Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

2,2,7,7-Tetramethyl-2,3,6,7-tetrahydrobenzofuro[7,6-*b*]furan

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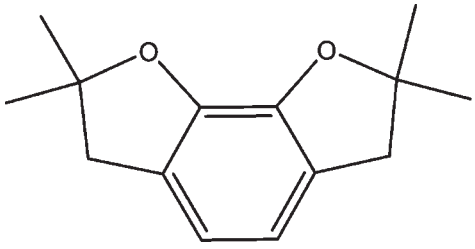
Received 28 January 2010; accepted 4 February 2010

Key indicators: single-crystal X-ray study; T = 173 K; mean σ(C–C) = 0.002 Å; R factor = 0.045; wR factor = 0.131; data-to-parameter ratio = 17.9.

The title compound, $C_{14}H_{18}O_2$, was obtained as a by-product during the preparation of carbofuran phenol. The two dihydrofuran rings are in envelope conformations.

Related literature

For chemical background and related structures, see: Xu *et al.* (2005); Li *et al.* (2009).



Experimental

Crystal data

$C_{14}H_{18}O_2$
M_r = 218.28
Monoclinic, $P2_1/n$
a = 8.7553 (6) Å
b = 6.0721 (4) Å
c = 23.2082 (17) Å
β = 92.186 (1)°
V = 1232.92 (15) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.08 mm^{-1}
T = 173 K
0.45 × 0.44 × 0.39 mm

Data collection

Bruker SMART 1000 CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2004)
T_{min} = 0.966, T_{max} = 0.971
5882 measured reflections
2662 independent reflections
1986 reflections with $I > 2\sigma(I)$
R_{int} = 0.022

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.045
$wR(F^2)$ = 0.131
S = 1.04
2662 reflections
149 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.25 e Å^{-3}
$\Delta\rho_{min}$ = −0.18 e Å^{-3}

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT-Plus* (Bruker, 2003); data reduction: *SAINT-Plus*; program(s) used to solve structure: *SHELXTL* (Sheldrick 2008); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

This work was supported by the Central University Basic Scientific Research Fund of Hunan University.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BT5185).

References

Bruker (2001). *SMART*. Bruker AXS Inc., Madison, Wisconsin, USA.
Bruker (2003). *SAINT-Plus*.Bruker AXS Inc., Madison, Wisconsin, USA.
Li, W.-S., Li, L. & Li, J.-S. (2009). *Acta Cryst.* E**65**, o2928.
Sheldrick, G. M. (2004). *SADABS*. University of Göttingen, Germany.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Xu, L.-Z., Yu, G.-P. & Yang, S.-H. (2005). *Acta Cryst.* E**61**, o1924–o1926.

supporting information

Acta Cryst. (2010). E**66**, o567 [doi:10.1107/S1600536810004423]

2,2,7,7-Tetramethyl-2,3,6,7-tetrahydrobenzofuro[7,6-*b*]furan

Xian-Fu Luo, Lin-Tao Yang, Yu Wang, Jian-Yu Zhang and Ai-Xi Hu

S1. Comment

Carbofuran phenol (systematic name: 2,2-dimethy-2,3-dihydrobenzofuran-7-ol) is an important intermediate to prepare Carbofuran (Xu *et al.*, 2005), Carbosulfan, Benfuracarb, Furathiocarb and other large tonnage carbamate pesticides. It also can be used as pharmaceutical intermediate, as a high value-added fine chemical product. The title compound, 2,2,7,7-tetramethyl-2,3,6,7-tetrahydrobenzofuro[7,6-b]furan, was obtained as a byproduct during the preparation of carbofuran phenol.

S2. Experimental

After distillation of carbofuran phenol, the fraction at 433.15 K(3.33 K Pa) was cooled to room temperature, then the precipitate was emerged. The solid was purified by recrystallization from saturated ethyl acetate solution, giving the title compound as a colourless crystalline solid. Single crystals suitable for X-ray diffraction were obtained by slow evaporation of an ethyl acetate solution at room temperature over a period of ten days. The identity of the title compound was confirmed by NMR and GC—MS spectroscopy.

S3. Refinement

All H atoms were placed in calculated positions, with C—H ranging from 0.95 Å to 0.99 Å and with *U*iso(H) = $1.5U_{eq}(C_{methyl})$ or *U*iso(H) = $1.2U_{eq}(C)$. The methyl groups were allowed to rotate but not to tip.

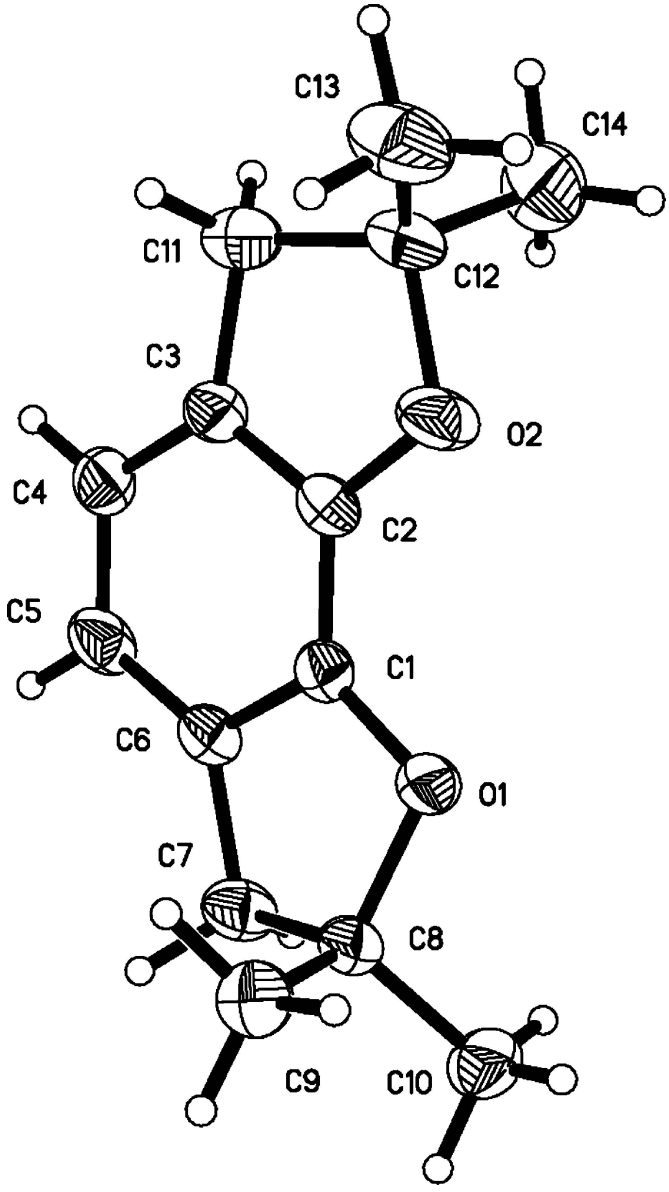


Figure 1

The structure of the title compound showing 30% probability displacement ellipsoids.

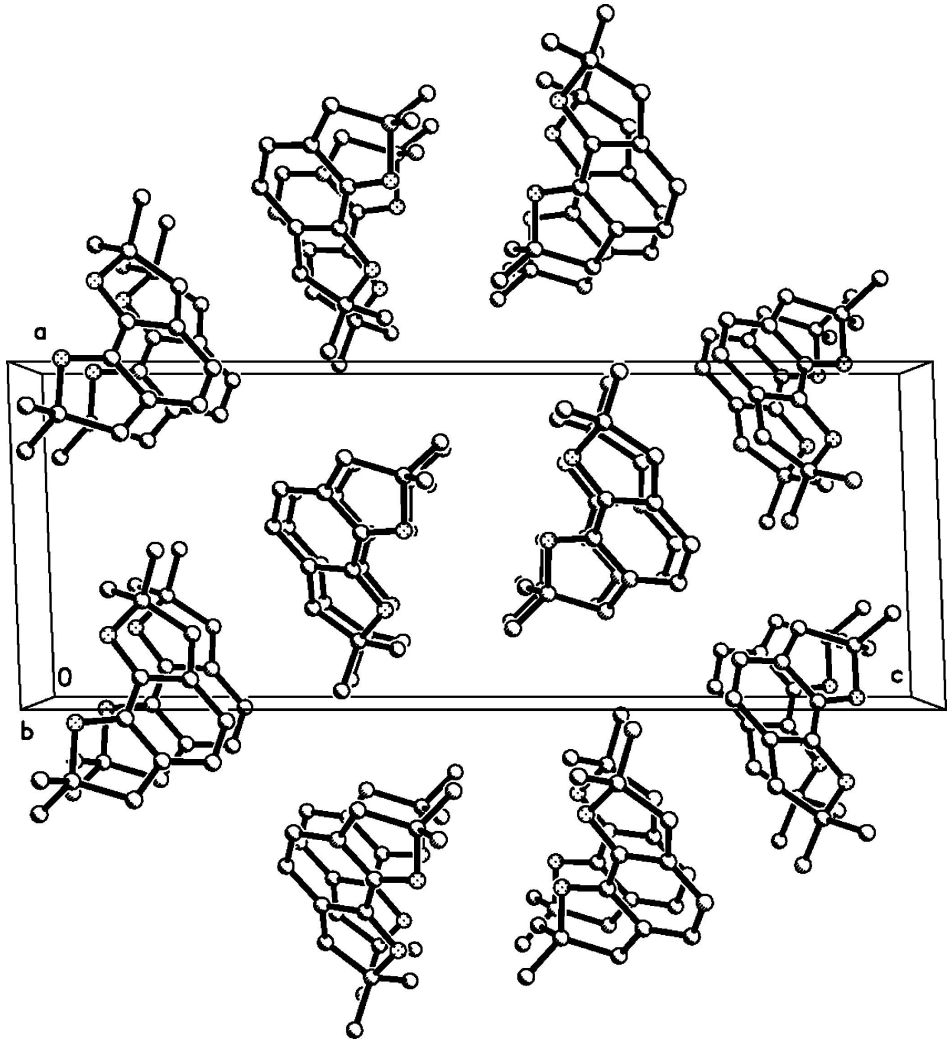


Figure 2

A packing diagram for the title compound. H atoms bonded to C atoms have been omitted for clarity.

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Crystal data

$C_{14}H_{18}O_2$
$M_r = 218.28$
Monoclinic, $P2_1/n$
Hall symbol: -P 2yn
$a = 8.7553$ (6) Å
$b = 6.0721$ (4) Å
$c = 23.2082$ (17) Å
$\beta = 92.186$ (1)°
$V = 1232.92$ (15) Å^3
$Z = 4$

$F(000) = 472$
$D_x = 1.176$ Mg m^{-3}
Melting point: 344.25 K
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 2852 reflections
$\theta = 2.5$–27.0°
$\mu = 0.08$ mm^{-1}
$T = 173$ K
Block, colourless
0.45 × 0.44 × 0.39 mm

Data collection

Bruker SMART 1000 CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2004)
T_{min} = 0.966, T_{max} = 0.971
5882 measured reflections
2662 independent reflections
1986 reflections with $I > 2\sigma(I)$
R_{int} = 0.022
θ_{max} = 27.0°, θ_{min} = 1.8°
h = −11→4
k = −7→7
l = −27→29

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.045
$wR(F^2)$ = 0.131
S = 1.04
2662 reflections
149 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
$w = 1/[\sigma^2(F_o^2) + (0.0698P)^2 + 0.2701P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.25 e Å^{-3}
$\Delta\rho_{min}$ = −0.18 e Å^{-3}

Special details

Experimental. ^{1}H NMR(300MHz, $CDCl_3$), delta: 1.49(s, 12H, CH_3); 2.99(s, 4H, CH_2); 6.63(s, 2H, C_6H_2). GC-MS(m/z): 218, 203, 185, 175, 161.

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 $F^2 > \sigma(F^2)$ 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}
C1	−0.00729 (15)	0.4208 (2)	0.12566 (5)	0.0263 (3)
C2	0.10104 (15)	0.2615 (2)	0.13845 (6)	0.0278 (3)
C3	0.09122 (16)	0.1360 (2)	0.18790 (6)	0.0303 (3)
C4	−0.02597 (17)	0.1695 (3)	0.22541 (6)	0.0362 (4)
H4	−0.0320	0.0825	0.2593	0.043*
C5	−0.13474 (16)	0.3316 (3)	0.21305 (6)	0.0370 (4)
H5	−0.2154	0.3568	0.2385	0.044*
C6	−0.12429 (15)	0.4558 (2)	0.16334 (6)	0.0284 (3)
C7	−0.22006 (17)	0.6411 (3)	0.13820 (6)	0.0344 (4)
H7A	−0.1985	0.7813	0.1587	0.041*
H7B	−0.3305	0.6076	0.1397	0.041*
C8	−0.16891 (15)	0.6516 (2)	0.07551 (6)	0.0288 (3)
C9	−0.26730 (18)	0.5101 (3)	0.03545 (7)	0.0365 (4)
H9A	−0.2232	0.5065	−0.0027	0.055*

H9B	−0.3707	0.5718	0.0322	0.055*
H9C	−0.2719	0.3601	0.0509	0.055*
C10	−0.1533 (2)	0.8823 (3)	0.05219 (8)	0.0426 (4)
H10A	−0.0817	0.9660	0.0772	0.064*
H10B	−0.2534	0.9549	0.0511	0.064*
H10C	−0.1148	0.8758	0.0131	0.064*
C11	0.21711 (19)	−0.0321 (3)	0.18774 (7)	0.0436 (4)
H11A	0.1756	−0.1821	0.1814	0.052*
H11B	0.2783	−0.0295	0.2245	0.052*
C12	0.31387 (17)	0.0417 (3)	0.13672 (7)	0.0361 (4)
C13	0.3435 (2)	−0.1428 (3)	0.09508 (9)	0.0541 (5)
H13A	0.2458	−0.2021	0.0800	0.081*
H13B	0.4015	−0.2597	0.1151	0.081*
H13C	0.4022	−0.0865	0.0631	0.081*
C14	0.4577 (2)	0.1601 (3)	0.15651 (8)	0.0552 (5)
H14A	0.5094	0.2176	0.1229	0.083*
H14B	0.5258	0.0573	0.1775	0.083*
H14C	0.4317	0.2822	0.1819	0.083*
O1	−0.01419 (11)	0.55235 (18)	0.07800 (4)	0.0333 (3)
O2	0.21901 (12)	0.2053 (2)	0.10432 (4)	0.0408 (3)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0245 (7)	0.0291 (7)	0.0254 (7)	−0.0008 (5)	0.0020 (5)	−0.0003 (5)
C2	0.0230 (7)	0.0322 (8)	0.0282 (7)	0.0018 (6)	0.0028 (5)	−0.0035 (6)
C3	0.0259 (7)	0.0317 (8)	0.0329 (7)	−0.0001 (6)	−0.0033 (5)	0.0010 (6)
C4	0.0309 (8)	0.0484 (9)	0.0295 (7)	−0.0024 (7)	0.0010 (6)	0.0120 (7)
C5	0.0260 (7)	0.0565 (10)	0.0288 (7)	0.0044 (7)	0.0065 (5)	0.0053 (7)
C6	0.0241 (7)	0.0341 (8)	0.0272 (7)	0.0020 (6)	0.0015 (5)	−0.0024 (6)
C7	0.0321 (8)	0.0383 (8)	0.0330 (8)	0.0093 (6)	0.0025 (6)	−0.0032 (6)
C8	0.0242 (7)	0.0285 (7)	0.0339 (7)	0.0040 (6)	0.0018 (5)	0.0005 (6)
C9	0.0379 (8)	0.0350 (8)	0.0364 (8)	−0.0012 (7)	−0.0006 (6)	−0.0028 (6)
C10	0.0421 (9)	0.0302 (8)	0.0547 (10)	−0.0037 (7)	−0.0068 (7)	0.0061 (7)
C11	0.0390 (9)	0.0416 (9)	0.0501 (10)	0.0108 (7)	0.0015 (7)	0.0082 (8)
C12	0.0266 (7)	0.0388 (9)	0.0425 (9)	0.0087 (6)	−0.0041 (6)	−0.0028 (7)
C13	0.0450 (10)	0.0521 (11)	0.0649 (12)	0.0124 (8)	−0.0022 (8)	−0.0167 (9)
C14	0.0455 (10)	0.0595 (12)	0.0595 (11)	−0.0107 (9)	−0.0124 (9)	0.0016 (9)
O1	0.0268 (5)	0.0397 (6)	0.0339 (5)	0.0067 (4)	0.0074 (4)	0.0099 (4)
O2	0.0341 (6)	0.0526 (7)	0.0365 (6)	0.0184 (5)	0.0103 (4)	0.0057 (5)

Geometric parameters (Å, º)

C1—O1	1.3639 (16)	C9—H9A	0.9800
C1—C2	1.3792 (19)	C9—H9B	0.9800
C1—C6	1.3882 (19)	C9—H9C	0.9800
C2—O2	1.3685 (16)	C10—H10A	0.9800
C2—C3	1.383 (2)	C10—H10B	0.9800

C3—C4	1.386 (2)	C10—H10C	0.9800
C3—C11	1.502 (2)	C11—C12	1.548 (2)
C4—C5	1.392 (2)	C11—H11A	0.9900
C4—H4	0.9500	C11—H11B	0.9900
C5—C6	1.384 (2)	C12—O2	1.4812 (18)
C5—H5	0.9500	C12—C14	1.507 (2)
C6—C7	1.508 (2)	C12—C13	1.509 (2)
C7—C8	1.540 (2)	C13—H13A	0.9800
C7—H7A	0.9900	C13—H13B	0.9800
C7—H7B	0.9900	C13—H13C	0.9800
C8—O1	1.4816 (16)	C14—H14A	0.9800
C8—C10	1.510 (2)	C14—H14B	0.9800
C8—C9	1.511 (2)	C14—H14C	0.9800

O1—C1—C2	126.45 (12)	H9A—C9—H9C	109.5
O1—C1—C6	114.22 (12)	H9B—C9—H9C	109.5
C2—C1—C6	119.32 (13)	C8—C10—H10A	109.5
O2—C2—C1	125.32 (13)	C8—C10—H10B	109.5
O2—C2—C3	114.54 (12)	H10A—C10—H10B	109.5
C1—C2—C3	120.07 (13)	C8—C10—H10C	109.5
C2—C3—C4	120.76 (13)	H10A—C10—H10C	109.5
C2—C3—C11	107.66 (13)	H10B—C10—H10C	109.5
C4—C3—C11	131.47 (14)	C3—C11—C12	103.19 (12)
C3—C4—C5	119.45 (13)	C3—C11—H11A	111.1
C3—C4—H4	120.3	C12—C11—H11A	111.1
C5—C4—H4	120.3	C3—C11—H11B	111.1
C6—C5—C4	119.37 (13)	C12—C11—H11B	111.1
C6—C5—H5	120.3	H11A—C11—H11B	109.1
C4—C5—H5	120.3	O2—C12—C14	106.33 (13)
C5—C6—C1	121.03 (13)	O2—C12—C13	106.23 (13)
C5—C6—C7	132.51 (13)	C14—C12—C13	112.80 (15)
C1—C6—C7	106.46 (12)	O2—C12—C11	105.66 (11)
C6—C7—C8	102.64 (11)	C14—C12—C11	112.36 (14)
C6—C7—H7A	111.2	C13—C12—C11	112.79 (15)
C8—C7—H7A	111.2	C12—C13—H13A	109.5
C6—C7—H7B	111.2	C12—C13—H13B	109.5
C8—C7—H7B	111.2	H13A—C13—H13B	109.5
H7A—C7—H7B	109.2	C12—C13—H13C	109.5
O1—C8—C10	107.24 (12)	H13A—C13—H13C	109.5
O1—C8—C9	106.96 (11)	H13B—C13—H13C	109.5
C10—C8—C9	111.36 (12)	C12—C14—H14A	109.5
O1—C8—C7	104.17 (10)	C12—C14—H14B	109.5
C10—C8—C7	114.23 (13)	H14A—C14—H14B	109.5
C9—C8—C7	112.23 (12)	C12—C14—H14C	109.5
C8—C9—H9A	109.5	H14A—C14—H14C	109.5
C8—C9—H9B	109.5	H14B—C14—H14C	109.5
H9A—C9—H9B	109.5	C1—O1—C8	106.37 (10)
C8—C9—H9C	109.5	C2—O2—C12	107.10 (11)

O1—C1—C2—O2	−1.5 (2)	C6—C7—C8—O1	−23.68 (14)
C6—C1—C2—O2	177.75 (13)	C6—C7—C8—C10	−140.34 (13)
O1—C1—C2—C3	−178.26 (13)	C6—C7—C8—C9	91.68 (14)
C6—C1—C2—C3	1.0 (2)	C2—C3—C11—C12	8.91 (17)
O2—C2—C3—C4	−177.69 (13)	C4—C3—C11—C12	−174.97 (15)
C1—C2—C3—C4	−0.6 (2)	C3—C11—C12—O2	−13.11 (16)
O2—C2—C3—C11	−1.07 (18)	C3—C11—C12—C14	102.42 (16)
C1—C2—C3—C11	176.00 (13)	C3—C11—C12—C13	−128.73 (14)
C2—C3—C4—C5	−0.1 (2)	C2—C1—O1—C8	165.26 (13)
C11—C3—C4—C5	−175.75 (16)	C6—C1—O1—C8	−14.05 (15)
C3—C4—C5—C6	0.3 (2)	C10—C8—O1—C1	144.79 (12)
C4—C5—C6—C1	0.1 (2)	C9—C8—O1—C1	−95.65 (13)
C4—C5—C6—C7	−179.27 (15)	C7—C8—O1—C1	23.36 (14)
O1—C1—C6—C5	178.59 (13)	C1—C2—O2—C12	175.32 (13)
C2—C1—C6—C5	−0.8 (2)	C3—C2—O2—C12	−7.79 (16)
O1—C1—C6—C7	−1.89 (16)	C14—C12—O2—C2	−106.65 (15)
C2—C1—C6—C7	178.75 (12)	C13—C12—O2—C2	132.97 (14)
C5—C6—C7—C8	−164.44 (16)	C11—C12—O2—C2	12.94 (16)
C1—C6—C7—C8	16.11 (15)		