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1-[2-(3,5-Difluorobenzoyloxy)phenyl]-ethanone

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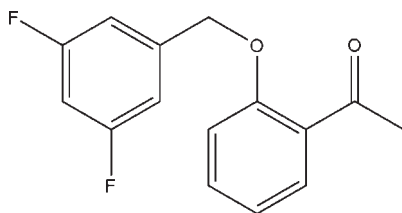
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Key indicators: single-crystal X-ray study; $T = 298$ K; mean $\sigma(\text{C}-\text{C}) = 0.004$ Å; R factor = 0.049; wR factor = 0.153; data-to-parameter ratio = 12.9.

In the title compound, $\text{C}_{15}\text{H}_{12}\text{F}_2\text{O}_2$, the dihedral angle between the aromatic rings is $70.43(4)^\circ$. The crystal packing exhibits no significantly short intermolecular contacts.

Related literature

For background to the Williamson reaction in organic synthesis, see: Dermer (1934). For a related structure, see: Ma *et al.* (2010).



Experimental

Crystal data

 $\text{C}_{15}\text{H}_{12}\text{F}_2\text{O}_2$ $M_r = 262.25$

Triclinic, $P\bar{1}$
 $a = 7.2808(7)$ Å
 $b = 7.9734(8)$ Å
 $c = 11.6466(12)$ Å
 $\alpha = 91.587(1)^\circ$
 $\beta = 106.559(2)^\circ$
 $\gamma = 95.343(1)^\circ$

$V = 644.22(11)$ Å³
 $Z = 2$
 Mo $K\alpha$ radiation
 $\mu = 0.11$ mm⁻¹
 $T = 298$ K
 $0.42 \times 0.38 \times 0.20$ mm

Data collection

Bruker SMART CD area-detector diffractometer
 Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
 $T_{\min} = 0.956$, $T_{\max} = 0.979$

3373 measured reflections
 2239 independent reflections
 1402 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.014$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.049$
 $wR(F^2) = 0.153$
 $S = 1.03$
 2239 reflections

173 parameters
 H-atom parameters constrained
 $\Delta\rho_{\max} = 0.21$ e Å⁻³
 $\Delta\rho_{\min} = -0.20$ e Å⁻³

Data collection: *SMART* (Bruker, 1996); cell refinement: *SAINTE* (Bruker, 1996); data reduction: *SAINTE*; 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*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: ZS2054).

References

- Bruker (1996). *SMART* and *SAINTE*. Bruker AXS Inc., Madison, Wisconsin, USA.
 Dermer, O. C. (1934). *Chem. Rev.* **14**, 385–430.
 Ma, Y.-T., Wang, J.-J., Liu, X.-W., Yang, S.-X. & Gao, J.-M. (2010). *Acta Cryst.* **E66**, o52.
 Sheldrick, G. M. (1996). *SADABS*. University of Göttingen, Germany.
 Sheldrick, G. M. (2008). *Acta Cryst.* **A64**, 112–122.

supplementary materials

Acta Cryst. (2010). E66, o2293 [doi:10.1107/S160053681003120X]

1-[2-(3,5-Difluorobenzoyloxy)phenyl]ethanone

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Comment

The Williamson reaction is a very useful transformation in organic synthesis since the products are of value in both industrial and academic applications. It usually involves the reaction of an alkali-metal salt of a hydroxy compound and an alkyl halide (Dermer, 1934). In the present paper, we present the structure of the title compound, C₁₅H₁₂F₂O₂ (I), which was synthesized by the reaction of 1-(2-hydroxyphenyl)ethanone, potassium carbonate and 3,5-difluorobenzyl bromide. We have previously reported the structure of a compound of this type (Ma *et al.*, 2010). In (I) (Fig. 1), the ethanone group is close to coplanar with the benzene ring [torsion angle C4–C3–C2–O1, 178.8 (2)°] while the dihedral angle between the aromatic rings is 70.43 (4)°. The crystal packing exhibits no significantly short intermolecular contacts.

Experimental

1-(2-Hydroxyphenyl)ethanone (4 mmol), potassium carbonate (8 mmol), 3,5-difluorobenzyl bromide (4 mmol), and 40 ml acetone were mixed in a 100 ml flask. After 3 h stirring at 331 K, the crude product was obtained. Crystals of (I) were obtained by recrystallization from *n*-hexane/ethyl acetate.

Refinement

The positions of all H atoms were determined geometrically and refined using a riding model with C–H = 0.93–0.97 Å and $U_{\text{iso}}(\text{methyl H}) = 1.5U_{\text{eq}}(\text{C})$ and $1.5U_{\text{eq}}$ for other H atoms.

Figures

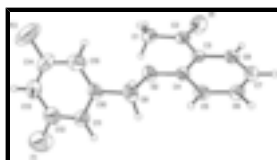


Fig. 1. The molecular structure of (I) with atom labels, and displacement ellipsoids drawn at the 30% probability level.

1-[2-(3,5-Difluorobenzoyloxy)phenyl]ethanone

Crystal data

C₁₅H₁₂F₂O₂

$M_r = 262.25$

Triclinic, *P*1

Hall symbol: -P 1

$a = 7.2808$ (7) Å

$b = 7.9734$ (8) Å

$Z = 2$

$F(000) = 272$

$D_x = 1.352$ Mg m⁻³

Melting point = 347–348 K

Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å

Cell parameters from 1136 reflections

supplementary materials

$c = 11.6466 (12) \text{ \AA}$	$\theta = 2.9\text{--}24.8^\circ$
$\alpha = 91.587 (1)^\circ$	$\mu = 0.11 \text{ mm}^{-1}$
$\beta = 106.559 (2)^\circ$	$T = 298 \text{ K}$
$\gamma = 95.343 (1)^\circ$	Plate, colorless
$V = 644.22 (11) \text{ \AA}^3$	$0.42 \times 0.38 \times 0.20 \text{ mm}$

Data collection

Bruker SMART CD area-detector diffractometer	2239 independent reflections
Radiation source: fine-focus sealed tube graphite	1402 reflections with $I > 2\sigma(I)$
φ and ω scans	$R_{\text{int}} = 0.014$
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	$\theta_{\text{max}} = 25.0^\circ$, $\theta_{\text{min}} = 1.8^\circ$
$T_{\text{min}} = 0.956$, $T_{\text{max}} = 0.979$	$h = -8 \rightarrow 8$
3373 measured reflections	$k = -9 \rightarrow 5$
	$l = -13 \rightarrow 13$

Refinement

Refinement on F^2	Primary atom site location: structure-invariant direct methods
Least-squares matrix: full	Secondary atom site location: difference Fourier map
$R[F^2 > 2\sigma(F^2)] = 0.049$	Hydrogen site location: inferred from neighbouring sites
$wR(F^2) = 0.153$	H-atom parameters constrained
$S = 1.03$	$w = 1/[\sigma^2(F_o^2) + (0.0702P)^2 + 0.1465P]$
2239 reflections	where $P = (F_o^2 + 2F_c^2)/3$
173 parameters	$(\Delta/\sigma)_{\text{max}} = 0.001$
0 restraints	$\Delta\rho_{\text{max}} = 0.21 \text{ e \AA}^{-3}$
	$\Delta\rho_{\text{min}} = -0.20 \text{ e \AA}^{-3}$

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	$U_{\text{iso}}^*/U_{\text{eq}}$
F1	0.1438 (3)	0.8789 (4)	0.0404 (2)	0.1646 (11)

F2	0.6758 (4)	0.6785 (4)	-0.0348 (2)	0.1880 (13)
O1	0.7550 (3)	0.2045 (2)	0.54230 (18)	0.0964 (7)
O2	0.7176 (2)	0.67975 (18)	0.41603 (13)	0.0629 (5)
C1	0.6631 (4)	0.3400 (3)	0.3629 (2)	0.0627 (7)
H1A	0.6321	0.2260	0.3292	0.094*
H1B	0.7598	0.3965	0.3321	0.094*
H1C	0.5495	0.3981	0.3416	0.094*
C2	0.7371 (3)	0.3388 (3)	0.4951 (2)	0.0564 (6)
C3	0.7914 (3)	0.4967 (3)	0.57422 (19)	0.0471 (5)
C4	0.7837 (3)	0.6620 (3)	0.53600 (19)	0.0481 (5)
C5	0.8396 (3)	0.7987 (3)	0.6196 (2)	0.0583 (6)
H5	0.8355	0.9080	0.5938	0.070*
C6	0.9010 (4)	0.7728 (3)	0.7398 (2)	0.0659 (7)
H6	0.9379	0.8650	0.7950	0.079*
C7	0.9086 (4)	0.6135 (3)	0.7795 (2)	0.0674 (7)
H7	0.9501	0.5966	0.8612	0.081*
C8	0.8540 (3)	0.4786 (3)	0.6971 (2)	0.0590 (6)
H8	0.8592	0.3703	0.7247	0.071*
C9	0.7050 (4)	0.8454 (3)	0.3733 (2)	0.0700 (7)
H9A	0.8327	0.9048	0.3882	0.084*
H9B	0.6320	0.9090	0.4138	0.084*
C10	0.6061 (4)	0.8257 (3)	0.2421 (2)	0.0587 (6)
C11	0.4210 (4)	0.8648 (3)	0.1996 (2)	0.0682 (7)
H11	0.3585	0.9069	0.2518	0.082*
C12	0.3289 (4)	0.8415 (4)	0.0801 (3)	0.0856 (9)
C13	0.4081 (5)	0.7794 (4)	-0.0002 (3)	0.0902 (9)
H13	0.3405	0.7632	-0.0811	0.108*
C14	0.5903 (6)	0.7418 (5)	0.0421 (3)	0.0977 (10)
C15	0.6927 (5)	0.7637 (4)	0.1624 (3)	0.0914 (9)
H15	0.8189	0.7365	0.1887	0.110*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
F1	0.0955 (15)	0.243 (3)	0.1260 (18)	0.0644 (17)	-0.0240 (13)	-0.0392 (17)
F2	0.187 (3)	0.284 (4)	0.1253 (19)	0.063 (2)	0.0883 (18)	-0.035 (2)
O1	0.163 (2)	0.0435 (11)	0.0898 (14)	0.0206 (11)	0.0447 (13)	0.0108 (10)
O2	0.0905 (12)	0.0383 (9)	0.0520 (10)	0.0087 (8)	0.0073 (8)	0.0027 (7)
C1	0.0713 (16)	0.0452 (13)	0.0708 (16)	0.0032 (11)	0.0215 (13)	-0.0080 (11)
C2	0.0622 (15)	0.0418 (13)	0.0717 (16)	0.0098 (10)	0.0285 (12)	0.0048 (11)
C3	0.0466 (12)	0.0453 (12)	0.0519 (13)	0.0099 (9)	0.0164 (10)	0.0051 (10)
C4	0.0481 (12)	0.0447 (12)	0.0507 (13)	0.0090 (9)	0.0117 (10)	0.0024 (10)
C5	0.0618 (15)	0.0463 (13)	0.0625 (15)	0.0097 (11)	0.0103 (12)	-0.0028 (11)
C6	0.0668 (16)	0.0672 (17)	0.0589 (16)	0.0124 (12)	0.0101 (12)	-0.0132 (12)
C7	0.0713 (17)	0.0807 (19)	0.0501 (14)	0.0160 (14)	0.0148 (12)	0.0049 (13)
C8	0.0598 (14)	0.0578 (15)	0.0646 (16)	0.0149 (11)	0.0227 (12)	0.0135 (12)
C9	0.0921 (19)	0.0431 (13)	0.0622 (15)	0.0051 (12)	0.0025 (13)	0.0081 (11)
C10	0.0715 (16)	0.0443 (13)	0.0597 (15)	0.0077 (11)	0.0166 (12)	0.0110 (11)

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C11	0.0706 (18)	0.0718 (17)	0.0618 (16)	0.0081 (13)	0.0189 (13)	-0.0016 (12)
C12	0.0740 (19)	0.099 (2)	0.075 (2)	0.0192 (16)	0.0052 (16)	-0.0020 (16)
C13	0.102 (2)	0.106 (2)	0.0591 (18)	0.0128 (19)	0.0171 (17)	0.0042 (16)
C14	0.117 (3)	0.117 (3)	0.076 (2)	0.022 (2)	0.052 (2)	-0.0050 (18)
C15	0.082 (2)	0.105 (2)	0.091 (2)	0.0324 (17)	0.0235 (17)	0.0040 (18)

Geometric parameters (Å, °)

F1—C12	1.359 (3)	C6—H6	0.9300
F2—C14	1.340 (4)	C7—C8	1.372 (3)
O1—C2	1.218 (3)	C7—H7	0.9300
O2—C4	1.358 (2)	C8—H8	0.9300
O2—C9	1.426 (3)	C9—C10	1.489 (3)
C1—C2	1.480 (3)	C9—H9A	0.9700
C1—H1A	0.9600	C9—H9B	0.9700
C1—H1B	0.9600	C10—C11	1.365 (3)
C1—H1C	0.9600	C10—C15	1.368 (4)
C2—C3	1.491 (3)	C11—C12	1.361 (4)
C3—C8	1.389 (3)	C11—H11	0.9300
C3—C4	1.404 (3)	C12—C13	1.336 (4)
C4—C5	1.390 (3)	C13—C14	1.342 (5)
C5—C6	1.371 (3)	C13—H13	0.9300
C5—H5	0.9300	C14—C15	1.384 (4)
C6—C7	1.365 (3)	C15—H15	0.9300
C4—O2—C9	118.86 (17)	C7—C8—H8	118.6
C2—C1—H1A	109.5	C3—C8—H8	118.6
C2—C1—H1B	109.5	O2—C9—C10	106.94 (18)
H1A—C1—H1B	109.5	O2—C9—H9A	110.3
C2—C1—H1C	109.5	C10—C9—H9A	110.3
H1A—C1—H1C	109.5	O2—C9—H9B	110.3
H1B—C1—H1C	109.5	C10—C9—H9B	110.3
O1—C2—C1	119.4 (2)	H9A—C9—H9B	108.6
O1—C2—C3	118.1 (2)	C11—C10—C15	118.4 (2)
C1—C2—C3	122.6 (2)	C11—C10—C9	119.5 (2)
C8—C3—C4	116.9 (2)	C15—C10—C9	122.0 (3)
C8—C3—C2	117.04 (19)	C12—C11—C10	119.4 (3)
C4—C3—C2	126.04 (19)	C12—C11—H11	120.3
O2—C4—C5	122.9 (2)	C10—C11—H11	120.3
O2—C4—C3	116.96 (18)	C13—C12—F1	118.0 (3)
C5—C4—C3	120.2 (2)	C13—C12—C11	123.8 (3)
C6—C5—C4	120.2 (2)	F1—C12—C11	118.2 (3)
C6—C5—H5	119.9	C12—C13—C14	116.5 (3)
C4—C5—H5	119.9	C12—C13—H13	121.7
C7—C6—C5	120.9 (2)	C14—C13—H13	121.7
C7—C6—H6	119.6	F2—C14—C13	118.8 (3)
C5—C6—H6	119.6	F2—C14—C15	118.6 (4)
C6—C7—C8	118.9 (2)	C13—C14—C15	122.6 (3)
C6—C7—H7	120.5	C10—C15—C14	119.2 (3)
C8—C7—H7	120.5	C10—C15—H15	120.4

C7—C8—C3	122.9 (2)	C14—C15—H15	120.4
O1—C2—C3—C8	-1.7 (3)	C2—C3—C8—C7	179.8 (2)
C1—C2—C3—C8	178.3 (2)	C4—O2—C9—C10	-173.0 (2)
O1—C2—C3—C4	178.8 (2)	O2—C9—C10—C11	107.6 (3)
C1—C2—C3—C4	-1.2 (3)	O2—C9—C10—C15	-70.1 (3)
C9—O2—C4—C5	0.1 (3)	C15—C10—C11—C12	-0.2 (4)
C9—O2—C4—C3	179.2 (2)	C9—C10—C11—C12	-177.9 (2)
C8—C3—C4—O2	-178.31 (19)	C10—C11—C12—C13	1.0 (5)
C2—C3—C4—O2	1.2 (3)	C10—C11—C12—F1	179.0 (3)
C8—C3—C4—C5	0.8 (3)	F1—C12—C13—C14	-179.2 (3)
C2—C3—C4—C5	-179.7 (2)	C11—C12—C13—C14	-1.1 (5)
O2—C4—C5—C6	178.5 (2)	C12—C13—C14—F2	179.7 (3)
C3—C4—C5—C6	-0.6 (3)	C12—C13—C14—C15	0.6 (5)
C4—C5—C6—C7	0.1 (4)	C11—C10—C15—C14	-0.3 (4)
C5—C6—C7—C8	0.1 (4)	C9—C10—C15—C14	177.3 (3)
C6—C7—C8—C3	0.2 (4)	F2—C14—C15—C10	-179.0 (3)
C4—C3—C8—C7	-0.6 (3)	C13—C14—C15—C10	0.1 (5)

Fig. 1

