Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

Methyl 4-(3-chloropropoxy)-3-methoxybenzoate

Min Zhang, Ran-Zhe Lu, Lu-Na Han, Bin Wang and Hai-Bo Wang*

College of Science, Nanjing University of Technology, Xinmofan Road No.5 Nanjing, Nanjing 210009, People's Republic of China
Correspondence e-mail: wanghaibo@njut.edu.cn

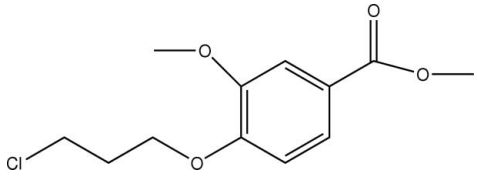
Received 18 December 2008; accepted 19 December 2008

Key indicators: single-crystal X-ray study; T = 294 K; mean σ(C–C) = 0.006 Å; R factor = 0.068; wR factor = 0.176; data-to-parameter ratio = 14.8.

In the title compound, $C_{12}H_{15}ClO_4$, the molecules are linked by C—H···O interactions.

Related literature

For general background, see: Knesl *et al.* (2006). For bond-length data, see: Allen *et al.* (1987).



Experimental

Crystal data

$C_{12}H_{15}ClO_4$
M_r = 258.69
Monoclinic, $P2_1/c$
a = 8.4980 (17) Å
b = 17.349 (4) Å
c = 8.8440 (18) Å
β = 106.46 (3)°
V = 1250.5 (5) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.31 mm^{-1}
T = 294 (2) K
0.30 × 0.20 × 0.10 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
Absorption correction: ψ scan (North *et al.*, 1968)
T_{min} = 0.914, T_{max} = 0.970
2431 measured reflections
2274 independent reflections
1575 reflections with $I > 2\sigma(I)$
R_{int} = 0.048
3 standard reflections
frequency: 120 min
intensity decay: 1%

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.068
$wR(F^2)$ = 0.176
S = 1.01
2274 reflections
154 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.35 e Å^{-3}
$\Delta\rho_{min}$ = −0.29 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
C1—H1B···O2^{i}	0.97	2.56	3.429 (6)	149
C2—H2A···O3^{ii}	0.97	2.41	3.358 (6)	164

Symmetry codes: (i) $-x+1, -y+1, -z+1$; (ii) $x+1, -y+\frac{3}{2}, z-\frac{1}{2}$.

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: *ORTEP-3 for Windows* (Farrugia, 1997) and *PLATON* (Spek, 2003); software used to prepare material for publication: *SHELXTL* (Sheldrick, 2008).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HK2602).

References

Allen, F. H., Kennard, O., Watson, D. G., Brammer, L., Orpen, A. G. & Taylor, R. (1987). *J. Chem. Soc. Perkin Trans. 2*, pp. S1–S19.
Enraf–Nonius (1989). *CAD-4 Software*. Enraf–Nonius, Delft. The Netherlands.
Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
Harms, K. & Wocadlo, S. (1995). *XCAD4*. University of Marburg, Germany.
Knesl, P., Roeseling, D. & Jordis, U. (2006). *Molecules*, **11**, 286–297.
North, A. C. T., Phillips, D. C. & Mathews, F. S. (1968). *Acta Cryst.* A**24**, 351–359.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Spek, A. L. (2003). *J. Appl. Cryst.* **36**, 7–13.

supplementary materials

Acta Cryst. (2009). E**65**, o231 [**doi:10.1107/S1600536808043274**]

Methyl 4-(3-chloropropoxy)-3-methoxybenzoate

M. Zhang, R.-Z. Lu, L.-N. Han, B. Wang and H.-B. Wang

Comment

As part of our ongoing studies on quinazoline derivatives (Knesl *et al.*, 2006), we report herein the crystal structure of the title compound.

In the molecule of the title compound (Fig 1), the bond lengths (Allen *et al.*, 1987) and angles are within normal ranges. Ring A (C4-C9) is, of course, planar. The intramolecular C-H···O hydrogen bond (Table 1) results in the formation of a five-membered ring B (O1/C1-C3/H1A), having envelope conformation with C2 atom displaced by -0.668 (3) Å from the plane of the other ring atoms.

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.

Experimental

For the preparation of the title compound, methyl 3-methoxy-4-hydroxybenzoate (55 mmol), 1-bromo-3-chloropropane (165 mmol) and potassium carbonate (275 mmol) were mixed with DMF (60 ml), and then the mixture was heated to reflux for 2 h. Reaction progress was monitored by TLC. After cooling and filtration, the title compound was obtained (yield; 93.7%, m.p. 384 K). Crystals suitable for X-ray analysis were obtained by slow evaporation of an ethyl acetate solution.

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.

Figures

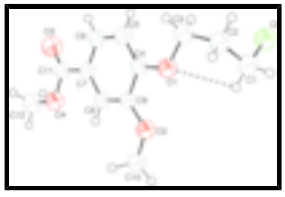


Fig. 1. The molecular structure of the title molecule, with the atom-numbering scheme. Hydrogen bond is shown as dashed line.

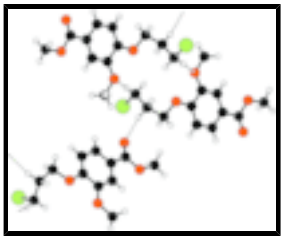


Fig. 2. A partial packing diagram of the title compound. Hydrogen bonds are shown as dashed lines.

Methyl 4-(3-chloropropoxy)-3-methoxybenzoate

Crystal data

$C_{12}H_{15}ClO_4$	F_{000} = 544
M_r = 258.69	D_x = 1.374 Mg m^{-3}
Monoclinic, $P2_1/c$	Melting point: 384 K
Hall symbol: -P 2ybc	Mo $K\alpha$ radiation λ = 0.71073 Å
a = 8.4980 (17) Å	Cell parameters from 25 reflections
b = 17.349 (4) Å	θ = 10–13º
c = 8.8440 (18) Å	μ = 0.31 mm^{-1}
β = 106.46 (3)º	T = 294 (2) K
V = 1250.5 (5) Å^3	Block, colorless
Z = 4	0.30 × 0.20 × 0.10 mm

Data collection

Enraf–Nonius CAD-4 diffractometer	R_{int} = 0.048
Radiation source: fine-focus sealed tube	θ_{max} = 25.3º
Monochromator: graphite	θ_{min} = 2.4º
T = 294(2) K	h = 0→10
ω/2θ scans	k = 0→20
Absorption correction: ψ scan (North *et al.*, 1968)	l = −10→10
T_{min} = 0.914, T_{max} = 0.970	3 standard reflections
2431 measured reflections	every 120 min
2274 independent reflections	intensity decay: 1%
1575 reflections with $I > 2\sigma(I)$	

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)]$ = 0.068	H-atom parameters constrained
$wR(F^2)$ = 0.176	$w = 1/[\sigma^2(F_o^2) + (0.050P)^2 + 3.3P]$ where $P = (F_o^2 + 2F_c^2)/3$
S = 1.01	$(\Delta/\sigma)_{max} < 0.001$

2274 reflections	$\Delta\rho_{max} = 0.35$ e Å^{-3}
154 parameters	$\Delta\rho_{min} = -0.29$ e Å^{-3}
Primary atom site location: structure-invariant direct methods	Extinction correction: none

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds 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 $F^2 > 2\text{sigma}(F^2)$ 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}
Cl	0.27901 (16)	0.58276 (8)	0.10729 (14)	0.0679 (4)
O1	0.3056 (3)	0.60776 (15)	0.5453 (3)	0.0480 (7)
O2	0.1798 (3)	0.50211 (15)	0.6779 (3)	0.0491 (7)
O3	−0.2908 (4)	0.74808 (19)	0.7610 (4)	0.0711 (10)
O4	−0.2715 (3)	0.62804 (17)	0.8531 (4)	0.0578 (8)
C1	0.4233 (6)	0.5519 (3)	0.2872 (5)	0.0580 (12)
H1A	0.3693	0.5170	0.3424	0.070*
H1B	0.5119	0.5239	0.2627	0.070*
C2	0.4929 (5)	0.6187 (3)	0.3921 (5)	0.0554 (11)
H2A	0.5409	0.6546	0.3337	0.066*
H2B	0.5803	0.6000	0.4807	0.066*
C3	0.3697 (5)	0.6615 (2)	0.4548 (5)	0.0532 (11)
H3A	0.4223	0.7043	0.5207	0.064*
H3B	0.2819	0.6817	0.3684	0.064*
C4	0.1809 (5)	0.6315 (2)	0.6028 (4)	0.0401 (9)
C5	0.1190 (5)	0.7053 (2)	0.5911 (5)	0.0488 (10)
H5A	0.1656	0.7438	0.5444	0.059*
C6	−0.0123 (5)	0.7224 (2)	0.6489 (5)	0.0489 (10)
H6A	−0.0547	0.7722	0.6390	0.059*
C7	−0.0814 (5)	0.6657 (2)	0.7216 (4)	0.0424 (9)
C8	−0.0174 (5)	0.5915 (2)	0.7335 (4)	0.0404 (9)
H8A	−0.0638	0.5533	0.7811	0.048*
C9	0.1129 (4)	0.5731 (2)	0.6768 (4)	0.0378 (8)
C10	0.1078 (5)	0.4411 (2)	0.7422 (5)	0.0495 (10)
H10A	0.1631	0.3937	0.7342	0.074*
H10B	−0.0061	0.4366	0.6849	0.074*
H10C	0.1176	0.4517	0.8511	0.074*
C11	−0.2236 (5)	0.6865 (2)	0.7781 (5)	0.0478 (10)

C12	−0.4095 (6)	0.6411 (3)	0.9117 (6)	0.0670 (14)
H12A	−0.4308	0.5957	0.9646	0.101*
H12B	−0.5039	0.6531	0.8255	0.101*
H12C	−0.3865	0.6835	0.9846	0.101*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl	0.0710 (8)	0.0750 (8)	0.0547 (7)	0.0031 (6)	0.0130 (6)	0.0007 (6)
O1	0.0477 (16)	0.0459 (16)	0.0542 (17)	−0.0020 (12)	0.0206 (13)	0.0082 (13)
O2	0.0481 (16)	0.0364 (15)	0.0657 (19)	0.0022 (12)	0.0211 (14)	0.0077 (13)
O3	0.079 (2)	0.057 (2)	0.086 (2)	0.0270 (17)	0.037 (2)	0.0083 (18)
O4	0.0487 (17)	0.0563 (19)	0.074 (2)	0.0088 (14)	0.0267 (16)	−0.0028 (16)
C1	0.060 (3)	0.055 (3)	0.062 (3)	0.006 (2)	0.022 (2)	0.006 (2)
C2	0.048 (2)	0.063 (3)	0.057 (3)	−0.003 (2)	0.018 (2)	0.008 (2)
C3	0.059 (3)	0.044 (2)	0.054 (3)	−0.009 (2)	0.012 (2)	0.004 (2)
C4	0.041 (2)	0.043 (2)	0.036 (2)	−0.0010 (17)	0.0103 (16)	−0.0003 (16)
C5	0.063 (3)	0.034 (2)	0.050 (2)	−0.0083 (19)	0.016 (2)	−0.0017 (18)
C6	0.056 (3)	0.035 (2)	0.051 (2)	0.0081 (18)	0.006 (2)	−0.0028 (18)
C7	0.043 (2)	0.042 (2)	0.041 (2)	0.0039 (17)	0.0092 (17)	−0.0063 (17)
C8	0.039 (2)	0.040 (2)	0.040 (2)	−0.0037 (16)	0.0073 (16)	0.0028 (17)
C9	0.041 (2)	0.0330 (19)	0.040 (2)	0.0027 (16)	0.0114 (16)	−0.0013 (16)
C10	0.057 (3)	0.034 (2)	0.062 (3)	0.0011 (18)	0.023 (2)	0.0053 (19)
C11	0.054 (2)	0.042 (2)	0.043 (2)	0.0044 (19)	0.0058 (19)	−0.0037 (18)
C12	0.052 (3)	0.083 (4)	0.073 (3)	0.007 (2)	0.030 (2)	−0.014 (3)

Geometric parameters (Å, °)

Cl—C1	1.794 (5)	C4—C5	1.378 (5)
O1—C3	1.433 (5)	C4—C9	1.415 (5)
O1—C4	1.362 (4)	C5—C6	1.385 (6)
O2—C9	1.355 (4)	C5—H5A	0.9300
O2—C10	1.420 (4)	C6—C7	1.394 (6)
O3—C11	1.201 (5)	C6—H6A	0.9300
O4—C11	1.336 (5)	C7—C8	1.390 (5)
O4—C12	1.429 (5)	C7—C11	1.478 (6)
C1—C2	1.498 (6)	C8—C9	1.377 (5)
C1—H1A	0.9700	C8—H8A	0.9300
C1—H1B	0.9700	C10—H10A	0.9600
C2—C3	1.512 (6)	C10—H10B	0.9600
C2—H2A	0.9700	C10—H10C	0.9600
C2—H2B	0.9700	C12—H12A	0.9600
C3—H3A	0.9700	C12—H12B	0.9600
C3—H3B	0.9700	C12—H12C	0.9600
C4—O1—C3	118.1 (3)	C5—C6—C7	120.5 (4)
C9—O2—C10	116.9 (3)	C5—C6—H6A	119.8
C11—O4—C12	117.1 (3)	C7—C6—H6A	119.8
Cl—C1—H1A	109.3	C8—C7—C6	118.9 (4)

Cl—C1—H1B	109.3	C8—C7—C11	122.7 (4)
C2—C1—Cl	111.6 (3)	C6—C7—C11	118.4 (4)
C2—C1—H1A	109.3	C9—C8—C7	121.6 (4)
C2—C1—H1B	109.3	C9—C8—H8A	119.2
H1A—C1—H1B	108.0	C7—C8—H8A	119.2
C1—C2—C3	114.5 (4)	O2—C9—C8	125.9 (3)
C1—C2—H2A	108.6	O2—C9—C4	115.4 (3)
C1—C2—H2B	108.6	C8—C9—C4	118.6 (3)
C3—C2—H2A	108.6	O2—C10—H10A	109.5
C3—C2—H2B	108.6	O2—C10—H10B	109.5
H2A—C2—H2B	107.6	H10A—C10—H10B	109.5
O1—C3—C2	107.3 (3)	O2—C10—H10C	109.5
O1—C3—H3A	110.3	H10A—C10—H10C	109.5
O1—C3—H3B	110.3	H10B—C10—H10C	109.5
C2—C3—H3A	110.3	O3—C11—O4	122.5 (4)
C2—C3—H3B	110.3	O3—C11—C7	125.4 (4)
H3A—C3—H3B	108.5	O4—C11—C7	112.1 (3)
O1—C4—C5	125.0 (3)	O4—C12—H12A	109.5
O1—C4—C9	114.8 (3)	O4—C12—H12B	109.5
C5—C4—C9	120.2 (4)	H12A—C12—H12B	109.5
C4—C5—C6	120.2 (4)	O4—C12—H12C	109.5
C4—C5—H5A	119.9	H12A—C12—H12C	109.5
C6—C5—H5A	119.9	H12B—C12—H12C	109.5
Cl—C1—C2—C3	65.9 (4)	C10—O2—C9—C4	−176.1 (3)
C4—O1—C3—C2	−174.3 (3)	C7—C8—C9—O2	−177.5 (4)
C1—C2—C3—O1	60.5 (5)	C7—C8—C9—C4	−0.9 (6)
C3—O1—C4—C5	−5.2 (6)	O1—C4—C9—O2	−0.9 (5)
C3—O1—C4—C9	173.8 (3)	C5—C4—C9—O2	178.2 (4)
O1—C4—C5—C6	177.6 (4)	O1—C4—C9—C8	−177.9 (3)
C9—C4—C5—C6	−1.3 (6)	C5—C4—C9—C8	1.2 (6)
C4—C5—C6—C7	1.1 (6)	C12—O4—C11—O3	0.8 (6)
C5—C6—C7—C8	−0.7 (6)	C12—O4—C11—C7	−179.4 (3)
C5—C6—C7—C11	−178.7 (4)	C8—C7—C11—O3	−174.5 (4)
C6—C7—C8—C9	0.6 (6)	C6—C7—C11—O3	3.4 (6)
C11—C7—C8—C9	178.5 (4)	C8—C7—C11—O4	5.6 (5)
C10—O2—C9—C8	0.6 (6)	C6—C7—C11—O4	−176.5 (4)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C1—H1A···O1	0.97	2.56	2.906 (5)	101
C1—H1B···O2^{i}	0.97	2.56	3.429 (6)	149
C2—H2A···O3^{ii}	0.97	2.41	3.358 (6)	164

Symmetry codes: (i) −*x*+1, −*y*+1, −*z*+1; (ii) *x*+1, −*y*+3/2, *z*−1/2.

Fig. 1

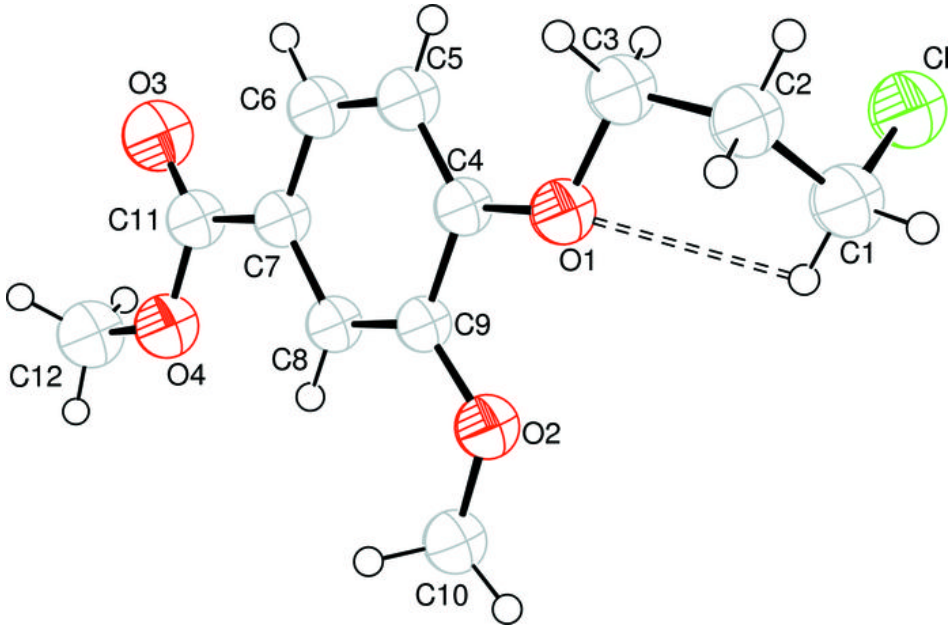


Fig. 2