1-(1,3-Benzodioxol-5-yl)ethanone

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In the title compound, $C_9H_8O_3$, the dihedral angle between the mean planes of the benzene and dioxole rings is 1.4 (8)°, with the dioxole group in a slightly distorted envelope configuration with the flap C atom displaced by 0.0645 Å from the plane through the other four atoms. In the crystal, weak intermolecular C—H···O hydrogen-bond interactions link the molecules into chains propagating in [011]. The crystal packing exhibits weak π–π interactions as evidenced by the relatively short distances [3.801 (9) Å] between the centroids of adjacent benzene rings.

Related literature

For the pharmaceutical properties of compounds containing the 1,3-dioxolyl group, see: Gabrielsen *et al.* (1992); Krause & Goeber (1972); Ma *et al.* (1987*a,b*); Ohta & Kimoto (1976); For bond-length data, see: Allen *et al.* (1987). For related structures, see: Jasinski *et al.* (2008); Yathirajan *et al.* (2007). For puckering parameters, see: Cremer & Pople (1975). For MOPAC AM1 calculations, see: Schmidt & Polik (2007).
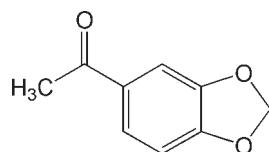
Experimental

Crystal data

$C_9H_8O_3$	b = 10.8445 (3) Å
M_r = 164.15	c = 7.5148 (3) Å
Monoclinic, $P2_1/c$	β = 105.973 (3)°
a = 9.4697 (3) Å	V = 741.93 (4) Å³
Z = 4	T = 200 K
Mo $K\alpha$ radiation	0.58 × 0.45 × 0.26 mm
μ = 0.11 mm⁻¹	

Data collection

Oxford Diffraction R Gemini diffractometer	12470 measured reflections
Absorption correction: multi-scan (*CrysAlis RED*; Oxford Diffraction, 2007) T_{min} = 0.909, T_{max} = 0.972	3061 independent reflections 2215 reflections with $I > 2\sigma(I)$ R_{int} = 0.024

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.045	110 parameters
$wR(F^2)$ = 0.133	H-atom parameters constrained
S = 1.03	$\Delta\rho_{max}$ = 0.39 e Å⁻³
3061 reflections	$\Delta\rho_{min}$ = −0.28 e Å⁻³

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

D—H···A	D—H	H···A	D···A	D—H···A
C3—H3A···O3^i	0.95	2.50	3.423 (1)	165

Symmetry code: (i) $-x + 1, y + \frac{1}{2}, -z + \frac{3}{2}$.

Data collection: *CrysAlis PRO* (Oxford Diffraction, 2007); cell refinement: *CrysAlis PRO*; data reduction: *CrysAlis PRO* 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: IM2174).

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S1. Comment

Acetophenone is the simplest aromatic ketone. It is used as a polymerization catalyst for the manufacture of olefins, as an intermediate for pharmaceuticals, agrochemicals and other organic compounds, as a drug to induce sleep and as a solvent for plastics, resins, cellulose ethers, and esters. Acetophenone and its derivatives are ingredients of flavor and fragrance for soaps, detergents, cosmetics, and perfumes as well as in foods, beverages, and tobacco. Many synthetic or naturally occurring compounds containing the 1,3-dioxolyl group are very important because of their pharmacological properties (Ma *et al.* 1987*a,b*; Ohta & Kimoto 1976; Krause & Goeber 1972; Gabrielsen *et al.* 1992). The crystal structure of 1,3-benzodioxol-5-ylmethanol (Yathirajan *et al.*, 2007) is reported. The title compound, (I), was used recently for the synthesis of (2E)-1-(1,3-benzodioxol-5-yl)-3-(4-chlorophenyl)prop-2-en-1-one and (2E)-1-(1,3-benzodioxol-5-yl)-3-(3,4-dimethoxyphenyl) prop-2-en-1-one (Jasinski *et al.*, 2008). In view of the importance of the title compound, $C_9H_8O_3$, (I), we report the crystal structure.

The molecular structure consists of an ethanoyl group bonded to a benzene group which is fused to a 1,3-dioxol ring in a nearly planar fashion (Fig. 1). The dihedral angle between the mean planes of the benzene and dioxol ring is 1.4 (8)°, as the dioxol group maintains itself in a slightly distorted envelope configuration (Cremer & Pople, 1975) with puckering parameters Q(2) and Phi(2) of 0.1020 and 34.7750, respectively. For an ideal envelope, Phi(2) has a value of k *x* 36. Bond lengths and bond angles are all within expected ranges (Allen *et al.* 1987).

Weak intermolecular C—H···O hydrogen bond interactions link the molecules into chains propagating in the [011] direction (Fig. 2). Crystal packing exhibits weak *Cg*2—*Cg*2 π-π interactions as evidenced by relatively short distances between the centroids of nearby aromatic rings (*Cg*2—*Cg*2: 3.8019 Å; slippage = 1.630 Å; 1 - *x*, -*y*, -*z*; *Cg*2 = ring centroid for C2—C7). A geometry optimized MOPAC AM1 computational calculation (Schmidt & Polik 2007) on (I) (AM1 (Austin Model 1 approximation), *in vacuo*, results in a completely planar molecule. This observation supports a suggestion that intermolecular forces influence the molecular conformation in the crystal.

S2. Experimental

The title compound (I) was obtained from Aldrich Chemical Company and was recrystallized from DMF by slow evaporation (m.p.: 360–362 K). Analysis for the title compound $C_9H_8O_3$: Found (calculated): C: 65.85 (65.91); H: 4.91(4.86).

S3. Refinement

All H atoms were placed in calculated positions and wer refined using the riding model with C—H = 0.95–0.98 Å, and with $U_{iso}(H) = 1.17–1.50 U_{eq}(C)$.
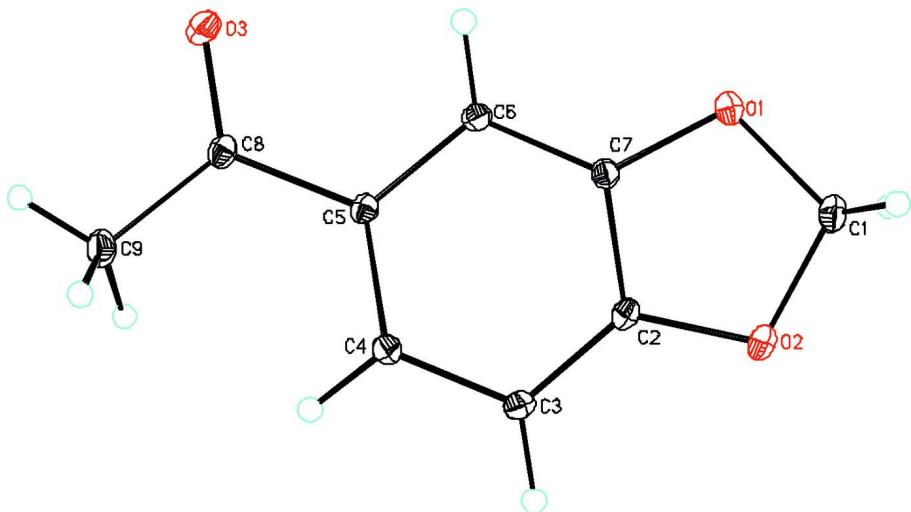
Figure 1

Molecular structure of (I), $C_9H_8O_3$, showing the atom labeling scheme and 50% probability displacement ellipsoids.
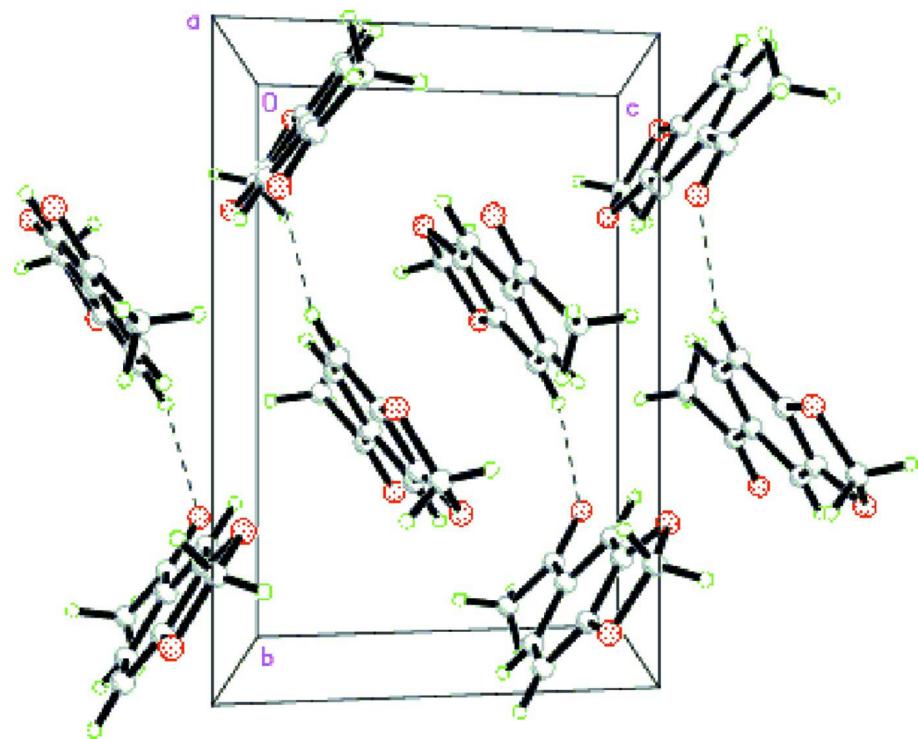
Figure 2

The molecular packing for (I) viewed down the *a* axis. Dashed lines indicate weak C—H···O intermolecular hydrogen bond interactions which link the molecule into chains propagating along the [011].

1-(1,3-Benzodioxol-5-yl)ethanone

Crystal data

$C_9H_8O_3$	Monoclinic, $P2_1/c$
$M_r = 164.15$	Hall symbol: -P 2ybc

$a = 9.4697$ (3) Å

$b = 10.8445$ (3) Å

$c = 7.5148$ (3) Å

$β = 105.973$ (3)°

$V = 741.93$ (4) Å^3

$Z = 4$

$F(000) = 344$

$D_x = 1.470$ Mg m^{-3}

Mo $Kα$ radiation, $λ = 0.71073$ Å

Cell parameters from 5168 reflections

$θ = 4.8–34.7°$

$μ = 0.11$ mm^{-1}

$T = 200$ K

Irregular plate, colorless

$0.58 × 0.45 × 0.26$ mm

Data collection

Oxford Diffraction R Gemini
    diffractometer

Radiation source: fine-focus sealed tube

Graphite monochromator

Detector resolution: 10.5081 pixels mm^{-1}

$φ$ and $ω$ scans

Absorption correction: multi-scan
    (*CrysAlis RED*; Oxford Diffraction, 2007)

$T_{min} = 0.909$, $T_{max} = 0.972$

12470 measured reflections

3061 independent reflections

2215 reflections with $I > 2σ(I)$

$R_{int} = 0.024$

$θ_{max} = 34.8°$, $θ_{min} = 4.9°$

$h = −14→14$

$k = −17→15$

$l = −11→11$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2σ(F^2)] = 0.045$

$wR(F^2) = 0.133$

$S = 1.03$

3061 reflections

110 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

$w = 1/[σ^2(F_o^2) + (0.0852P)^2]$
    where $P = (F_o^2 + 2F_c^2)/3$

$(Δ/σ)_{max} < 0.001$

$Δρ_{max} = 0.39$ e Å^{-3}

$Δρ_{min} = −0.28$ 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 $F^2 > σ(F^2)$ 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	$U_{iso}*/U_{eq}$
O1	0.19844 (7)	0.26081 (7)	0.45409 (11)	0.03260 (19)
O2	0.12787 (7)	0.42705 (7)	0.60401 (10)	0.02944 (17)
O3	0.76739 (8)	0.26971 (7)	0.62415 (11)	0.03273 (18)
C1	0.07627 (10)	0.31692 (10)	0.50175 (15)	0.0315 (2)
H1A	−0.0023	0.3370	0.3882	0.038*
H1B	0.0357	0.2596	0.5777	0.038*
C2	0.27739 (9)	0.42276 (8)	0.64441 (11)	0.02067 (17)
C3	0.37564 (9)	0.50450 (8)	0.74958 (12)	0.02310 (18)

H3A	0.3448	0.5732	0.8079	0.028*
C4	0.52406 (9)	0.48140 (8)	0.76650 (12)	0.02156 (17)
H4A	0.5960	0.5353	0.8397	0.026*
C5	0.56965 (8)	0.38161 (8)	0.67914 (11)	0.01868 (16)
C6	0.46499 (9)	0.30025 (8)	0.56785 (12)	0.02042 (17)
H6A	0.4936	0.2329	0.5048	0.024*
C7	0.32064 (9)	0.32334 (8)	0.55556 (11)	0.02051 (17)
C8	0.72805 (9)	0.35800 (8)	0.70045 (12)	0.02201 (18)
C9	0.84055 (10)	0.44275 (10)	0.82066 (14)	0.0293 (2)
H9A	0.9373	0.4250	0.8032	0.044*
H9B	0.8436	0.4298	0.9507	0.044*
H9C	0.8139	0.5286	0.7863	0.044*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0185 (3)	0.0348 (4)	0.0419 (4)	−0.0031 (3)	0.0041 (3)	−0.0136 (3)
O2	0.0162 (3)	0.0337 (4)	0.0373 (4)	0.0036 (2)	0.0055 (3)	−0.0046 (3)
O3	0.0236 (3)	0.0311 (4)	0.0461 (4)	0.0029 (3)	0.0140 (3)	−0.0052 (3)
C1	0.0180 (4)	0.0394 (5)	0.0362 (5)	−0.0029 (4)	0.0061 (4)	−0.0063 (4)
C2	0.0166 (3)	0.0237 (4)	0.0217 (4)	0.0032 (3)	0.0054 (3)	0.0019 (3)
C3	0.0227 (4)	0.0221 (4)	0.0248 (4)	0.0030 (3)	0.0071 (3)	−0.0028 (3)
C4	0.0204 (4)	0.0209 (4)	0.0227 (4)	−0.0010 (3)	0.0048 (3)	−0.0013 (3)
C5	0.0172 (3)	0.0191 (4)	0.0201 (4)	0.0006 (3)	0.0056 (3)	0.0024 (3)
C6	0.0203 (4)	0.0188 (4)	0.0230 (4)	0.0013 (3)	0.0075 (3)	−0.0009 (3)
C7	0.0178 (3)	0.0210 (4)	0.0217 (4)	−0.0014 (3)	0.0036 (3)	−0.0005 (3)
C8	0.0186 (3)	0.0227 (4)	0.0261 (4)	0.0004 (3)	0.0084 (3)	0.0039 (3)
C9	0.0186 (4)	0.0328 (5)	0.0353 (5)	−0.0031 (3)	0.0056 (3)	−0.0003 (4)

Geometric parameters (Å, º)

O1—C7	1.3765 (10)	C4—C5	1.3945 (12)
O1—C1	1.4370 (12)	C4—H4A	0.9500
O2—C2	1.3648 (10)	C5—C6	1.4157 (11)
O2—C1	1.4314 (12)	C5—C8	1.4862 (11)
O3—C8	1.2256 (11)	C6—C7	1.3676 (11)
C1—H1A	0.9900	C6—H6A	0.9500
C1—H1B	0.9900	C8—C9	1.5053 (12)
C2—C3	1.3679 (12)	C9—H9A	0.9800
C2—C7	1.3879 (12)	C9—H9B	0.9800
C3—C4	1.3985 (11)	C9—H9C	0.9800
C3—H3A	0.9500		
C7—O1—C1	105.37 (7)	C4—C5—C8	121.13 (7)
C2—O2—C1	105.72 (7)	C6—C5—C8	118.56 (7)
O2—C1—O1	107.93 (7)	C7—C6—C5	116.75 (8)
O2—C1—H1A	110.1	C7—C6—H6A	121.6
O1—C1—H1A	110.1	C5—C6—H6A	121.6

O2—C1—H1B	110.1	C6—C7—O1	128.28 (8)
O1—C1—H1B	110.1	C6—C7—C2	122.11 (8)
H1A—C1—H1B	108.4	O1—C7—C2	109.56 (7)
O2—C2—C3	127.26 (8)	O3—C8—C5	120.79 (8)
O2—C2—C7	110.18 (7)	O3—C8—C9	120.11 (8)
C3—C2—C7	122.51 (8)	C5—C8—C9	119.09 (8)
C2—C3—C4	116.31 (8)	C8—C9—H9A	109.5
C2—C3—H3A	121.8	C8—C9—H9B	109.5
C4—C3—H3A	121.8	H9A—C9—H9B	109.5
C5—C4—C3	121.99 (8)	C8—C9—H9C	109.5
C5—C4—H4A	119.0	H9A—C9—H9C	109.5
C3—C4—H4A	119.0	H9B—C9—H9C	109.5
C4—C5—C6	120.31 (7)		
C2—O2—C1—O1	−10.84 (10)	C5—C6—C7—C2	1.19 (13)
C7—O1—C1—O2	10.94 (10)	C1—O1—C7—C6	175.70 (9)
C1—O2—C2—C3	−175.94 (9)	C1—O1—C7—C2	−6.94 (10)
C1—O2—C2—C7	6.63 (10)	O2—C2—C7—C6	177.78 (8)
O2—C2—C3—C4	−178.36 (8)	C3—C2—C7—C6	0.21 (14)
C7—C2—C3—C4	−1.22 (13)	O2—C2—C7—O1	0.22 (10)
C2—C3—C4—C5	0.83 (13)	C3—C2—C7—O1	−177.35 (8)
C3—C4—C5—C6	0.55 (13)	C4—C5—C8—O3	179.58 (8)
C3—C4—C5—C8	−179.39 (8)	C6—C5—C8—O3	−0.36 (12)
C4—C5—C6—C7	−1.54 (12)	C4—C5—C8—C9	0.81 (12)
C8—C5—C6—C7	178.40 (7)	C6—C5—C8—C9	−179.12 (8)
C5—C6—C7—O1	178.26 (8)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C3—H3*A*···O3[i]	0.95	2.50	3.423 (1)	165

Symmetry code: (i) −*x*+1, *y*+1/2, −*z*+3/2.