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Ethyl 4-fluoro-3-nitrobenzoate

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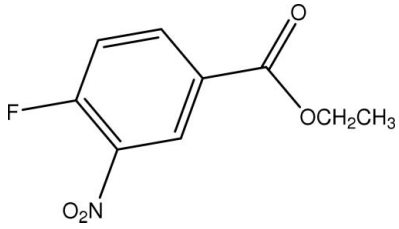
Key indicators: single-crystal X-ray study; T = 100 K; mean σ(C–C) = 0.002 Å; R factor = 0.045; wR factor = 0.139; data-to-parameter ratio = 21.1.

In the title compound, $C_9H_8FNO_4$, C—H···O intermolecular interactions form dimers with $R_2^2(10)$ motifs. These dimers are arranged into chains parallel to the b axis and the chains are stacked down the c axis.

Related literature

For general background, see: Ishida *et al.* (2006); Rida *et al.* (2005); Mohd. Maidin, Abdul Rahim, Abdul Hamid *et al.* (2008). For bond-length data, see: Allen *et al.* (1987). For related structures, see: Mohd. Maidin, Abdul Rahim, Osman *et al.* (2008); Li *et al.* (2008, 2009). For details of hydrogen-bond motifs, see: Bernstein *et al.* (1995). For details on the stability of the temperature controller, see: Cosier & Glazer (1986).



Experimental

Crystal data

$C_9H_8FNO_4$
M_r = 213.16
Monoclinic, $P2_1/c$
a = 9.9246 (3) Å
b = 13.2883 (3) Å
c = 6.9310 (2) Å
β = 94.410 (2)°
V = 911.36 (4) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.14 mm^{-1}
T = 100 K
0.55 × 0.22 × 0.09 mm

Data collection

Bruker SMART APEXII CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2005)
T_{min} = 0.929, T_{max} = 0.988
12540 measured reflections
2913 independent reflections
2411 reflections with $I > 2\sigma(I)$
R_{int} = 0.028

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.045
$wR(F^2)$ = 0.139
S = 1.11
2913 reflections
138 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.55 e Å^{-3}
$\Delta\rho_{min}$ = −0.36 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
C1—H1A···O4^{i}	0.93	2.44	3.2380 (15)	144

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

Data collection: *APEX2* (Bruker, 2005); cell refinement: *SAINT* (Bruker, 2005); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 2008); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL* and *PLATON* (Spek, 2009).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: AT2723).

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supplementary materials

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Ethyl 4-fluoro-3-nitrobenzoate

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Comment

The nitro benzoic acid intermediates are convenient starting materials for the synthesis of various biologically active heterocycles *e.g.* benzimidazoles (Ishida *et al.*, 2006) and benzoxazoles (Rida *et al.*, 2005). As a part of our ongoing studies on new nitro benzoic acid derivatives (Mohd. Maidin, Abdul Rahim, Abdul Hamid *et al.*, 2008), we have synthesized the title compound as an intermediate and report its structure here.

The bond lengths (Allen *et al.*, 1987) and angles observed in (I) are within normal ranges and are consistent with other related structures (Mohd. Maidin, Abdul Rahim, Osman *et al.*, 2008; Li *et al.*, 2009; Li *et al.*, 2008). The C1—H1A···O4^{i} intermolecular interactions (Table 2) linked the molecules into dimers forming 10-membered rings with $R^2_2(10)$ motifs (Bernstein *et al.*, 1995). In the crystal structure, these dimers are arranged into chains parallel to the *b* axis. The chains are stacked down the *c* axis (Fig. 2).

Experimental

For the preparation of the title compound, 4-fluoro-3-nitro-benzoic acid (5.0 g, 0.027 mol) was refluxed in absolute ethanol (50 ml) and conc. H_2SO_4 (2.0 ml) for 8 h. Upon reaction completion, ethanol was evaporated and the reaction mixture was diluted with water. The aqueous layer was extracted with ethyl acetate (25 *x* 2 ml). The combined organic layer was collected and dried over anhydrous $MgSO_4$. The solvent was removed under reduced pressure to afford yellow oil as the crude product. Recrystallization with hot ethyl acetate and petroleum ether (60–80) yielded colourless crystals that were found suitable for X-ray analysis.

Refinement

All the H atoms were positioned geometrically and refined using a riding model with C—H = 0.93Å for aromatic and 0.96Å for CH_3. The U_{iso} values were constrained to be -1.5U_{eq} of the carrier atom for the methyl H atoms and -1.2U_{equ} for the remaining hydrogen atoms. The rotating model group was considered for the methyl group.

Figures

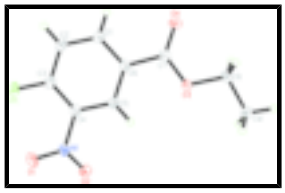

Fig. 1. The asymmetric unit of the title compound, showing 50% probability displacement ellipsoids and the atom numbering scheme.

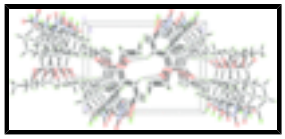

Fig. 2. The crystal packing of the title compound, viewed down the *c* axis. Intermolecular hydrogen bondings are shown as dotted lines.

Ethyl 4-fluoro-3-nitrobenzoate

Crystal data

$C_9H_8FNO_4$	F_{000} = 440
M_r = 213.16	D_x = 1.554 Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: -P 2ybc	Cell parameters from 5027 reflections
a = 9.9246 (3) Å	θ = 2.6–30.8º
b = 13.2883 (3) Å	μ = 0.14 mm^{-1}
c = 6.9310 (2) Å	T = 100 K
β = 94.410 (2)º	Block, colourless
V = 911.36 (4) Å^3	0.55 × 0.22 × 0.09 mm
Z = 4	

Data collection

Bruker SMART APEXII CCD area-detector diffractometer	2913 independent reflections
Radiation source: sealed tube	2411 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.028
T = 100 K	θ_{max} = 31.1º
φ and ω scans	θ_{min} = 2.1º
Absorption correction: multi-scan (SADABS; Bruker, 2005)	h = −14→14
T_{min} = 0.929, T_{max} = 0.988	k = −18→19
12540 measured reflections	l = −10→10

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)]$ = 0.045	$w = 1/[\sigma^2(F_o^2) + (0.0772P)^2 + 0.1955P]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2)$ = 0.139	$(\Delta/\sigma)_{max} < 0.001$
S = 1.11	$\Delta\rho_{max}$ = 0.55 e Å^{-3}
2913 reflections	$\Delta\rho_{min}$ = −0.36 e Å^{-3}
138 parameters	Extinction correction: SHELXL, $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$

Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Extinction coefficient: 0.018 (4)

Special details

Experimental. The crystal was placed in the cold stream of an Oxford Cryosystems Cobra open-flow nitrogen cryostat (Cosier & Glazer, 1986) operating at 100.0 (1) K.

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 (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
F1	0.04351 (7)	0.10679 (6)	0.18019 (12)	0.0241 (2)
O1	0.07165 (9)	0.29746 (7)	0.29028 (14)	0.0240 (2)
O2	−0.04092 (9)	0.39394 (6)	0.08249 (15)	0.0247 (2)
O3	−0.52330 (8)	0.30897 (6)	−0.00372 (14)	0.0203 (2)
O4	−0.58597 (9)	0.14638 (7)	−0.02358 (16)	0.0276 (2)
N1	−0.02296 (10)	0.31401 (7)	0.16902 (15)	0.0172 (2)
C1	−0.31523 (12)	0.08546 (9)	0.06256 (18)	0.0197 (2)
H1A	−0.3804	0.0356	0.0410	0.024*
C2	−0.18146 (12)	0.05833 (9)	0.10726 (19)	0.0211 (3)
H2A	−0.1566	−0.0091	0.1139	0.025*
C3	−0.08581 (11)	0.13301 (9)	0.14173 (17)	0.0181 (2)
C4	−0.12280 (11)	0.23409 (8)	0.12960 (16)	0.0154 (2)
C5	−0.25648 (11)	0.26145 (8)	0.08154 (16)	0.0154 (2)
H5A	−0.2807	0.3290	0.0710	0.018*
C6	−0.35338 (11)	0.18648 (8)	0.04945 (17)	0.0161 (2)
C7	−0.49936 (11)	0.21032 (9)	0.00323 (18)	0.0179 (2)
C8	−0.66549 (12)	0.33789 (10)	−0.0398 (2)	0.0231 (3)
H8A	−0.7170	0.3145	0.0648	0.028*
H8B	−0.7035	0.3081	−0.1597	0.028*
C9	−0.67127 (13)	0.45039 (10)	−0.0529 (2)	0.0280 (3)
H9A	−0.7638	0.4716	−0.0713	0.042*
H9B	−0.6233	0.4725	−0.1603	0.042*
H9C	−0.6304	0.4791	0.0646	0.042*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}

F1	0.0168 (3)	0.0239 (4)	0.0312 (4)	0.0077 (3)	0.0003 (3)	0.0020 (3)
O1	0.0153 (4)	0.0295 (5)	0.0264 (5)	0.0006 (3)	−0.0037 (3)	0.0008 (4)
O2	0.0228 (4)	0.0174 (4)	0.0333 (5)	−0.0018 (3)	−0.0025 (4)	0.0042 (4)
O3	0.0123 (4)	0.0155 (4)	0.0324 (5)	0.0009 (3)	−0.0017 (3)	0.0002 (3)
O4	0.0191 (4)	0.0176 (4)	0.0453 (6)	−0.0038 (3)	−0.0041 (4)	0.0012 (4)
N1	0.0141 (4)	0.0180 (5)	0.0196 (5)	0.0007 (3)	0.0017 (3)	−0.0015 (4)
C1	0.0201 (5)	0.0148 (5)	0.0242 (6)	−0.0008 (4)	0.0010 (4)	−0.0002 (4)
C2	0.0229 (6)	0.0140 (5)	0.0263 (6)	0.0037 (4)	0.0015 (5)	0.0004 (4)
C3	0.0166 (5)	0.0185 (5)	0.0192 (5)	0.0049 (4)	0.0015 (4)	0.0006 (4)
C4	0.0144 (5)	0.0158 (5)	0.0159 (5)	0.0007 (4)	0.0010 (4)	0.0001 (4)
C5	0.0144 (5)	0.0149 (5)	0.0169 (5)	0.0018 (4)	0.0014 (4)	0.0005 (4)
C6	0.0148 (5)	0.0152 (5)	0.0181 (5)	0.0005 (4)	0.0004 (4)	−0.0001 (4)
C7	0.0157 (5)	0.0162 (5)	0.0216 (5)	−0.0003 (4)	0.0005 (4)	0.0003 (4)
C8	0.0119 (5)	0.0218 (6)	0.0350 (7)	0.0007 (4)	−0.0013 (4)	0.0022 (5)
C9	0.0188 (6)	0.0213 (6)	0.0426 (8)	0.0040 (4)	−0.0050 (5)	−0.0051 (5)

Geometric parameters (Å, °)

F1—C3	1.3368 (13)	C3—C4	1.3932 (16)
O1—N1	1.2308 (13)	C4—C5	1.3912 (14)
O2—N1	1.2261 (13)	C5—C6	1.3906 (15)
O3—C7	1.3326 (14)	C5—H5A	0.9300
O3—C8	1.4653 (13)	C6—C7	1.4935 (15)
O4—C7	1.2127 (14)	C8—C9	1.4984 (19)
N1—C4	1.4638 (15)	C8—H8A	0.9700
C1—C2	1.3876 (16)	C8—H8B	0.9700
C1—C6	1.3959 (16)	C9—H9A	0.9600
C1—H1A	0.9300	C9—H9B	0.9600
C2—C3	1.3814 (17)	C9—H9C	0.9600
C2—H2A	0.9300		
C7—O3—C8	115.52 (9)	C5—C6—C1	119.85 (10)
O2—N1—O1	124.29 (10)	C5—C6—C7	122.00 (10)
O2—N1—C4	117.80 (9)	C1—C6—C7	118.14 (10)
O1—N1—C4	117.89 (10)	O4—C7—O3	124.15 (11)
C2—C1—C6	120.96 (11)	O4—C7—C6	123.28 (11)
C2—C1—H1A	119.5	O3—C7—C6	112.57 (9)
C6—C1—H1A	119.5	O3—C8—C9	107.69 (9)
C3—C2—C1	119.01 (11)	O3—C8—H8A	110.2
C3—C2—H2A	120.5	C9—C8—H8A	110.2
C1—C2—H2A	120.5	O3—C8—H8B	110.2
F1—C3—C2	118.93 (10)	C9—C8—H8B	110.2
F1—C3—C4	120.52 (10)	H8A—C8—H8B	108.5
C2—C3—C4	120.51 (10)	C8—C9—H9A	109.5
C5—C4—C3	120.56 (10)	C8—C9—H9B	109.5
C5—C4—N1	118.32 (10)	H9A—C9—H9B	109.5
C3—C4—N1	121.11 (10)	C8—C9—H9C	109.5
C6—C5—C4	119.08 (10)	H9A—C9—H9C	109.5
C6—C5—H5A	120.5	H9B—C9—H9C	109.5
C4—C5—H5A	120.5		

C6—C1—C2—C3	0.93 (19)	N1—C4—C5—C6	−178.04 (10)
C1—C2—C3—F1	−178.41 (11)	C4—C5—C6—C1	−1.07 (18)
C1—C2—C3—C4	−0.74 (19)	C4—C5—C6—C7	178.12 (10)
F1—C3—C4—C5	177.27 (10)	C2—C1—C6—C5	−0.02 (19)
C2—C3—C4—C5	−0.36 (18)	C2—C1—C6—C7	−179.25 (11)
F1—C3—C4—N1	−3.44 (17)	C8—O3—C7—O4	2.20 (18)
C2—C3—C4—N1	178.93 (11)	C8—O3—C7—C6	−177.35 (10)
O2—N1—C4—C5	−31.36 (16)	C5—C6—C7—O4	−179.29 (12)
O1—N1—C4—C5	146.88 (11)	C1—C6—C7—O4	−0.09 (19)
O2—N1—C4—C3	149.33 (12)	C5—C6—C7—O3	0.26 (16)
O1—N1—C4—C3	−32.42 (16)	C1—C6—C7—O3	179.47 (11)
C3—C4—C5—C6	1.27 (17)	C7—O3—C8—C9	−177.07 (11)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C1—H1A···O4^{i}	0.93	2.44	3.2380 (15)	144

Symmetry codes: (i) −*x*−1, −*y*, −*z*.

Fig. 1

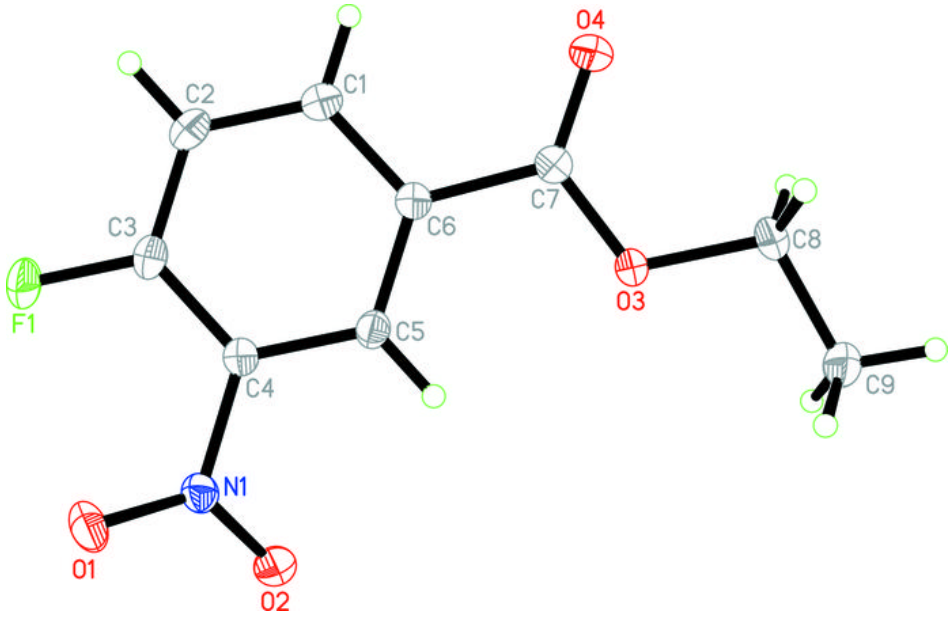

Fig. 2