Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

3-(4-Methoxyphenyl)-6,7-dihydro-1*H*-furo[3,4-*c*]pyran-4(3*H*)-one

Jingyi Zhang,[a]* Ye An,[a] Yikai Zhang[b] and Guobing Shi[a]*

[a]Department of Pharmacy, General Hospital of Shenyang Military Command, Shenyang 110016, People's Republic of China, and [b]Department of Medicinal Chemistry, School of Pharmacy, Second Military Medical University, Shanghai 200433, People's Republic of China
Correspondence e-mail: hu829Zh213@yahoo.com.cn, sgsgb@126.com

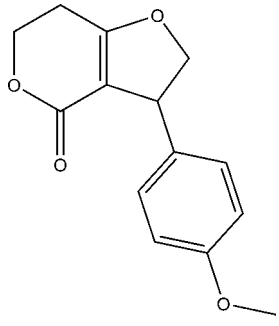
Received 13 November 2012; accepted 16 November 2012

Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.004 Å; R factor = 0.074; wR factor = 0.227; data-to-parameter ratio = 13.0.

In the title compound, $C_{14}H_{14}O_4$, the dihedral angle between the hydrofuran and benzene rings is 88.41 (15)°. The hydropyran ring adopts an envelope conformation, with the O-bound methylene C atom as the flap. In the crystal, weak aromatic π–π stacking is observed [centroid–centroid separation = 3.848 (2) Å].

Related literature

For medicinal background, see: Wang *et al.* (2011).



Experimental

Crystal data

$C_{14}H_{14}O_4$
M_r = 246.25
Monoclinic, $P2_1/n$
a = 7.240 (3) Å
b = 8.635 (4) Å
c = 19.545 (8) Å
β = 97.352 (6)°
V = 1212.0 (9) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.10 mm^{-1}
T = 293 K
0.25 × 0.25 × 0.20 mm

Data collection

Bruker SMART CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2002)
T_{min} = 0.976, T_{max} = 0.981
4872 measured reflections
2127 independent reflections
1494 reflections with $I > 2\sigma(I)$
R_{int} = 0.104

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.074
$wR(F^2)$ = 0.227
S = 1.06
2127 reflections
164 parameters
6 restraints
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.55 e Å^{-3}
$\Delta\rho_{min}$ = −0.31 e Å^{-3}

Data collection: *SMART* (Bruker, 2002); cell refinement: *SAINT* (Bruker, 2002); data reduction: *SAINT*; 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: *SHELXTL*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HB6991).

References

Bruker (2002). *SMART*, *SAINT* and *SADABS*. Bruker AXS Inc., Madison, Wisconsin, USA.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Wang, T. T., Liu, J., Zhong, H. Y., Chen, H., Lv, Z. L., Zhang, Y. K., Zhang, M. F., Geng, D. P., Niu, C. J., Li, Y. M. & Li, K. (2011). *Bioorg. Med. Chem. Lett.* **21**, 3381–3383.

supporting information

Acta Cryst. (2012). E**68**, o3451 [doi:10.1107/S160053681204723X]

3-(4-Methoxyphenyl)-6,7-dihydro-1*H*-furo[3,4-*c*]pyran-4(3*H*)-one

Jingyi Zhang, Ye An, Yikai Zhang and Guobing Shi

S1. Experimental

NaH (60% in mineral oil, 24 mmol) was added to a solution of but-2-yne-1,4-diol (2.58 g, 30 mmol) in THF (50 ml) under nitrogen, and the solution was stirred for 5 min at 20 C. Diethyl 2-(4-methoxybenzylidene)malonate (5.56 g, 20 mmol) and CuI (0.38 g, 2 mmol) were then added successively. When consumption of the starting materials was observed by TLC, the reaction mixture was added 3% HCl solution until the PH value was 7. Then the mixture was extracted with CH_2Cl_2 (30 ml × 3). The combined organic layers were dried and solids were combined. The solid (0.973 g, 3 mmol) subsequently was reacted with 20% KOH in EtOH/THF (15/15 ml) at room temperature for 6 h. Then the reaction mixture was diluted with CH_2Cl_2 (30 ml) and washed with saturated Na_2CO_3, brine and dried with MgSO4. The mixture was purified with silica gel column chromagraphy. The Trans-form compounds could be obtained. Yield: 10%. *M*. p.: 407 K.

S2. Refinement

All hydrogen atoms were placed in calculated positions using a riding model, with d (C—H) = 0.93 Å for aromatic, 0.97 Å for CH2 and 0.96 Å for CH3 groups, and with U_{iso} (H) = 1.2 U_{eq}(C).

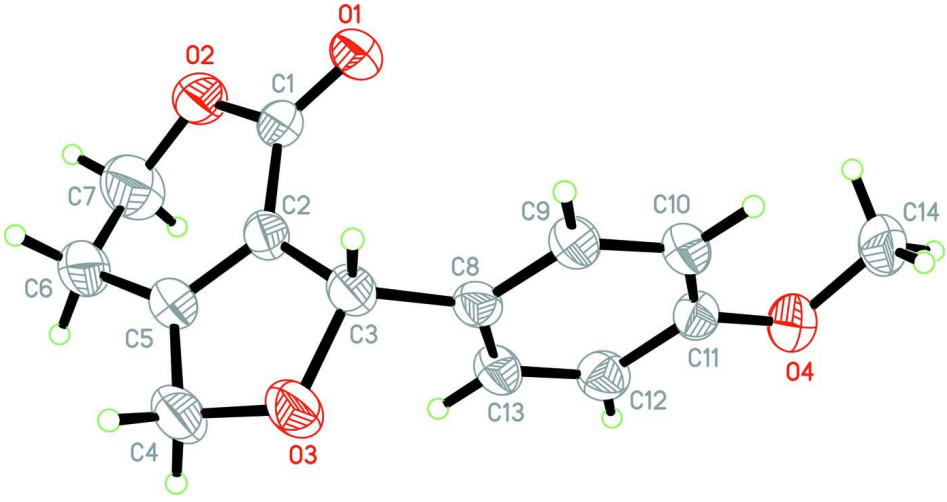


Figure 1

The molecular structure of the title compound, with 30% probability displacement ellipsoids for non-H atoms.

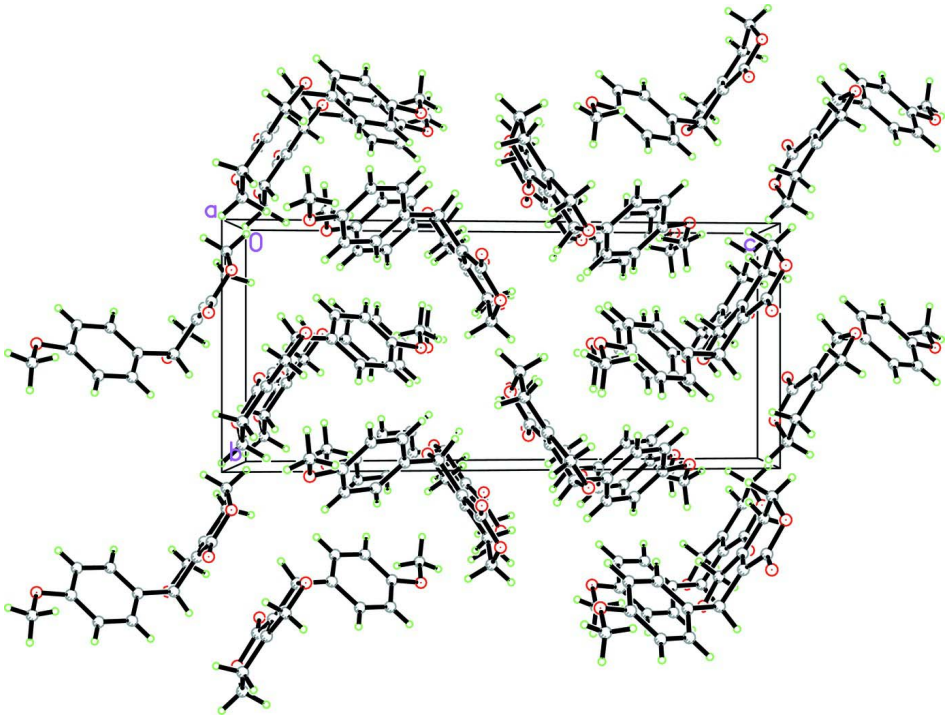


Figure 2

A view of the unit-cell contents for the title compound.

3-(4-Methoxyphenyl)-6,7-dihydro-1*H*-furo[3,4-*c*]pyran-4(3*H*) -one

Crystal data

$C_{14}H_{14}O_4$
$M_r = 246.25$
Monoclinic, $P2_1/n$
Hall symbol: -P 2yn
$a = 7.240$ (3) Å
$b = 8.635$ (4) Å
$c = 19.545$ (8) Å
$\beta = 97.352$ (6)°
$V = 1212.0$ (9) Å^3
$Z = 4$

$F(000) = 520$
$D_x = 1.350$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 735 reflections
$\theta = 2.6$–24.3°
$\mu = 0.10$ mm^{-1}
$T = 293$ K
Prism, colorless
0.25 × 0.25 × 0.20 mm

Data collection

Bruker SMART CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
phi and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2002)
$T_{min} = 0.976$, $T_{max} = 0.981$

4872 measured reflections
2127 independent reflections
1494 reflections with $I > 2\sigma(I)$
$R_{int} = 0.104$
$\theta_{max} = 25.0°$, $\theta_{min} = 2.6°$
$h = -8 \rightarrow 7$
$k = -10 \rightarrow 10$
$l = -23 \rightarrow 19$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.074$
$wR(F^2) = 0.227$
$S = 1.06$
2127 reflections
164 parameters
6 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
$w = 1/[\sigma^2(F_o^2) + (0.1396P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.55$ e Å^{-3}
$\Delta\rho_{min} = -0.31$ 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}
O1	0.3995 (3)	0.6361 (2)	0.04179 (10)	0.0677 (6)
O2	0.5812 (3)	0.8161 (3)	0.00885 (11)	0.0771 (7)
O3	0.9242 (3)	0.4487 (2)	0.14388 (12)	0.0777 (7)
O4	0.2905 (3)	0.5087 (3)	0.33985 (12)	0.0800 (7)
C1	0.5550 (4)	0.6850 (3)	0.04378 (13)	0.0541 (7)
C2	0.7206 (3)	0.6116 (3)	0.07879 (13)	0.0542 (7)
C3	0.7270 (3)	0.4668 (3)	0.12146 (15)	0.0569 (7)
H3	0.6841	0.3796	0.0915	0.068*
C4	1.0291 (4)	0.5583 (4)	0.1116 (2)	0.0805 (10)
H4A	1.1033	0.5076	0.0803	0.097*
H4B	1.1116	0.6153	0.1457	0.097*
C5	0.8909 (4)	0.6629 (3)	0.07366 (15)	0.0622 (8)
C6	0.9218 (4)	0.8021 (4)	0.03314 (19)	0.0838 (10)
H6A	1.0260	0.8605	0.0563	0.101*
H6B	0.9517	0.7719	−0.0120	0.101*
C7	0.7547 (6)	0.8987 (5)	0.0253 (3)	0.1143 (15)
H7A	0.7502	0.9555	0.0679	0.137*
H7B	0.7654	0.9740	−0.0108	0.137*
C8	0.6177 (3)	0.4702 (3)	0.18107 (14)	0.0525 (7)
C9	0.4688 (4)	0.3726 (3)	0.18314 (15)	0.0593 (7)
H9	0.4417	0.2997	0.1483	0.071*
C10	0.3579 (4)	0.3798 (3)	0.23563 (16)	0.0646 (8)
H10	0.2584	0.3119	0.2360	0.077*
C11	0.3952 (4)	0.4871 (3)	0.28691 (15)	0.0593 (7)
C12	0.5476 (4)	0.5848 (3)	0.28669 (16)	0.0657 (8)

H12	0.5769	0.6556	0.3223	0.079*
C13	0.6550 (4)	0.5765 (3)	0.23382 (15)	0.0613 (8)
H13	0.7550	0.6439	0.2335	0.074*
C14	0.1150 (5)	0.4338 (5)	0.33449 (19)	0.0881 (11)
H14A	0.0466	0.4550	0.2901	0.132*
H14B	0.0464	0.4716	0.3699	0.132*
H14C	0.1330	0.3241	0.3398	0.132*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0474 (11)	0.0838 (13)	0.0720 (13)	0.0007 (9)	0.0075 (9)	−0.0005 (10)
O2	0.0686 (13)	0.0824 (14)	0.0777 (14)	−0.0039 (10)	−0.0007 (11)	0.0275 (11)
O3	0.0481 (11)	0.0835 (13)	0.1019 (17)	0.0080 (9)	0.0110 (11)	0.0278 (12)
O4	0.0760 (15)	0.0989 (16)	0.0681 (14)	−0.0059 (12)	0.0208 (11)	0.0058 (11)
C1	0.0532 (16)	0.0636 (15)	0.0458 (14)	−0.0009 (12)	0.0077 (11)	−0.0025 (12)
C2	0.0493 (14)	0.0612 (14)	0.0532 (15)	−0.0073 (11)	0.0109 (12)	−0.0019 (12)
C3	0.0467 (14)	0.0597 (14)	0.0643 (16)	−0.0031 (11)	0.0076 (12)	0.0027 (13)
C4	0.0480 (16)	0.091 (2)	0.103 (2)	−0.0051 (14)	0.0124 (16)	0.0236 (19)
C5	0.0490 (15)	0.0732 (17)	0.0647 (17)	−0.0080 (12)	0.0085 (12)	0.0031 (14)
C6	0.067 (2)	0.098 (2)	0.086 (2)	−0.0250 (17)	0.0082 (17)	0.0274 (19)
C7	0.092 (3)	0.104 (3)	0.144 (4)	−0.017 (2)	0.006 (3)	0.053 (3)
C8	0.0444 (14)	0.0499 (12)	0.0615 (16)	−0.0012 (10)	0.0007 (12)	0.0103 (12)
C9	0.0571 (16)	0.0583 (14)	0.0620 (16)	−0.0110 (12)	0.0051 (13)	0.0006 (13)
C10	0.0543 (15)	0.0695 (16)	0.0691 (18)	−0.0151 (13)	0.0048 (13)	0.0096 (15)
C11	0.0540 (16)	0.0656 (15)	0.0590 (17)	0.0025 (12)	0.0095 (13)	0.0131 (13)
C12	0.0650 (17)	0.0652 (16)	0.0653 (17)	−0.0068 (13)	0.0018 (14)	−0.0034 (14)
C13	0.0507 (15)	0.0629 (15)	0.0696 (18)	−0.0127 (12)	0.0058 (13)	0.0028 (14)
C14	0.068 (2)	0.114 (3)	0.087 (2)	−0.0002 (18)	0.0279 (18)	0.022 (2)

Geometric parameters (Å, º)

O1—C1	1.198 (3)	C6—H6A	0.9700
O2—C1	1.348 (3)	C6—H6B	0.9700
O2—C7	1.445 (4)	C7—H7A	0.9700
O3—C4	1.411 (4)	C7—H7B	0.9700
O3—C3	1.447 (3)	C8—C9	1.374 (4)
O4—C11	1.371 (4)	C8—C13	1.381 (4)
O4—C14	1.418 (4)	C9—C10	1.383 (4)
C1—C2	1.448 (4)	C9—H9	0.9300
C2—C5	1.326 (4)	C10—C11	1.366 (4)
C2—C3	1.501 (4)	C10—H10	0.9300
C3—C8	1.490 (4)	C11—C12	1.390 (4)
C3—H3	0.9800	C12—C13	1.372 (4)
C4—C5	1.476 (4)	C12—H12	0.9300
C4—H4A	0.9700	C13—H13	0.9300
C4—H4B	0.9700	C14—H14A	0.9600
C5—C6	1.472 (4)	C14—H14B	0.9600

C6—C7	1.461 (5)	C14—H14C	0.9600
C1—O2—C7	118.5 (2)	O2—C7—C6	115.2 (3)
C4—O3—C3	111.1 (2)	O2—C7—H7A	108.5
C11—O4—C14	117.3 (3)	C6—C7—H7A	108.5
O1—C1—O2	118.2 (2)	O2—C7—H7B	108.5
O1—C1—C2	125.4 (3)	C6—C7—H7B	108.5
O2—C1—C2	116.4 (2)	H7A—C7—H7B	107.5
C5—C2—C1	122.7 (3)	C9—C8—C13	117.7 (3)
C5—C2—C3	111.0 (2)	C9—C8—C3	120.6 (2)
C1—C2—C3	126.3 (2)	C13—C8—C3	121.6 (2)
O3—C3—C8	111.6 (2)	C8—C9—C10	121.8 (3)
O3—C3—C2	102.55 (19)	C8—C9—H9	119.1
C8—C3—C2	115.9 (2)	C10—C9—H9	119.1
O3—C3—H3	108.9	C11—C10—C9	119.7 (3)
C8—C3—H3	108.9	C11—C10—H10	120.1
C2—C3—H3	108.9	C9—C10—H10	120.1
O3—C4—C5	105.4 (2)	C10—C11—O4	124.7 (3)
O3—C4—H4A	110.7	C10—C11—C12	119.4 (3)
C5—C4—H4A	110.7	O4—C11—C12	115.9 (3)
O3—C4—H4B	110.7	C13—C12—C11	119.8 (3)
C5—C4—H4B	110.7	C13—C12—H12	120.1
H4A—C4—H4B	108.8	C11—C12—H12	120.1
C2—C5—C6	121.4 (3)	C12—C13—C8	121.5 (3)
C2—C5—C4	109.5 (3)	C12—C13—H13	119.3
C6—C5—C4	129.1 (2)	C8—C13—H13	119.3
C7—C6—C5	110.0 (3)	O4—C14—H14A	109.5
C7—C6—H6A	109.7	O4—C14—H14B	109.5
C5—C6—H6A	109.7	H14A—C14—H14B	109.5
C7—C6—H6B	109.7	O4—C14—H14C	109.5
C5—C6—H6B	109.7	H14A—C14—H14C	109.5
H6A—C6—H6B	108.2	H14B—C14—H14C	109.5