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(2E)-3-(3-Bromo-4-methoxyphenyl)-1-(4-fluorophenyl)prop-2-en-1-oneGrzegorz Dutkiewicz,^a B. P. Siddaraju,^b H. S. Yathirajan,^b
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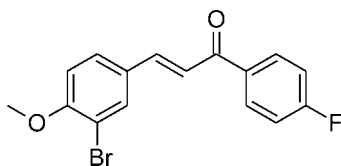
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Key indicators: single-crystal X-ray study; $T = 295$ K; mean $\sigma(\text{C}-\text{C}) = 0.005$ Å; R factor = 0.044; wR factor = 0.092; data-to-parameter ratio = 15.9.

In the title compound, $\text{C}_{16}\text{H}_{12}\text{BrFO}_2$, the dihedral angle between the aromatic rings is $23.75(12)^\circ$ and the dihedral angle between the prop-2-en-1-one fragment and the fluorobenzene ring is $20.9(2)^\circ$. In the crystal, only van der Waals interactions occur.

Related literature

For the normal probability plot test, see: Abrahams & Keve (1971); Cromer (1974). For the influence of the substituents on the geometry of the phenyl ring, see: Domenicano & Murray-Rust (1979); Domenicano (1988). For a closely related structure, see: Dutkiewicz *et al.* (2011).



Experimental

Crystal data

$\text{C}_{16}\text{H}_{12}\text{BrFO}_2$	$V = 1391.3(6) \text{ \AA}^3$
$M_r = 335.17$	$Z = 4$
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation
$a = 11.056(2) \text{ \AA}$	$\mu = 2.96 \text{ mm}^{-1}$
$b = 4.1110(15) \text{ \AA}$	$T = 295 \text{ K}$
$c = 30.825(5) \text{ \AA}$	$0.5 \times 0.4 \times 0.15 \text{ mm}$
$\beta = 96.76(2)^\circ$	

Data collection

Agilent Xcalibur Eos diffractometer	6983 measured reflections
Absorption correction: multi-scan (<i>CrysAlis PRO</i> ; Agilent, 2010)	2887 independent reflections
$T_{\min} = 0.507$, $T_{\max} = 1.000$	1918 reflections with $I > 2\sigma(I)$
	$R_{\text{int}} = 0.031$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.044$	182 parameters
$wR(F^2) = 0.092$	H-atom parameters constrained
$S = 1.02$	$\Delta\rho_{\text{max}} = 0.32 \text{ e \AA}^{-3}$
2887 reflections	$\Delta\rho_{\text{min}} = -0.52 \text{ e \AA}^{-3}$

Data collection: *CrysAlis PRO* (Agilent, 2010); cell refinement: *CrysAlis PRO*; data reduction: *CrysAlis PRO*; program(s) used to solve structure: *SIR92* (Altomare *et al.*, 1993); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXL97*.

BPS thanks the UOM for the research facilities.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: DN2670).

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supporting information

Acta Cryst. (2011). E67, o1090 [doi:10.1107/S1600536811012505]

(2E)-3-(3-Bromo-4-methoxyphenyl)-1-(4-fluorophenyl)prop-2-en-1-one

Grzegorz Dutkiewicz, B. P. Siddaraju, H. S. Yathirajan, B. Narayana and Maciej Kubicki

S1. Comment

As a part of our ongoing studies on chalcone derivatives (*e.g.* Dutkiewicz *et al.*, 2011) we have synthesized (2E)-3-(3-Bromo-4-methoxyphenyl)-1-(4-fluorophenyl)prop-2-en-1-one (**I**, Scheme 1).

The geometry of the molecule of **I** is very similar to that of previously reported (2E)-3-(3-Bromo-4-methoxyphenyl)-1-(4-methoxyphenyl)prop-2-en-1-one (Dutkiewicz *et al.*, 2011). The bond lengths and angles in both compounds are very similar; a majority of them differ by less than 3σ , and even the results of the normal probability plot test (Abrahams & Keve, 1971; International Tables for X-ray Crystallography, vol. IV (Cromer, 1974) confirm that the differences between the molecules are mainly of statistic nature. The correlation coefficient R^2 between the set of experimental differences between the geometrical parameters and the theoretical values for pure statistical distribution is 0.97 for the bond lengths (excluding C14—C141 and C14—F14 bonds, of course) and 0.94 - for angles. From the normal probability plot for bond angles it is obvious that the largest, and certainly not random, differences are observed within the phenyl ring with different substituents. This is consistent with the old observation of Domenicano and Murray-Rust (1979) that substituents to the phenyl ring influence much more intraannular bond angles than the bond lengths. The nature of substituents causes completely different bond angles pattern within the phenyl ring, in agreement with the values given by Domenicano (1988) which highlight quite opposite natures of methyl and fluorine groups.

More significant differences are observed at the level of torsion angles, that means that the overall conformations of both compounds differ. The shape of these molecules can be described by the dihedral angles between three planar fragments (*cf.* Fig. 1) 1-bromo-2-methoxyphenyl ring (A), the central prop-2-en-1-one chain (B), and the fluoro-phenyl ring (C). In both cases the dihedral angles between A and B planes are comparable, and in both cases the bridging chain is not significantly tilted out of the plane of the A ring, the orientations of rings C are really different: it is almost coplanar with the B - bridge for methyl derivative while it makes with the plane of prop-2-en-1-one group the significant dihedral angle of $20.9(2)^\circ$ in **I**. The comparison of both molecules, fitted onto the central C1O1C2C3 plane, is shown in Fig. 2.

In the structure of **I**, contrary to 1-(4-methoxyphenyl) derivative, where we observed quite a rich structure of weak interactions, there are virtually no specific interactions which might play a role in the designing of the crystal structure. Therefore only close packing requirements and van der Waals forces are involved in the crystal structure.

S2. Experimental

3-Bromo-4-methoxybenzaldehyde (2.15 g, 0.01 mol) was mixed with 1-(4-fluorophenyl)ethanone (1.38 g, 0.01 mol) and dissolved in ethanol (40 ml). To the solution, 4 ml of KOH (50%) was added. The reaction mixture was stirred for 6–10 h. The resulting crude solid was filtered, washed successively with distilled water and finally recrystallized from ethanol (95%) to give the pure chalcones. Crystals suitable for x-ray diffraction studies were grown by the slow evaporation from acetone solution (mp.: 140°C). Composition: Found (Calculated): C₁₆H₁₂BrFO₂:C: 57.26 (57.34); H: 3.57 (3.61)

S3. Refinement

The hydrogen were placed geometrically, in idealized positions, and refined as rigid groups with their $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$ with distances C—H = 0.93Å of the appropriate carrier atom ($U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{C})$ with distances C—H = 0.96Å for methyl H).

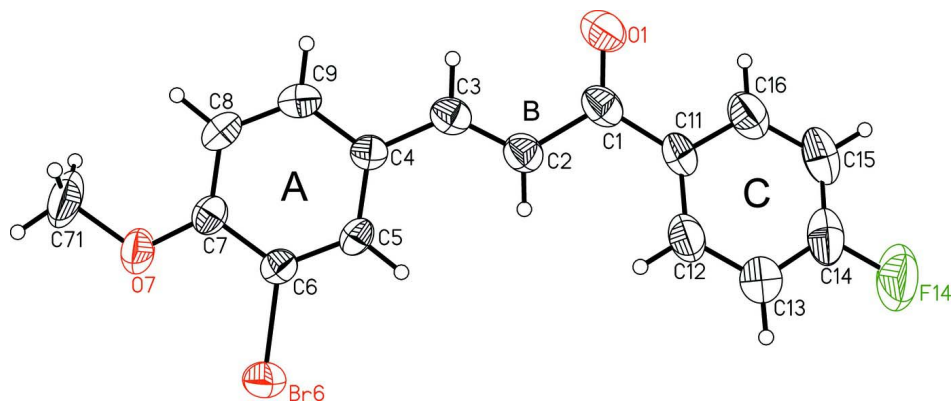


Figure 1

Anisotropic ellipsoid representation of the compound **I** together with atom labeling scheme. The ellipsoids are drawn at 50% probability level, hydrogen atoms are shown as spheres of arbitrary radii.

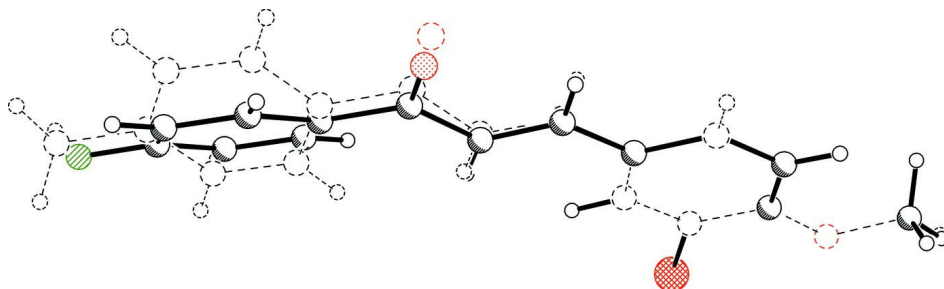


Figure 2

The comparison of the molecules of **I** (solid) and its 4-methoxy analogue (Dutkiewicz *et al.*, 2011; solid); the central prop-1-ene-3-one fragments were fitted onto one another (*SHELXTL* (Sheldrick, 2008)).

(2*E*)-3-(3-Bromo-4-methoxyphenyl)-1-(4-fluorophenyl)prop-2-en-1-one

Crystal data

$\text{C}_{16}\text{H}_{12}\text{BrFO}_2$

$M_r = 335.17$

Monoclinic, $P2_1/c$

Hall symbol: -P 2ybc

$a = 11.056 (2) \text{ \AA}$

$b = 4.1110 (15) \text{ \AA}$

$c = 30.825 (5) \text{ \AA}$

$\beta = 96.76 (2)^\circ$

$V = 1391.3 (6) \text{ \AA}^3$

$Z = 4$

$F(000) = 672$

$D_x = 1.600 \text{ Mg m}^{-3}$

Mo $K\alpha$ radiation, $\lambda = 0.71073 \text{ \AA}$

Cell parameters from 2417 reflections

$\theta = 3.1\text{--}28.0^\circ$

$\mu = 2.96 \text{ mm}^{-1}$

$T = 295 \text{ K}$

Prism, colourless

$0.5 \times 0.4 \times 0.15 \text{ mm}$

Data collection

Agilent Xcalibur Eos diffractometer	6983 measured reflections 2887 independent reflections
Radiation source: Enhance (Mo) X-ray Source	1918 reflections with $I > 2\sigma(I)$
Graphite monochromator	$R_{\text{int}} = 0.031$
Detector resolution: 16.1544 pixels mm^{-1}	$\theta_{\text{max}} = 28.0^\circ$, $\theta_{\text{min}} = 3.1^\circ$
ω scans	$h = -13 \rightarrow 14$
Absorption correction: multi-scan (<i>CrysAlis PRO</i> ; Agilent, 2010)	$k = -5 \rightarrow 5$
$T_{\text{min}} = 0.507$, $T_{\text{max}} = 1.000$	$l = -38 \rightarrow 39$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.044$	H-atom parameters constrained
$wR(F^2) = 0.092$	$w = 1/[\sigma^2(F_o^2) + (0.045P)^2]$
$S = 1.02$	where $P = (F_o^2 + 2F_c^2)/3$
2887 reflections	$(\Delta/\sigma)_{\text{max}} = 0.001$
182 parameters	$\Delta\rho_{\text{max}} = 0.32 \text{ e } \text{\AA}^{-3}$
0 restraints	$\Delta\rho_{\text{min}} = -0.52 \text{ e } \text{\AA}^{-3}$
Primary atom site location: structure-invariant direct methods	

Special details

Geometry. All s.u.'s (except the s.u. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell s.u.'s are taken into account individually in the estimation of s.u.'s in distances, angles and torsion angles; correlations between s.u.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell s.u.'s is used for estimating s.u.'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 $F^2 > 2\sigma(F^2)$ 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)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
C1	0.1287 (3)	0.1073 (8)	0.61784 (11)	0.0453 (9)
O1	0.0290 (2)	0.2293 (7)	0.60968 (8)	0.0649 (7)
C2	0.1926 (3)	0.1062 (8)	0.66297 (11)	0.0427 (8)
H2	0.2639	-0.0139	0.6689	0.051*
C3	0.1509 (3)	0.2716 (8)	0.69512 (11)	0.0397 (8)
H3	0.0814	0.3956	0.6873	0.048*
C4	0.1999 (3)	0.2836 (7)	0.74082 (10)	0.0328 (7)
C5	0.3100 (3)	0.1388 (7)	0.75684 (10)	0.0340 (7)
H5	0.3557	0.0294	0.7379	0.041*
C6	0.3511 (3)	0.1577 (7)	0.80038 (11)	0.0342 (7)
Br6	0.50370 (3)	-0.02669 (8)	0.820926 (12)	0.05081 (15)
C7	0.2842 (3)	0.3147 (7)	0.83016 (10)	0.0370 (8)
O7	0.3326 (2)	0.3123 (6)	0.87212 (7)	0.0539 (7)
C71	0.2665 (4)	0.4821 (10)	0.90272 (13)	0.0834 (15)
H71A	0.1882	0.3821	0.9033	0.125*
H71B	0.2558	0.7053	0.8939	0.125*

H71C	0.3114	0.4722	0.9313	0.125*
C8	0.1740 (3)	0.4534 (7)	0.81422 (12)	0.0434 (8)
H8	0.1263	0.5534	0.8333	0.052*
C9	0.1346 (3)	0.4439 (7)	0.77032 (12)	0.0414 (8)
H9	0.0622	0.5477	0.7600	0.050*
C11	0.1903 (3)	-0.0519 (8)	0.58326 (10)	0.0414 (8)
C12	0.3150 (3)	-0.1039 (9)	0.58687 (12)	0.0549 (10)
H12	0.3634	-0.0397	0.6121	0.066*
C13	0.3682 (4)	-0.2492 (10)	0.55362 (13)	0.0653 (11)
H13	0.4519	-0.2840	0.5562	0.078*
C14	0.2958 (4)	-0.3405 (10)	0.51699 (13)	0.0647 (11)
F14	0.3475 (3)	-0.4875 (6)	0.48435 (9)	0.1010 (9)
C15	0.1731 (4)	-0.2938 (10)	0.51172 (13)	0.0664 (11)
H15	0.1257	-0.3590	0.4863	0.080*
C16	0.1211 (3)	-0.1476 (10)	0.54502 (12)	0.0579 (10)
H16	0.0375	-0.1121	0.5418	0.069*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.044 (2)	0.050 (2)	0.040 (2)	-0.0048 (18)	-0.0020 (16)	0.0113 (17)
O1	0.0470 (15)	0.096 (2)	0.0495 (17)	0.0107 (15)	-0.0012 (12)	0.0108 (15)
C2	0.040 (2)	0.0474 (19)	0.040 (2)	-0.0022 (16)	0.0025 (16)	0.0054 (17)
C3	0.0342 (18)	0.0413 (19)	0.043 (2)	-0.0046 (15)	0.0030 (15)	0.0054 (17)
C4	0.0280 (16)	0.0331 (17)	0.0376 (19)	-0.0056 (14)	0.0059 (14)	0.0018 (15)
C5	0.0352 (18)	0.0330 (16)	0.0360 (19)	-0.0028 (15)	0.0135 (14)	-0.0030 (15)
C6	0.0311 (17)	0.0309 (16)	0.041 (2)	-0.0039 (14)	0.0074 (14)	-0.0027 (15)
Br6	0.0406 (2)	0.0546 (2)	0.0557 (2)	0.01012 (18)	-0.00083 (15)	-0.00365 (19)
C7	0.0423 (19)	0.0339 (17)	0.036 (2)	0.0000 (16)	0.0111 (15)	0.0006 (15)
O7	0.0669 (17)	0.0631 (16)	0.0318 (15)	0.0137 (13)	0.0064 (12)	-0.0071 (12)
C71	0.125 (4)	0.090 (3)	0.037 (2)	0.046 (3)	0.014 (2)	-0.013 (2)
C8	0.0422 (19)	0.041 (2)	0.050 (2)	0.0063 (17)	0.0174 (16)	-0.0053 (17)
C9	0.0318 (17)	0.0398 (18)	0.052 (2)	0.0035 (15)	0.0049 (15)	0.0017 (17)
C11	0.048 (2)	0.0445 (19)	0.0297 (18)	-0.0072 (17)	-0.0026 (15)	0.0058 (16)
C12	0.060 (2)	0.069 (2)	0.034 (2)	-0.004 (2)	-0.0011 (17)	-0.0014 (19)
C13	0.062 (3)	0.084 (3)	0.049 (3)	0.009 (2)	0.006 (2)	-0.003 (2)
C14	0.097 (4)	0.059 (2)	0.037 (2)	0.002 (3)	0.009 (2)	-0.003 (2)
F14	0.141 (2)	0.112 (2)	0.0519 (16)	0.0258 (17)	0.0209 (15)	-0.0175 (15)
C15	0.086 (3)	0.074 (3)	0.036 (2)	-0.015 (3)	-0.007 (2)	-0.004 (2)
C16	0.053 (2)	0.075 (3)	0.043 (2)	-0.011 (2)	-0.0043 (18)	0.006 (2)

Geometric parameters (Å, °)

C1—O1	1.210 (4)	C71—H71B	0.9600
C1—C11	1.483 (5)	C71—H71C	0.9600
C1—C2	1.484 (5)	C8—C9	1.372 (5)
C2—C3	1.328 (4)	C8—H8	0.9300
C2—H2	0.9300	C9—H9	0.9300

C3—C4	1.449 (4)	C11—C16	1.385 (5)
C3—H3	0.9300	C11—C12	1.387 (4)
C4—C9	1.391 (4)	C12—C13	1.377 (5)
C4—C5	1.393 (4)	C12—H12	0.9300
C5—C6	1.367 (4)	C13—C14	1.358 (5)
C5—H5	0.9300	C13—H13	0.9300
C6—C7	1.402 (4)	C14—F14	1.357 (4)
C6—Br6	1.890 (3)	C14—C15	1.361 (5)
C7—O7	1.340 (4)	C15—C16	1.372 (5)
C7—C8	1.383 (4)	C15—H15	0.9300
O7—C71	1.440 (4)	C16—H16	0.9300
C71—H71A	0.9600		
O1—C1—C11	121.2 (3)	H71A—C71—H71C	109.5
O1—C1—C2	121.1 (3)	H71B—C71—H71C	109.5
C11—C1—C2	117.7 (3)	C9—C8—C7	120.1 (3)
C3—C2—C1	122.0 (3)	C9—C8—H8	119.9
C3—C2—H2	119.0	C7—C8—H8	119.9
C1—C2—H2	119.0	C8—C9—C4	122.0 (3)
C2—C3—C4	128.3 (3)	C8—C9—H9	119.0
C2—C3—H3	115.8	C4—C9—H9	119.0
C4—C3—H3	115.8	C16—C11—C12	118.0 (3)
C9—C4—C5	118.0 (3)	C16—C11—C1	118.9 (3)
C9—C4—C3	119.2 (3)	C12—C11—C1	123.1 (3)
C5—C4—C3	122.8 (3)	C13—C12—C11	121.0 (3)
C6—C5—C4	119.9 (3)	C13—C12—H12	119.5
C6—C5—H5	120.0	C11—C12—H12	119.5
C4—C5—H5	120.0	C14—C13—C12	118.5 (4)
C5—C6—C7	122.0 (3)	C14—C13—H13	120.8
C5—C6—Br6	119.1 (2)	C12—C13—H13	120.8
C7—C6—Br6	118.9 (2)	F14—C14—C13	118.8 (4)
O7—C7—C8	125.5 (3)	F14—C14—C15	118.4 (4)
O7—C7—C6	116.6 (3)	C13—C14—C15	122.8 (4)
C8—C7—C6	117.9 (3)	C14—C15—C16	118.1 (4)
C7—O7—C71	117.0 (3)	C14—C15—H15	120.9
O7—C71—H71A	109.5	C16—C15—H15	120.9
O7—C71—H71B	109.5	C15—C16—C11	121.6 (4)
H71A—C71—H71B	109.5	C15—C16—H16	119.2
O7—C71—H71C	109.5	C11—C16—H16	119.2
O1—C1—C2—C3	-8.4 (5)	C7—C8—C9—C4	3.2 (5)
C11—C1—C2—C3	172.8 (3)	C5—C4—C9—C8	-2.0 (4)
C1—C2—C3—C4	177.2 (3)	C3—C4—C9—C8	177.6 (3)
C2—C3—C4—C9	-172.8 (3)	O1—C1—C11—C16	-19.2 (5)
C2—C3—C4—C5	6.7 (5)	C2—C1—C11—C16	159.6 (3)
C9—C4—C5—C6	-0.2 (4)	O1—C1—C11—C12	159.3 (3)
C3—C4—C5—C6	-179.7 (3)	C2—C1—C11—C12	-21.8 (5)
C4—C5—C6—C7	1.2 (4)	C16—C11—C12—C13	-0.6 (5)

C4—C5—C6—Br6	-177.6 (2)	C1—C11—C12—C13	-179.2 (3)
C5—C6—C7—O7	178.8 (3)	C11—C12—C13—C14	0.1 (6)
Br6—C6—C7—O7	-2.3 (4)	C12—C13—C14—F14	-179.2 (3)
C5—C6—C7—C8	-0.2 (5)	C12—C13—C14—C15	0.2 (6)
Br6—C6—C7—C8	178.7 (2)	F14—C14—C15—C16	179.4 (4)
C8—C7—O7—C71	-3.1 (5)	C13—C14—C15—C16	0.1 (6)
C6—C7—O7—C71	178.0 (3)	C14—C15—C16—C11	-0.6 (6)
O7—C7—C8—C9	179.2 (3)	C12—C11—C16—C15	0.9 (6)
C6—C7—C8—C9	-2.0 (5)	C1—C11—C16—C15	179.5 (3)
