Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

3-Bromo-4-dibenzylamino-5-methoxy-furan-2(5*H*)-one

Jian-Hua Fu, Zhao-Yang Li, Zhao-Yang Wang* and Rui-Rong Ye

School of Chemistry and Environment, South China Normal University, Guangzhou 510006, People's Republic of China
Correspondence e-mail: wangwangzhaoyang@tom.com

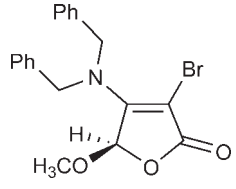
Received 30 January 2010; accepted 2 March 2010

Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.005 Å; R factor = 0.036; wR factor = 0.089; data-to-parameter ratio = 15.7.

In the the title compound, $C_{19}H_{18}BrNO_3$, the furanone ring is almost planar [maximum atomic deviation = 0.019 (3) Å] and is nearly perpendicular to the two phenyl rings, making dihedral angles of 88.96 (17) and 87.71 (17)°. Intermolecular C—H···O hydrogen bonding is present in the crystal structure.

Related literature

2(5*H*)-Furanone is the simplest sub-unit of a large class of five-membered heterocyclic carbonyl compounds, see: Reva *et al.* (2008). The title compound is a derivative of 4-amino-2(5*H*)-furanone. For the biological activity of 4-amino-2(5*H*)-furanones, see: Kimura *et al.* (2000); Tanoury *et al.* (2008). For the synthesis, see: Toshiyuki & Yoshikazu (1955).



Experimental

Crystal data

$C_{19}H_{18}BrNO_3$
M_r = 388.25
Orthorhombic, *Pbca*
a = 15.756 (2) Å
b = 11.2475 (14) Å
c = 19.779 (3) Å
V = 3505.2 (8) Å^3
Z = 8
Mo $K\alpha$ radiation
μ = 2.36 mm^{-1}
T = 298 K
0.23 × 0.20 × 0.16 mm

Data collection

Bruker APEXII area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.613, T_{max} = 0.704
18029 measured reflections
3429 independent reflections
2028 reflections with $I > 2\sigma(I)$
R_{int} = 0.054

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.036
$wR(F^2)$ = 0.089
S = 1.00
3429 reflections
218 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.30 e Å^{-3}
$\Delta\rho_{min}$ = −0.27 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
C16—H16···O3^{i}	0.98	2.49	3.396 (4)	154

Symmetry code: (i) $-x, y-\frac{1}{2}, -z+\frac{1}{2}$.

Data collection: *APEX2* (Bruker, 2008); cell refinement: *SAINT* (Bruker, 2008); data reduction: *SAINT*; 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); software used to prepare material for publication: *SHELXL97*.

The work was supported by the National Natural Science Foundation of China (grant No. 20772035) and the Natural Science Foundation of Guangdong Province, China (grant No. 5300082).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: XU2724).

References

Bruker (2008). *APEX2* and *SAINT*. Bruker AXS Inc., Madison, Wisconsin, USA.
Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
Kimura, Y., Mizuno, T., Kawano, T., Okada, K. & Shimad, A. (2000). *Phytochemistry*, **53**, 829–831.
Reva, I., Nowak, M. J., Lapinski, L. & Fausto, R. (2008). *Chem. Phys. Lett.* **452**, 20–28.
Sheldrick, G. M. (1996). *SADABS*. University of Göttingen, Germany.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Tanoury, G. J., Chen, M. Z., Dong, Y., Forslund, R. E. & Magdziak, D. (2008). *Org. Lett.* **10**, 185–188.
Toshiyuki, S. & Yoshikazu, H. (1955). *Nippon Kagaku Kaishi*, **58**, 692–693.

supporting information

Acta Cryst. (2010). E**66**, o769 [doi:10.1107/S1600536810008007]

3-Bromo-4-dibenzylamino-5-methoxyfuran-2(5*H*)-one

Jian-Hua Fu, Zhao-Yang Li, Zhao-Yang Wang and Rui-Rong Ye

S1. Comment

2(5H)-Furanone is a simplest sub-unit of a large class of five membered heterocyclic carbonyl compounds (Reva *et al.*, 2008). At the same time, 4-amino-2(5H)-furanone is an attractive moiety in chemical, pharmaceutical and agrochemical research. Many 4-amino-2(5H)-furanones have been patented as prodrugs or insecticides and herbicides (Kimura *et al.*, 2000; Tanoury *et al.*, 2008).

Attracted by versatile 4-amino-2(5H)-furanones, we synthesized the title compound with 3,4-dibromo-5-methoxy-furan-2(5H)-one and dibenzylamine in the presence of potassium fluoride *via* the tandem asymmetric Michael addition-elimination reaction. With 2(5H)-furanone moiety and polyfunctional groups (carboxyl, amino, halo), the title compound is expected to be a biologically active product.

The structure of the title compound (I) is illustrated in Fig. 1. The title compound contains a five-membered furanone ring and two six-membered benzene rings. The furanone ring is approximately planar.

S2. Experimental

The precursor 3,4-dibromo-5-methoxyfuran-2(5H)-furanone was prepared according to the literature procedure (Toshiyuki & Yoshikazu, 1955).

After the mixture of dibenzylamine (2 mmol) and potassium fluoride (6 mmol) was dissolved in absolute tetrahydro-furan (2 ml) under nitrogen atmosphere, dichloromethane solution of 3,4-dibromo-5-methoxyfuran-2(5H)-furanone (2.0 mmol) was added. The residual liquid was dissolved in dichloromethane. The reaction was carried out under the stirring at room temperature for 48 h. Once the reaction was complete, the solvents were removed under reduced pressure. The residual solid was dissolved in dichloromethane. Then the combined organic layers from extraction were concentrated under reduced pressure, and the crude product was purified by silica gel column chromatography with the gradient mixture of petroleum ether and ethyl acetate to give the product yielding (I) 0.6224 g (80.2%).

S3. Refinement

H atoms were positioned in calculated positions with C—H = 0.93-0.98 Å and were refined using a riding model, with $U_{iso}(H) = 1.5U_{eq}(C)$ for methyl and $1.2U_{eq}(C)$ for the others.

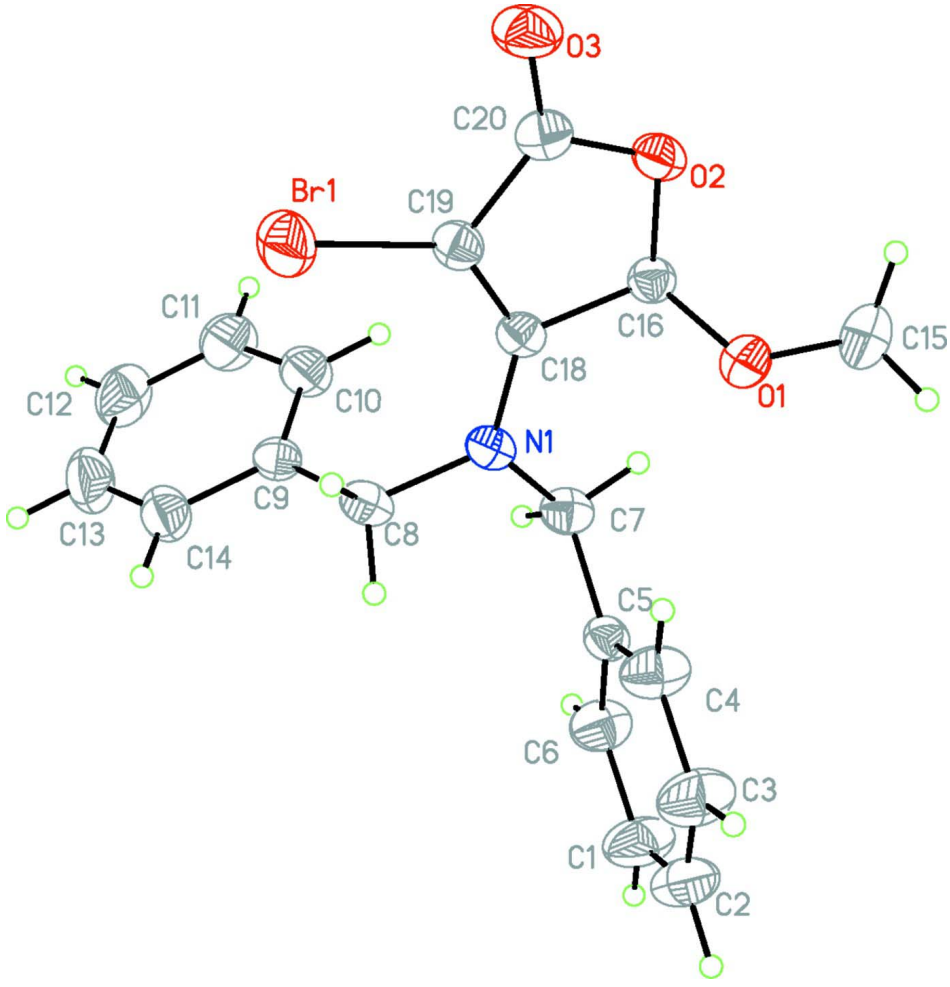


Figure 1

View of the title compound showing the atom-labelling scheme. Ellipsoids are drawn at the 50% probability level.

3-Bromo-4-dibenzylamino-5-methoxyfuran-2(5*H*)-one

Crystal data

$C_{19}H_{18}BrNO_3$
$M_r = 388.25$
Orthorhombic, *Pbca*
Hall symbol: -P 2ac 2ab
$a = 15.756\ (2)$ Å
$b = 11.2475\ (14)$ Å
$c = 19.779\ (3)$ Å
$V = 3505.2\ (8)$ Å^3
$Z = 8$
$F(000) = 1584$
$D_x = 1.471$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 2798 reflections
$\theta = 2.5$–$21.2°$
$\mu = 2.36$ mm^{-1}
$T = 298$ K
Block, colourless
0.23 × 0.20 × 0.16 mm

Data collection

Bruker APEXII area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
φ and ω scan
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{min} = 0.613$, $T_{max} = 0.704$
18029 measured reflections
3429 independent reflections

2028 reflections with $I > 2\sigma(I)$
$R_{int} = 0.054$
$\theta_{max} = 26.0°$, $\theta_{min} = 2.1°$
$h = -19 \rightarrow 18$
$k = -13 \rightarrow 13$
$l = -10 \rightarrow 24$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.036$
$wR(F^2) = 0.089$
$S = 1.00$
3429 reflections
218 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
$w = 1/[\sigma^2(F_o^2) + (0.0297P)^2 + 1.9449P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.002$
$\Delta\rho_{max} = 0.30$ e Å^{-3}
$\Delta\rho_{min} = -0.27$ e Å^{-3}

Special details

Experimental. ^{1}H NMR (400 MHz, $CDCl_3$, TMS): 3.52 (3H, *s*, CH, CH_3), 4.53 (2H, *d*, CH, CH_2), 4.90 (2H, *d*, CH, CH_2), 5.74 (1H, *s*, CH), 7.21-7.42 (10H, *m*, CH, Ar-H);

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)

	x	*y*	*z*	U_{iso}*/U_{eq}
Br1	0.09981 (2)	1.14889 (3)	0.35209 (2)	0.07708 (16)
C9	0.24254 (19)	0.8956 (3)	0.36299 (14)	0.0447 (7)
C5	0.29710 (17)	0.8590 (3)	0.16073 (14)	0.0440 (7)
C8	0.25104 (19)	0.9658 (3)	0.29832 (14)	0.0506 (8)
H8A	0.2423	1.0493	0.3083	0.061*
H8B	0.3085	0.9570	0.2815	0.061*
C6	0.3624 (2)	0.7779 (3)	0.15594 (17)	0.0689 (10)
H6	0.3598	0.7075	0.1805	0.083*
C7	0.22153 (18)	0.8294 (2)	0.20442 (15)	0.0495 (8)
H7A	0.1754	0.8027	0.1757	0.059*
H7B	0.2365	0.7644	0.2344	0.059*
C10	0.1799 (2)	0.8127 (3)	0.37322 (17)	0.0618 (9)
H10	0.1395	0.7997	0.3398	0.074*
C4	0.3031 (2)	0.9608 (3)	0.12329 (18)	0.0674 (10)
H4	0.2598	1.0168	0.1252	0.081*
C14	0.3011 (2)	0.9133 (3)	0.41324 (17)	0.0625 (9)
H14	0.3436	0.9696	0.4072	0.075*
C11	0.1764 (2)	0.7486 (3)	0.43241 (19)	0.0750 (11)
H11	0.1335	0.6928	0.4388	0.090*
C13	0.2981 (3)	0.8493 (4)	0.47231 (18)	0.0828 (12)

H13	0.3385	0.8623	0.5058	0.099*
C12	0.2358 (3)	0.7666 (4)	0.48199 (18)	0.0806 (11)
H12	0.2337	0.7230	0.5219	0.097*
C2	0.4371 (3)	0.9017 (4)	0.0793 (2)	0.0818 (12)
H2	0.4844	0.9168	0.0525	0.098*
C1	0.4314 (2)	0.7997 (4)	0.1153 (2)	0.0858 (12)
H1	0.4747	0.7438	0.1125	0.103*
C3	0.3730 (3)	0.9814 (4)	0.0826 (2)	0.0840 (12)
H3	0.3760	1.0509	0.0573	0.101*
N1	0.19202 (15)	0.9304 (2)	0.24524 (12)	0.0448 (6)
O1	0.08872 (13)	0.95812 (19)	0.12416 (10)	0.0558 (6)
O3	−0.06901 (14)	1.1434 (2)	0.26344 (12)	0.0691 (6)
O2	−0.02363 (12)	0.99727 (18)	0.19541 (10)	0.0532 (5)
C16	0.05326 (18)	0.9313 (3)	0.18643 (14)	0.0434 (7)
H16	0.0426	0.8457	0.1904	0.052*
C18	0.11283 (18)	0.9735 (2)	0.24190 (14)	0.0400 (7)
C20	−0.0134 (2)	1.0746 (3)	0.24854 (16)	0.0497 (8)
C19	0.06917 (18)	1.0562 (2)	0.27812 (14)	0.0446 (7)
C15	0.0499 (3)	0.8984 (4)	0.07008 (18)	0.0898 (13)
H15A	0.0518	0.8142	0.0780	0.135*
H15B	0.0794	0.9166	0.0289	0.135*
H15C	−0.0082	0.9235	0.0663	0.135*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Br1	0.0724 (3)	0.0756 (3)	0.0833 (3)	0.0017 (2)	−0.0117 (2)	−0.0326 (2)
C9	0.0377 (17)	0.0467 (16)	0.0498 (19)	0.0101 (14)	−0.0009 (15)	−0.0007 (14)
C5	0.0398 (17)	0.0478 (17)	0.0445 (18)	0.0011 (15)	−0.0038 (13)	0.0051 (15)
C8	0.0364 (17)	0.0554 (19)	0.060 (2)	−0.0035 (15)	−0.0035 (15)	0.0059 (16)
C6	0.059 (2)	0.074 (2)	0.073 (2)	0.0192 (19)	0.0072 (19)	0.026 (2)
C7	0.0452 (18)	0.0435 (18)	0.060 (2)	0.0056 (14)	0.0007 (15)	0.0084 (15)
C10	0.053 (2)	0.075 (2)	0.057 (2)	−0.0080 (18)	−0.0030 (16)	0.0139 (18)
C4	0.060 (2)	0.057 (2)	0.085 (3)	0.0059 (17)	0.0155 (19)	0.020 (2)
C14	0.061 (2)	0.068 (2)	0.059 (2)	0.0024 (18)	−0.0100 (18)	−0.0032 (19)
C11	0.079 (3)	0.081 (3)	0.066 (2)	−0.006 (2)	0.005 (2)	0.017 (2)
C13	0.088 (3)	0.102 (3)	0.058 (3)	0.008 (3)	−0.022 (2)	−0.005 (2)
C12	0.097 (3)	0.091 (3)	0.054 (2)	0.015 (3)	0.002 (2)	0.019 (2)
C2	0.059 (2)	0.103 (3)	0.083 (3)	−0.005 (2)	0.023 (2)	0.006 (3)
C1	0.060 (2)	0.102 (3)	0.095 (3)	0.026 (2)	0.024 (2)	0.017 (3)
C3	0.080 (3)	0.075 (3)	0.098 (3)	−0.007 (2)	0.026 (2)	0.030 (2)
N1	0.0362 (14)	0.0476 (14)	0.0506 (15)	−0.0009 (12)	−0.0020 (11)	0.0026 (12)
O1	0.0549 (13)	0.0668 (14)	0.0457 (13)	−0.0002 (11)	0.0038 (11)	0.0049 (11)
O3	0.0516 (13)	0.0647 (14)	0.0909 (18)	0.0162 (12)	0.0008 (12)	−0.0133 (13)
O2	0.0393 (12)	0.0610 (13)	0.0594 (14)	0.0064 (10)	−0.0041 (10)	−0.0071 (11)
C16	0.0411 (17)	0.0447 (17)	0.0442 (18)	−0.0011 (14)	0.0014 (14)	0.0013 (14)
C18	0.0381 (17)	0.0366 (16)	0.0452 (17)	−0.0082 (13)	0.0016 (13)	0.0085 (13)
C20	0.048 (2)	0.0440 (18)	0.057 (2)	−0.0009 (16)	0.0058 (16)	0.0004 (16)

C19	0.0427 (17)	0.0406 (16)	0.0506 (18)	−0.0044 (14)	−0.0017 (14)	−0.0012 (14)
C15	0.086 (3)	0.133 (4)	0.050 (2)	−0.001 (3)	0.004 (2)	−0.015 (2)

Geometric parameters (Å, º)

Br1—C19	1.860 (3)	C11—H11	0.9300
C9—C14	1.371 (4)	C13—C12	1.366 (5)
C9—C10	1.372 (4)	C13—H13	0.9300
C9—C8	1.509 (4)	C12—H12	0.9300
C5—C4	1.367 (4)	C2—C3	1.351 (5)
C5—C6	1.378 (4)	C2—C1	1.353 (5)
C5—C7	1.508 (4)	C2—H2	0.9300
C8—N1	1.458 (3)	C1—H1	0.9300
C8—H8A	0.9700	C3—H3	0.9300
C8—H8B	0.9700	N1—C18	1.340 (3)
C6—C1	1.374 (5)	O1—C16	1.386 (3)
C6—H6	0.9300	O1—C15	1.404 (4)
C7—N1	1.469 (3)	O3—C20	1.206 (3)
C7—H7A	0.9700	O2—C20	1.374 (3)
C7—H7B	0.9700	O2—C16	1.432 (3)
C10—C11	1.376 (4)	C16—C18	1.520 (4)
C10—H10	0.9300	C16—H16	0.9800
C4—C3	1.383 (5)	C18—C19	1.361 (4)
C4—H4	0.9300	C20—C19	1.441 (4)
C14—C13	1.373 (5)	C15—H15A	0.9600
C14—H14	0.9300	C15—H15B	0.9600
C11—C12	1.371 (5)	C15—H15C	0.9600
C14—C9—C10	118.4 (3)	C13—C12—H12	120.3
C14—C9—C8	118.6 (3)	C11—C12—H12	120.3
C10—C9—C8	123.0 (3)	C3—C2—C1	119.1 (4)
C4—C5—C6	117.7 (3)	C3—C2—H2	120.4
C4—C5—C7	123.4 (3)	C1—C2—H2	120.4
C6—C5—C7	118.9 (3)	C2—C1—C6	120.8 (4)
N1—C8—C9	114.3 (2)	C2—C1—H1	119.6
N1—C8—H8A	108.7	C6—C1—H1	119.6
C9—C8—H8A	108.7	C2—C3—C4	120.8 (4)
N1—C8—H8B	108.7	C2—C3—H3	119.6
C9—C8—H8B	108.7	C4—C3—H3	119.6
H8A—C8—H8B	107.6	C18—N1—C8	122.1 (2)
C1—C6—C5	120.8 (3)	C18—N1—C7	123.2 (2)
C1—C6—H6	119.6	C8—N1—C7	113.9 (2)
C5—C6—H6	119.6	C16—O1—C15	113.4 (2)
N1—C7—C5	113.2 (2)	C20—O2—C16	108.9 (2)
N1—C7—H7A	108.9	O1—C16—O2	109.8 (2)
C5—C7—H7A	108.9	O1—C16—C18	108.9 (2)
N1—C7—H7B	108.9	O2—C16—C18	105.7 (2)
C5—C7—H7B	108.9	O1—C16—H16	110.7

H7A—C7—H7B	107.7	O2—C16—H16	110.7
C9—C10—C11	120.7 (3)	C18—C16—H16	110.7
C9—C10—H10	119.6	N1—C18—C19	133.8 (3)
C11—C10—H10	119.6	N1—C18—C16	119.9 (2)
C5—C4—C3	120.7 (3)	C19—C18—C16	106.3 (2)
C5—C4—H4	119.7	O3—C20—O2	120.5 (3)
C3—C4—H4	119.7	O3—C20—C19	130.5 (3)
C9—C14—C13	121.2 (3)	O2—C20—C19	109.0 (3)
C9—C14—H14	119.4	C18—C19—C20	109.9 (3)
C13—C14—H14	119.4	C18—C19—Br1	131.8 (2)
C12—C11—C10	120.2 (4)	C20—C19—Br1	118.2 (2)
C12—C11—H11	119.9	O1—C15—H15A	109.5
C10—C11—H11	119.9	O1—C15—H15B	109.5
C12—C13—C14	120.0 (4)	H15A—C15—H15B	109.5
C12—C13—H13	120.0	O1—C15—H15C	109.5
C14—C13—H13	120.0	H15A—C15—H15C	109.5
C13—C12—C11	119.4 (4)	H15B—C15—H15C	109.5

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C16—H16···O3^{i}	0.98	2.49	3.396 (4)	154

Symmetry code: (i) −*x*, *y*−1/2, −*z*+1/2.