

Acta Crystallographica Section E

Structure Reports

Online

ISSN 1600-5368

Methyl 4-butoxy-3-methoxybenzoate

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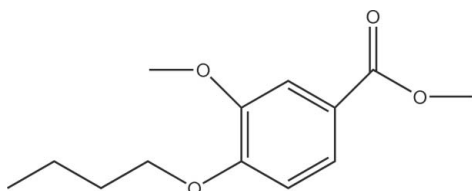
Received 13 January 2009; accepted 24 January 2009

Key indicators: single-crystal X-ray study; $T = 293$ K; mean $\sigma(\text{C}-\text{C}) = 0.004$ Å;
 R factor = 0.064; wR factor = 0.168; data-to-parameter ratio = 14.9.

The title compound, $\text{C}_{13}\text{H}_{18}\text{O}_4$, is an intermediate product in the synthesis of quinazoline derivatives. Crystal structure analysis shows that the benzene–butoxy $\text{C}_{\text{ar}}-\text{O}-\text{C}$ torsion angle is $175.3(2)^\circ$ and that the benzene–methoxycarbonyl $\text{C}_{\text{ar}}-\text{C}-\text{O}-\text{C}$ torsion angle is $175.2(2)^\circ$. Torsion angles close to 180° indicate that the molecule is almost planar.

Related literature

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



Experimental

Crystal data

$\text{C}_{13}\text{H}_{18}\text{O}_4$	$\gamma = 79.26(3)^\circ$
$M_r = 238.27$	$V = 632.3(2) \text{ \AA}^3$
Triclinic, $P\bar{1}$	$Z = 2$
$a = 7.9660(16) \text{ \AA}$	Mo $K\alpha$ radiation
$b = 9.1630(18) \text{ \AA}$	$\mu = 0.09 \text{ mm}^{-1}$
$c = 10.143(2) \text{ \AA}$	$T = 293(2) \text{ K}$
$\alpha = 64.80(2)^\circ$	$0.30 \times 0.20 \times 0.10 \text{ mm}$
$\beta = 70.96(3)^\circ$	

Data collection

Enraf–Nonius CAD-4 diffractometer	2294 independent reflections
Absorption correction: ψ scan (North <i>et al.</i> , 1968)	1567 reflections with $I > 2\sigma(I)$
$T_{\text{min}} = 0.973$, $T_{\text{max}} = 0.991$	$R_{\text{int}} = 0.067$
2474 measured reflections	3 standard reflections every 200 reflections
	intensity decay: 1%

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.064$	154 parameters
$wR(F^2) = 0.168$	H-atom parameters constrained
$S = 1.00$	$\Delta\rho_{\text{max}} = 0.18 \text{ e \AA}^{-3}$
2294 reflections	$\Delta\rho_{\text{min}} = -0.21 \text{ e \AA}^{-3}$

Data collection: *CAD-4 EXPRESS* (Enraf–Nonius, 1994); cell refinement: *CAD-4 EXPRESS*; 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* (Sheldrick, 2008); software used to prepare material for publication: *PLATON* (Spek, 2003).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: WK2098).

References

- Allen, F. H., Kennard, O., Watson, D. G., Brammer, L., Orpen, A. G. & Taylor, R. (1987). *J. Chem. Soc. Perkin Trans. 2*, pp. S1–19.
- Enraf–Nonius (1994). *CAD-4 EXPRESS*. Enraf–Nonius, Delft, The Netherlands.
- 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.* **A24**, 351–359.
- Sheldrick, G. M. (2008). *Acta Cryst.* **A64**, 112–122.
- Spek, A. L. (2003). *J. Appl. Cryst.* **36**, 7–13.

supporting information

Acta Cryst. (2009). E65, o434 [doi:10.1107/S1600536809003080]

Methyl 4-butoxy-3-methoxybenzoate

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S1. 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.

S2. Experimental

For the preparation of the title compound, methyl 3-methoxy-4-hydroxybenzoate (55 mmol), 1-bromobutane (110 mmol) and potassium carbonate (165 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 92%, m.p. 317 K). Crystals suitable for X-ray analysis were obtained by slow evaporation of an ethyl acetate 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 $U_{\text{iso}}(\text{H}) = xU_{\text{eq}}(\text{C})$, where $x = 1.5$ for methyl H and $x = 1.2$ for all other H atoms.

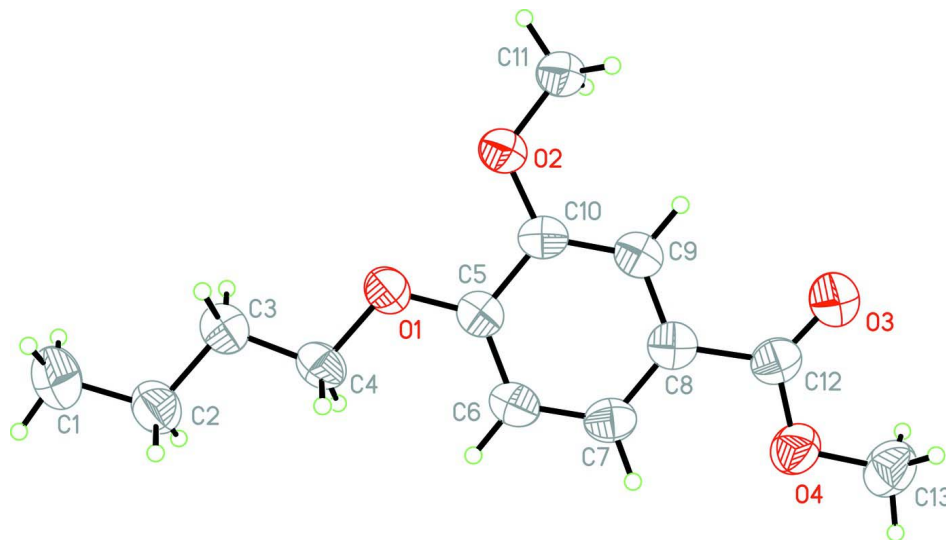


Figure 1

The molecular structure of the title molecule, with the atom-numbering scheme.

Methyl 4-butoxy-3-methoxybenzoate*Crystal data*C₁₃H₁₈O₄ $M_r = 238.27$ Triclinic, $P\bar{1}$

Hall symbol: -P 1

 $a = 7.9660$ (16) Å $b = 9.1630$ (18) Å $c = 10.143$ (2) Å $\alpha = 64.80$ (2)° $\beta = 70.96$ (3)° $\gamma = 79.26$ (3)° $V = 632.3$ (2) Å³ $Z = 2$ $F(000) = 256$ $D_x = 1.252$ Mg m⁻³

Melting point: 317 K

Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å

Cell parameters from 25 reflections

 $\theta = 9\text{--}12^\circ$ $\mu = 0.09$ mm⁻¹ $T = 293$ K

Block, colourless

 $0.30 \times 0.20 \times 0.10$ mm*Data collection*

Enraf–Nonius CAD-4

diffractometer

Radiation source: fine-focus sealed tube

Graphite monochromator

 $\omega/2\theta$ scansAbsorption correction: ψ scan(North *et al.*, 1968) $T_{\min} = 0.973$, $T_{\max} = 0.991$

2474 measured reflections

2294 independent reflections

1567 reflections with $I > 2\sigma(I)$ $R_{\text{int}} = 0.067$ $\theta_{\max} = 25.3^\circ$, $\theta_{\min} = 2.3^\circ$ $h = 0\text{--}9$ $k = -10\text{--}11$ $l = -11\text{--}12$

3 standard reflections every 200 reflections

intensity decay: 1%

*Refinement*Refinement on F^2

Least-squares matrix: full

 $R[F^2 > 2\sigma(F^2)] = 0.064$ $wR(F^2) = 0.168$ $S = 1.00$

2294 reflections

154 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

 $w = 1/[\sigma^2(F_o^2) + (0.07P)^2 + 0.43P]$ where $P = (F_o^2 + 2F_c^2)/3$ $(\Delta/\sigma)_{\max} < 0.001$ $\Delta\rho_{\max} = 0.18$ e Å⁻³ $\Delta\rho_{\min} = -0.21$ e Å⁻³*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 $F^2 > 2\sigma(F^2)$ 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)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
O1	0.3147 (2)	0.6241 (2)	0.05115 (19)	0.0488 (5)
O2	0.0656 (2)	0.4271 (2)	0.15700 (19)	0.0516 (5)

O3	0.0117 (3)	0.1304 (3)	0.7289 (2)	0.0733 (7)
O4	0.1887 (2)	0.2708 (2)	0.75552 (19)	0.0570 (5)
C1	0.6296 (5)	1.0667 (4)	-0.4050 (3)	0.0749 (10)
H1A	0.7158	1.1438	-0.4358	0.112*
H1B	0.5201	1.1226	-0.4245	0.112*
H1C	0.6732	0.9990	-0.4612	0.112*
C2	0.5977 (4)	0.9645 (4)	-0.2387 (3)	0.0606 (8)
H2A	0.5569	1.0348	-0.1838	0.073*
H2B	0.7101	0.9117	-0.2204	0.073*
C3	0.4645 (3)	0.8369 (3)	-0.1746 (3)	0.0473 (6)
H3A	0.3493	0.8884	-0.1865	0.057*
H3B	0.5016	0.7675	-0.2305	0.057*
C4	0.4487 (4)	0.7377 (3)	-0.0111 (3)	0.0493 (7)
H4A	0.5619	0.6806	0.0002	0.059*
H4B	0.4189	0.8078	0.0438	0.059*
C5	0.2767 (3)	0.5331 (3)	0.2032 (3)	0.0410 (6)
C6	0.3595 (3)	0.5399 (3)	0.3000 (3)	0.0495 (7)
H6A	0.4504	0.6098	0.2622	0.059*
C7	0.3085 (3)	0.4438 (3)	0.4524 (3)	0.0477 (6)
H7A	0.3639	0.4505	0.5172	0.057*
C8	0.1752 (3)	0.3373 (3)	0.5099 (3)	0.0443 (6)
C9	0.0934 (3)	0.3285 (3)	0.4133 (3)	0.0438 (6)
H9A	0.0051	0.2559	0.4516	0.053*
C10	0.1400 (3)	0.4256 (3)	0.2606 (3)	0.0401 (6)
C11	-0.0929 (4)	0.3439 (4)	0.2120 (3)	0.0564 (8)
H11A	-0.1314	0.3542	0.1278	0.085*
H11B	-0.1841	0.3895	0.2749	0.085*
H11C	-0.0703	0.2317	0.2702	0.085*
C12	0.1161 (3)	0.2332 (3)	0.6753 (3)	0.0468 (6)
C13	0.1295 (4)	0.1866 (4)	0.9164 (3)	0.0661 (8)
H13A	0.1890	0.2239	0.9638	0.099*
H13B	0.1562	0.0729	0.9425	0.099*
H13C	0.0034	0.2066	0.9511	0.099*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0478 (10)	0.0490 (11)	0.0479 (10)	-0.0110 (8)	-0.0085 (8)	-0.0176 (8)
O2	0.0480 (10)	0.0598 (12)	0.0497 (10)	-0.0140 (9)	-0.0114 (8)	-0.0211 (9)
O3	0.0858 (16)	0.0749 (15)	0.0559 (12)	-0.0289 (13)	-0.0188 (11)	-0.0135 (11)
O4	0.0592 (12)	0.0667 (13)	0.0460 (10)	-0.0054 (10)	-0.0141 (9)	-0.0226 (9)
C1	0.076 (2)	0.065 (2)	0.066 (2)	-0.0173 (18)	-0.0084 (16)	-0.0119 (16)
C2	0.0603 (18)	0.0566 (18)	0.0607 (17)	-0.0145 (15)	-0.0097 (14)	-0.0198 (14)
C3	0.0466 (15)	0.0402 (15)	0.0547 (15)	0.0003 (12)	-0.0074 (11)	-0.0241 (12)
C4	0.0486 (15)	0.0425 (15)	0.0589 (16)	-0.0092 (12)	-0.0099 (12)	-0.0228 (12)
C5	0.0390 (13)	0.0368 (13)	0.0485 (13)	0.0017 (11)	-0.0093 (10)	-0.0215 (11)
C6	0.0443 (15)	0.0510 (16)	0.0575 (16)	-0.0088 (12)	-0.0119 (12)	-0.0245 (13)
C7	0.0444 (15)	0.0497 (16)	0.0563 (15)	0.0033 (12)	-0.0201 (12)	-0.0254 (12)

C8	0.0394 (13)	0.0425 (14)	0.0528 (15)	0.0072 (11)	-0.0137 (11)	-0.0236 (12)
C9	0.0403 (14)	0.0397 (14)	0.0536 (15)	0.0013 (11)	-0.0115 (11)	-0.0228 (11)
C10	0.0361 (13)	0.0417 (14)	0.0498 (14)	0.0055 (11)	-0.0125 (10)	-0.0274 (11)
C11	0.0539 (17)	0.0673 (19)	0.0517 (15)	-0.0164 (15)	-0.0143 (12)	-0.0217 (14)
C12	0.0418 (14)	0.0487 (16)	0.0534 (15)	0.0075 (12)	-0.0184 (12)	-0.0235 (12)
C13	0.0680 (19)	0.079 (2)	0.0490 (16)	-0.0016 (17)	-0.0149 (14)	-0.0253 (15)

Geometric parameters (Å, °)

O1—C5	1.364 (3)	C4—H4B	0.9700
O1—C4	1.427 (3)	C5—C6	1.375 (3)
O2—C10	1.359 (3)	C5—C10	1.411 (3)
O2—C11	1.423 (3)	C6—C7	1.376 (4)
O3—C12	1.198 (3)	C6—H6A	0.9300
O4—C12	1.317 (3)	C7—C8	1.385 (3)
O4—C13	1.429 (3)	C7—H7A	0.9300
C1—C2	1.501 (4)	C8—C9	1.376 (3)
C1—H1A	0.9600	C8—C12	1.495 (4)
C1—H1B	0.9600	C9—C10	1.379 (3)
C1—H1C	0.9600	C9—H9A	0.9300
C2—C3	1.510 (4)	C11—H11A	0.9600
C2—H2A	0.9700	C11—H11B	0.9600
C2—H2B	0.9700	C11—H11C	0.9600
C3—C4	1.488 (4)	C13—H13A	0.9600
C3—H3A	0.9700	C13—H13B	0.9600
C3—H3B	0.9700	C13—H13C	0.9600
C4—H4A	0.9700		
C5—O1—C4	116.99 (19)	C5—C6—C7	120.4 (2)
C10—O2—C11	117.84 (19)	C5—C6—H6A	119.8
C12—O4—C13	116.2 (2)	C7—C6—H6A	119.8
C2—C1—H1A	109.5	C6—C7—C8	120.5 (2)
C2—C1—H1B	109.5	C6—C7—H7A	119.8
H1A—C1—H1B	109.5	C8—C7—H7A	119.8
C2—C1—H1C	109.5	C9—C8—C7	119.4 (2)
H1A—C1—H1C	109.5	C9—C8—C12	119.2 (2)
H1B—C1—H1C	109.5	C7—C8—C12	121.4 (2)
C1—C2—C3	115.2 (3)	C8—C9—C10	121.2 (2)
C1—C2—H2A	108.5	C8—C9—H9A	119.4
C3—C2—H2A	108.5	C10—C9—H9A	119.4
C1—C2—H2B	108.5	O2—C10—C9	125.5 (2)
C3—C2—H2B	108.5	O2—C10—C5	115.7 (2)
H2A—C2—H2B	107.5	C9—C10—C5	118.9 (2)
C4—C3—C2	111.0 (2)	O2—C11—H11A	109.5
C4—C3—H3A	109.4	O2—C11—H11B	109.5
C2—C3—H3A	109.4	H11A—C11—H11B	109.5
C4—C3—H3B	109.4	O2—C11—H11C	109.5
C2—C3—H3B	109.4	H11A—C11—H11C	109.5

H3A—C3—H3B	108.0	H11B—C11—H11C	109.5
O1—C4—C3	110.7 (2)	O3—C12—O4	124.0 (2)
O1—C4—H4A	109.5	O3—C12—C8	123.9 (2)
C3—C4—H4A	109.5	O4—C12—C8	112.0 (2)
O1—C4—H4B	109.5	O4—C13—H13A	109.5
C3—C4—H4B	109.5	O4—C13—H13B	109.5
H4A—C4—H4B	108.1	H13A—C13—H13B	109.5
O1—C5—C6	125.3 (2)	O4—C13—H13C	109.5
O1—C5—C10	115.1 (2)	H13A—C13—H13C	109.5
C6—C5—C10	119.7 (2)	H13B—C13—H13C	109.5
C1—C2—C3—C4	177.4 (3)	C11—O2—C10—C5	-168.8 (2)
C5—O1—C4—C3	-175.3 (2)	C8—C9—C10—O2	-179.1 (2)
C2—C3—C4—O1	176.4 (2)	C8—C9—C10—C5	1.3 (4)
C4—O1—C5—C6	-1.4 (4)	O1—C5—C10—O2	0.2 (3)
C4—O1—C5—C10	178.3 (2)	C6—C5—C10—O2	179.9 (2)
O1—C5—C6—C7	179.0 (2)	O1—C5—C10—C9	179.8 (2)
C10—C5—C6—C7	-0.7 (4)	C6—C5—C10—C9	-0.4 (4)
C5—C6—C7—C8	1.0 (4)	C13—O4—C12—O3	-3.1 (4)
C6—C7—C8—C9	-0.2 (4)	C13—O4—C12—C8	175.2 (2)
C6—C7—C8—C12	-179.3 (2)	C9—C8—C12—O3	7.1 (4)
C7—C8—C9—C10	-0.9 (4)	C7—C8—C12—O3	-173.9 (3)
C12—C8—C9—C10	178.1 (2)	C9—C8—C12—O4	-171.2 (2)
C11—O2—C10—C9	11.6 (4)	C7—C8—C12—O4	7.8 (4)
