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3-Ethylindan-1-one

Marcel Sonneck, Anke Spannenberg, Sebastian Wohlrab and Tim Peppel*

Leibniz-Institut für Katalyse e. V. an der Universität Rostock, Albert-Einstein-Strasse 29a, 18059 Rostock, Germany. *Correspondence e-mail: tim.peppel@catalysis.de

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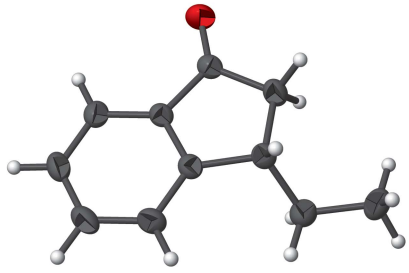
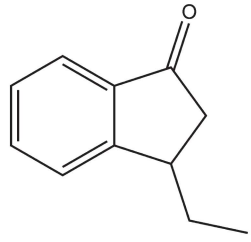
The title compound, $C_{11}H_{12}O$, has been prepared as a side product in the attempted room-temperature synthesis of (*E*)-1-phenylpent-2-en-1-one. The molecular structure consists of an approximately planar indanone core (r.m.s. deviation = 0.042 Å) with the ethyl group protruding from this plane. In the crystal, centrosymmetrically related molecules are linked into dimers by pairs of C—H···O hydrogen bonds, forming rings of $R_2^2(10)$ graph-set motif. The dimers are further connected by C—H···π interactions into chains running parallel to [$\bar{1}$01].

3D view



Chemical scheme



Structure description

In recent years, new Cu-based complexes suitable for the photocatalytic water-splitting reaction have attracted increasing attention due to their application in sustainable hydrogen-storage technologies (Chen *et al.*, 2017). As part of ongoing efforts to synthesize feasible new ligands for these Cu-based complexes (Sonneck *et al.*, 2015, 2016), the title compound was obtained as a side product in the attempted synthesis of the precursor compound (*E*)-1-phenylpent-2-en-1-one in moderate yield (30%).

The title compound 3-ethylindan-1-one is a racemic ring-closure product of (*E*)-1-phenylpent-2-en-1-one and the asymmetric unit consists of one indanone molecule (Fig. 1). The indanone ring system is nearly planar [r.m.s. deviation = 0.042 Å; maximum displacement 0.1082 (12) Å for atom C2] with the ethyl group protruding from this plane. All bond lengths and angles are in expected ranges and the C=O bond equals 1.2138 (13) Å. The structure exhibits a typical geometry that corresponds well with that of the parent structure 1-indanone (Morin *et al.*, 1974; Peña Ruiz *et al.*, 2004).

In the crystal structure, centrosymmetric molecules are linked into dimers through pairs of C—H···O hydrogen bonds (Table 1), forming rings of $R_2^2(10)$ graph-set motif. The dimers are further connected by C—H···π interactions, forming chains parallel to [$\bar{1}$01].

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

*Cg*1 is the centroid of the C4–C9 ring

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C8—H8···O1^{i}	0.95	2.52	3.3960 (15)	154
C10—H10*B*···*Cg*1^{ii}	0.99	2.96	3.7752 (13)	141

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

Synthesis and crystallization

The title compound was obtained as a racemic side product during an attempted room temperature synthesis of (*E*)-1-phenylpent-2-en-1-one in 30% yield (Ansell & Whitfield, 1968, 1971).

Dry $AlCl_3$ (41.67 g, 312.53 mmol, 1.5 eq.) was suspended in benzene (81.38 g, 1.04 mol, 5.0 eq.) in a 500 ml two-necked round-bottom flask at 0°C. (*E*)-Pent-2-enoyl chloride (24.70 g, 208.35 mmol, 1.0 eq.) was added to this suspension dropwise and the remaining solution was further stirred for seven days at 25°C. Afterwards, the solution was poured onto HCl/ice (150 g/50 g), the organic phase was separated and the aqueous phase was extracted with ethyl acetate until it was colorless. The combined organic phases were reduced to a total volume of 150 ml and extracted with brine, afterwards with portions of 10% NaOH solution (250 ml) and again with brine. The organic phase was dried over Na_2SO_4 and the solvent was removed under diminished pressure. The resulting crude product was distilled *in vacuo* to yield a slightly yellow liquid (10.0 g, 30%, m.p. 289 K). Single crystals were obtained from a distilled sample spontaneously at −30°C over one week.

Analytic data for 3-ethylindan-1-one: m.p. 16°C, b.p. 105°C (6 mbar), ^{1}H NMR (400 MHz, $CDCl_3$): δ (p.p.m.): 7.70–7.65 (*m*, 1H, ArH); 7.57–7.51 (*m*, 1H, ArH); 7.47–7.43 (*m*, 1H, ArH); 7.36–7.27 (*m*, 1H, ArH); 3.31–3.23 (*m*, 1H); 2.82–2.74 (*m*, 1H); 2.35–2.26 (*m*, 1H); 1.95–1.86 (*m*, 1H); 1.53–1.44 (*m*, 1H); 0.95–0.89 (*m*, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ (p.p.m.): 206.3 (CO), 158.6, 136.8 (C); 134.5, 127.4, 125.6, 123.4 (CH); 42.5 (CH_2), 39.6 (CH), 28.6 (CH_2); 11.6 (CH_3); MS (EI, 70 eV): *m*/*z* = 160 (M^+, 33), 133 (10), 132 (100), 131 (70), 115 (15), 103 (46), 77 (34), 51 (12); HRMS (ESI–TOF/MS): calculated for $C_{11}H_{12}O$ ($[M+H]^+$) 174.10392, found 174.10366; EA for $C_{11}H_{12}O$ % (calc.): C 82.57 (82.46); H 7.62 (7.55).

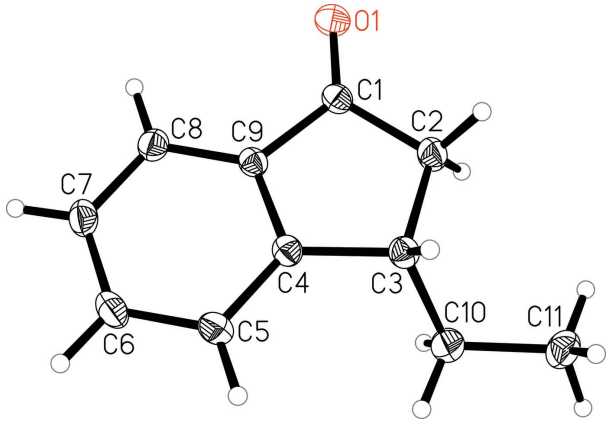


Figure 1
The molecular structure of the title compound, with displacement ellipsoids drawn at the 30% probability level.

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{11}H_{12}O$
M_r	160.21
Crystal system, space group	Monoclinic, $P2_1/c$
Temperature (K)	150
a, *b*, *c* (Å)	9.0852 (2), 6.4314 (2), 15.5196 (4)
β (°)	102.6361 (10)
V (Å^3)	884.85 (4)
Z	4
Radiation type	Mo *K*α
μ (mm^{-1})	0.08
Crystal size (mm)	0.51 × 0.45 × 0.29
Data collection	
Diffractometer	Bruker APEXII CCD
Absorption correction	Multi-scan (*SADABS*; Bruker, 2014)
T_{min}, T_{max}	0.92, 0.98
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	10725, 2134, 1793
R_{int}	0.019
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.660
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, *S*	0.039, 0.111, 1.07
No. of reflections	2134
No. of parameters	110
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.34, −0.16

Computer programs: *APEX2* (Bruker, 2014), *SAINT* (Bruker, 2013), *XP* in *SHELXTL* and *SHELXS97* (Sheldrick, 2008), *SHELXL2014* (Sheldrick, 2015) and *publCIF* (Westrip, 2010).

Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2.

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full crystallographic data

IUCrData (2017). **2**, x171685 [https://doi.org/10.1107/S2414314617016856]

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Crystal data

$C_{11}H_{12}O$
M_r = 160.21
Monoclinic, $P2_1/c$
a = 9.0852 (2) Å
b = 6.4314 (2) Å
c = 15.5196 (4) Å
β = 102.6361 (10)°
V = 884.85 (4) Å^3
Z = 4
$F(000)$ = 344
D_x = 1.203 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 5279 reflections
θ = 2.7–30.7°
μ = 0.08 mm^{-1}
T = 150 K
Prism, colourless
0.51 × 0.45 × 0.29 mm

Data collection

Bruker APEXII CCD diffractometer
Radiation source: fine-focus sealed tube
Detector resolution: 8.3333 pixels mm^{-1}
φ and ω scans
Absorption correction: multi-scan (SADABS; Bruker, 2014)
T_{min} = 0.92, T_{max} = 0.98
10725 measured reflections
2134 independent reflections
1793 reflections with $I > 2\sigma(I)$
R_{int} = 0.019
θ_{max} = 28.0°, θ_{min} = 2.3°
h = −11→11
k = −8→7
l = −20→20

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.039
$wR(F^2)$ = 0.111
S = 1.07
2134 reflections
110 parameters
0 restraints
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0531P)^2 + 0.2028P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.34 e Å^{-3}
$\Delta\rho_{min}$ = −0.16 e Å^{-3}

Special details

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. The H atoms were placed in idealized positions with d(C—H) = 0.95–1.00 Å (CH), 0.99 Å (CH_2), 0.98 Å (CH_3) and refined using a riding model, with U_{iso}(H) fixed at 1.2 U_{eq}(C) for CH, CH_2 or 1.5 U_{eq}(C) for CH_3. A rotating model was used for the methyl group.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
C1	0.16973 (11)	0.67197 (17)	0.07020 (7)	0.0286 (2)
C2	0.24409 (13)	0.48447 (18)	0.12097 (7)	0.0344 (3)
H2A	0.1796	0.4281	0.1592	0.041*
H2B	0.3429	0.5236	0.1586	0.041*
C3	0.26520 (12)	0.32138 (17)	0.05201 (7)	0.0288 (2)
H3	0.1920	0.2053	0.0520	0.035*
C4	0.22155 (11)	0.43841 (17)	−0.03458 (6)	0.0276 (2)
C5	0.22969 (13)	0.3713 (2)	−0.11876 (7)	0.0354 (3)
H5	0.2658	0.2360	−0.1276	0.042*
C6	0.18401 (13)	0.5057 (2)	−0.18932 (7)	0.0384 (3)
H6	0.1901	0.4619	−0.2469	0.046*
C7	0.12954 (12)	0.7031 (2)	−0.17778 (7)	0.0376 (3)
H7	0.0988	0.7921	−0.2273	0.045*
C8	0.11967 (12)	0.77122 (19)	−0.09466 (7)	0.0335 (3)
H8	0.0818	0.9056	−0.0861	0.040*
C9	0.16722 (11)	0.63589 (17)	−0.02383 (6)	0.0265 (2)
C10	0.42424 (13)	0.2319 (2)	0.06797 (8)	0.0404 (3)
H10A	0.4314	0.1386	0.0183	0.049*
H10B	0.4967	0.3471	0.0686	0.049*
C11	0.46888 (14)	0.1111 (2)	0.15406 (8)	0.0435 (3)
H11A	0.4648	0.2033	0.2038	0.065*
H11B	0.5716	0.0574	0.1604	0.065*
H11C	0.3989	−0.0051	0.1535	0.065*
O1	0.12284 (10)	0.82406 (14)	0.10197 (5)	0.0405 (2)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0289 (5)	0.0301 (6)	0.0270 (5)	−0.0029 (4)	0.0065 (4)	−0.0032 (4)
C2	0.0437 (6)	0.0338 (6)	0.0251 (5)	0.0023 (5)	0.0063 (4)	−0.0007 (4)
C3	0.0310 (5)	0.0280 (5)	0.0273 (5)	−0.0011 (4)	0.0063 (4)	−0.0002 (4)
C4	0.0261 (5)	0.0309 (6)	0.0258 (5)	−0.0017 (4)	0.0055 (4)	−0.0004 (4)
C5	0.0397 (6)	0.0376 (6)	0.0302 (5)	0.0015 (5)	0.0102 (4)	−0.0055 (4)
C6	0.0382 (6)	0.0539 (8)	0.0244 (5)	−0.0026 (5)	0.0095 (4)	−0.0019 (5)
C7	0.0339 (6)	0.0502 (8)	0.0288 (5)	0.0015 (5)	0.0074 (4)	0.0106 (5)
C8	0.0312 (5)	0.0352 (6)	0.0344 (5)	0.0024 (4)	0.0081 (4)	0.0056 (5)
C9	0.0245 (5)	0.0305 (6)	0.0249 (5)	−0.0020 (4)	0.0060 (4)	−0.0008 (4)
C10	0.0370 (6)	0.0472 (7)	0.0379 (6)	0.0082 (5)	0.0101 (5)	0.0056 (5)
C11	0.0379 (6)	0.0454 (7)	0.0451 (7)	0.0091 (5)	0.0043 (5)	0.0097 (5)
O1	0.0504 (5)	0.0356 (5)	0.0363 (4)	0.0062 (4)	0.0115 (4)	−0.0077 (3)

Geometric parameters (Å, º)

C1—O1	1.2138 (13)	C6—C7	1.3882 (18)
C1—C9	1.4730 (14)	C6—H6	0.9500

C1—C2	1.5161 (16)	C7—C8	1.3836 (17)
C2—C3	1.5402 (15)	C7—H7	0.9500
C2—H2A	0.9900	C8—C9	1.3945 (15)
C2—H2B	0.9900	C8—H8	0.9500
C3—C4	1.5155 (14)	C10—C11	1.5217 (16)
C3—C10	1.5244 (15)	C10—H10A	0.9900
C3—H3	1.0000	C10—H10B	0.9900
C4—C9	1.3858 (15)	C11—H11A	0.9800
C4—C5	1.3931 (14)	C11—H11B	0.9800
C5—C6	1.3859 (16)	C11—H11C	0.9800
C5—H5	0.9500		

O1—C1—C9	126.78 (10)	C7—C6—H6	119.3
O1—C1—C2	125.87 (9)	C8—C7—C6	120.49 (11)
C9—C1—C2	107.35 (9)	C8—C7—H7	119.8
C1—C2—C3	106.83 (8)	C6—C7—H7	119.8
C1—C2—H2A	110.4	C7—C8—C9	117.72 (11)
C3—C2—H2A	110.4	C7—C8—H8	121.1
C1—C2—H2B	110.4	C9—C8—H8	121.1
C3—C2—H2B	110.4	C4—C9—C8	122.29 (10)
H2A—C2—H2B	108.6	C4—C9—C1	109.57 (9)
C4—C3—C10	112.74 (9)	C8—C9—C1	128.14 (10)
C4—C3—C2	103.28 (8)	C11—C10—C3	113.36 (10)
C10—C3—C2	113.70 (9)	C11—C10—H10A	108.9
C4—C3—H3	109.0	C3—C10—H10A	108.9
C10—C3—H3	109.0	C11—C10—H10B	108.9
C2—C3—H3	109.0	C3—C10—H10B	108.9
C9—C4—C5	119.36 (10)	H10A—C10—H10B	107.7
C9—C4—C3	112.30 (9)	C10—C11—H11A	109.5
C5—C4—C3	128.34 (10)	C10—C11—H11B	109.5
C6—C5—C4	118.64 (11)	H11A—C11—H11B	109.5
C6—C5—H5	120.7	C10—C11—H11C	109.5
C4—C5—H5	120.7	H11A—C11—H11C	109.5
C5—C6—C7	121.49 (10)	H11B—C11—H11C	109.5
C5—C6—H6	119.3		

O1—C1—C2—C3	172.80 (10)	C5—C4—C9—C8	0.11 (15)
C9—C1—C2—C3	−7.95 (11)	C3—C4—C9—C8	−179.72 (9)
C1—C2—C3—C4	7.82 (11)	C5—C4—C9—C1	−179.79 (9)
C1—C2—C3—C10	130.32 (10)	C3—C4—C9—C1	0.38 (12)
C10—C3—C4—C9	−128.37 (10)	C7—C8—C9—C4	−0.64 (16)
C2—C3—C4—C9	−5.23 (11)	C7—C8—C9—C1	179.25 (10)
C10—C3—C4—C5	51.81 (15)	O1—C1—C9—C4	−175.94 (10)
C2—C3—C4—C5	174.95 (11)	C2—C1—C9—C4	4.82 (11)
C9—C4—C5—C6	0.52 (16)	O1—C1—C9—C8	4.16 (18)
C3—C4—C5—C6	−179.68 (10)	C2—C1—C9—C8	−175.08 (10)
C4—C5—C6—C7	−0.64 (17)	C4—C3—C10—C11	−179.14 (10)
C5—C6—C7—C8	0.11 (18)	C2—C3—C10—C11	63.73 (14)

C6—C7—C8—C9	0.52 (17)

Hydrogen-bond geometry (Å, º)

Cg1 is the centroid of the C4–C9 ring

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C8—H8···O1^{i}	0.95	2.52	3.3960 (15)	154
C10—H10*B*···*Cg*1^{ii}	0.99	2.96	3.7752 (13)	141

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