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Ethyl 4-chloro-3-nitrobenzoate

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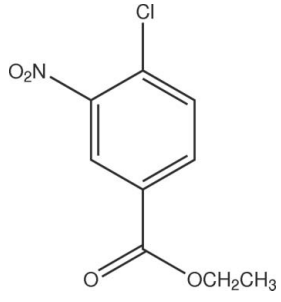
Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.003 Å; R factor = 0.046; wR factor = 0.130; data-to-parameter ratio = 14.6.

In the molecule of the title compound, $C_9H_8ClNO_4$, an intramolecular C—H···O hydrogen bond results in the formation of a planar five-membered ring, which is nearly coplanar with the adjacent six-membered ring, the rings being oriented at a dihedral angle of 4.40 (3)°. In the crystal structure, intermolecular C—H···O hydrogen bonds link the molecules.

Related literature

For related literature, see: Jönsson *et al.* (2004). For bond-length data, see: Allen *et al.* (1987).



Experimental

Crystal data

$C_9H_8ClNO_4$
M_r = 229.61
Monoclinic, $C2/c$
a = 12.930 (3) Å
b = 7.4820 (15) Å
c = 20.945 (4) Å
β = 92.11 (3)°
V = 2024.9 (7) Å^3
Z = 8
Mo $K\alpha$ radiation
μ = 0.37 mm^{-1}
T = 298 (2) K
0.40 × 0.30 × 0.10 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
Absorption correction: ψ scan (North *et al.*, 1968)
T_{min} = 0.866, T_{max} = 0.964
1984 measured reflections
1984 independent reflections
1449 reflections with $I > 2\sigma(I)$
3 standard reflections
frequency: 120 min
intensity decay: none

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.045
$wR(F^2)$ = 0.130
S = 1.06
1984 reflections
136 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.16 e Å^{-3}
$\Delta\rho_{min}$ = −0.21 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
C2—H2B···O2	0.97	2.29	2.706 (3)	104
C8—H8A···O2^{i}	0.93	2.53	3.357 (3)	148

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

Data collection: *CAD-4 Software* (Enraf–Nonius, 1989); cell refinement: *CAD-4 Software*; data reduction: *XCAD4* (Harms & Wocadlo, 1995); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Siemens, 1996); software used to prepare material for publication: *SHELXTL*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HK2403).

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supporting information

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Ethyl 4-chloro-3-nitrobenzoate

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S1. Comment

Some derivatives of benzoic acid are important chemical materials. As part of our ongoing studies, we synthesized the title compound, (I), and report herein its crystal structure.

In the molecule of (I), (Fig. 1) the bond lengths and angles are within normal ranges (Allen *et al.*, 1987). The intramolecular C—H···O hydrogen bond (Table 1) results in the formation of a planar five-membered ring B (C2/H2B/C3/O1/O2). Ring A (C4—C9) is, of course, planar and the dihedral angle between them is A/B = 4.40 (3)°. So, rings A and B are also nearly co-planar.

In the crystal structure, intermolecular C—H···O hydrogen bonds (Table 1) link the molecules (Fig. 2), in which they may be effective in the stabilization of the structure.

S2. Experimental

For the preparation of the title compound, 4-chloro-3-nitrobenzoic acid (35.0 g, 174 mmol) was suspended in ethanol (150 ml) and cooled to 273 K. Concentrated sulfuric acid (15 ml) was slowly added with stirring, and then the mixture was heated under reflux for 17 h. Upon cooling to room temperature, a precipitate formed, which was collected by filtration and washed with cold ethanol (2 × 50 ml) and hexane (2 × 50 ml) to afford the ethyl ester as a white solid (yield; 29.9 g, 75%) (Daniel *et al.*, 2004). Crystals of (I) suitable for X-ray analysis were obtained by slow evaporation of a methanol solution.

S3. Refinement

H atoms were positioned geometrically, with C—H = 0.93, 0.97 and 0.96 Å for aromatic, methylene and methyl H, respectively, and constrained to ride on their parent atoms, with U_{iso}(H) = xU_{eq}(C), where x = 1.5 for methyl H, and x = 1.2 for all other H atoms.

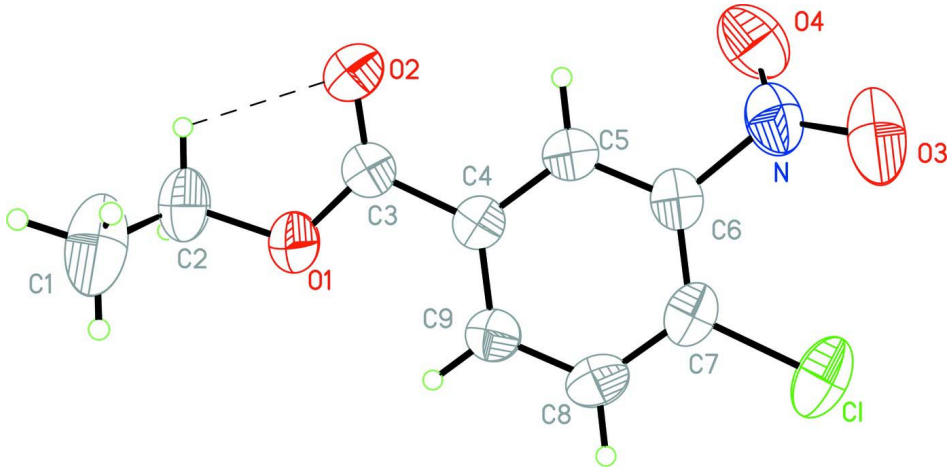

Figure 1

The molecular structure of the title molecule, with the atom-numbering scheme. Displacement ellipsoids are drawn at the 30% probability level.

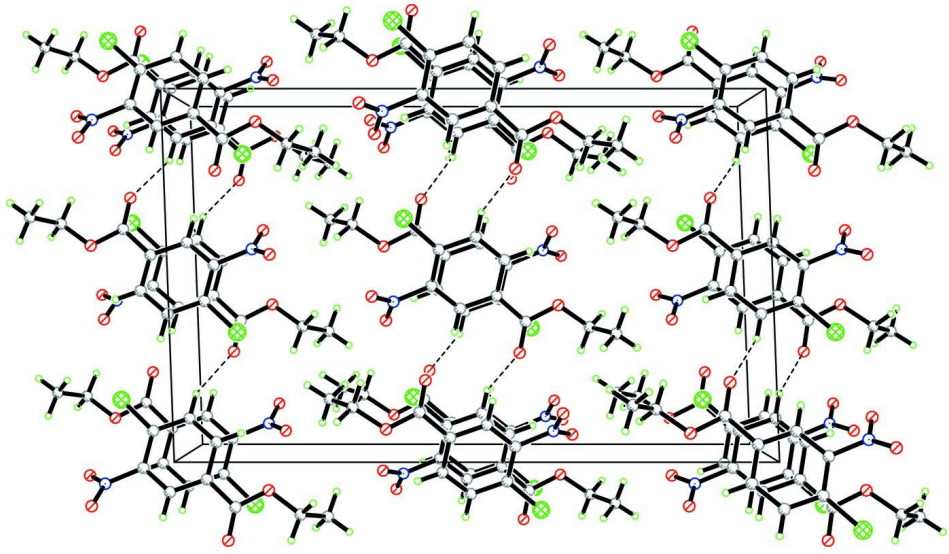

Figure 2

A packing diagram of (I). Hydrogen bonds are shown as dashed lines.

Ethyl 4-chloro-3-nitrobenzoate

Crystal data

$C_9H_8ClNO_4$
$M_r = 229.61$
Monoclinic, $C2/c$
Hall symbol: -C 2yc
$a = 12.930\ (3)$ Å
$b = 7.4820\ (15)$ Å
$c = 20.945\ (4)$ Å
$\beta = 92.11\ (3)°$
$V = 2024.9\ (7)$ Å^3
$Z = 8$

$F(000) = 944$
$D_x = 1.506$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 25 reflections
$\theta = 9–14°$
$\mu = 0.37$ mm^{-1}
$T = 298$ K
Block, colorless
0.40 × 0.30 × 0.10 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
$\omega/2\theta$ scans
Absorption correction: ψ scan (North *et al.*, 1968)
T_{min} = 0.866, T_{max} = 0.964
1984 measured reflections
1984 independent reflections
1449 reflections with $I > 2\sigma(I)$
R_{int} = 0.000
θ_{max} = 26.0°, θ_{min} = 2.0°
$h = -15 \rightarrow 15$
$k = 0 \rightarrow 9$
$l = 0 \rightarrow 25$
3 standard reflections every 120 min
intensity decay: none

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.045
$wR(F^2)$ = 0.130
S = 1.06
1984 reflections
136 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
$w = 1/[\sigma^2(F_o^2) + (0.06P)^2 + 1.5P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.16 e Å^{-3}
$\Delta\rho_{min}$ = −0.21 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}
Cl	1.15365 (6)	0.38995 (10)	0.39251 (4)	0.0722 (3)
O1	0.91716 (14)	0.1218 (3)	0.65000 (8)	0.0663 (6)
O2	0.78374 (14)	0.0870 (3)	0.58085 (9)	0.0627 (5)
O3	0.9430 (2)	0.3802 (4)	0.33433 (11)	0.1031 (9)
O4	0.87451 (19)	0.1219 (3)	0.35272 (10)	0.0825 (7)
N	0.92750 (19)	0.2507 (4)	0.36829 (10)	0.0628 (6)
C1	0.8393 (3)	0.2219 (5)	0.74482 (16)	0.0967 (12)
H1A	0.7999	0.1862	0.7807	0.145*
H1B	0.8023	0.3127	0.7210	0.145*
H1C	0.9051	0.2681	0.7598	0.145*
C2	0.8556 (3)	0.0672 (5)	0.70361 (13)	0.0801 (10)
H2A	0.8912	−0.0264	0.7277	0.096*
H2B	0.7894	0.0207	0.6879	0.096*
C3	0.87120 (18)	0.1287 (3)	0.59279 (11)	0.0427 (5)
C4	0.94310 (16)	0.1938 (3)	0.54379 (10)	0.0387 (5)

C5	0.90841 (17)	0.1923 (3)	0.48080 (10)	0.0408 (5)
H5A	0.8426	0.1492	0.4700	0.049*
C6	0.97087 (18)	0.2546 (3)	0.43393 (11)	0.0444 (5)
C7	1.06993 (18)	0.3170 (3)	0.44914 (11)	0.0459 (6)
C8	1.10485 (18)	0.3167 (3)	0.51210 (12)	0.0489 (6)
H8A	1.1711	0.3576	0.5229	0.059*
C9	1.04200 (17)	0.2561 (3)	0.55916 (11)	0.0446 (5)
H9A	1.0660	0.2569	0.6016	0.054*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl	0.0766 (5)	0.0632 (5)	0.0794 (5)	0.0006 (4)	0.0387 (4)	0.0099 (4)
O1	0.0616 (11)	0.0952 (15)	0.0424 (10)	−0.0104 (10)	0.0052 (8)	0.0096 (9)
O2	0.0497 (10)	0.0774 (13)	0.0612 (11)	−0.0127 (9)	0.0065 (8)	0.0051 (9)
O3	0.130 (2)	0.116 (2)	0.0628 (13)	−0.0096 (17)	0.0003 (14)	0.0378 (14)
O4	0.1026 (17)	0.0833 (16)	0.0601 (12)	0.0024 (14)	−0.0146 (11)	−0.0164 (11)
N	0.0721 (15)	0.0704 (16)	0.0461 (12)	0.0122 (13)	0.0057 (11)	0.0025 (12)
C1	0.109 (3)	0.109 (3)	0.074 (2)	0.028 (2)	0.037 (2)	0.012 (2)
C2	0.088 (2)	0.107 (3)	0.0461 (15)	−0.011 (2)	0.0176 (14)	0.0144 (16)
C3	0.0456 (13)	0.0368 (12)	0.0455 (13)	0.0046 (10)	0.0015 (10)	−0.0005 (10)
C4	0.0422 (11)	0.0301 (11)	0.0441 (12)	0.0041 (9)	0.0044 (9)	−0.0015 (9)
C5	0.0397 (11)	0.0347 (11)	0.0480 (13)	0.0031 (9)	0.0012 (9)	−0.0023 (10)
C6	0.0526 (13)	0.0385 (12)	0.0424 (12)	0.0085 (10)	0.0053 (10)	0.0005 (10)
C7	0.0517 (13)	0.0340 (12)	0.0531 (14)	0.0057 (10)	0.0157 (11)	0.0025 (10)
C8	0.0412 (12)	0.0402 (13)	0.0657 (16)	−0.0015 (10)	0.0054 (11)	−0.0038 (11)
C9	0.0424 (12)	0.0443 (13)	0.0472 (13)	0.0021 (10)	0.0009 (10)	−0.0032 (10)

Geometric parameters (Å, º)

Cl—C7	1.724 (2)	C2—H2B	0.9700
O1—C3	1.319 (3)	C3—C4	1.492 (3)
O1—C2	1.459 (3)	C4—C5	1.378 (3)
O2—C3	1.191 (3)	C4—C9	1.388 (3)
N—O3	1.222 (3)	C5—C6	1.375 (3)
N—O4	1.220 (3)	C5—H5A	0.9300
N—C6	1.466 (3)	C6—C7	1.389 (3)
C1—C2	1.463 (5)	C7—C8	1.378 (4)
C1—H1A	0.9600	C8—C9	1.377 (3)
C1—H1B	0.9600	C8—H8A	0.9300
C1—H1C	0.9600	C9—H9A	0.9300
C2—H2A	0.9700		
C3—O1—C2	118.0 (2)	C5—C4—C9	119.3 (2)
O4—N—O3	124.9 (3)	C5—C4—C3	117.84 (19)
O4—N—C6	117.3 (2)	C9—C4—C3	122.8 (2)
O3—N—C6	117.7 (3)	C6—C5—C4	120.1 (2)
C2—C1—H1A	109.5	C6—C5—H5A	119.9

C2—C1—H1B	109.5	C4—C5—H5A	119.9
H1A—C1—H1B	109.5	C5—C6—C7	120.7 (2)
C2—C1—H1C	109.5	C5—C6—N	116.6 (2)
H1A—C1—H1C	109.5	C7—C6—N	122.6 (2)
H1B—C1—H1C	109.5	C8—C7—C6	119.1 (2)
O1—C2—C1	109.1 (3)	C8—C7—Cl	117.85 (19)
O1—C2—H2A	109.9	C6—C7—Cl	123.07 (19)
C1—C2—H2A	109.9	C9—C8—C7	120.3 (2)
O1—C2—H2B	109.9	C9—C8—H8A	119.9
C1—C2—H2B	109.9	C7—C8—H8A	119.9
H2A—C2—H2B	108.3	C8—C9—C4	120.5 (2)
O2—C3—O1	125.0 (2)	C8—C9—H9A	119.8
O2—C3—C4	123.5 (2)	C4—C9—H9A	119.8
O1—C3—C4	111.5 (2)		

C3—O1—C2—C1	−109.8 (3)	C3—C4—C5—C6	178.6 (2)
C2—O1—C3—O2	−3.0 (4)	C5—C4—C9—C8	0.3 (3)
C2—O1—C3—C4	177.6 (2)	C3—C4—C9—C8	−179.2 (2)
O4—N—C6—C5	−38.6 (3)	C4—C5—C6—C7	1.0 (3)
O3—N—C6—C5	138.0 (3)	C4—C5—C6—N	−179.2 (2)
O4—N—C6—C7	141.2 (3)	C5—C6—C7—C8	−0.3 (3)
O3—N—C6—C7	−42.2 (3)	N—C6—C7—C8	179.8 (2)
O2—C3—C4—C5	−5.1 (3)	C5—C6—C7—Cl	177.82 (17)
O1—C3—C4—C5	174.2 (2)	N—C6—C7—Cl	−2.0 (3)
O2—C3—C4—C9	174.4 (2)	C6—C7—C8—C9	−0.3 (3)
O1—C3—C4—C9	−6.2 (3)	Cl—C7—C8—C9	−178.58 (18)
C9—C4—C5—C6	−1.0 (3)	C7—C8—C9—C4	0.3 (4)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C2—H2*B*···O2	0.97	2.29	2.706 (3)	104
C8—H8*A*···O2^{i}	0.93	2.53	3.357 (3)	148

Symmetry code: (i) *x*+1/2, *y*+1/2, *z*.