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(*E*)-2-(2-Fluorobenzylidene)cyclooctanone

Shen-Lin Huang, Yu-Lin Zhu and Yuan-Jiang Pan*

Department of Chemistry, Zhejiang University, Hangzhou 310027, People's Republic of China

Correspondence e-mail: cheyjpan@zju.edu.cn

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The title compound, $C_{15}H_{17}FO$, was synthesized directly from the condensation of cyclooctanone with 2-fluorobenzaldehyde, catalysed effectively by palladium in the presence of trimethylsilyl chloride (TMSCl). The structure contains a boat–chair eight-membered ring and a benzene ring. The packing of the molecules in the crystal structure is mainly due to C—H$\cdots\pi$ interactions and C—H$\cdots$O hydrogen bonds.

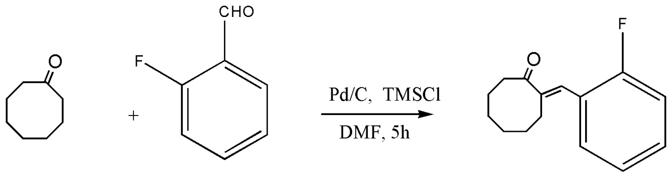
Key indicators

Single-crystal X-ray study
T = 287 K
Mean σ(C–C) = 0.003 Å
R factor = 0.032
wR factor = 0.073
Data-to-parameter ratio = 10.1

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Comment

The aldol condensation reaction, which is performed in the presence of strong acids or bases, is one of the most useful reactions in organic chemistry. Pd/TMSCl (TMSCl is trimethylsilyl chloride) was used as a catalytic system for an aldol condensation reaction, and the title compound, (I), was obtained in excellent yields (Zhou & Pan, 2004).



(I)

Fig. 1 shows the molecular structure of (I). It contains one eight-membered cyclooctanone ring which adopts a boat–chair conformation and a benzene ring. In general, the

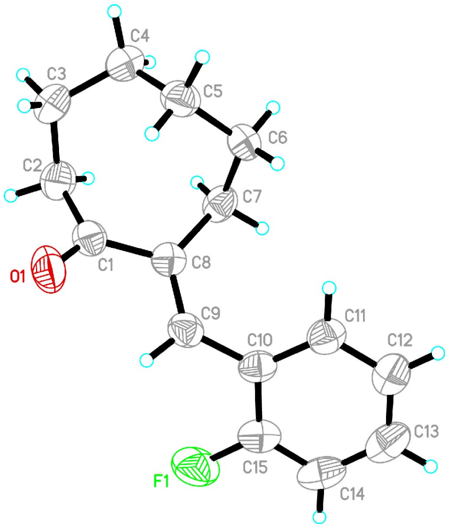


Figure 1
A view of the molecule of (I), with the atomic numbering scheme. Displacement ellipsoids are drawn at the 50% probability level and H atoms are shown as small spheres of arbitrary radii.

cyclooctanone ring adopts two types of conformations: (i) a crown conformation and (ii) a boat–chair conformation (Allinger & Greenberg, 1959). The boat–chair conformation is favourable for the cyclooctanone ring of (I), because of the substituent group containing a C=C double bond and a benzene ring.

The C1—C8—C9—C10 torsion angle of 179.00 (17)° describes the *E* configuration of the molecule about the C8=C9 bond. The O1—C1—C8—C9 torsion angle value of 29.4 (3)° and normal C1=O1 and C8=C9 lengths (Table 1) indicate the absence of conjugation between these double bonds. Also, the C8—C9—C10—C11 torsion angle of 41.5 (3)°, and the dihedral angle between the C8=C9—C10 plane and benzene ring of 40.5 (3)° show that the C8=C9 bond does not conjugate with the benzene ring either.

A network of intermolecular C—H···O interactions (Fig. 2), as well as C—H···π interactions, provide strong packing directives in the structure of (I). Benzene ring atoms C11 and C14 are hydrogen bonded to atom O1 of symmetry-related molecules at ($1 - x$, $1 - y$, $z - \frac{1}{2}$) and ($\frac{1}{2} + x$, $\frac{3}{2} - y$, z), respectively (Table 2). In addition, atom C4 is involved in two separate C—H···π interactions, with benzene ring of symmetry-related molecules at ($x - \frac{1}{2}$, $\frac{1}{2} - y$, z) and (x, $y - 1$, z) (Table 2).

Experimental

A solution of cyclooctanone (1 mmol), 2-fluorobenzaldehyde (1 mmol), and TMSCl (1.1 mmol) in DMF (1 ml) with 2 mol% of palladium was heated under 348 K for 5 h. A crystalline product precipitated directly after the whole reaction mixture was placed in a refrigerator overnight. It was isolated by filtration, washed with ethanol and dried, giving 81% yield. Single crystals suitable for X-ray structure analysis were obtained by slow evaporation of a DMF and methanol solution at room temperature.

Crystal data

$C_{15}H_{17}FO$
$M_r = 232.29$
Orthorhombic, $Pna2_1$
$a = 12.291$ (1) Å
$b = 8.294$ (1) Å
$c = 12.273$ (1) Å
$V = 1251.2$ (2) Å^3
$Z = 4$
$D_x = 1.233$ Mg m^{-3}

Mo $K\alpha$ radiation
Cell parameters from 39 reflections
$\theta = 3.0$–$14.6°$
$\mu = 0.09$ mm^{-1}
$T = 287$ (2) K
Block, colorless
0.50 × 0.50 × 0.46 mm

Data collection

Siemens *P*4 diffractometer
ω scans
Absorption correction: none
1904 measured reflections
1560 independent reflections
1207 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.009$

$\theta_{\max} = 27.9°$
$h = -1 \rightarrow 16$
$k = 0 \rightarrow 10$
$l = 0 \rightarrow 16$
3 standard reflections every 97 reflections intensity decay: 2.7%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.032$
$wR(F^2) = 0.073$
$S = 0.94$
1560 reflections
154 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0429P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.11$ e Å^{-3}
$\Delta\rho_{\min} = -0.10$ e Å^{-3}
Extinction correction: *SHELXL*97
Extinction coefficient: 0.033 (3)

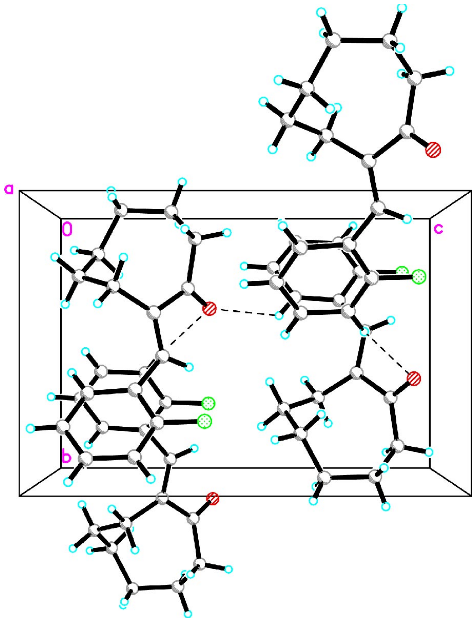


Figure 2
The crystal packing of (I), viewed down the *a* axis. Dashed lines indicate hydrogen bonds.

Table 1
Selected geometric parameters (Å, °).

F1—C15	1.357 (2)	C8—C9	1.339 (2)
O1—C1	1.219 (2)	C9—C10	1.472 (3)
C1—C8	1.491 (3)		
O1—C1—C8	120.46 (17)	O1—C1—C2	119.27 (19)
C8—C1—C2—C3	−99.3 (2)	C4—C5—C6—C7	−57.8 (2)
C1—C2—C3—C4	69.8 (3)	C5—C6—C7—C8	−53.3 (2)
C2—C3—C4—C5	−63.1 (3)	C2—C1—C8—C7	27.7 (2)
C3—C4—C5—C6	103.5 (2)	C6—C7—C8—C1	74.9 (2)

Table 2
Hydrogen-bonding and C—H···π geometry (Å, °).

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C11—H11···O1$^{\rm i}$	0.93	2.47	3.189 (3)	134
C14—H14···O1$^{\rm ii}$	0.93	2.48	3.408 (3)	173
C4—H4*A*···*CgP*$^{\rm iii}$	0.97	2.85	3.738 (3)	153
C4—H4*B*···*CgP*$^{\rm iv}$	0.97	2.90	3.829 (2)	160

Symmetry codes: (i) $1 - x, 1 - y, z - \frac{1}{2}$; (ii) $\frac{1}{2} + x, \frac{3}{2} - y, z$; (iii) $x - \frac{1}{2}, \frac{3}{2} - y, z$; (iv) $x, y - 1, z$. *CgP* denotes the centre of gravity of the benzene ring.

H atoms were placed in calculated positions (C—H = 0.93–0.97 Å) and refined as riding atoms with $U_{\rm iso}$(H) set equal to $1.2U_{\rm eq}$(carrier atom). Since the structure contains only light atoms, the intensities of Friedel opposites were not measured.

Data collection: *XSCANS* (Siemens, 1994); cell refinement: *XSCANS*; data reduction: *SHELXTL* (Siemens, 1991); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

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