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(*E*)-3-(3,4-Difluorophenyl)-1-(3,4-dimethoxyphenyl)prop-2-en-1-one

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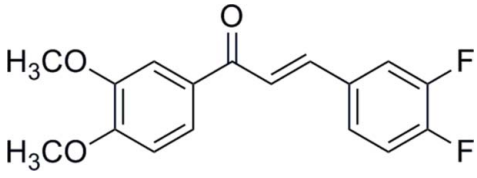
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.002 Å; disorder in main residue; R factor = 0.043; wR factor = 0.129; data-to-parameter ratio = 14.3.

In the title compound, $C_{17}H_{14}F_2O_3$, the dihedral angle between the benzene rings is 20.56 (8)° and the H atoms at the central propenone group are *trans* configured. One of the F atoms is disordered over two positions (occupancy ratio 0.57:0.43) and was refined using a split model. In the crystal, the molecules are linked into centrosymmetrical dimers and are further connected into a three-dimensional network *via* weak C—H···O interactions.

Related literature

For related structures, see: Peng *et al.*(2010); Wu *et al.* (2010, 2011, 2012*b*). For background to and applications of chalcones, see: Boumendjel *et al.* (2008); Kumar *et al.* (2011); Wu *et al.* (2011, 2012*a*); Zhang *et al.* (2011).



Experimental

Crystal data

$C_{17}H_{14}F_2O_3$
M_r = 304.28
Monoclinic, $P2_1/n$
a = 8.7444 (9) Å
b = 8.4832 (9) Å
c = 19.829 (2) Å
β = 94.053 (2)°
V = 1467.2 (3) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.11 mm^{-1}
T = 293 K
0.21 × 0.15 × 0.11 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2002)
T_{min} = 0.977, T_{max} = 0.988
8592 measured reflections
2876 independent reflections
2121 reflections with $I > 2\sigma(I)$
R_{int} = 0.028

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.043
$wR(F^2)$ = 0.129
S = 1.04
2876 reflections
201 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.19 e Å^{-3}
$\Delta\rho_{min}$ = −0.15 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
C1—H1···O1^{i}	0.93	2.44	3.321 (2)	159
C5—H5···O2^{ii}	0.93	2.60	3.2769 (15)	130
C5—H5···O3^{ii}	0.93	2.49	3.3950 (15)	164

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

Data collection: *SMART* (Bruker, 2002); cell refinement: *SAINT* (Bruker, 2002); data reduction: *SAINT*; 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: NC2310).

References

Boumendjel, A., Boccard, J., Carrupt, P. A., Nicolle, E., Blanc, M., Geze, A., Choisnard, L., Wouessidjewe, D., Matera, E. L. & Dumontet, C. (2008). *J. Med. Chem.* **51**, 2307–2310.
Bruker (2002). *SMART*, *SAINT* and *SADABS*. Bruker AXS Inc., Madison, Wisconsin, USA.
Kumar, V., Kumar, S., Hassan, M., Wu, H., Thimmulappa, R. K., Kumar, A., Sharma, S. K., Parmar, V. S., Biswal, S. & Malhotra, S. V. (2011). *J. Med. Chem.* **54**, 4147–4159.
Peng, J., Xu, H., Li, Z., Zhang, Y. & Wu, J. (2010). *Acta Cryst.* E**66**, o1156–o1157.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Wu, X., Cai, X., Zheng, X., Zhang, Z. & Ye, X. (2010). *Acta Cryst.* E**66**, o3015.
Wu, J. Z., Jiang, X., Zhao, C. G., Li, X. K. & Yang, S. L. (2012*b*). *Z. Kristallogr. New Cryst. Struct.* **227**, 215–216.
Wu, J. Z., Li, J. L., Cai, Y. P., Pan, Y., Ye, F. Q., Zhang, Y. L., Zhao, Y. J., Yang, S. L., Li, X. K. & Liang, G. (2011). *J. Med. Chem.* **54**, 8110–8123.
Wu, J. Z., Wang, C., Cai, Y. P., Peng, J., Liang, D. L., Zhao, Y. J., Yang, S. L., Li, X. K., Wu, X. P. & Liang, G. (2012*a*). *Med. Chem. Res.* **21**, 444–452.
Zhang, H. J., Qian, Y., Zhu, D. D., Yang, X. G. & Zhu, H. L. (2011). *Eur. J. Med. Chem.* **46**, 4702–4708.

supporting information

Acta Cryst. (2013). E**69**, o960 [doi:10.1107/S1600536813013767]

(*E*)-3-(3,4-Difluorophenyl)-1-(3,4-dimethoxyphenyl)prop-2-en-1-one

He-Ping Zhu, Peng-Tian Yu, Zhe Wang, Sheng-Li Yang and Zhi-Guo Liu

S1. Comment

The title compound is a biologically active derivative of chalcone. Chalcones constitute an important group of natural products and some of them possess a wide range of biological activities including anti-inflammatory and anti-tumor (Boumendjel *et al.*, 2008; Kumar *et al.*, 2011; Zhang *et al.*, 2011). As part of our ongoing studies on chalcones (Wu *et al.*, 2012*a*,*b*; 2011), the title compound was synthesized and its crystal structure is reported here.

In the crystal structure, the dihedral angle between the mean planes of the difluorophenyl and the dimethoxyphenyl rings amount to 20.56 (8)°. The H atoms of the central propenone group are *trans* configurated. The two methoxy groups attached to C13 and C14 are almost coplanar with the benzene ring.

S2. Experimental

1-(3,4-difluorophenyl) ethanone (0.01 mol) and 2,3-dimethoxybenzaldehyde (0.01 mol) were dissolved in methanol (50 ml). Sodium hydroxide (5 ml, 20%) was added drop wised to the solution, which was stirred at ambient temperature. The content of the flask were poured into ice-cold water, and the resulting crude solid was collected by filtration. The compound was purified by flash column and single crystals were obtained by slow evaporation from an ethanol/ dichloromethane solution (1:2, *v*/*v*) at 293 K.

S3. Refinement

The hydrogen atoms were positioned with idalized geometry and refined isotropic with $U_{iso}(H) = 1.2\ U_{eq}(C)$ (1.5 for methyl H atoms) using a riding model. One of the fluorine atoms was disordered over two positions and was refined using a split model with sof = 0.55 and 0.45.

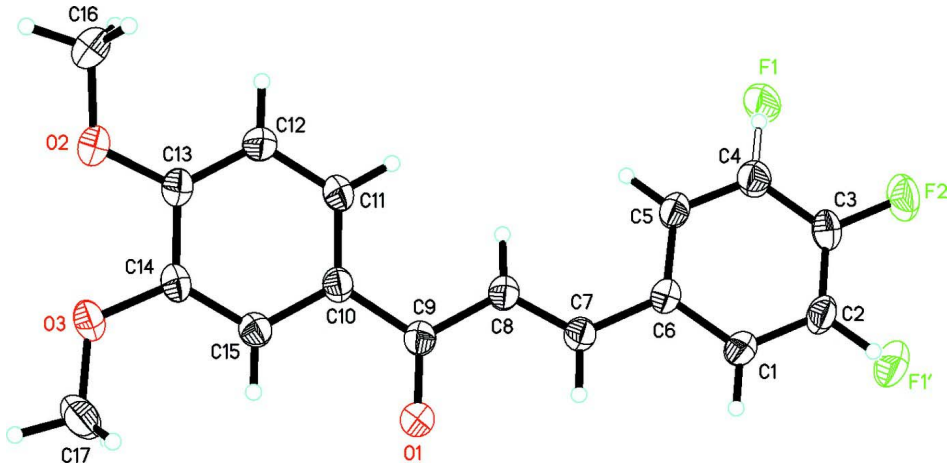

Figure 1

Crystal structure of the title compound with labeling and displacement ellipsoids drawn at the 30% probability level. Disordering is shown as full and open bonds.

(*E*)-3-(3,4-Difluorophenyl)-1-(3,4-dimethoxyphenyl)prop-2-en-1-one

Crystal data

$C_{17}H_{14}F_2O_3$
$M_r = 304.28$
Monoclinic, $P2_1/n$
$a = 8.7444$ (9) Å
$b = 8.4832$ (9) Å
$c = 19.829$ (2) Å
$\beta = 94.053$ (2)°
$V = 1467.2$ (3) Å^3
$Z = 4$
$F(000) = 632$
$D_x = 1.377$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 2015 reflections
$\theta = 5.0$–$54.2°$
$\mu = 0.11$ mm^{-1}
$T = 293$ K
Prismatic, colorless
0.21 × 0.15 × 0.11 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
phi and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2002)
$T_{min} = 0.977$, $T_{max} = 0.988$
8592 measured reflections
2876 independent reflections
2121 reflections with $I > 2\sigma(I)$
$R_{int} = 0.028$
$\theta_{max} = 26.0°$, $\theta_{min} = 2.6°$
$h = -10 \rightarrow 10$
$k = -10 \rightarrow 10$
$l = -24 \rightarrow 20$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.043$
$wR(F^2) = 0.129$
$S = 1.04$
2876 reflections
201 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
$w = 1/[\sigma^2(F_o^2) + (0.0703P)^2 + 0.091P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.19$ e Å^{-3}
$\Delta\rho_{min} = -0.15$ 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 $F^2 > \sigma(F^2)$ 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 (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}	Occ. (<1)
F1	−0.0102 (2)	−0.0304 (3)	0.14625 (12)	0.0862 (7)	0.57
F1′	−0.1205 (3)	0.2840 (4)	−0.03983 (14)	0.0844 (9)	0.43
F2	−0.21164 (11)	0.07757 (14)	0.05038 (6)	0.0743 (4)	
O1	0.67825 (13)	0.44604 (16)	0.07214 (6)	0.0643 (4)	
O2	1.05520 (12)	0.37085 (16)	0.35153 (5)	0.0569 (3)	
O3	1.13490 (13)	0.49922 (18)	0.24315 (6)	0.0711 (4)	
C1	0.12377 (18)	0.3023 (2)	0.01589 (8)	0.0512 (4)	
H1	0.1536	0.3772	−0.0148	0.061*	
C2	−0.02383 (9)	0.24437 (11)	0.01024 (4)	0.0546 (4)	
H2	−0.0955	0.2809	−0.0253	0.065*	0.57
C3	−0.06758 (9)	0.13470 (11)	0.05544 (4)	0.0520 (4)	
C4	0.03440 (9)	0.08176 (11)	0.10642 (4)	0.0557 (4)	
H4′	0.0024	0.0047	0.1379	0.067*	0.43
C5	0.18143 (9)	0.13853 (11)	0.11244 (4)	0.0520 (4)	
H5	0.2498	0.1023	0.1471	0.062*	
C6	0.22850 (16)	0.24987 (19)	0.06709 (8)	0.0431 (4)	
C7	0.38359 (17)	0.3175 (2)	0.07232 (8)	0.0457 (4)	
H7	0.4064	0.3889	0.0389	0.055*	
C8	0.49391 (17)	0.2877 (2)	0.11943 (8)	0.0505 (4)	
H8	0.4769	0.2139	0.1528	0.061*	
C9	0.64366 (17)	0.3684 (2)	0.12068 (8)	0.0474 (4)	
C10	0.74973 (16)	0.35850 (19)	0.18235 (8)	0.0441 (4)	
C11	0.71003 (19)	0.2904 (2)	0.24136 (9)	0.0588 (5)	
H11	0.6145	0.2429	0.2426	0.071*	
C12	0.80881 (19)	0.2909 (2)	0.29899 (9)	0.0587 (5)	
H12	0.7792	0.2439	0.3384	0.070*	
C13	0.95050 (17)	0.36056 (19)	0.29820 (8)	0.0459 (4)	
C14	0.99356 (16)	0.42987 (19)	0.23785 (8)	0.0458 (4)	
C15	0.89489 (17)	0.42856 (18)	0.18126 (8)	0.0449 (4)	
H15	0.9242	0.4746	0.1416	0.054*	
C16	1.0133 (2)	0.3151 (3)	0.41527 (9)	0.0700 (6)	
H16A	0.9196	0.3648	0.4263	0.105*	
H16B	1.0931	0.3399	0.4494	0.105*	
H16C	0.9990	0.2030	0.4132	0.105*	
C17	1.1804 (2)	0.5882 (3)	0.18825 (10)	0.0762 (6)	

H17A	1.1838	0.5214	0.1493	0.114*
H17B	1.2802	0.6320	0.1993	0.114*
H17C	1.1083	0.6719	0.1786	0.114*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
F1	0.0593 (11)	0.0901 (16)	0.1083 (16)	−0.0182 (10)	0.0008 (10)	0.0475 (13)
F1′	0.0602 (14)	0.111 (2)	0.0770 (17)	−0.0076 (14)	−0.0306 (13)	0.0194 (16)
F2	0.0443 (5)	0.0795 (8)	0.0971 (9)	−0.0160 (5)	−0.0097 (5)	0.0008 (6)
O1	0.0498 (7)	0.0841 (10)	0.0580 (7)	−0.0110 (6)	−0.0045 (6)	0.0218 (7)
O2	0.0488 (6)	0.0697 (8)	0.0502 (7)	−0.0076 (6)	−0.0095 (5)	−0.0012 (6)
O3	0.0497 (7)	0.1028 (11)	0.0602 (8)	−0.0332 (7)	−0.0014 (6)	0.0012 (7)
C1	0.0492 (9)	0.0576 (11)	0.0454 (9)	−0.0017 (8)	−0.0057 (7)	0.0055 (8)
C2	0.0453 (9)	0.0650 (12)	0.0510 (10)	0.0007 (8)	−0.0140 (8)	0.0014 (8)
C3	0.0369 (8)	0.0538 (10)	0.0644 (10)	−0.0046 (7)	−0.0044 (7)	−0.0089 (8)
C4	0.0490 (9)	0.0506 (11)	0.0668 (11)	−0.0037 (8)	−0.0002 (8)	0.0092 (8)
C5	0.0431 (9)	0.0564 (11)	0.0550 (10)	0.0024 (8)	−0.0076 (7)	0.0088 (8)
C6	0.0403 (8)	0.0445 (9)	0.0436 (8)	0.0022 (7)	−0.0032 (6)	−0.0026 (7)
C7	0.0432 (8)	0.0498 (10)	0.0438 (9)	−0.0001 (7)	0.0007 (7)	−0.0001 (7)
C8	0.0420 (8)	0.0550 (10)	0.0534 (10)	−0.0056 (7)	−0.0052 (7)	0.0080 (8)
C9	0.0404 (8)	0.0498 (10)	0.0514 (9)	−0.0002 (7)	−0.0006 (7)	0.0047 (8)
C10	0.0381 (8)	0.0437 (9)	0.0498 (9)	−0.0031 (7)	−0.0014 (7)	0.0007 (7)
C11	0.0442 (9)	0.0694 (12)	0.0616 (11)	−0.0195 (8)	−0.0055 (8)	0.0140 (9)
C12	0.0544 (10)	0.0688 (12)	0.0515 (10)	−0.0157 (9)	−0.0053 (8)	0.0157 (8)
C13	0.0422 (8)	0.0449 (9)	0.0496 (9)	−0.0010 (7)	−0.0050 (7)	−0.0035 (7)
C14	0.0364 (8)	0.0472 (10)	0.0533 (9)	−0.0065 (7)	0.0005 (7)	−0.0070 (7)
C15	0.0430 (8)	0.0454 (9)	0.0465 (9)	−0.0033 (7)	0.0041 (7)	−0.0017 (7)
C16	0.0654 (11)	0.0901 (15)	0.0523 (11)	−0.0033 (10)	−0.0120 (9)	0.0085 (10)
C17	0.0639 (12)	0.0933 (16)	0.0735 (13)	−0.0337 (11)	0.0201 (10)	−0.0104 (11)

Geometric parameters (Å, º)

F1—C4	1.313 (2)	C7—H7	0.9300
F1′—C2	1.302 (2)	C8—C9	1.476 (2)
F2—C3	1.3468 (12)	C8—H8	0.9300
O1—C9	1.2220 (19)	C9—C10	1.484 (2)
O2—C13	1.3516 (17)	C10—C11	1.371 (2)
O2—C16	1.421 (2)	C10—C15	1.403 (2)
O3—C14	1.3662 (18)	C11—C12	1.383 (2)
O3—C17	1.405 (2)	C11—H11	0.9300
C1—C2	1.3784 (18)	C12—C13	1.374 (2)
C1—C6	1.391 (2)	C12—H12	0.9300
C1—H1	0.9300	C13—C14	1.408 (2)
C2—C3	1.3650	C14—C15	1.366 (2)
C2—H2	0.9600	C15—H15	0.9300
C3—C4	1.3753	C16—H16A	0.9600
C4—C5	1.3703	C16—H16B	0.9600

C4—H4′	0.9600	C16—H16C	0.9600
C5—C6	1.3866 (18)	C17—H17A	0.9600
C5—H5	0.9300	C17—H17B	0.9600
C6—C7	1.469 (2)	C17—H17C	0.9600
C7—C8	1.319 (2)		

C13—O2—C16	118.12 (13)	C8—C9—C10	119.32 (14)
C14—O3—C17	118.42 (14)	C11—C10—C15	118.39 (14)
C2—C1—C6	120.61 (15)	C11—C10—C9	123.07 (14)
C2—C1—H1	119.7	C15—C10—C9	118.47 (14)
C6—C1—H1	119.7	C10—C11—C12	121.53 (15)
F1′—C2—C3	118.67 (13)	C10—C11—H11	119.2
F1′—C2—C1	121.57 (16)	C12—C11—H11	119.2
C3—C2—C1	119.62 (8)	C13—C12—C11	120.20 (16)
C3—C2—H2	120.2	C13—C12—H12	119.9
C1—C2—H2	120.2	C11—C12—H12	119.9
F2—C3—C2	120.03 (6)	O2—C13—C12	125.48 (15)
F2—C3—C4	119.45 (6)	O2—C13—C14	115.52 (13)
C2—C3—C4	120.5	C12—C13—C14	118.99 (14)
F1—C4—C5	121.27 (9)	O3—C14—C15	125.60 (15)
F1—C4—C3	118.29 (9)	O3—C14—C13	114.15 (13)
C5—C4—C3	120.3	C15—C14—C13	120.22 (13)
C5—C4—H4′	119.8	C14—C15—C10	120.68 (15)
C3—C4—H4′	119.8	C14—C15—H15	119.7
C4—C5—C6	120.17 (6)	C10—C15—H15	119.7
C4—C5—H5	119.9	O2—C16—H16A	109.5
C6—C5—H5	119.9	O2—C16—H16B	109.5
C5—C6—C1	118.75 (13)	H16A—C16—H16B	109.5
C5—C6—C7	122.34 (12)	O2—C16—H16C	109.5
C1—C6—C7	118.89 (14)	H16A—C16—H16C	109.5
C8—C7—C6	126.89 (15)	H16B—C16—H16C	109.5
C8—C7—H7	116.6	O3—C17—H17A	109.5
C6—C7—H7	116.6	O3—C17—H17B	109.5
C7—C8—C9	121.78 (15)	H17A—C17—H17B	109.5
C7—C8—H8	119.1	O3—C17—H17C	109.5
C9—C8—H8	119.1	H17A—C17—H17C	109.5
O1—C9—C8	120.35 (14)	H17B—C17—H17C	109.5
O1—C9—C10	120.29 (14)		

C6—C1—C2—F1′	−175.4 (2)	O1—C9—C10—C11	171.47 (18)
C6—C1—C2—C3	0.14 (19)	C8—C9—C10—C11	−6.5 (3)
F1′—C2—C3—F2	−4.45 (17)	O1—C9—C10—C15	−5.4 (2)
C1—C2—C3—F2	179.90 (13)	C8—C9—C10—C15	176.53 (15)
F1′—C2—C3—C4	175.63 (17)	C15—C10—C11—C12	0.5 (3)
C1—C2—C3—C4	−0.03 (9)	C9—C10—C11—C12	−176.42 (17)
F2—C3—C4—F1	3.98 (15)	C10—C11—C12—C13	0.0 (3)
C2—C3—C4—F1	−176.10 (14)	C16—O2—C13—C12	−4.5 (3)
F2—C3—C4—C5	−179.96 (7)	C16—O2—C13—C14	174.74 (16)

C2—C3—C4—C5	0.0	C11—C12—C13—O2	178.70 (16)
F1—C4—C5—C6	175.92 (18)	C11—C12—C13—C14	−0.5 (3)
C3—C4—C5—C6	−0.02 (8)	C17—O3—C14—C15	5.4 (3)
C4—C5—C6—C1	0.13 (17)	C17—O3—C14—C13	−172.30 (16)
C4—C5—C6—C7	178.41 (10)	O2—C13—C14—O3	−0.9 (2)
C2—C1—C6—C5	−0.2 (2)	C12—C13—C14—O3	178.39 (16)
C2—C1—C6—C7	−178.53 (13)	O2—C13—C14—C15	−178.80 (14)
C5—C6—C7—C8	−2.0 (3)	C12—C13—C14—C15	0.5 (2)
C1—C6—C7—C8	176.24 (16)	O3—C14—C15—C10	−177.60 (16)
C6—C7—C8—C9	−177.52 (15)	C13—C14—C15—C10	0.0 (2)
C7—C8—C9—O1	−11.8 (3)	C11—C10—C15—C14	−0.5 (2)
C7—C8—C9—C10	166.18 (16)	C9—C10—C15—C14	176.55 (15)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C1—H1···O1^{i}	0.93	2.44	3.321 (2)	159
C5—H5···O2^{ii}	0.93	2.60	3.2769 (15)	130
C5—H5···O3^{ii}	0.93	2.49	3.3950 (15)	164

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