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Ethyl 4-chloro-3,5-dinitrobenzoate

Hao Wu, Min-Hao Xie, Ya-Ling Liu, Yong-Jun He and Pei Zou*

Jiangsu Institute of Nuclear Medicine, Wuxi 214063, People's Republic of China
Correspondence e-mail: zou-pei@163.com

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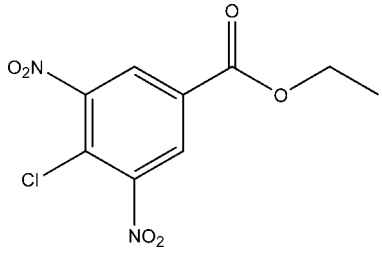
Key indicators: single-crystal X-ray study; T = 133 K; mean σ(C–C) = 0.003 Å; R factor = 0.041; wR factor = 0.098; data-to-parameter ratio = 15.5.

In the title compound, $C_9H_7ClN_2O_6$, the nitro groups and the ester group make dihedral angles of 44.0 (1), 89.6 (1) and 164.1 (1)°, respectively, with the benzene ring. In the crystal, molecules are linked through weak C—H···O hydrogen-bonding interactions. Molecules are stacked *via* π–π interactions about inversion centers, with a centroid–centroid distance of 3.671 (2) Å.

Related literature

For applications of the title compound as a herbicide and a related structure, see: Liu *et al.* (2010).



Experimental

Crystal data

$C_9H_7ClN_2O_6$ M_r = 274.62
Monoclinic, $P2_1/c$
a = 7.744 (2) Å
b = 21.389 (6) Å
c = 7.241 (2) Å
β = 110.504 (4)°
V = 1123.3 (5) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.36 mm^{-1}
T = 133 K
0.30 × 0.20 × 0.10 mm

Data collection

Rigaku SPIDER diffractometer
Absorption correction: multi-scan (*ABSCOR*; Higashi, 1995)
T_{min} = 0.899, T_{max} = 0.965
8777 measured reflections
2549 independent reflections
1939 reflections with $I > 2\sigma(I)$
R_{int} = 0.034

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.041
$wR(F^2)$ = 0.098
S = 1.00
2549 reflections
164 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.51 e Å^{-3}
$\Delta\rho_{min}$ = −0.32 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
C5—H5···O2^{i}	0.95	2.32	3.157 (3)	147

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

Data collection: *RAPID-AUTO* (Rigaku, 2004); cell refinement: *RAPID-AUTO*; data reduction: *RAPID-AUTO*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); 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: PV2450).

References

Higashi, T. (1995). *ABSCOR*. Rigaku Corporation, Tokyo, Japan.
Liu, Y.-L., Zou, P., Xie, M.-H., Wu, H. & He, Y.-J. (2010). *Acta Cryst.* E**66**, o62.
Rigaku (2004). *RAPID-AUTO*. Rigaku Corporation, Tokyo, Japan.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.

supporting information

Acta Cryst. (2011). E**67**, o2917 [doi:10.1107/S160053681103978X]

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S1. Comment

The title compound is useful as a herbicide (Liu *et al.*, 2010). In the title molecule (Fig. 1), two nitro groups (O3/N1/O4 and O5/N2/O6) attached at C2 and C4 and the ester group (O1/C7/O2) attached at C6 form dihedral angles of 44.0 (1), 89.6 (1) and 164.1 (1)°, respectively, with the mean plane of the benzene ring (C1–C6). In the cyrstal structure, the molecules are linked through weak C—H···O hydrogen bonding interactions. The molecules are stacked *via* π-π interactions, about inversion centers with the ring centroid-centroid distance of 3.671 (2) Å.

S2. Experimental

A sample of commercial ethyl 4-chloro-3,5-dinitrobenzoate (Aldrich) was crystalized by slow evaporation of a solution in methanol yielding colorless chunky crystals after several days.

S3. Refinement

H atoms were placed in calculated positions with C—H = 0.99, 0.98 or 0.95 Å for methylene, methyl or aryl type H-atoms, respectively, and were refined in a riding mode with U_{iso}(H) = 1.2 or 1.5U_{eq}(C).

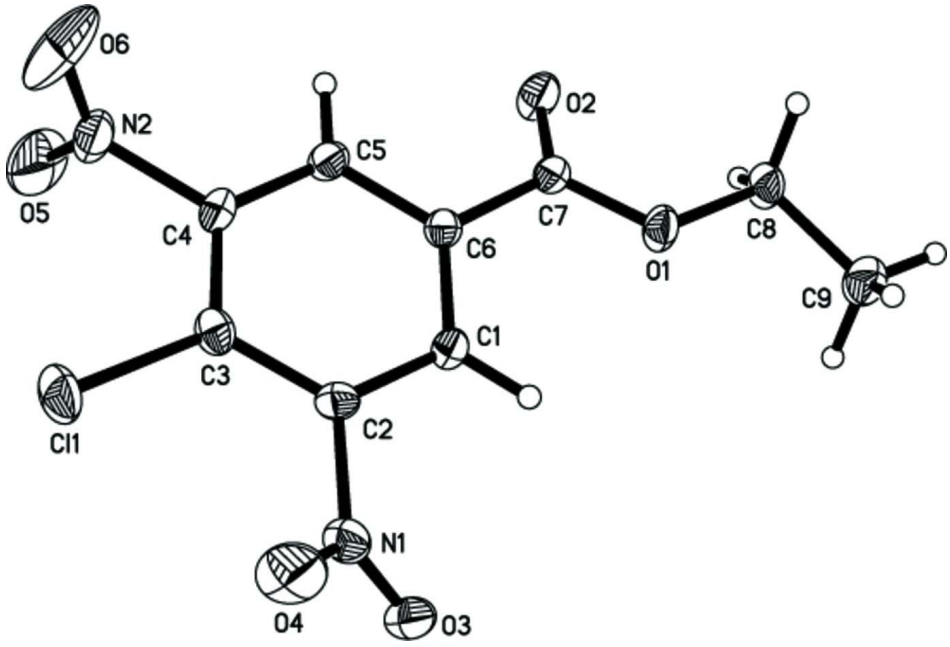

Figure 1

A view of the title compound with the atomic numbering scheme. Displacement ellipsoids were drawn at the 50% probability level.

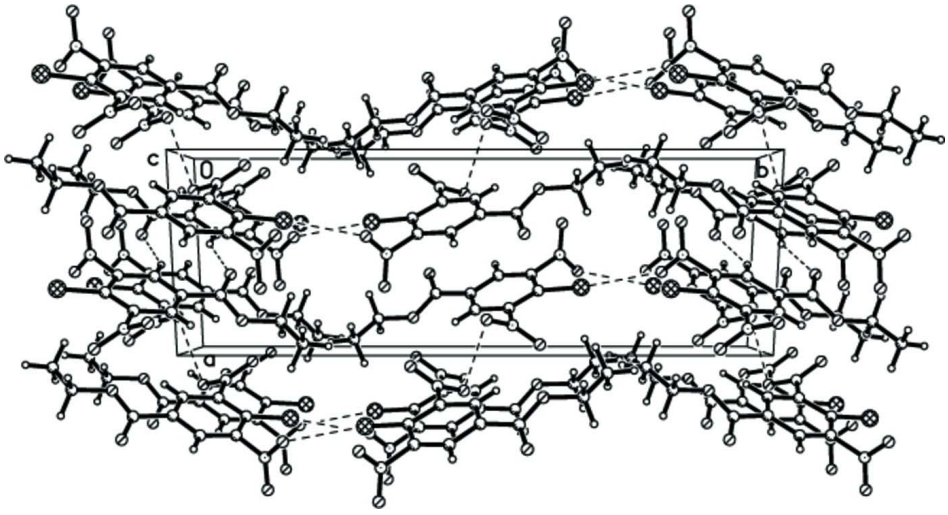

Figure 2

Part of the packing of the title compound, viewed down the c direction; dashed lines indicate hydrogen bonds.

Ethyl 4-chloro-3,5-dinitrobenzoate

Crystal data

$C_9H_7ClN_2O_6$
$M_r = 274.62$
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
$a = 7.744$ (2) Å
$b = 21.389$ (6) Å
$c = 7.241$ (2) Å
$\beta = 110.504$ (4)°
$V = 1123.3$ (5) Å^3
$Z = 4$
$F(000) = 560$
$D_x = 1.624$ Mg m^{-3}
Melting point: 357(2) K
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 2718 reflections
$\theta = 3.2$–$27.5°$
$\mu = 0.36$ mm^{-1}
$T = 133$ K
Block, colorless
0.30 × 0.20 × 0.10 mm

Data collection

Rigaku SPIDER diffractometer
Radiation source: Rotating Anode
Graphite monochromator
ω scans
Absorption correction: multi-scan (*ABSCOR*; Higashi, 1995)
$T_{min} = 0.899$, $T_{max} = 0.965$
8777 measured reflections
2549 independent reflections
1939 reflections with $I > 2\sigma(I)$
$R_{int} = 0.034$
$\theta_{max} = 27.5°$, $\theta_{min} = 3.8°$
$h = -8 \rightarrow 10$
$k = -27 \rightarrow 27$
$l = -9 \rightarrow 9$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.041$
$wR(F^2) = 0.098$
$S = 1.00$
2549 reflections
164 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
$w = 1/[\sigma^2(F_o^2) + (0.0496P)^2 + 0.269P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.51$ e Å^{-3}
$\Delta\rho_{min} = -0.32$ 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	0.66615 (7)	0.68101 (2)	0.67393 (8)	0.02902 (15)
O1	0.82698 (18)	0.39122 (6)	0.43011 (19)	0.0229 (3)
O2	0.6015 (2)	0.41837 (6)	0.14870 (19)	0.0283 (3)
O3	0.82102 (19)	0.51915 (7)	1.00581 (19)	0.0269 (3)
O4	0.9456 (2)	0.60842 (7)	0.9835 (2)	0.0402 (4)
O5	0.3393 (3)	0.65336 (10)	0.2213 (3)	0.0610 (6)
O6	0.5882 (3)	0.68236 (8)	0.1760 (3)	0.0562 (6)
N1	0.8513 (2)	0.56285 (8)	0.9112 (2)	0.0237 (4)
N2	0.5021 (3)	0.64949 (8)	0.2496 (2)	0.0286 (4)
C1	0.7812 (2)	0.49987 (8)	0.6145 (3)	0.0172 (4)
H1	0.8417	0.4660	0.6966	0.021*
C2	0.7714 (2)	0.55765 (8)	0.6956 (3)	0.0177 (4)
C3	0.6846 (2)	0.60877 (8)	0.5805 (3)	0.0189 (4)
C4	0.6034 (2)	0.59805 (8)	0.3785 (3)	0.0194 (4)
C5	0.6100 (3)	0.54110 (8)	0.2923 (3)	0.0194 (4)
H5	0.5530	0.5357	0.1539	0.023*
C6	0.7015 (2)	0.49173 (8)	0.4111 (3)	0.0166 (4)
C7	0.7039 (2)	0.43024 (8)	0.3140 (3)	0.0178 (4)
C8	0.8358 (3)	0.32869 (8)	0.3507 (3)	0.0232 (4)
H8A	0.9010	0.3303	0.2551	0.028*
H8B	0.7101	0.3121	0.2829	0.028*
C9	0.9385 (3)	0.28811 (10)	0.5225 (3)	0.0317 (5)
H9A	1.0599	0.3064	0.5927	0.048*
H9B	0.9540	0.2463	0.4752	0.048*
H9C	0.8689	0.2851	0.6121	0.048*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.0369 (3)	0.0164 (2)	0.0335 (3)	0.0007 (2)	0.0121 (2)	−0.00696 (19)
O1	0.0279 (8)	0.0157 (6)	0.0196 (7)	0.0058 (5)	0.0016 (6)	−0.0021 (5)
O2	0.0401 (9)	0.0187 (7)	0.0173 (7)	0.0037 (6)	−0.0012 (6)	−0.0029 (5)
O3	0.0299 (8)	0.0321 (8)	0.0197 (7)	0.0041 (6)	0.0102 (6)	0.0041 (6)
O4	0.0530 (10)	0.0306 (9)	0.0267 (8)	−0.0122 (7)	0.0010 (7)	−0.0100 (6)
O5	0.0495 (12)	0.0778 (15)	0.0620 (13)	0.0438 (10)	0.0275 (10)	0.0343 (11)
O6	0.0571 (12)	0.0344 (10)	0.0572 (12)	−0.0177 (8)	−0.0049 (9)	0.0259 (9)

N1	0.0249 (9)	0.0255 (9)	0.0190 (8)	0.0027 (7)	0.0058 (7)	−0.0032 (7)
N2	0.0392 (11)	0.0175 (9)	0.0226 (9)	0.0054 (8)	0.0027 (8)	0.0015 (7)
C1	0.0177 (9)	0.0149 (8)	0.0176 (9)	0.0010 (7)	0.0046 (7)	0.0016 (7)
C2	0.0189 (10)	0.0195 (9)	0.0140 (9)	−0.0019 (7)	0.0048 (7)	−0.0009 (7)
C3	0.0194 (10)	0.0146 (9)	0.0233 (9)	−0.0019 (7)	0.0081 (8)	−0.0029 (7)
C4	0.0211 (10)	0.0151 (9)	0.0211 (9)	0.0026 (7)	0.0063 (8)	0.0042 (7)
C5	0.0224 (10)	0.0180 (9)	0.0164 (9)	0.0001 (7)	0.0051 (8)	0.0015 (7)
C6	0.0180 (9)	0.0146 (8)	0.0172 (8)	−0.0006 (7)	0.0062 (7)	−0.0001 (7)
C7	0.0210 (10)	0.0153 (9)	0.0179 (9)	0.0010 (7)	0.0077 (8)	0.0017 (7)
C8	0.0300 (11)	0.0140 (9)	0.0230 (10)	0.0032 (8)	0.0062 (8)	−0.0029 (7)
C9	0.0426 (13)	0.0225 (11)	0.0285 (11)	0.0077 (9)	0.0104 (10)	0.0031 (8)

Geometric parameters (Å, º)

Cl1—C3	1.7125 (19)	C2—C3	1.396 (3)
O1—C7	1.323 (2)	C3—C4	1.394 (3)
O1—C8	1.467 (2)	C4—C5	1.378 (3)
O2—C7	1.209 (2)	C5—C6	1.389 (2)
O3—N1	1.229 (2)	C5—H5	0.9500
O4—N1	1.220 (2)	C6—C7	1.495 (2)
O5—N2	1.207 (2)	C8—C9	1.497 (3)
O6—N2	1.213 (2)	C8—H8A	0.9900
N1—C2	1.468 (2)	C8—H8B	0.9900
N2—C4	1.478 (2)	C9—H9A	0.9800
C1—C2	1.382 (2)	C9—H9B	0.9800
C1—C6	1.394 (2)	C9—H9C	0.9800
C1—H1	0.9500		
C7—O1—C8	116.64 (14)	C4—C5—H5	120.6
O4—N1—O3	124.79 (17)	C6—C5—H5	120.6
O4—N1—C2	118.86 (16)	C5—C6—C1	120.02 (16)
O3—N1—C2	116.32 (16)	C5—C6—C7	117.72 (16)
O5—N2—O6	126.00 (19)	C1—C6—C7	122.22 (16)
O5—N2—C4	116.76 (18)	O2—C7—O1	124.99 (17)
O6—N2—C4	117.18 (18)	O2—C7—C6	122.60 (16)
C2—C1—C6	119.39 (16)	O1—C7—C6	112.40 (15)
C2—C1—H1	120.3	O1—C8—C9	106.70 (16)
C6—C1—H1	120.3	O1—C8—H8A	110.4
C1—C2—C3	122.25 (17)	C9—C8—H8A	110.4
C1—C2—N1	117.00 (16)	O1—C8—H8B	110.4
C3—C2—N1	120.72 (16)	C9—C8—H8B	110.4
C4—C3—C2	116.31 (16)	H8A—C8—H8B	108.6
C4—C3—Cl1	119.57 (14)	C8—C9—H9A	109.5
C2—C3—Cl1	124.07 (15)	C8—C9—H9B	109.5
C5—C4—C3	123.11 (17)	H9A—C9—H9B	109.5
C5—C4—N2	117.89 (17)	C8—C9—H9C	109.5
C3—C4—N2	118.99 (16)	H9A—C9—H9C	109.5
C4—C5—C6	118.89 (17)	H9B—C9—H9C	109.5

C6—C1—C2—C3	−0.3 (3)	O5—N2—C4—C3	−90.7 (2)
C6—C1—C2—N1	177.67 (16)	O6—N2—C4—C3	92.1 (2)
O4—N1—C2—C1	136.26 (18)	C3—C4—C5—C6	0.1 (3)
O3—N1—C2—C1	−41.8 (2)	N2—C4—C5—C6	−178.79 (17)
O4—N1—C2—C3	−45.8 (3)	C4—C5—C6—C1	1.4 (3)
O3—N1—C2—C3	136.14 (17)	C4—C5—C6—C7	179.03 (16)
C1—C2—C3—C4	1.6 (3)	C2—C1—C6—C5	−1.3 (3)
N1—C2—C3—C4	−176.24 (16)	C2—C1—C6—C7	−178.82 (16)
C1—C2—C3—Cl1	179.17 (14)	C8—O1—C7—O2	−1.0 (3)
N1—C2—C3—Cl1	1.3 (3)	C8—O1—C7—C6	177.99 (15)
C2—C3—C4—C5	−1.5 (3)	C5—C6—C7—O2	−15.2 (3)
Cl1—C3—C4—C5	−179.18 (15)	C1—C6—C7—O2	162.35 (18)
C2—C3—C4—N2	177.30 (17)	C5—C6—C7—O1	165.76 (16)
Cl1—C3—C4—N2	−0.4 (2)	C1—C6—C7—O1	−16.6 (2)
O5—N2—C4—C5	88.2 (2)	C7—O1—C8—C9	−163.27 (17)
O6—N2—C4—C5	−89.0 (2)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C5—H5···O2^{i}	0.95	2.32	3.157 (3)	147

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