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5-Chloro-2-(4-chlorophenyl)-7-methyl-3-methylsulfinyl-1-benzofuran

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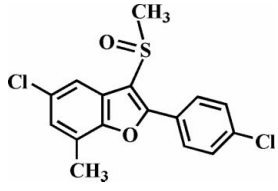
Key indicators: single-crystal X-ray study; T = 173 K; mean σ(C–C) = 0.002 Å; R factor = 0.031; wR factor = 0.089; data-to-parameter ratio = 16.5.

In the title compound, $C_{16}H_{12}Cl_2O_2S$, the O atom and the methyl group of the methylsulfinyl substituent lie on opposite sides of the plane through the benzofuran fragment. The 4-chlorophenyl ring is rotated out of the benzofuran plane, as indicated by the dihedral angle of 15.91 (4)°. In the crystal, molecules are linked into chains along the b axis by weak intermolecular C—H···O hydrogen bonds.

Related literature

For the crystal structures of similar 2-(4-fluorophenyl)-5-halo-3-methylsulfinyl-1-benzofuran derivatives, see: Choi *et al.* (2009, 2010*a*,*b*). For the biological activity of benzofuran compounds, see: Aslam *et al.* (2006); Galal *et al.* (2009); Khan *et al.* (2005). For natural products with benzofuran rings, see: Akgul & Anil (2003); Soekamto *et al.* (2003).



Experimental

Crystal data

$C_{16}H_{12}Cl_2O_2S$ M_r = 339.22
Triclinic, $P\overline{1}$
a = 7.538 (2) Å
b = 9.734 (2) Å
c = 11.277 (3) Å
α = 72.525 (2)°
β = 88.846 (3)°
γ = 70.058 (2)°
V = 738.8 (3) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.58 mm^{-1}
T = 173 K
0.50 × 0.50 × 0.45 mm

Data collection

Bruker SMART APEXII CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2009)
T_{min} = 0.760, T_{max} = 0.780
6331 measured reflections
3170 independent reflections
2879 reflections with $I > 2\sigma(I)$
R_{int} = 0.017

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.031
$wR(F^2)$ = 0.089
S = 1.03
3170 reflections
192 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.67 e Å^{-3}
$\Delta\rho_{min}$ = −0.33 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
C15—H15A···O2^{i}	0.96	2.52	3.216 (2)	130

Symmetry code: (i) $x, y-1, z$.

Data collection: *APEX2* (Bruker, 2009); cell refinement: *SAINT* (Bruker, 2009); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3* (Farrugia, 1997) and *DIAMOND* (Brandenburg, 1998); software used to prepare material for publication: *SHELXL97*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HG2648).

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supporting information

Acta Cryst. (2010). E**66**, o706 [doi:10.1107/S1600536810006823]

5-Chloro-2-(4-chlorophenyl)-7-methyl-3-methylsulfinyl-1-benzofuran

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S1. Comment

Compounds containing benzofuran skeleton display significant biological activities such as antifungal (Aslam *et al.*, 2006), antitumor and antiviral (Galal *et al.*, 2009), antimicrobial (Khan *et al.*, 2005) properties. These compounds are widely occurring in nature (Akgul & Anil, 2003; Soekamto *et al.*, 2003). As a part of our ongoing studies of the effect of side chain substituents on the solid state structures of 2-(4-fluorophenyl)-5-halo-3-methylsulfinyl-1-benzofuran analogues (Choi *et al.*, 2009, 2010*a,b*), we report the crystal structure of the title compound (Fig. 1).

The benzofuran unit is essentially planar, with a mean deviation of 0.013 (1) Å from the least-squares plane defined by the nine constituent atoms. The dihedral angle formed by the plane of the benzofuran and the 4-chlorophenyl ring is 15.91 (4)°. The crystal packing (Fig. 2) is stabilized by a weak intermolecular C—H···O hydrogen bond between the methyl H atom and the oxygen of the S═O unit, with a C15—H15A···O2^{i} (Table 1).

S2. Experimental

77% 3-Chloroperoxybenzoic acid (247 mg, 1.1 mmol) was added in small portions to a stirred solution of 5-chloro-2-(4-chlorophenyl)-7-methyl-3-methylsulfanyl-1-benzofuran (323 mg, 1.0 mmol) in dichloromethane (30 mL) at 273 K. After being stirred at room temperature for 4h, the mixture was washed with saturated sodium bicarbonate solution and the organic layer was separated, dried over magnesium sulfate, filtered and concentrated at reduced pressure. The residue was purified by column chromatography (hexane–ethyl acetate, 1:1 v/v) to afford the title compound as a colorless solid [yield 79%, m.p. 456–457 K; R_f = 0.62 (hexane–ethyl acetate, 1:1 v/v)]. Single crystals suitable for X-ray diffraction were prepared by slow evaporation of a solution of the title compound in tetrahydrofuran at room temperature.

S3. Refinement

All H atoms were positioned geometrically and refined using a riding model, with C—H = 0.93 Å for aryl and 0.96 Å for methyl H atoms. U_{iso}(H) = 1.2U_{eq}(C) for aryl and 1.5U_{eq}(C) for methyl H atoms.

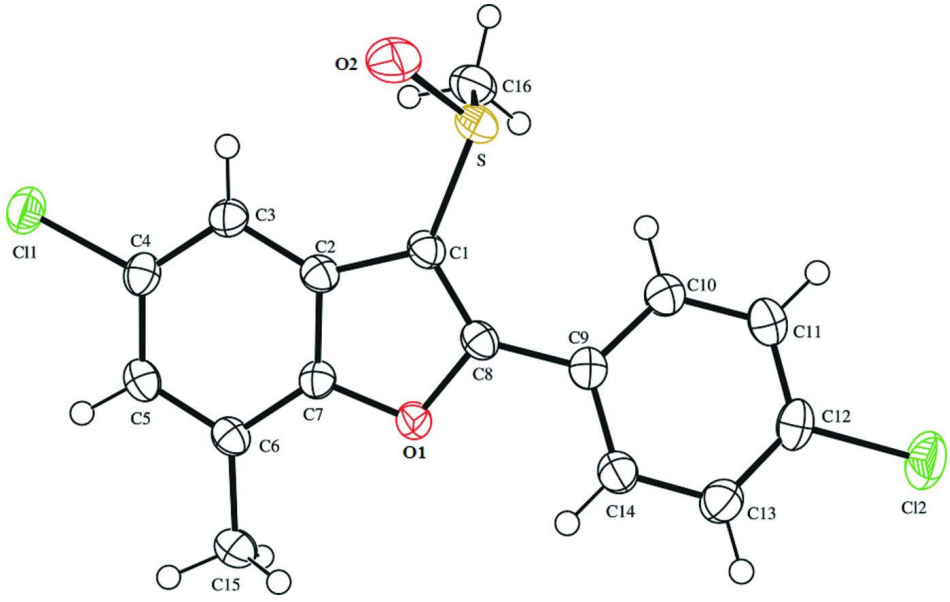

Figure 1

The molecular structure of the title compound with the atom numbering scheme. Displacement ellipsoids are drawn at the 50 % probability level. H atoms are presented as a small spheres of arbitrary radius.

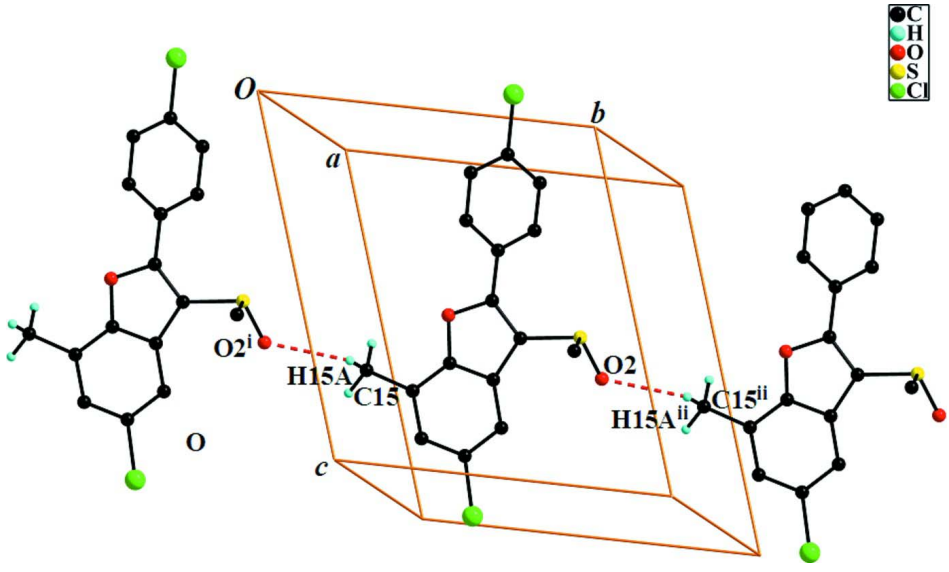

Figure 2

C—H···O interactions (dotted lines) in the crystal structure of the title compound. [Symmetry codes: i) *x*, *y* - 1, *z*; ii) *x*, *y* + 1, *z*.]

5-Chloro-2-(4-chlorophenyl)-7-methyl-3-methylsulfinyl-1-benzofuran

Crystal data

$C_{16}H_{12}Cl_2O_2S$
M_r = 339.22
Triclinic, $P\overline{1}$
Hall symbol: -P 1

a = 7.538 (2) Å
b = 9.734 (2) Å
c = 11.277 (3) Å
α = 72.525 (2)°

β = 88.846 (3)°
γ = 70.058 (2)°
V = 738.8 (3) Å^3
Z = 2
F(000) = 348
D_x = 1.525 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å

Cell parameters from 5112 reflections
θ = 2.3–27.5°
μ = 0.58 mm^{-1}
T = 173 K
Block, colourless
0.50 × 0.50 × 0.45 mm

Data collection

Bruker SMART APEXII CCD diffractometer
Radiation source: Rotating Anode
Bruker HELIOS graded multilayer optics monochromator
Detector resolution: 10.0 pixels mm^{-1}
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2009)

T_{min} = 0.760, T_{max} = 0.780
6331 measured reflections
3170 independent reflections
2879 reflections with $I > 2\sigma(I)$
R_{int} = 0.017
θ_{max} = 27.0°, θ_{min} = 2.3°
h = −9→9
k = −12→12
l = −14→14

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.031
$wR(F^2)$ = 0.089
S = 1.03
3170 reflections
192 parameters
0 restraints
Primary atom site location: structure-invariant direct methods

Secondary atom site location: difference Fourier map
Hydrogen site location: difference Fourier map
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0425P)^2 + 0.4419P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.67 e Å^{-3}
$\Delta\rho_{min}$ = −0.33 e Å^{-3}

Special details

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

	x	y	z	U_{iso}*/U_{eq}
Cl1	0.54735 (6)	0.25715 (5)	1.03989 (4)	0.03540 (12)
Cl2	−0.06257 (7)	0.78823 (6)	−0.05751 (4)	0.04700 (14)
S	0.19164 (6)	0.77859 (5)	0.56104 (4)	0.03206 (12)
O2	0.3263 (2)	0.78670 (16)	0.65093 (15)	0.0479 (4)
O1	0.31330 (15)	0.36506 (12)	0.52197 (10)	0.0257 (2)
C1	0.2500 (2)	0.58258 (17)	0.57346 (15)	0.0256 (3)
C2	0.3368 (2)	0.45451 (18)	0.68431 (15)	0.0251 (3)
C3	0.3874 (2)	0.43701 (19)	0.80827 (15)	0.0276 (3)
H3	0.3628	0.5210	0.8373	0.033*

C4	0.4757 (2)	0.28803 (19)	0.88482 (15)	0.0276 (3)
C5	0.5149 (2)	0.15889 (18)	0.84399 (15)	0.0277 (3)
H5	0.5761	0.0613	0.8998	0.033*
C6	0.4640 (2)	0.17381 (18)	0.72181 (15)	0.0259 (3)
C7	0.3749 (2)	0.32444 (18)	0.64609 (14)	0.0240 (3)
C8	0.2391 (2)	0.52362 (17)	0.47881 (15)	0.0251 (3)
C9	0.1655 (2)	0.58997 (18)	0.34785 (15)	0.0260 (3)
C10	0.0411 (2)	0.7420 (2)	0.29918 (16)	0.0320 (4)
H10	0.0039	0.8025	0.3516	0.038*
C11	−0.0275 (2)	0.8040 (2)	0.17491 (16)	0.0336 (4)
H11	−0.1079	0.9060	0.1432	0.040*
C12	0.0253 (2)	0.7123 (2)	0.09853 (16)	0.0321 (4)
C13	0.1472 (2)	0.5606 (2)	0.14326 (17)	0.0340 (4)
H13	0.1808	0.5003	0.0905	0.041*
C14	0.2181 (2)	0.50046 (19)	0.26736 (16)	0.0295 (3)
H14	0.3016	0.3994	0.2978	0.035*
C15	0.4973 (3)	0.03952 (19)	0.67375 (17)	0.0342 (4)
H15A	0.3806	0.0217	0.6680	0.051*
H15B	0.5895	−0.0505	0.7299	0.051*
H15C	0.5430	0.0616	0.5926	0.051*
C16	−0.0290 (3)	0.8027 (2)	0.63063 (19)	0.0402 (4)
H16A	−0.0092	0.7243	0.7099	0.060*
H16B	−0.1193	0.7945	0.5767	0.060*
H16C	−0.0764	0.9021	0.6424	0.060*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.0418 (2)	0.0380 (2)	0.0257 (2)	−0.01599 (18)	−0.00416 (16)	−0.00618 (17)
Cl2	0.0449 (3)	0.0568 (3)	0.0281 (2)	−0.0095 (2)	−0.00846 (19)	−0.0063 (2)
S	0.0416 (2)	0.0211 (2)	0.0340 (2)	−0.01348 (17)	0.00087 (18)	−0.00631 (16)
O2	0.0564 (9)	0.0358 (7)	0.0589 (9)	−0.0222 (6)	−0.0092 (7)	−0.0175 (7)
O1	0.0296 (6)	0.0221 (5)	0.0243 (5)	−0.0094 (4)	−0.0009 (4)	−0.0053 (4)
C1	0.0278 (7)	0.0208 (7)	0.0280 (8)	−0.0102 (6)	0.0006 (6)	−0.0052 (6)
C2	0.0253 (7)	0.0227 (7)	0.0289 (8)	−0.0111 (6)	0.0017 (6)	−0.0070 (6)
C3	0.0320 (8)	0.0257 (8)	0.0276 (8)	−0.0131 (6)	−0.0001 (6)	−0.0082 (6)
C4	0.0273 (8)	0.0315 (8)	0.0248 (8)	−0.0137 (6)	0.0000 (6)	−0.0059 (6)
C5	0.0257 (7)	0.0241 (8)	0.0296 (8)	−0.0088 (6)	0.0000 (6)	−0.0028 (6)
C6	0.0244 (7)	0.0225 (7)	0.0303 (8)	−0.0095 (6)	0.0019 (6)	−0.0061 (6)
C7	0.0241 (7)	0.0250 (7)	0.0245 (7)	−0.0112 (6)	0.0009 (6)	−0.0069 (6)
C8	0.0240 (7)	0.0215 (7)	0.0289 (8)	−0.0091 (6)	0.0012 (6)	−0.0052 (6)
C9	0.0247 (7)	0.0277 (8)	0.0262 (8)	−0.0125 (6)	0.0020 (6)	−0.0057 (6)
C10	0.0319 (8)	0.0309 (8)	0.0301 (8)	−0.0074 (7)	0.0007 (7)	−0.0094 (7)
C11	0.0298 (8)	0.0307 (8)	0.0325 (9)	−0.0066 (7)	−0.0017 (7)	−0.0031 (7)
C12	0.0272 (8)	0.0420 (10)	0.0241 (8)	−0.0136 (7)	−0.0016 (6)	−0.0041 (7)
C13	0.0346 (9)	0.0372 (9)	0.0308 (9)	−0.0114 (7)	0.0008 (7)	−0.0128 (7)
C14	0.0299 (8)	0.0272 (8)	0.0303 (8)	−0.0101 (6)	−0.0003 (6)	−0.0071 (7)
C15	0.0419 (9)	0.0230 (8)	0.0351 (9)	−0.0088 (7)	0.0016 (7)	−0.0082 (7)

C16	0.0454 (10)	0.0299 (9)	0.0426 (10)	−0.0085 (8)	0.0067 (8)	−0.0131 (8)

Geometric parameters (Å, º)

Cl1—C4	1.7468 (17)	C8—C9	1.457 (2)
Cl2—C12	1.7358 (17)	C9—C10	1.399 (2)
S—O2	1.4862 (14)	C9—C14	1.402 (2)
S—C1	1.7672 (16)	C10—C11	1.380 (2)
S—C16	1.797 (2)	C10—H10	0.9300
O1—C7	1.3782 (19)	C11—C12	1.380 (3)
O1—C8	1.3794 (18)	C11—H11	0.9300
C1—C8	1.371 (2)	C12—C13	1.387 (3)
C1—C2	1.444 (2)	C13—C14	1.383 (2)
C2—C7	1.395 (2)	C13—H13	0.9300
C2—C3	1.401 (2)	C14—H14	0.9300
C3—C4	1.380 (2)	C15—H15A	0.9600
C3—H3	0.9300	C15—H15B	0.9600
C4—C5	1.403 (2)	C15—H15C	0.9600
C5—C6	1.389 (2)	C16—H16A	0.9600
C5—H5	0.9300	C16—H16B	0.9600
C6—C7	1.389 (2)	C16—H16C	0.9600
C6—C15	1.504 (2)		
O2—S—C1	107.20 (8)	C10—C9—C8	121.71 (15)
O2—S—C16	106.67 (10)	C14—C9—C8	119.94 (14)
C1—S—C16	97.03 (8)	C11—C10—C9	121.30 (16)
C7—O1—C8	106.66 (12)	C11—C10—H10	119.3
C8—C1—C2	107.57 (14)	C9—C10—H10	119.3
C8—C1—S	126.90 (12)	C10—C11—C12	118.87 (16)
C2—C1—S	125.25 (12)	C10—C11—H11	120.6
C7—C2—C3	119.54 (14)	C12—C11—H11	120.6
C7—C2—C1	104.68 (14)	C11—C12—C13	121.64 (16)
C3—C2—C1	135.77 (14)	C11—C12—Cl2	119.29 (14)
C4—C3—C2	116.18 (14)	C13—C12—Cl2	119.07 (14)
C4—C3—H3	121.9	C14—C13—C12	119.06 (16)
C2—C3—H3	121.9	C14—C13—H13	120.5
C3—C4—C5	123.34 (15)	C12—C13—H13	120.5
C3—C4—Cl1	118.85 (13)	C13—C14—C9	120.76 (16)
C5—C4—Cl1	117.80 (12)	C13—C14—H14	119.6
C6—C5—C4	121.32 (15)	C9—C14—H14	119.6
C6—C5—H5	119.3	C6—C15—H15A	109.5
C4—C5—H5	119.3	C6—C15—H15B	109.5
C7—C6—C5	114.60 (14)	H15A—C15—H15B	109.5
C7—C6—C15	121.71 (15)	C6—C15—H15C	109.5
C5—C6—C15	123.68 (14)	H15A—C15—H15C	109.5
O1—C7—C6	124.10 (14)	H15B—C15—H15C	109.5
O1—C7—C2	110.89 (13)	S—C16—H16A	109.5
C6—C7—C2	125.01 (15)	S—C16—H16B	109.5

C1—C8—O1	110.18 (14)	H16A—C16—H16B	109.5
C1—C8—C9	134.67 (15)	S—C16—H16C	109.5
O1—C8—C9	115.15 (13)	H16A—C16—H16C	109.5
C10—C9—C14	118.35 (15)	H16B—C16—H16C	109.5
O2—S—C1—C8	−144.91 (15)	C1—C2—C7—O1	−1.46 (17)
C16—S—C1—C8	105.17 (16)	C3—C2—C7—C6	−1.0 (2)
O2—S—C1—C2	28.29 (16)	C1—C2—C7—C6	178.09 (15)
C16—S—C1—C2	−81.64 (15)	C2—C1—C8—O1	0.14 (17)
C8—C1—C2—C7	0.79 (17)	S—C1—C8—O1	174.32 (11)
S—C1—C2—C7	−173.51 (12)	C2—C1—C8—C9	178.83 (16)
C8—C1—C2—C3	179.69 (17)	S—C1—C8—C9	−7.0 (3)
S—C1—C2—C3	5.4 (3)	C7—O1—C8—C1	−1.03 (16)
C7—C2—C3—C4	0.8 (2)	C7—O1—C8—C9	180.00 (12)
C1—C2—C3—C4	−177.97 (17)	C1—C8—C9—C10	−16.6 (3)
C2—C3—C4—C5	0.0 (2)	O1—C8—C9—C10	162.01 (14)
C2—C3—C4—Cl1	178.74 (12)	C1—C8—C9—C14	163.90 (17)
C3—C4—C5—C6	−0.7 (2)	O1—C8—C9—C14	−17.5 (2)
Cl1—C4—C5—C6	−179.45 (12)	C14—C9—C10—C11	−0.7 (2)
C4—C5—C6—C7	0.5 (2)	C8—C9—C10—C11	179.83 (15)
C4—C5—C6—C15	−178.23 (15)	C9—C10—C11—C12	1.4 (3)
C8—O1—C7—C6	−177.99 (14)	C10—C11—C12—C13	−1.0 (3)
C8—O1—C7—C2	1.57 (16)	C10—C11—C12—Cl2	179.16 (13)
C5—C6—C7—O1	179.82 (13)	C11—C12—C13—C14	−0.3 (3)
C15—C6—C7—O1	−1.4 (2)	Cl2—C12—C13—C14	179.60 (13)
C5—C6—C7—C2	0.3 (2)	C12—C13—C14—C9	1.1 (3)
C15—C6—C7—C2	179.10 (15)	C10—C9—C14—C13	−0.6 (2)
C3—C2—C7—O1	179.43 (13)	C8—C9—C14—C13	178.90 (15)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C15—H15*A*···O2^{i}	0.96	2.52	3.216 (2)	130

Symmetry code: (i) *x*, *y*−1, *z*.