Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

2-(3-Bromo-4-butoxyphenyl)-3,4-dimethyl-5-(3,4,5-trimethoxyphenyl)furan

Run-Ling Wang,[a,b] Ji-Xian Shi,[a]* Xiu-Fang Zheng[a] and Ye Zhou[b]

[a]College of Pharmaceuticals and Biotechnology, Tianjin University, Tianjin 300072, People's Republic of China, and [b]School of Pharmaceutical Sciences, Tianjin Medical University, Tianjin 300070, People's Republic of China

Correspondence e-mail: jxshi@eyou.com

Key indicators

Single-crystal X-ray study
T = 294 K
Mean σ(C–C) = 0.005 Å
R factor = 0.042
wR factor = 0.110
Data-to-parameter ratio = 14.3

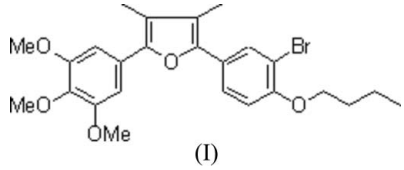
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Received 4 July 2005
Accepted 13 July 2005
Online 20 July 2005

The title compound, $C_{25}H_{29}BrO_5$, was synthesized by the cyclization of 1-(3-bromo-4-butoxyphenyl)-2,3-dimethyl-4-(3,4,5-trimethoxyphenyl)butane-1,4-dione with a 5% solution of hydrogen chloride in dried methanol. The aromatic rings make dihedral angles of 4.8 (3) and 7.1 (3)° with the furan ring. The bond lengths are unexceptional.

Comment

Much attention has been paid to diaryl-substituted heterocycles (Khanna *et al.*, 1997; Penning *et al.*, 1997; Wu-Wong *et al.*, 2001), since research findings indicate that the diaryl-heterocycle system is associated with high biological activities, *e.g.* as selective COX-2 inhibitors (Portevin *et al.*, 2000) and antitumor agents (Szczepankiewicz *et al.*, 2001). In view of this, we have recently focused on the preparation of diaryl-substituted furans. A new compound, *viz*. 2-(3-bromo-4-butoxyphenyl)-3,4-dimethyl-5-(3,4,5-trimethoxyphenyl)furan, (I), has been synthesized by the reaction of 1-(3-bromo-4-butoxyphenyl)-2,3-dimethyl-4-(3,4,5-trimethoxyphenyl)butane-1,4-dione in the presence of hydrogen chloride in dried methanol (Perry *et al.*, 1972). An X-ray crystal structure determination of (I) was carried out and the results are presented here.



(I)

The molecular structure of (I) is illustrated in Fig. 1. The aromatic rings C5–C10 (*A*) and C13–C18 (*B*) make dihedral angles of 4.8 (3) and 7.1 (3)°, respectively, with the furan ring; rings A and *B* are inclined to each other at an angle of 7.1 (3)°. The bond lengths are unexceptional.

Experimental

A 5% solution of hydrogen chloride in dried methanol (15 ml) was added dropwise to a boiling solution of 1-(3-bromo-4-butoxyphenyl)-2,3-dimethyl-4-(3,4,5-trimethoxyphenyl)butane-1,4-dione (1.48 mmol) in dichloromethane (7.0 ml). After about 15 min, the solution was cooled, producing crude crystals (yield 85.2%). Single crystals suitable for crystallographic analysis were obtained by slow evaporation of a dichloromethane–methanol solution (m.p. 375 K). ^{1}H NMR (DMSO): δ 7.85–6.88 (*m*, 5H), 4.06 (*t*, 2H), 3.93 (*s*, 6H), 3.90 (*s*, 3H), 2.22 (*s*, 3H), 2.20 (*s*, 3H), 1.81–1.87 (*m*, 2H), 1.53–1.59 (*m*, 2H), 1.01 (*t*, 3H).

Crystal data

$C_{25}H_{29}BrO_5$	$Z = 2$
$M_r = 489.39$	$D_x = 1.392$ Mg m^{-3}
Triclinic, $P\overline{1}$	Mo $K\alpha$ radiation
$a = 8.9854$ (17) Å	Cell parameters from 1888 reflections
$b = 11.955$ (2) Å	$\theta = 2.4$–$23.7°$
$c = 12.210$ (2) Å	$\mu = 1.79$ mm^{-1}
$\alpha = 71.179$ (3)°	$T = 294$ (2) K
$\beta = 70.188$ (3)°	Block, colorless
$\gamma = 84.085$ (3)°	0.42 × 0.38 × 0.22 mm
$V = 1168.0$ (4) Å^3	

Data collection

Bruker SMART 1000 CCD area-detector diffractometer	4100 independent reflections
	2555 reflections with $I > 2\sigma(I)$
φ and ω scans	$R_{int} = 0.026$
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)	$\theta_{max} = 25.0°$
	$h = -10 \rightarrow 9$
$T_{min} = 0.438$, $T_{max} = 0.674$	$k = -13 \rightarrow 14$
5993 measured reflections	$l = -14 \rightarrow 9$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0479P)^2 + 0.1271P]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)] = 0.042$	
$wR(F^2) = 0.110$	$(\Delta/\sigma)_{max} = 0.001$
$S = 1.02$	$\Delta\rho_{max} = 0.35$ e Å^{-3}
4100 reflections	$\Delta\rho_{min} = -0.42$ e Å^{-3}
287 parameters	Extinction correction: *SHELXL97*
H-atom parameters constrained	Extinction coefficient: 0.0140 (15)

All H atoms were positioned geometrically and refined as riding (C—H = 0.93–0.97 Å). For CH and CH_2 groups, U_{iso}(H) values were set equal to $1.2U_{eq}$(carrier atom) and for the methyl groups they were set equal to $1.5U_{eq}$(carrier atom).

Data collection: *SMART* (Bruker, 1997); cell refinement: *SAINT* (Bruker, 1997); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1997); software used to prepare material for publication: *SHELXTL*.

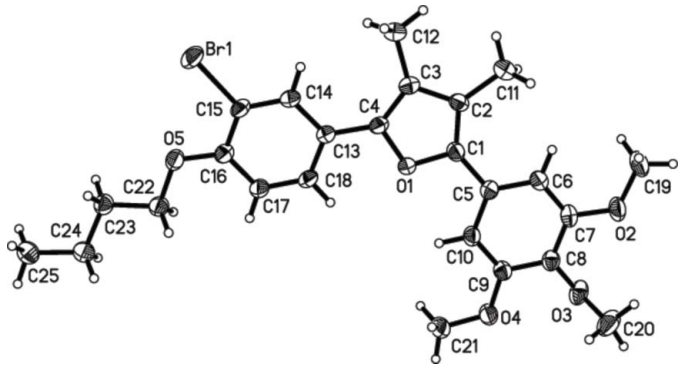


Figure 1
View of the molecule of (I) showing the atom-labeling scheme. Displacement ellipsoids are drawn at the 30% probability level.

The authors acknowledge support from the Basic Research Project (No, 2002CCA01500) of the Most, and from the National Young Scholar Award of the NSFC (No. 30125043).

References

Bruker (1997). *SMART*, *SAINT* and *SHELXTL*. Bruker AXS Inc., Madison, Wisconsin, USA.
Khanna, I. K., Weier, R. M., Yu, Y., Collins, P. W., Miyashiro, J. M., Koboldt, C. M. & Veenhuizen, A. W. (1997). *J. Med. Chem.* **40**, 1619–1633.
Penning, T. D., Talley, J. J., Bertenshaw, S. R., Carter, J. S. & Collins, C. P. (1997). *J. Med. Chem.* **40**, 1347–1365.
Perry, C. W., Kalnins, M. V. & Deitcher, K. H. (1972). *J. Org. Chem.* **37**, 4371–4376.
Portevin, B., Tordjman, C., Pastoureau, P., Bonnet, J. & De Nanteuil, G. (2000). *J. Med. Chem.* **43**, 4582–4593.
Sheldrick, G. M. (1996). *SADABS*. University of Göttingen, Germany.
Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.
Szczepankiewicz, B. G., Liu, G., Jae, H.-S., Tasker, A. S., Gunawardana, , J. W., von Geldern, T. W., Gwaltney, S. J. R., Gehrke, L., Chiou, W. J., Credo, R. B., Alder, J. D., Nukkala, M. A., Zielinski, N. A., Jarvis, K., Mollison, K. *et al.* (2001). *J. Med. Chem.* **44**, 4416–4430.
Wu-Wong, J. R., Alder, J. D., Alder, L., Burns, D. J., Han, E. K.-H., Credo, B., Tahir, S. K. & Dayton, B. D. (2001). *Cancer Res.* **61**, 1486–1487.