

(E)-4-(1,3-Benzodioxol-5-yl)but-3-en-2-oneS. Sarveswari,^a V. Vijayakumar,^a Priya Susan Mathew,^a Rafael Mendoza-Meroño^b and Santiago García-Granda^{b*}^aOrganic Chemistry Division, School of Advanced Sciences, VIT University, Vellore 632 014, India, and ^bDepartamento de Química Física y Analítica, Facultad de Química, Universidad de Oviedo – CINN, C/ Julián Clavería 8, 33006 Oviedo, Asturias, Spain

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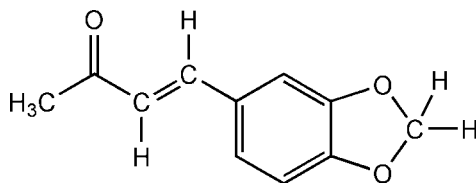
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Key indicators: single-crystal X-ray study; $T = 293$ K; mean $\sigma(\text{C}-\text{C}) = 0.002$ Å; R factor = 0.036; wR factor = 0.094; data-to-parameter ratio = 10.4.

In the title compound, $\text{C}_{11}\text{H}_{10}\text{O}_3$, the benzodioxole ring adopts a flattened [puckering parameters: $q_2 = 0.107$ (2) Å, $\varphi_2 = 160$ (1)°] envelope conformation with the methylene C atom as the flap. The crystal packing features chains, parallel to the c axis, composed of dimers connected by weak C—H⋯O hydrogen bonds and extending in layers in the bc plane.

Related literature

For the synthesis of chalcones, see: Loh *et al.* (2010). For a related structure, see: Gao & Ng (2006).

**Experimental***Crystal data*

$\text{C}_{11}\text{H}_{10}\text{O}_3$
 $M_r = 190.19$
 Monoclinic, $P2_1/c$
 $a = 5.3469$ (3) Å
 $b = 16.4849$ (8) Å
 $c = 10.5475$ (6) Å
 $\beta = 99.183$ (5)°

$V = 917.77$ (9) Å³
 $Z = 4$
 Cu $K\alpha$ radiation
 $\mu = 0.83$ mm⁻¹
 $T = 293$ K
 $0.18 \times 0.11 \times 0.09$ mm

Data collection

Oxford Diffraction Xcalibur Ruby Gemini diffractometer
 Absorption correction: multi-scan (*CrysAlis PRO*; Oxford Diffraction, 2009)
 $T_{\min} = 0.950$, $T_{\max} = 1.000$
 5375 measured reflections
 1737 independent reflections
 1121 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.029$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.036$
 $wR(F^2) = 0.094$
 $S = 0.92$
 1737 reflections
 167 parameters
 All H-atom parameters refined
 $\Delta\rho_{\max} = 0.12$ e Å⁻³
 $\Delta\rho_{\min} = -0.15$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

| $D-H\cdots A$ | $D-H$ | $H\cdots A$ | $D\cdots A$ | $D-H\cdots A$ |
|-----------------------|----------|-------------|-------------|---------------|
| $C7-H7\cdots O1^i$ | 0.95 (2) | 2.56 (2) | 3.489 (2) | 166 (1) |
| $C9-H9\cdots O1^i$ | 0.93 (2) | 2.60 (2) | 3.517 (2) | 171 (1) |
| $C8-H8\cdots O2^{ii}$ | 0.93 (2) | 2.90 (2) | 3.819 (2) | 177 (1) |

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

Data collection: *CrysAlis PRO* (Oxford Diffraction, 2009); cell refinement: *CrysAlis PRO*; data reduction: *CrysAlis PRO*; program(s) used to solve structure: *SIR92* (Altomare *et al.*, 1994); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: NG5110).

References

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supplementary materials

Acta Cryst. (2011). E67, o583 [doi:10.1107/S1600536811004077]

(E)-4-(1,3-Benzodioxol-5-yl)but-3-en-2-one

S. Sarveswari, V. Vijayakumar, P. S. Mathew, R. Mendoza-Meroño and S. García-Granda

Comment

Chalcones and its heterocyclic analogs have been used as intermediates in organic synthesis and exhibit diverse biological activities such as antimicrobial and cytotoxic agents. From a chemistry point of view, an important feature of chalcones and their heteroanalogs is the ability to act as activated unsaturated systems in conjugated addition reactions of carbanions. In continuation with our interest in the synthesis of chalcones (Loh *et al.*, 2010) herein we report the structure of the title compound (I).

In the title compound (I) the spatial arrangement of the keto group C(10)=O(3) and the olefinic double bond C(8)=C(9) with respect to the single bond C9—C10 is *trans*, as indicated the C(8)—C(9)—C(10)—O(3) torsion angle value(-176.10 (18)°). The C(8)=C(9) (1.325 (2) Å), C(9)—C(10) (1.459 (2) Å) and C10=O3 (1.225 (2) Å) distances values are similar of the structures previously reported (Gao and Ng, 2006).

Plane A is referred to C(8)/C(9)/C(10)/O(3) atoms (maximum deviation C(9) 0.0229 (17) Å). The dihedral angle between C(2)/C(7) benzene ring (maximum deviation C(4) -0.0040 (18) Å) and *plane A* is 7.25 (10)°. In benzodioxole ring C(1) is displaced from mean plane by 0.1351 (22) Å, forming a flattened envelope conformation with C(1) as the flap atom. The packing in the crystal structure is dominated by molecular chains made of dimers connected by C—H—O weak hydrogen bonds and extended along *bc* plane.

Insert scheme 1.

The asymmetric unit consists of a single molecule (I), shown in Figure 1.

Experimental

A mixture of acetone (3.0 g 0.005M) and benzo[*d*][1,3]dioxole-5-carbaldehyde (1.5 g 0.01M) and a catalytic amount of KOH in distilled ethanol was stirred for about 12 h, the resulting mixture was concentrated to remove ethanol then poured on to ice and neutralized with dilute acetic acid. The resultant solid was filtered, dried and purified by column chromatography using 1:1 mixture of ethyl acetate and petroleum ether. Recrystallized from acetone; Yield: 49% and m.pt: 412–414 K.

Refinement

At the end of the refinement the highest peak in the electron density was 0.124 e Å⁻³, while the deepest hole was -0.154 e Å⁻³.

Figures

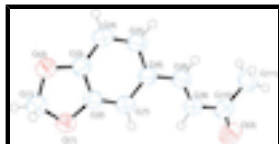


Fig. 1. A view of (I), showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level.

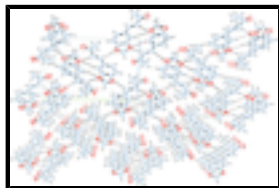


Fig. 2. Packing diagram viewed parallel to the *bc* plane. Hydrogen bonds are indicated by dashed lines.

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Crystal data

$C_{11}H_{10}O_3$

$M_r = 190.19$

Monoclinic, $P2_1/c$

Hall symbol: -P 2ybc

$a = 5.3469$ (3) Å

$b = 16.4849$ (8) Å

$c = 10.5475$ (6) Å

$\beta = 99.183$ (5)°

$V = 917.77$ (9) Å³

$Z = 4$

$F(000) = 400$

$D_x = 1.376$ Mg m⁻³

Melting point: 413 K

Cu $K\alpha$ radiation, $\lambda = 1.54184$ Å

Cell parameters from 1941 reflections

$\theta = 4.2$ – 70.6 °

$\mu = 0.83$ mm⁻¹

$T = 293$ K

Prismatic, yellow

$0.18 \times 0.11 \times 0.09$ mm

Data collection

Oxford Diffraction Xcalibur Ruby Gemini diffractometer

Radiation source: fine-focus sealed tube graphite

Detector resolution: 10.2673 pixels mm⁻¹

ω scans

Absorption correction: multi-scan (*Crys.Alis PRO*; Oxford Diffraction, 2009)

$T_{\min} = 0.950$, $T_{\max} = 1.000$

5375 measured reflections

1737 independent reflections

1121 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.029$

$\theta_{\max} = 70.5$ °, $\theta_{\min} = 5.0$ °

$h = -5$ → 6

$k = -14$ → 19

$l = -12$ → 11

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.036$

Primary atom site location: structure-invariant direct methods

Secondary atom site location: difference Fourier map

Hydrogen site location: inferred from neighbouring sites

| | |
|-------------------|--|
| $wR(F^2) = 0.094$ | All H-atom parameters refined |
| $S = 0.92$ | $w = 1/[\sigma^2(F_o^2) + (0.0547P)^2]$ |
| 1737 reflections | where $P = (F_o^2 + 2F_c^2)/3$ |
| 167 parameters | $(\Delta/\sigma)_{\max} < 0.001$ |
| 0 restraints | $\Delta\rho_{\max} = 0.12 \text{ e } \text{\AA}^{-3}$ |
| | $\Delta\rho_{\min} = -0.15 \text{ e } \text{\AA}^{-3}$ |

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 (\AA^2)

| | x | y | z | $U_{\text{iso}}^*/U_{\text{eq}}$ |
|------|-------------|--------------|---------------|----------------------------------|
| O2 | 0.5552 (2) | 0.23540 (7) | -0.17522 (12) | 0.0686 (3) |
| O1 | 0.5790 (2) | 0.09846 (7) | -0.13128 (11) | 0.0669 (4) |
| C2 | 0.4130 (3) | 0.13463 (9) | -0.06159 (15) | 0.0528 (4) |
| C6 | 0.1246 (3) | 0.14906 (9) | 0.08521 (14) | 0.0528 (4) |
| C5 | 0.1132 (3) | 0.23186 (10) | 0.05728 (16) | 0.0597 (4) |
| C7 | 0.2809 (3) | 0.09884 (10) | 0.02303 (16) | 0.0556 (4) |
| C3 | 0.3994 (3) | 0.21645 (9) | -0.08768 (15) | 0.0557 (4) |
| C8 | -0.0220 (3) | 0.11602 (10) | 0.17895 (16) | 0.0563 (4) |
| C4 | 0.2514 (3) | 0.26731 (10) | -0.02911 (17) | 0.0627 (4) |
| C9 | -0.0146 (3) | 0.04080 (10) | 0.22361 (17) | 0.0591 (4) |
| O3 | -0.1186 (3) | -0.06111 (8) | 0.35856 (14) | 0.0857 (4) |
| C10 | -0.1536 (3) | 0.00895 (11) | 0.32109 (16) | 0.0632 (4) |
| C11 | -0.3362 (5) | 0.06204 (16) | 0.3750 (3) | 0.0802 (6) |
| C1 | 0.6491 (4) | 0.16001 (11) | -0.2141 (2) | 0.0683 (5) |
| H8 | -0.129 (3) | 0.1522 (10) | 0.2107 (17) | 0.065 (5)* |
| H7 | 0.295 (3) | 0.0424 (10) | 0.0405 (15) | 0.058 (4)* |
| H4 | 0.242 (3) | 0.3245 (11) | -0.0461 (17) | 0.074 (5)* |
| H9 | 0.085 (3) | 0.0018 (11) | 0.1929 (15) | 0.069 (5)* |
| H5 | 0.007 (3) | 0.2651 (10) | 0.0987 (15) | 0.061 (4)* |
| H1A | 0.567 (3) | 0.1472 (11) | -0.304 (2) | 0.079 (6)* |
| H1B | 0.839 (4) | 0.1638 (11) | -0.2067 (17) | 0.078 (5)* |
| H11A | -0.415 (6) | 0.0290 (19) | 0.423 (3) | 0.156 (12)* |
| H11C | -0.247 (5) | 0.1005 (18) | 0.433 (3) | 0.148 (12)* |
| H11B | -0.442 (6) | 0.0918 (19) | 0.316 (3) | 0.147 (12)* |

supplementary materials

Atomic displacement parameters (\AA^2)

| | U^{11} | U^{22} | U^{33} | U^{12} | U^{13} | U^{23} |
|-----|-------------|-------------|-------------|-------------|-------------|-------------|
| O2 | 0.0755 (7) | 0.0532 (7) | 0.0818 (8) | 0.0007 (5) | 0.0272 (6) | 0.0100 (6) |
| O1 | 0.0752 (8) | 0.0532 (7) | 0.0789 (8) | 0.0086 (5) | 0.0323 (6) | 0.0055 (6) |
| C2 | 0.0533 (8) | 0.0466 (8) | 0.0587 (9) | 0.0033 (7) | 0.0092 (7) | -0.0001 (7) |
| C6 | 0.0540 (8) | 0.0482 (9) | 0.0560 (9) | 0.0025 (7) | 0.0082 (7) | -0.0004 (7) |
| C5 | 0.0658 (10) | 0.0485 (9) | 0.0663 (10) | 0.0081 (8) | 0.0148 (8) | -0.0030 (8) |
| C7 | 0.0602 (9) | 0.0430 (9) | 0.0643 (10) | 0.0042 (7) | 0.0121 (7) | 0.0024 (8) |
| C3 | 0.0569 (9) | 0.0497 (9) | 0.0605 (10) | -0.0023 (7) | 0.0090 (7) | 0.0046 (7) |
| C8 | 0.0565 (9) | 0.0530 (10) | 0.0594 (9) | 0.0041 (7) | 0.0094 (7) | -0.0033 (8) |
| C4 | 0.0719 (10) | 0.0427 (9) | 0.0745 (11) | 0.0031 (8) | 0.0144 (8) | 0.0047 (8) |
| C9 | 0.0610 (9) | 0.0497 (10) | 0.0684 (10) | 0.0019 (7) | 0.0153 (8) | -0.0020 (8) |
| O3 | 0.1042 (10) | 0.0586 (8) | 0.0963 (10) | -0.0024 (7) | 0.0216 (8) | 0.0171 (7) |
| C10 | 0.0642 (10) | 0.0564 (10) | 0.0673 (10) | -0.0067 (8) | 0.0048 (8) | 0.0047 (8) |
| C11 | 0.0771 (13) | 0.0829 (15) | 0.0875 (16) | 0.0063 (12) | 0.0340 (12) | 0.0146 (14) |
| C1 | 0.0753 (12) | 0.0575 (10) | 0.0766 (13) | 0.0014 (9) | 0.0260 (10) | 0.0071 (9) |

Geometric parameters (\AA , $^\circ$)

| | | | |
|----------|-------------|---------------|-------------|
| O2—C3 | 1.3746 (19) | C8—C9 | 1.325 (2) |
| O2—C1 | 1.425 (2) | C8—H8 | 0.926 (18) |
| O1—C2 | 1.3755 (18) | C4—H4 | 0.960 (18) |
| O1—C1 | 1.428 (2) | C9—C10 | 1.459 (2) |
| C2—C7 | 1.358 (2) | C9—H9 | 0.927 (18) |
| C2—C3 | 1.376 (2) | O3—C10 | 1.225 (2) |
| C6—C5 | 1.396 (2) | C10—C11 | 1.490 (3) |
| C6—C7 | 1.410 (2) | C11—H11A | 0.90 (3) |
| C6—C8 | 1.462 (2) | C11—H11C | 0.95 (3) |
| C5—C4 | 1.390 (2) | C11—H11B | 0.92 (3) |
| C5—H5 | 0.944 (17) | C1—H1A | 1.005 (19) |
| C7—H7 | 0.948 (17) | C1—H1B | 1.009 (19) |
| C3—C4 | 1.366 (2) | | |
| C3—O2—C1 | 105.93 (12) | C3—C4—H4 | 122.5 (11) |
| C2—O1—C1 | 105.90 (12) | C5—C4—H4 | 121.1 (11) |
| C7—C2—O1 | 127.63 (14) | C8—C9—C10 | 126.69 (16) |
| C7—C2—C3 | 122.73 (14) | C8—C9—H9 | 120.7 (11) |
| O1—C2—C3 | 109.63 (13) | C10—C9—H9 | 112.6 (11) |
| C5—C6—C7 | 119.01 (14) | O3—C10—C9 | 119.83 (17) |
| C5—C6—C8 | 119.81 (14) | O3—C10—C11 | 120.43 (17) |
| C7—C6—C8 | 121.17 (14) | C9—C10—C11 | 119.74 (17) |
| C4—C5—C6 | 122.66 (16) | C10—C11—H11A | 105 (2) |
| C4—C5—H5 | 118.7 (10) | C10—C11—H11C | 110.3 (18) |
| C6—C5—H5 | 118.6 (10) | H11A—C11—H11C | 106 (2) |
| C2—C7—C6 | 117.36 (15) | C10—C11—H11B | 115.3 (19) |
| C2—C7—H7 | 121.4 (9) | H11A—C11—H11B | 115 (3) |
| C6—C7—H7 | 121.3 (9) | H11C—C11—H11B | 106 (3) |

| | | | |
|-------------|--------------|---------------|--------------|
| C4—C3—O2 | 128.40 (14) | O2—C1—O1 | 107.73 (14) |
| C4—C3—C2 | 121.79 (15) | O2—C1—H1A | 109.7 (11) |
| O2—C3—C2 | 109.80 (13) | O1—C1—H1A | 108.3 (11) |
| C9—C8—C6 | 126.90 (16) | O2—C1—H1B | 108.7 (10) |
| C9—C8—H8 | 117.3 (11) | O1—C1—H1B | 110.9 (10) |
| C6—C8—H8 | 115.8 (11) | H1A—C1—H1B | 111.5 (15) |
| C3—C4—C5 | 116.44 (16) | | |
| C1—O1—C2—C7 | 174.79 (18) | C7—C2—C3—O2 | 179.18 (15) |
| C1—O1—C2—C3 | -6.33 (18) | O1—C2—C3—O2 | 0.24 (18) |
| C7—C6—C5—C4 | -0.4 (3) | C5—C6—C8—C9 | -173.77 (17) |
| C8—C6—C5—C4 | 178.81 (16) | C7—C6—C8—C9 | 5.4 (3) |
| O1—C2—C7—C6 | 178.83 (15) | O2—C3—C4—C5 | -179.36 (16) |
| C3—C2—C7—C6 | 0.1 (2) | C2—C3—C4—C5 | -0.6 (3) |
| C5—C6—C7—C2 | 0.0 (2) | C6—C5—C4—C3 | 0.7 (3) |
| C8—C6—C7—C2 | -179.22 (15) | C6—C8—C9—C10 | 177.30 (16) |
| C1—O2—C3—C4 | -175.19 (18) | C8—C9—C10—O3 | -176.10 (17) |
| C1—O2—C3—C2 | 5.96 (19) | C8—C9—C10—C11 | 3.5 (3) |
| C7—C2—C3—C4 | 0.3 (3) | C3—O2—C1—O1 | -9.8 (2) |
| O1—C2—C3—C4 | -178.69 (15) | C2—O1—C1—O2 | 9.91 (19) |

Hydrogen-bond geometry (\AA , $^\circ$)

| $D-H\cdots A$ | $D-H$ | $H\cdots A$ | $D\cdots A$ | $D-H\cdots A$ |
|---------------------------------|----------|-------------|-------------|---------------|
| C7—H7 \cdots O1 ⁱ | 0.95 (2) | 2.56 (2) | 3.489 (2) | 166 (1) |
| C9—H9 \cdots O1 ⁱ | 0.93 (2) | 2.60 (2) | 3.517 (2) | 171 (1) |
| C8—H8 \cdots O2 ⁱⁱ | 0.93 (2) | 2.90 (2) | 3.819 (2) | 177 (1) |

Symmetry codes: (i) $-x+1, -y, -z$; (ii) $x-1, -y+1/2, z+1/2$.

Fig. 1

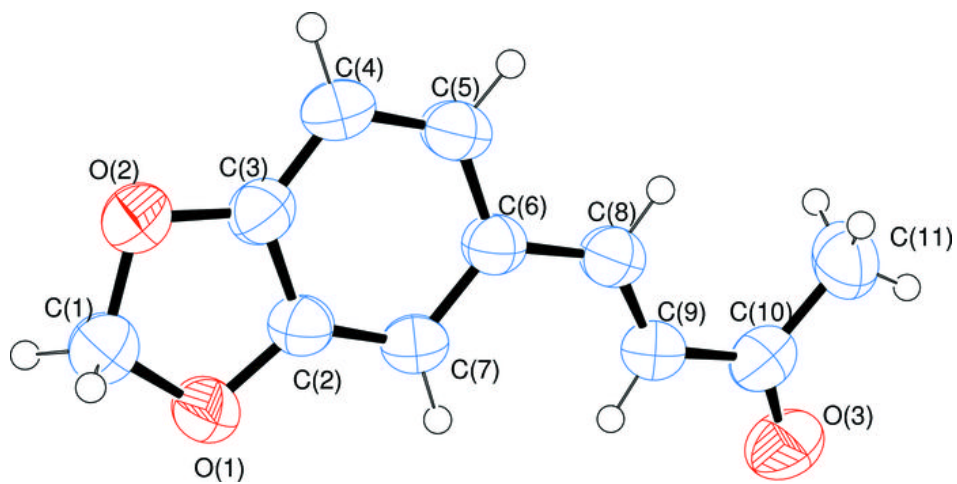


Fig. 2

