
C14	0.0249 (6)	0.0238 (6)	0.0155 (5)	0.0013 (5)	−0.0002 (4)	0.0005 (4)
C15	0.0214 (5)	0.0297 (6)	0.0189 (5)	−0.0001 (5)	−0.0034 (4)	0.0004 (5)
C16	0.0201 (5)	0.0298 (6)	0.0204 (5)	0.0009 (5)	0.0006 (4)	0.0031 (5)

Geometric parameters (Å, º)

Cl1—C2	1.7245 (16)	C4—H4	0.9500
N1—O1	1.2149 (17)	C5—C6	1.3827 (19)
N1—O2	1.2210 (16)	C5—H5	0.9500
N1—C14	1.4650 (16)	C6—H6	0.9500
O3—C7	1.3710 (16)	C11—C12	1.3842 (18)
O3—C11	1.3906 (14)	C11—C16	1.3849 (18)
O4—C7	1.1923 (17)	C12—C13	1.3873 (18)
C1—C6	1.394 (2)	C12—H12	0.9500
C1—C2	1.4001 (18)	C13—C14	1.3839 (18)
C1—C7	1.4901 (17)	C13—H13	0.9500
C2—C3	1.394 (2)	C14—C15	1.3842 (18)
C3—C4	1.376 (3)	C15—C16	1.3839 (17)
C3—H3	0.9500	C15—H15	0.9500
C4—C5	1.386 (2)	C16—H16	0.9500
O1—N1—O2	123.23 (12)	O4—C7—O3	122.88 (12)
O1—N1—C14	118.15 (12)	O4—C7—C1	127.74 (12)
O2—N1—C14	118.63 (12)	O3—C7—C1	109.38 (11)
C7—O3—C11	118.99 (10)	C12—C11—C16	122.23 (11)
C6—C1—C2	118.10 (12)	C12—C11—O3	121.12 (11)
C6—C1—C7	119.68 (12)	C16—C11—O3	116.45 (11)
C2—C1—C7	122.21 (13)	C11—C12—C13	118.78 (12)
C3—C2—C1	120.43 (14)	C11—C12—H12	120.6
C3—C2—Cl1	117.03 (11)	C13—C12—H12	120.6
C1—C2—Cl1	122.49 (11)	C14—C13—C12	118.56 (12)
C4—C3—C2	120.22 (14)	C14—C13—H13	120.7
C4—C3—H3	119.9	C12—C13—H13	120.7
C2—C3—H3	119.9	C13—C14—C15	122.93 (11)
C3—C4—C5	120.12 (13)	C13—C14—N1	118.30 (12)
C3—C4—H4	119.9	C15—C14—N1	118.77 (11)
C5—C4—H4	119.9	C16—C15—C14	118.20 (11)
C6—C5—C4	119.75 (15)	C16—C15—H15	120.9
C6—C5—H5	120.1	C14—C15—H15	120.9
C4—C5—H5	120.1	C15—C16—C11	119.28 (12)

C5—C6—C1	121.36 (14)	C15—C16—H16	120.4
C5—C6—H6	119.3	C11—C16—H16	120.4
C1—C6—H6	119.3		

C6—C1—C2—C3	1.5 (2)	C7—O3—C11—C12	54.46 (18)
C7—C1—C2—C3	−177.88 (13)	C7—O3—C11—C16	−130.52 (13)
C6—C1—C2—Cl1	178.88 (11)	C16—C11—C12—C13	1.0 (2)
C7—C1—C2—Cl1	−0.49 (19)	O3—C11—C12—C13	175.74 (12)
C1—C2—C3—C4	−1.2 (2)	C11—C12—C13—C14	−0.81 (19)
Cl1—C2—C3—C4	−178.77 (12)	C12—C13—C14—C15	0.1 (2)
C2—C3—C4—C5	−0.2 (2)	C12—C13—C14—N1	−179.49 (12)
C3—C4—C5—C6	1.4 (2)	O1—N1—C14—C13	−176.12 (15)
C4—C5—C6—C1	−1.1 (2)	O2—N1—C14—C13	4.49 (19)
C2—C1—C6—C5	−0.3 (2)	O1—N1—C14—C15	4.3 (2)
C7—C1—C6—C5	179.08 (14)	O2—N1—C14—C15	−175.11 (13)
C11—O3—C7—O4	7.1 (2)	C13—C14—C15—C16	0.4 (2)
C11—O3—C7—C1	−173.56 (11)	N1—C14—C15—C16	−179.97 (12)
C6—C1—C7—O4	162.13 (16)	C14—C15—C16—C11	−0.3 (2)
C2—C1—C7—O4	−18.5 (2)	C12—C11—C16—C15	−0.5 (2)
C6—C1—C7—O3	−17.14 (18)	O3—C11—C16—C15	−175.43 (12)
C2—C1—C7—O3	162.22 (12)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C4—H4···O1^{i}	0.95	2.48	3.3368 (19)	150

Symmetry code: (i) *x*, *y*, *z*−1.