Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

Methyl 4-chloro-3-nitrobenzoate

Bo-Nian Liu,[a] Shi-Gui Tang,[b] Hao-Yuan Li,[a] Ye-Ming Xu[a] and Cheng Guo[a]*

[a]College of Science, Nanjing University of Technology, Xinmofan Road No. 5, Nanjing 210009, People's Republic of China, and [b]College of Life Sciences and Pharmaceutical Engineering, Nanjing University of Technology, Nanjing 210009, People's Republic of China
Correspondence e-mail: guocheng@njut.edu.cn

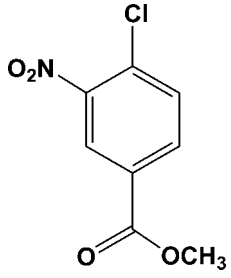
Received 19 November 2007; accepted 16 December 2007

Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.004 Å; R factor = 0.045; wR factor = 0.142; data-to-parameter ratio = 13.9.

In the title compound, $C_8H_6ClNO_4$, the molecules are linked by C—H···O interactions to form a chain parallel to the a axis. The chains are further connected by slipped π–π stacking between symmetry-related benzene rings, with a centroid-to-centroid distance of 3.646 (2) Å and an interplanar distance of 3.474 Å, resulting in an offset of 1.106 Å.

Related literature

For related literature, see: de Souza *et al.* (2006); Jin & Xiao (2005); Spiniello & White (2003); Jönssen *et al.* (2004); Andrews & Ladlow (2003).



Experimental

Crystal data

$C_8H_6ClNO_4$
M_r = 215.59
Triclinic, $P\overline{1}$
a = 7.338 (1) Å
b = 7.480 (1) Å
c = 9.715 (2) Å
α = 98.39 (3)°
β = 94.89 (3)°
γ = 118.95 (3)°
V = 454.1 (2) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.41 mm^{-1}
T = 293 (2) K
0.40 × 0.10 × 0.10 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
Absorption correction: ψ scan (North *et al.*, 1968)
T_{min} = 0.854, T_{max} = 0.961
1918 measured reflections
1773 independent reflections
1389 reflections with $I > 2\sigma(I)$
R_{int} = 0.019
3 standard reflections
every 200 reflections
intensity decay: none

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.046
$wR(F^2)$ = 0.142
S = 1.12
1773 reflections
128 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.21 e Å^{-3}
$\Delta\rho_{min}$ = −0.24 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}	0.93	2.47	3.272 (3)	145

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

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, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEPIII* (Burnett & Johnson, 1996), *ORTEP-3 for Windows* (Farrugia, 1997) and *PLATON* (Spek, 2003); software used to prepare material for publication: *SHELXL97*.

The authors thank the Center for Testing and Analysis, Nanjing University, for support.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: DN2295).

References

Andrews, S. P. & Ladlow, M. (2003). *J. Org. Chem.* **68**, 5525–5533.
Burnett, M. N. & Johnson, C. K. (1996). *ORTEPIII*. Report ORNL-6895. Oak Ridge National Laboratory, Tennessee, USA.
Enraf–Nonius (1989). *CAD-4 Software*. Version 5.0. Enraf–Nonius, Delft, The Netherlands.
Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
Harms, K. & Wocadlo, S. (1995). *XCAD4*. University of Marburg, Germany.
Jin, L.-F. & Xiao, F.-P. (2005). *Acta Cryst.* E**61**, o1237–o1238.
Jönssen, D., Warrington, B. H. & Ladlow, M. (2004). *J. Comb. Chem.* **6**, 584–595.
North, A. C. T., Phillips, D. C. & Mathews, F. S. (1968). *Acta Cryst.* A**24**, 351–359.
Sheldrick, G. M. (1997). *SHELXL97* and *SHELXS97*. University of Göttingen, Germany.
Souza, M. V. N. de, Vasconcelos, T. R. A., Wardell, S. M. S. V., Wardell, J. L., Low, J. N. & Glidewell, C. (2006). *Acta Cryst.* C**62**, o295–o298.
Spek, A. L. (2003). *J. Appl. Cryst.* **36**, 7–13.
Spiniello, M. & White, J. M. (2003). *Org. Biomol. Chem.* **1**, 3094–3096.

  doi:10.1107/S1600536807067219

supporting information

Acta Cryst. (2008). E**64**, o456 [doi:10.1107/S1600536807067219]

Methyl 4-chloro-3-nitrobenzoate

Bo-Nian Liu, Shi-Gui Tang, Hao-Yuan Li, Ye-Ming Xu and Cheng Guo

S1. Comment

Some derivatives of benzoic acid are important chemical materials. We report here the crystal structure of the title compound, (I).

In compound (I) the nitro group is twisted with respect to the phenyl ring making a dihedral angle of 45.4 (1)° (Fig. 1). Similar twisted conformations are observed in related structures where the aryl ring bears nitro and halide adjacent to each other (de Souza *et al.*, 2006; Spiniello & White, 2003), whereas a planar conformation is observed in other case (Jin & Xiao, 2005).

The molecules of (I) are linked by C—H···O interactions to form a chain parallel to the *a* axis (Table 1, Fig. 2). The chains are further connected by slippest *π*–*π* stacking between symmetry related phenyl rings with a centroit to centroid distance *Cg*1···*Cg*1i (Symmetry code: (i) 1 - *x*, 1 - *y*, 1 - *z*) of 3.646 (2) Å and an interplanar distance of 3.474 Å resulting in an offset of 1.106 Å.

S2. Experimental

4-Chloro-3-nitrobenzoic acid (35.0 g, 0.174 mol) was suspended in methanol (150 ml) and cooled to 0°. Concentrated sulfuric acid (15 ml) was slowly added with stirring, and then the mixture was heated at reflux for 17 h. Upon cooling to room temperature, a precipitate formed, which was collected by filtration and washed with cold methanol (2*50 ml) and hexane (2*50 ml) to afford the methyl ester as a white solid (31.8 g, 85%) (Andrews & Ladlow, 2003; Jönssen *et al.*, 2004). Pure compound (I) was obstained by crystallizing from methanol. Crystals of (I) suitable for X-ray diffraction were obstained by slow evaporation of an methanol solution.

S3. Refinement

All H atoms were placed geometrically and treated as riding on their parent C atoms with C—H = 0.93 Å (Caromatic) and 0.96 Å (Cmethyl) with U_{iso}(H) = 1.2(Caromatic) or 1.5(methyl)U_{eq} (C).

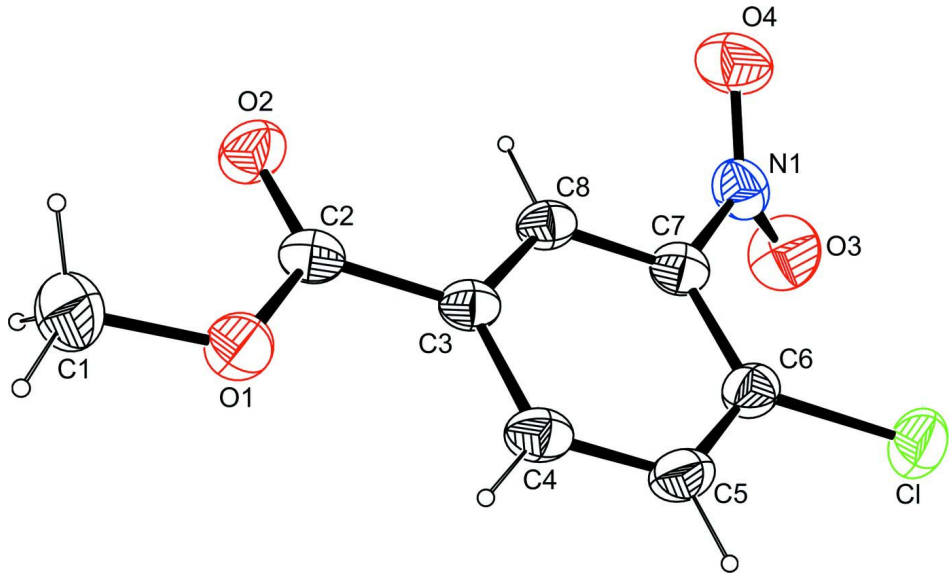


Figure 1

The molecular structure of (I), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level. H atoms are represented as small spheres of arbitrary radii.

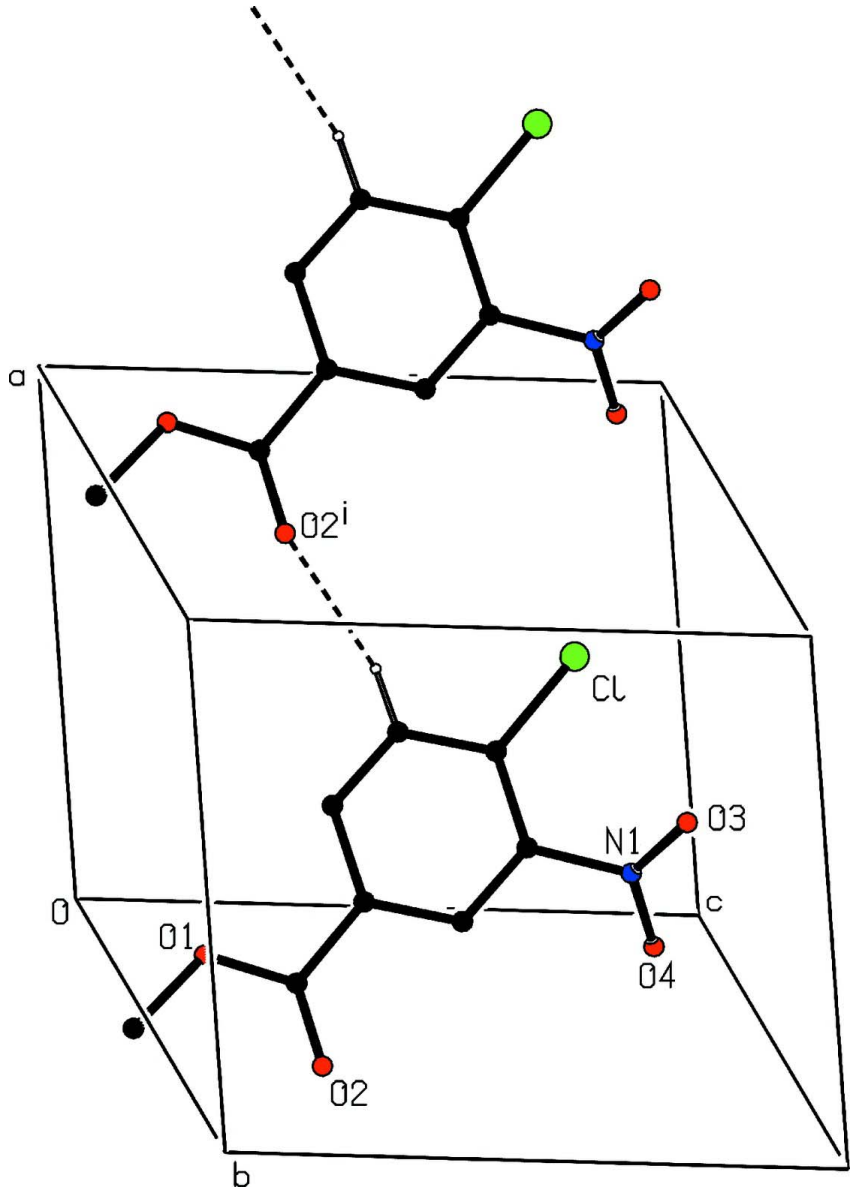


Figure 2

Partial packing view of (I) showing the formation of the chain through C—H···O hydrogen bonds indicated as dashed lines. H atoms not involved in hydrogen bondings have been omitted for clarity. [Symmetry code: (i) 1 + *x*, *y*, *z*]

Methyl 4-chloro-3-nitrobenzoate

Crystal data

$C_8H_6ClNO_4$
$M_r = 215.59$
Triclinic, $P\overline{1}$
Hall symbol: -P 1
$a = 7.338$ (1) Å
$b = 7.480$ (1) Å
$c = 9.715$ (2) Å
$\alpha = 98.39$ (3)°
$\beta = 94.89$ (3)°
$\gamma = 118.95$ (3)°
$V = 454.1$ (2) Å^3
$Z = 2$
$F(000) = 220$
$D_x = 1.577$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 25 reflections
$\theta = 9$–13°
$\mu = 0.41$ mm^{-1}

T = 293 K
Box, colourless

0.40 × 0.10 × 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} = 0.854, T_{max} = 0.961
1918 measured reflections

1773 independent reflections
1389 reflections with $I > 2\sigma(I)$
R_{int} = 0.019
θ_{max} = 26.0°, θ_{min} = 2.2°
h = 0→9
k = −9→8
l = −11→11
3 standard reflections every 200 reflections
intensity decay: none

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.046
$wR(F^2)$ = 0.142
S = 1.12
1773 reflections
128 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
$w = 1/[\sigma^2(F_o^2) + (0.0588P)^2 + 0.2181P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.21 e Å^{-3}
$\Delta\rho_{min}$ = −0.24 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}
C1	−0.2034 (5)	0.0805 (6)	0.0613 (3)	0.0722 (9)
H1A	−0.3435	−0.0080	0.0779	0.108*
H1B	−0.1832	0.0171	−0.0248	0.108*
H1C	−0.1857	0.2143	0.0536	0.108*
C2	−0.0585 (4)	0.1907 (4)	0.3056 (3)	0.0454 (6)
C3	0.1131 (4)	0.2225 (4)	0.4166 (3)	0.0417 (6)
C4	0.2701 (4)	0.1747 (4)	0.3866 (3)	0.0502 (7)
H4	0.2672	0.1180	0.2941	0.060*
C5	0.4285 (4)	0.2108 (4)	0.4925 (3)	0.0536 (7)
H5	0.5306	0.1769	0.4709	0.064*
C6	0.4376 (4)	0.2965 (4)	0.6299 (3)	0.0497 (6)
C7	0.2808 (4)	0.3452 (4)	0.6588 (3)	0.0459 (6)
C8	0.1197 (4)	0.3055 (4)	0.5547 (3)	0.0425 (6)

H8	0.0146	0.3344	0.5771	0.051*
Cl	0.63808 (12)	0.33572 (14)	0.75859 (9)	0.0731 (3)
N1	0.2849 (4)	0.4450 (4)	0.8022 (2)	0.0569 (6)
O1	−0.0490 (3)	0.1079 (3)	0.1782 (2)	0.0591 (5)
O2	−0.1910 (3)	0.2373 (3)	0.3279 (2)	0.0626 (6)
O3	0.4525 (4)	0.5930 (4)	0.8665 (2)	0.0838 (7)
O4	0.1169 (4)	0.3786 (4)	0.8445 (2)	0.0801 (7)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.075 (2)	0.089 (2)	0.0466 (17)	0.0412 (19)	0.0021 (15)	0.0024 (15)
C2	0.0449 (14)	0.0383 (13)	0.0488 (15)	0.0175 (11)	0.0148 (11)	0.0063 (11)
C3	0.0406 (13)	0.0343 (12)	0.0489 (14)	0.0170 (10)	0.0144 (11)	0.0086 (10)
C4	0.0533 (15)	0.0490 (15)	0.0560 (16)	0.0298 (13)	0.0225 (13)	0.0108 (12)
C5	0.0475 (15)	0.0572 (16)	0.0691 (18)	0.0328 (13)	0.0226 (13)	0.0198 (14)
C6	0.0412 (13)	0.0462 (14)	0.0633 (17)	0.0207 (12)	0.0122 (12)	0.0191 (12)
C7	0.0466 (14)	0.0388 (13)	0.0491 (15)	0.0187 (11)	0.0126 (11)	0.0085 (11)
C8	0.0399 (13)	0.0363 (12)	0.0532 (15)	0.0199 (10)	0.0147 (11)	0.0084 (10)
Cl	0.0564 (5)	0.0836 (6)	0.0821 (6)	0.0363 (4)	0.0021 (4)	0.0278 (4)
N1	0.0682 (16)	0.0606 (15)	0.0451 (13)	0.0349 (13)	0.0112 (12)	0.0106 (11)
O1	0.0626 (12)	0.0725 (13)	0.0444 (10)	0.0400 (11)	0.0080 (9)	−0.0024 (9)
O2	0.0584 (12)	0.0832 (15)	0.0574 (12)	0.0468 (11)	0.0118 (9)	0.0049 (10)
O3	0.0794 (16)	0.0785 (16)	0.0661 (15)	0.0294 (13)	−0.0085 (12)	−0.0115 (12)
O4	0.0839 (17)	0.0938 (18)	0.0647 (14)	0.0446 (14)	0.0324 (13)	0.0128 (12)

Geometric parameters (Å, º)

C1—O1	1.450 (4)	C4—H4	0.9300
C1—H1A	0.9600	C5—C6	1.375 (4)
C1—H1B	0.9600	C5—H5	0.9300
C1—H1C	0.9600	C6—C7	1.402 (4)
C2—O2	1.207 (3)	C6—Cl	1.725 (3)
C2—O1	1.324 (3)	C7—C8	1.370 (4)
C2—C3	1.486 (4)	C7—N1	1.471 (3)
C3—C8	1.380 (4)	C8—H8	0.9300
C3—C4	1.402 (3)	N1—O3	1.215 (3)
C4—C5	1.376 (4)	N1—O4	1.220 (3)
O1—C1—H1A	109.5	C6—C5—H5	119.6
O1—C1—H1B	109.5	C4—C5—H5	119.6
H1A—C1—H1B	109.5	C5—C6—C7	118.1 (3)
O1—C1—H1C	109.5	C5—C6—Cl	118.7 (2)
H1A—C1—H1C	109.5	C7—C6—Cl	123.1 (2)
H1B—C1—H1C	109.5	C8—C7—C6	121.6 (2)
O2—C2—O1	123.3 (3)	C8—C7—N1	117.2 (2)
O2—C2—C3	124.1 (2)	C6—C7—N1	121.2 (2)
O1—C2—C3	112.7 (2)	C7—C8—C3	120.1 (2)

C8—C3—C4	118.7 (2)	C7—C8—H8	120.0
C8—C3—C2	118.5 (2)	C3—C8—H8	120.0
C4—C3—C2	122.8 (2)	O3—N1—O4	125.0 (3)
C5—C4—C3	120.7 (2)	O3—N1—C7	117.8 (2)
C5—C4—H4	119.6	O4—N1—C7	117.1 (2)
C3—C4—H4	119.6	C2—O1—C1	116.9 (2)
C6—C5—C4	120.8 (2)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C5—H5···O2^{i}	0.93	2.47	3.272 (3)	145

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