Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

Second monoclinic form of (*E*)-3-(4-fluorophenyl)-1-phenylprop-2-en-1-one

Saira N. Arias-Ruiz,[a] Nancy Romero,[a] Carlos E. Lobato-García,[a] Abraham Gómez-Rivera[a] and Angel Mendoza[b]*

[a]División Académica de Ciencias Básicas, Universidad Juárez Autónoma de Tabasco, AP 24, 86690 Cunduacán, Tab., Mexico, and [b]Centro de Química, Instituto de Ciencias, Benemérita Universidad Autónoma de Puebla, 72570 Puebla, Pue., Mexico
Correspondence e-mail: angel.mendoza@correo.buap.mx

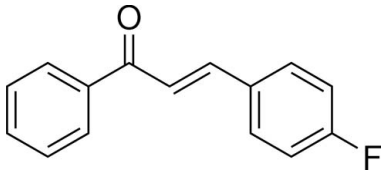
Received 11 September 2013; accepted 12 October 2013

Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.002 Å; R factor = 0.045; wR factor = 0.131; data-to-parameter ratio = 14.7.

The unit-cell dimensions and space group of the second monoclinic polymorph of the title compound, $C_{15}H_{11}FO$, differ from those of the previously reported form [Jing (2009). *Acta Cryst.* E**65**, o2515]. The title compound shows an *E* conformation of the C=C bond with the 4-fluorophenyl group opposite to the benzoyl group. The torsion angle of between the planes of the 4-fluorophenyl and benzoyl groups is 10.53 (6)°. In the crystal, weak C—H···O and C—H···F interactions form a cross-linked packing motif, building sheets parallel to ($\bar{1}$02).

Related literature

For the first monoclinic polymorph of the title compound, see: Jing (2009). For related crystal structures, see: Li *et al.* (1992); Li & Su (1994); For biological properties reports of chalcones, see: Foresti *et al.* (2005); Nowakowska (2007); Kouskoura *et al.* (2008); Zhang *et al.* (2010); Doan & Tran (2011). For solvent-free synthesis of chalcones, see: Srivastava (2008); Krishnakumar & Swaminathan (2011); Thirunarayanan *et al.* (2012). For applications of chalcones in organic synthesis, see: Prakash *et al.* (2009); Bandgar *et al.* (2009).



Experimental

Crystal data

$C_{15}H_{11}FO$
M_r = 226.24
Monoclinic, $P2_1/c$
a = 8.6925 (4) Å
b = 5.9266 (2) Å
c = 22.6456 (9) Å
β = 95.423 (4)°
V = 1161.41 (8) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.09 mm^{-1}
T = 293 K
0.59 × 0.15 × 0.07 mm

Data collection

Oxford Diffraction Xcalibur (Atlas, Gemini) diffractometer
Absorption correction: analytical (*CrysAlis PRO*; Oxford Diffraction, 2009)
T_{min} = 0.993, T_{max} = 0.999
22101 measured reflections
2276 independent reflections
1471 reflections with $I > 2\sigma(I)$
R_{int} = 0.044

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.045
$wR(F^2)$ = 0.131
S = 1.01
2276 reflections
155 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.11 e Å^{-3}
$\Delta\rho_{min}$ = −0.11 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
C5—H5···O1^{i}	0.93	2.49	3.244 (2)	138
C13—H13···F1^{ii}	0.93	2.68	3.465 (2)	142

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

Data collection: *CrysAlis PRO* (Oxford Diffraction, 2009); cell refinement: *CrysAlis PRO*; data reduction: *CrysAlis PRO*; program(s) used to solve structure: *SHELXS2013* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL2013* (Sheldrick, 2008); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 2012); software used to prepare material for publication: *WinGX* (Farrugia, 2012).

The authors wish to acknowledge CONACyT–Gobierno del Estado Tabasco and the Universidad Juárez Autónoma de Tabasco for financial support *via* projects TAB-2009-C18–122141 and UJAT-2011-C07–22, respectively.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BH2484).

References

Bandgar, B. P., Gawande, S. S., Bodade, R. G., Gawande, N. M. & Khobragade, C. N. (2009). *Bioorg. Med. Chem.* **17**, 8168–8173.
Doan, T. N. & Tran, D. T. (2011). *Pharmacol. Pharmacy*, **2**, 282–288.
Farrugia, L. J. (2012). *J. Appl. Cryst.* **45**, 849–854.
Foresti, R., Hoque, M., Monti, D., Green, C. J. & Motterlini, R. J. (2005). *J. Pharmacol. Exp. Ther.* **312**, 686–693.
Jing, L.-H. (2009). *Acta Cryst.* E**65**, o2515.
Kouskoura, M., Hadjipavlou-Litina, D. & Giakoumakou, M. (2008). *Med. Chem.* **4**, 586–596.
Krishnakumar, B. & Swaminathan, M. (2011). *J. Mol. Catal. A Chem.* **350**, 16–25.
Li, Z., Pa, F. & Su, G. (1992). *Acta Cryst.* C**48**, 712–714.
Li, Z. & Su, G. (1994). *Acta Cryst.* C**50**, 126–127.
Nowakowska, Z. (2007). *Eur. J. Med. Chem.* **42**, 125–137.
Oxford Diffraction (2009). *CrysAlis PRO*. Oxford Diffraction Ltd, Yarnton, England.
Prakash, O., Kumar, R. & Sehrawat, R. (2009). *Eur. J. Med. Chem.* **44**, 1763–1767.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Srivastava, Y. K. (2008). *Rasayan J. Chem.* **1**, 884–886.

Thirunarayanan, G., Mayavel, P. & Thirumurthy, K. (2012). *Spectrochim. Acta Part A*, **91**, 18–22.

Zhang, X.-W., Zhao, D.-H., Quan, Y.-C., Sun, L.-P., Yin, X.-M. & Guan, L.-P. (2010). *Med. Chem. Res.* **19**, 403–412.

supplementary materials

Acta Cryst. (2013). E**69**, o1694–o1695 [doi:10.1107/S1600536813028079]

Second monoclinic form of (*E*)-3-(4-fluorophenyl)-1-phenylprop-2-en-1-one

Saira N. Arias-Ruiz, Nancy Romero, Carlos E. Lobato-García, Abraham Gómez-Rivera and Angel Mendoza

1. Comment

Chalcones, like the title compound, are highly interesting materials due to their antioxidant, antibacterial, antifungal, antitumor and anti-inflammatory properties (Zhang *et al.*, 2010; Kouskoura *et al.*, 2008; Nowakowska, 2007; Foresti *et al.*, 2005 and Doan & Tran, 2011). The title compound was obtained by a green Claisen-Schimdt condensation of 4-fluorobenzaldehyde with acetophenone. This reaction was carried out by a free solvent and microwave assisted method, with *p*-toluenesulfonic acid as catalyst (Thirunarayanan *et al.*, 2012; Krishnakumar & Swaminathan, 2011; Srivastava, 2008). Also, chalcones are valuable intermediates in organic synthesis because of the *α*,*β*-unsaturated carbonyl system, which is considered as a key building block (Prakash *et al.*, 2009; Bandgar *et al.*, 2009).

The crystal structure of the title compound, $C_{15}H_{11}FO$, has been reported previously at 93 K (Jing, 2009). However, here we report the structure of a second monoclinic polymorph, which was elucidated at 293 (2) K. The new polymorph crystallizes in space group $P2_1/c$, which is different from the previous space group, *Cc*. The cell parameters of the current monoclinic polymorph vary significantly from the earlier form [*a* = 24.926 (9), *b* = 5.6940 (19), *c* = 7.749 (3) Å and *β* = 94.747 (5)°]. Furthermore, two more halo-chalcones were reported previously: 4-bromochalcone [Li *et al.*, 1992; unit-cell parameters: *a* = 29.027 (7), *b* = 7.26 (2), *c* = 5.917 (3) Å and *β* = 101.38 (3)°] and 4-chlorochalcone [Li & Su, 1994; unit-cell parameters: *a* = 8.211 (2), *b* = 5.869 (2), *c* = 25.291 (5) Å and *β* = 99.18 °]. Both compounds show similar cell parameters to current polymorphs of the F-chalcone, however the space group is $P2_1/c$ for the Cl derivative and *Cc* for the Br-chalcone, both characterized at room temperature.

The title compound shows a configuration *E* on the C═C bond with *p*-fluorophenyl group opposite to the 1-phenyl-ketone. Torsion angle of *p*-fluorophenyl to 1-phenylketone group is 10.53 (6)°. The crystal packing presents two intermolecular interactions of the type hydrogen bond (Table 1), C5—H5···O1 and C13—H13···F1 with the symmetry codes (i) -*x* + 1, -*y*, -*z* and (ii) *x* + 1, -*y* + 3/2, *z* + 1/2, respectively. These interactions form a cross-linked crystal packing, building sheets parallel to the $(\bar{1}02)$ plane.

2. Experimental

To a mixture of acetophenone (1.15 mmol) and 4-fluorobenzaldehyde (1.15 mmol) in dry media, 0.1 g of *p*-TsOH was added and the mixture was irradiated in a microwave oven at 480 W for 10 min. Completion of the reaction was tested by thin layer chromatography (TLC), and the crude product was purified by column chromatography on silica gel (eluent: hexane), to afford a yellow solid, (*E*)-3-(4-fluorophenyl)-1-phenylprop-2-en-1-one, in 85.4% yield, m. p. 79 °C. The pure product was recrystallized from hexane. The microwave oven VICHI: MW-600 (600 W) MOD: MICV/Vichy/F-814 was used; all the chemicals were purchased from Sigma-Aldrich and were used without further preparation. Spectroscopic analysis: ν_{max}/cm^{-1} (neat KBr)= 3438, 1661, 1604, 1588, 1509, 1217, 1016, 829, 772. ^{1}H-NMR (400 MHz, $CDCl_3$): *δ* (p.p.m.) = 7.09 (tt, *J*= 2.0, 8.4 Hz, 2H), 7.46 (d, *J*=16 Hz, 1H), 7.49 (dd, *J*= 6.4, 8.4 Hz, 2H), 7.56 (dt, *J*=1.2, 6.4 Hz, 1H),

7.61 (ddt, *J*=2.0, 5.2, 8.4 Hz, 2H), 7.76 (d, *J*=16 Hz, 1H), 8.01 (dd, *J*=1.2, 8.4 Hz, 2H). ^{13}C-NMR (101 MHz, $CDCl_3$): δ (p.p.m.) = 115.91, 116.13, 121.58, 128.38 (2 C), 128.56 (2 C), 130.22, 130.31, 132.77, 137.98, 143.38, 162.69, 165.19, 190.15.

3. Refinement

H atoms linked to C atoms were placed in idealized positions and refined as riding on their parent atoms, with C—H = 0.93 Å and with U_{iso}(H) = 1.2 U_{eq}(carrier C).

Computing details

Data collection: *CrysAlis PRO* (Oxford Diffraction, 2009); cell refinement: *CrysAlis PRO* (Oxford Diffraction, 2009); data reduction: *CrysAlis PRO* (Oxford Diffraction, 2009); program(s) used to solve structure: *SHELXS2013* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL2013* (Sheldrick, 2008); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 2012); software used to prepare material for publication: *WinGX* (Farrugia, 2012).

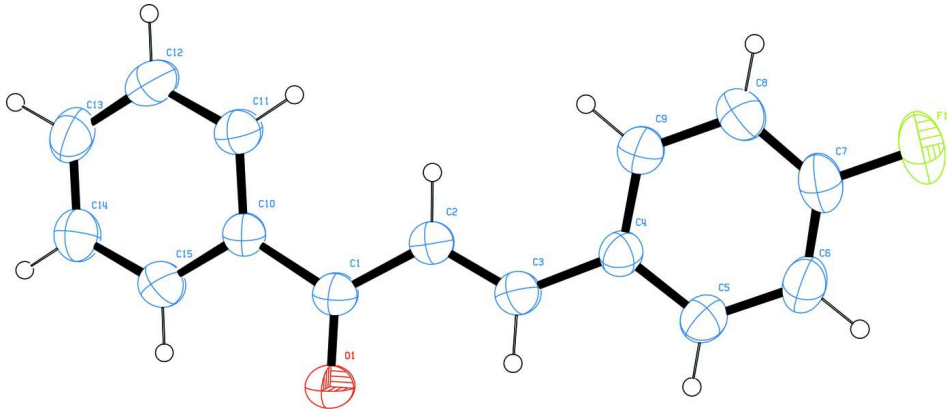


Figure 1

The molecular structure of title compound, with 30% probability displacement ellipsoids for non-H atoms.

(*E*)-3-(4-Fluorophenyl)-1-phenylprop-2-en-1-one

Crystal data

$C_{15}H_{11}FO$
M_r = 226.24
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
a = 8.6925 (4) Å
b = 5.9266 (2) Å
c = 22.6456 (9) Å
β = 95.423 (4)°
V = 1161.41 (8) Å^3
Z = 4

$F(000)$ = 472
D_x = 1.294 Mg m^{-3}
Melting point: 352 K
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 4193 reflections
θ = 3.6–23.2°
μ = 0.09 mm^{-1}
T = 293 K
Prism, colourless
0.59 × 0.15 × 0.07 mm

Data collection

Oxford Diffraction Xcalibur (Atlas, Gemini) diffractometer
Graphite monochromator
Detector resolution: 10.5564 pixels mm^{-1}
ω scans

Absorption correction: analytical (*CrysAlis PRO*; Oxford Diffraction, 2009)
T_{min} = 0.993, T_{max} = 0.999
22101 measured reflections
2276 independent reflections
1471 reflections with $I > 2\sigma(I)$

R_{int} = 0.044
θ_{max} = 26.1°, θ_{min} = 2.8°
h = −10→10
k = −7→7
l = −27→27

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.045
$wR(F^2)$ = 0.131
S = 1.01
2276 reflections
155 parameters
0 restraints
0 constraints
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
$w = 1/[\sigma^2(F_o^2) + (0.0624P)^2 + 0.1205P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.11 e Å^{-3}
$\Delta\rho_{min}$ = −0.11 e Å^{-3}
Extinction correction: *SHELXL2013*
Extinction coefficient: 0.0047 (18)

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

	x	*y*	*z*	U_{iso}*/U_{eq}
C2	0.48635 (19)	0.3169 (3)	0.11626 (7)	0.0677 (5)
H2	0.4743	0.4587	0.1328	0.081*
C4	0.31624 (17)	0.4186 (3)	0.02499 (7)	0.0603 (4)
O1	0.58960 (17)	−0.0451 (2)	0.13196 (6)	0.0977 (5)
C3	0.41537 (18)	0.2726 (3)	0.06357 (7)	0.0657 (4)
H3	0.4308	0.1283	0.0491	0.079*
C1	0.58376 (19)	0.1497 (3)	0.14973 (7)	0.0680 (5)
F1	0.02919 (14)	0.8135 (2)	−0.08612 (6)	0.1173 (5)
C10	0.67648 (18)	0.2155 (3)	0.20579 (7)	0.0620 (4)
C8	0.1728 (2)	0.7614 (3)	0.00570 (9)	0.0815 (5)
H8	0.1411	0.9022	0.018	0.098*
C5	0.2652 (2)	0.3477 (3)	−0.03154 (7)	0.0733 (5)
H5	0.2962	0.2074	−0.0445	0.088*
C9	0.2692 (2)	0.6298 (3)	0.04281 (8)	0.0735 (5)
H9	0.3034	0.6827	0.0804	0.088*
C7	0.1246 (2)	0.6831 (3)	−0.04919 (9)	0.0791 (5)
C6	0.1692 (2)	0.4803 (3)	−0.06940 (8)	0.0818 (6)
H6	0.1363	0.4317	−0.1076	0.098*
C15	0.7800 (2)	0.0620 (3)	0.23197 (9)	0.0910 (6)
H15	0.7907	−0.0775	0.214	0.109*
C11	0.6644 (2)	0.4203 (3)	0.23317 (8)	0.0818 (5)
H11	0.5962	0.5282	0.2162	0.098*
C13	0.8525 (2)	0.3131 (4)	0.31101 (8)	0.0909 (6)
H13	0.9105	0.3453	0.3466	0.109*
C12	0.7521 (3)	0.4682 (4)	0.28558 (9)	0.0928 (6)
H12	0.7425	0.6077	0.3037	0.111*
C14	0.8678 (3)	0.1097 (4)	0.28394 (10)	0.1066 (8)
H14	0.9376	0.0036	0.3007	0.128*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C2	0.0750 (11)	0.0601 (10)	0.0658 (10)	0.0067 (8)	−0.0043 (8)	−0.0066 (8)
C4	0.0562 (9)	0.0621 (9)	0.0614 (9)	−0.0056 (8)	−0.0005 (7)	−0.0005 (8)
O1	0.1263 (12)	0.0693 (8)	0.0905 (9)	0.0259 (7)	−0.0263 (8)	−0.0201 (7)
C3	0.0668 (10)	0.0602 (9)	0.0688 (10)	0.0014 (8)	−0.0003 (8)	−0.0053 (8)
C1	0.0740 (11)	0.0621 (10)	0.0665 (10)	0.0091 (8)	−0.0008 (8)	−0.0073 (8)
F1	0.1054 (9)	0.1264 (10)	0.1130 (9)	0.0174 (7)	−0.0265 (7)	0.0378 (8)
C10	0.0658 (10)	0.0610 (9)	0.0587 (9)	0.0044 (8)	0.0028 (7)	−0.0012 (7)
C8	0.0795 (12)	0.0741 (12)	0.0888 (13)	0.0132 (10)	−0.0026 (10)	0.0048 (10)
C5	0.0779 (11)	0.0716 (11)	0.0683 (11)	−0.0064 (9)	−0.0043 (9)	−0.0043 (9)
C9	0.0758 (11)	0.0729 (11)	0.0699 (11)	0.0071 (9)	−0.0032 (9)	−0.0054 (9)
C7	0.0642 (11)	0.0878 (13)	0.0824 (13)	−0.0012 (10)	−0.0073 (9)	0.0216 (11)
C6	0.0807 (12)	0.0935 (14)	0.0675 (11)	−0.0128 (11)	−0.0132 (9)	0.0056 (10)
C15	0.1073 (15)	0.0710 (11)	0.0887 (13)	0.0170 (11)	−0.0233 (11)	−0.0078 (10)
C11	0.0900 (13)	0.0775 (12)	0.0751 (11)	0.0183 (10)	−0.0077 (10)	−0.0150 (9)
C13	0.1022 (15)	0.1003 (15)	0.0659 (11)	−0.0081 (13)	−0.0140 (10)	−0.0003 (11)
C12	0.1098 (16)	0.0867 (13)	0.0791 (13)	0.0040 (12)	−0.0051 (12)	−0.0251 (11)
C14	0.1241 (18)	0.0903 (14)	0.0954 (15)	0.0148 (13)	−0.0433 (13)	−0.0021 (12)

Geometric parameters (Å, º)

C2—C3	1.317 (2)	C5—C6	1.383 (2)
C2—C1	1.467 (2)	C5—H5	0.93
C2—H2	0.93	C9—H9	0.93
C4—C5	1.380 (2)	C7—C6	1.356 (3)
C4—C9	1.388 (2)	C6—H6	0.93
C4—C3	1.453 (2)	C15—C14	1.370 (3)
O1—C1	1.2255 (19)	C15—H15	0.93
C3—H3	0.93	C11—C12	1.378 (2)
C1—C10	1.490 (2)	C11—H11	0.93
F1—C7	1.3615 (19)	C13—C12	1.358 (3)
C10—C11	1.372 (2)	C13—C14	1.365 (3)
C10—C15	1.374 (2)	C13—H13	0.93
C8—C7	1.356 (3)	C12—H12	0.93
C8—C9	1.372 (2)	C14—H14	0.93
C8—H8	0.93		
C3—C2—C1	122.11 (15)	C4—C9—H9	119.5
C3—C2—H2	118.9	C8—C7—C6	122.65 (17)
C1—C2—H2	118.9	C8—C7—F1	119.12 (19)
C5—C4—C9	117.80 (15)	C6—C7—F1	118.23 (18)
C5—C4—C3	119.76 (15)	C7—C6—C5	118.00 (17)
C9—C4—C3	122.44 (15)	C7—C6—H6	121
C2—C3—C4	128.77 (15)	C5—C6—H6	121
C2—C3—H3	115.6	C14—C15—C10	121.52 (18)
C4—C3—H3	115.6	C14—C15—H15	119.2
O1—C1—C2	120.43 (15)	C10—C15—H15	119.2
O1—C1—C10	119.37 (15)	C10—C11—C12	120.78 (17)

C2—C1—C10	120.20 (14)	C10—C11—H11	119.6
C11—C10—C15	117.77 (16)	C12—C11—H11	119.6
C11—C10—C1	123.94 (15)	C12—C13—C14	119.57 (18)
C15—C10—C1	118.29 (15)	C12—C13—H13	120.2
C7—C8—C9	118.99 (18)	C14—C13—H13	120.2
C7—C8—H8	120.5	C13—C12—C11	120.46 (18)
C9—C8—H8	120.5	C13—C12—H12	119.8
C4—C5—C6	121.60 (17)	C11—C12—H12	119.8
C4—C5—H5	119.2	C13—C14—C15	119.88 (19)
C6—C5—H5	119.2	C13—C14—H14	120.1
C8—C9—C4	120.94 (17)	C15—C14—H14	120.1
C8—C9—H9	119.5		

C1—C2—C3—C4	−179.76 (16)	C9—C8—C7—C6	−0.8 (3)
C5—C4—C3—C2	173.35 (17)	C9—C8—C7—F1	−179.91 (16)
C9—C4—C3—C2	−6.9 (3)	C8—C7—C6—C5	1.4 (3)
C3—C2—C1—O1	−7.3 (3)	F1—C7—C6—C5	−179.52 (16)
C3—C2—C1—C10	172.77 (16)	C4—C5—C6—C7	−0.7 (3)
O1—C1—C10—C11	−171.95 (19)	C11—C10—C15—C14	0.5 (3)
C2—C1—C10—C11	8.0 (3)	C1—C10—C15—C14	−179.2 (2)
O1—C1—C10—C15	7.7 (3)	C15—C10—C11—C12	−0.8 (3)
C2—C1—C10—C15	−172.33 (18)	C1—C10—C11—C12	178.88 (17)
C9—C4—C5—C6	−0.6 (2)	C14—C13—C12—C11	0.8 (3)
C3—C4—C5—C6	179.19 (16)	C10—C11—C12—C13	0.1 (3)
C7—C8—C9—C4	−0.5 (3)	C12—C13—C14—C15	−1.1 (4)
C5—C4—C9—C8	1.2 (3)	C10—C15—C14—C13	0.4 (4)
C3—C4—C9—C8	−178.60 (16)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C5—H5···O1^{i}	0.93	2.49	3.244 (2)	138
C13—H13···F1^{ii}	0.93	2.68	3.465 (2)	142

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