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Isopropyl 2-(5-chloro-3-methylsulfinyl-1-benzofuran-2-yl)acetate

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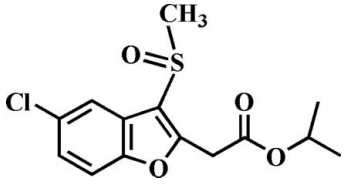
Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.005 Å; R factor = 0.048; wR factor = 0.124; data-to-parameter ratio = 14.1.

In the title compound, $C_{14}H_{15}ClO_4S$, the S atom has a distorted trigonal-pyramidal coordination. The O atom and the methyl group of the methylsulfinyl substituent lie on opposite sides of the benzofuran ring system. The crystal structure is stabilized by intermolecular aromatic π–π interactions between the benzene rings of neighbouring molecules [centroid–centroid distance = 4.057 (3) Å], and by C—H$\cdots\pi$ interactions between a methyl H atom and the benzene ring of an adjacent molecule.

Related literature

For the crystal structures of similar compounds, see: Choi *et al.* (2008*a*,*b*).



Experimental

Crystal data

$C_{14}H_{15}ClO_4S$
M_r = 314.77
Triclinic, $P\overline{1}$
a = 7.8824 (6) Å
b = 10.0352 (8) Å
c = 10.9004 (8) Å
α = 69.254 (1)°
β = 81.662 (1)°
γ = 67.703 (1)°
V = 745.98 (10) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.41 mm^{-1}
T = 298 (2) K
0.50 × 0.40 × 0.15 mm

Data collection

Bruker SMART CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1999)
T_{min} = 0.816, T_{max} = 0.939
5508 measured reflections
2602 independent reflections
2354 reflections with $I > 2\sigma(I)$
R_{int} = 0.017

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.048
$wR(F^2)$ = 0.124
S = 1.12
2602 reflections
184 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.49 e Å^{-3}
$\Delta\rho_{min}$ = −0.29 e Å^{-3}

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

D—H$\cdots A$	D—H	H$\cdots A$	$D\cdots A$	D—H$\cdots A$
C13—H13$B\cdots Cg^{i}$	0.96	2.78	3.515 (3)	134

Symmetry code: (i) $x, y+1, z$. Cg is the centroid of the C2–C7 benzene ring.

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT* (Bruker, 2001); 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: NG2521).

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supporting information

Acta Cryst. (2009). E**65**, o3 [doi:10.1107/S1600536808039937]

Isopropyl 2-(5-chloro-3-methylsulfinyl-1-benzofuran-2-yl)acetate

Hong Dae Choi, Pil Ja Seo, Byeng Wha Son and Uk Lee

S1. Comment

As a part of our ongoing research on the synthesis and structure of isopropyl 2-(5-halo-3-methylsulfinyl-1-benzofuran-2-yl)acetate analogues, we have recently described the crystal structures of isopropyl 2-(5-bromo-3-methylsulfinyl-1-benzofuran-2-yl)acetate (Choi *et al.*, 2008a) and isopropyl 2-(5-iodo-3-methylsulfinyl-1-benzofuran-2-yl)acetate (Choi *et al.*, 2008b). Here we report the crystal structure of the title compound, isopropyl 2-(5-chloro-3-methylsulfinyl-1-benzofuran-2-yl)acetate (Fig. 1).

The benzofuran unit is essentially planar, with a mean deviation of 0.012 (2) Å from the least-squares plane defined by the nine constituent atoms. The molecular packing is stabilized by aromatic π—π stacking interactions between the benzene rings of adjacent molecules. The Cg···Cg^{ii} distance is 4.057 (3) Å (Fig. 2; Cg is the centroid of C2—C7 benzene ring; symmetry code as in Fig. 2). The molecular packing is further stabilized by C—H···π interactions between a methyl H atom of isoproyl group and the benzene ring of the benzofuran uint, with a C13—H13B···Cg^{i} separation of 2.78 Å (Fig. 2 and Table 1; Cg is the centroid of the C2-C7 benzene ring).

S2. Experimental

77% 3-chloroperoxybenzoic acid (173 mg, 0.77 mmol) was added in small portions to a stirred solution of isopropyl 2-(5-chloro-3-methylsulfanyl-1-benzofuran-2-yl)acetate (209 mg, 0.7 mmol) in dichloromethane (30 ml) at 273 K. After being stirred for 3 h at room temperature, the mixture was washed with saturated sodium bicarbonate solution and the organic layer was separated, dried over magnesium sulfate, filtered and concentrated in vacuum. The residue was purified by column chromatography (hexane-ethyl acetate, 1:2 v/v) to afford the title compound as a colorless solid [yield 83%, m.p. 422–423 K; R_f = 0.52 (hexane-ethyl acetate, 1;2 v/v)]. Single crystals suitable for X-ray diffraction were prepared by evaporation of a solution of the title compound in acetone at room temperature. Spectroscopic analysis: ^{1}H NMR ($CDCl_3$, 400 MHz) δ 1.28 (d, J = 6.20 Hz, 6H), 3.08 (s, 3H), 4.0 (s, 2H), 5.02-5.08 (m, 1H), 7.35 (dd, J = 8.76 Hz and J = 1.84 Hz, 1H), 7.45 (d, J = 8.76 Hz, 1H), 7.95 (d, J = 1.84 Hz, 1H); EI-MS 316 [M+2], 314 [M^+].

S3. Refinement

All H atoms were geometrically positioned and refined using a riding model, with C—H = 0.93 Å for the aryl, 0.97 Å for the methylene, 0.98 Å for the methine, and 0.96 Å for the methyl H atoms. *U*iso(H) = 1.2*U*eq(C) for the aryl, methine and methylene H atoms, and 1.5*U*eq(C) for methyl H atoms.

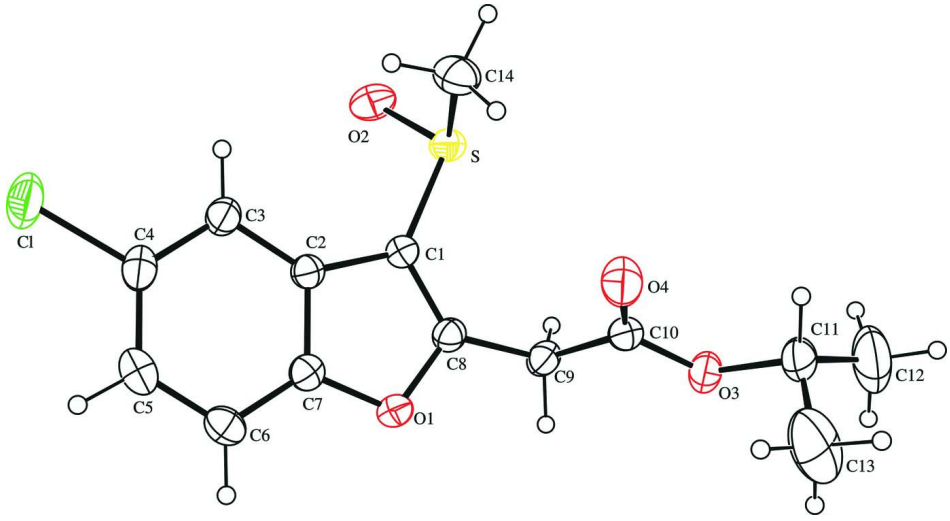

Figure 1

The molecular structure of the title compound, showing displacement ellipsoids drawn at the 30% probability level.

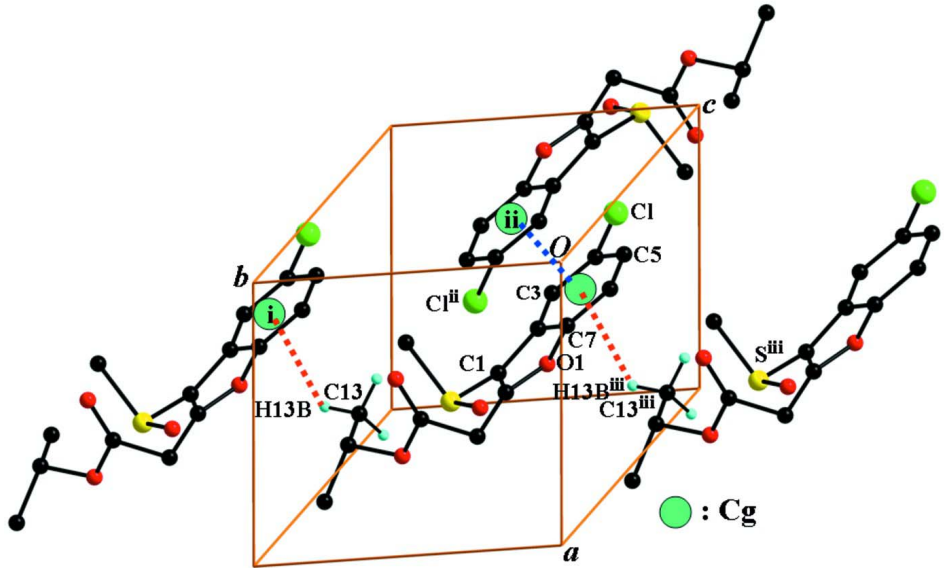

Figure 2

π—π and C—H···π interactions (dotted lines) in the title compound. Cg denotes ring centroid.[Symmetry code: (i) x, y+1, z; (ii) -x+1, -y+1, -z+2; (iii) x, y-1, z.]

Isopropyl 2-(5-chloro-3-methylsulfinyl-1-benzofuran-2-yl)acetate

Crystal data

$C_{14}H_{15}ClO_4S$
M_r = 314.77
Triclinic, $P\overline{1}$
Hall symbol: -P 1
a = 7.8824 (6) Å
b = 10.0352 (8) Å
c = 10.9004 (8) Å
α = 69.254 (1)°
β = 81.662 (1)°
γ = 67.703 (1)°
V = 745.98 (10) Å^3
Z = 2
F(000) = 328
D_x = 1.401 Mg m^{-3}

Melting point = 422–423 K
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 4167 reflections
θ = 2.5–28.3°
μ = 0.41 mm^{-1}
T = 298 K
Plate, colorless
0.50 × 0.40 × 0.15 mm

Data collection

Bruker SMART CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
Detector resolution: 10.0 pixels mm^{-1}
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1999)
T_{min} = 0.816, T_{max} = 0.939
5508 measured reflections
2602 independent reflections
2354 reflections with $I > 2\sigma(I)$
R_{int} = 0.017
θ_{max} = 25.0°, θ_{min} = 2.5°
h = −9→9
k = −11→11
l = −12→12

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.048
$wR(F^2)$ = 0.125
S = 1.12
2602 reflections
184 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
$w = 1/[\sigma^2(F_o^2) + (0.0534P)^2 + 0.4971P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.49 e Å^{-3}
$\Delta\rho_{min}$ = −0.29 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}
S	0.74726 (9)	0.60225 (8)	0.54010 (6)	0.0480 (2)
Cl	0.29574 (12)	0.21443 (10)	0.87346 (10)	0.0816 (3)
O1	0.8348 (2)	0.45903 (19)	0.91914 (16)	0.0435 (4)
O2	0.7488 (3)	0.4832 (3)	0.48756 (19)	0.0637 (6)
O3	1.0254 (3)	0.8446 (2)	0.7284 (2)	0.0582 (5)
O4	0.7414 (3)	0.8557 (2)	0.7070 (2)	0.0674 (6)
C1	0.7475 (3)	0.5246 (3)	0.7125 (2)	0.0393 (5)
C2	0.6447 (3)	0.4337 (3)	0.7973 (2)	0.0378 (5)
C3	0.5137 (3)	0.3799 (3)	0.7797 (3)	0.0462 (6)
H3	0.4686	0.4043	0.6972	0.055*
C4	0.4550 (4)	0.2888 (3)	0.8910 (3)	0.0510 (7)
C5	0.5171 (4)	0.2520 (3)	1.0159 (3)	0.0544 (7)

H5	0.4720	0.1905	1.0878	0.065*
C6	0.6450 (4)	0.3061 (3)	1.0341 (3)	0.0486 (6)
H6	0.6878	0.2832	1.1170	0.058*
C7	0.7062 (3)	0.3959 (3)	0.9229 (2)	0.0408 (5)
C8	0.8579 (3)	0.5352 (3)	0.7894 (2)	0.0409 (5)
C9	0.9910 (3)	0.6147 (3)	0.7601 (3)	0.0455 (6)
H9A	1.0667	0.5947	0.6856	0.055*
H9B	1.0708	0.5735	0.8347	0.055*
C10	0.9005 (4)	0.7845 (3)	0.7302 (2)	0.0437 (6)
C11	0.9615 (5)	1.0095 (3)	0.7059 (4)	0.0697 (9)
H11	0.8528	1.0642	0.6503	0.084*
C12	1.1212 (8)	1.0569 (5)	0.6388 (5)	0.1199 (18)
H12A	1.0856	1.1656	0.6142	0.180*
H12B	1.1570	1.0267	0.5619	0.180*
H12C	1.2226	1.0085	0.6979	0.180*
C13	0.9213 (8)	1.0315 (4)	0.8347 (5)	0.1195 (19)
H13A	0.8255	0.9938	0.8777	0.179*
H13B	0.8825	1.1381	0.8233	0.179*
H13C	1.0296	0.9770	0.8872	0.179*
C14	0.5183 (4)	0.7385 (4)	0.5211 (3)	0.0671 (8)
H14A	0.4977	0.7998	0.4304	0.101*
H14B	0.5018	0.8027	0.5728	0.101*
H14C	0.4327	0.6861	0.5499	0.101*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S	0.0489 (4)	0.0589 (4)	0.0385 (4)	−0.0261 (3)	−0.0038 (3)	−0.0100 (3)
Cl	0.0694 (5)	0.0748 (6)	0.1092 (7)	−0.0480 (5)	−0.0073 (5)	−0.0132 (5)
O1	0.0486 (10)	0.0464 (9)	0.0398 (9)	−0.0184 (8)	−0.0057 (7)	−0.0154 (7)
O2	0.0685 (13)	0.0804 (14)	0.0515 (12)	−0.0250 (11)	−0.0047 (9)	−0.0322 (11)
O3	0.0622 (12)	0.0429 (10)	0.0740 (13)	−0.0240 (9)	−0.0176 (10)	−0.0120 (9)
O4	0.0501 (12)	0.0510 (11)	0.0946 (16)	−0.0161 (10)	−0.0120 (11)	−0.0139 (11)
C1	0.0427 (13)	0.0388 (12)	0.0383 (13)	−0.0157 (10)	−0.0035 (10)	−0.0124 (10)
C2	0.0390 (12)	0.0345 (12)	0.0401 (13)	−0.0113 (10)	−0.0023 (10)	−0.0134 (10)
C3	0.0438 (14)	0.0438 (14)	0.0532 (15)	−0.0174 (11)	−0.0051 (11)	−0.0146 (12)
C4	0.0428 (14)	0.0421 (14)	0.0682 (18)	−0.0179 (11)	−0.0002 (12)	−0.0153 (13)
C5	0.0541 (16)	0.0437 (14)	0.0553 (16)	−0.0172 (13)	0.0089 (13)	−0.0084 (12)
C6	0.0509 (15)	0.0428 (14)	0.0432 (14)	−0.0102 (12)	0.0007 (11)	−0.0111 (11)
C7	0.0404 (13)	0.0353 (12)	0.0457 (13)	−0.0099 (10)	−0.0016 (10)	−0.0155 (10)
C8	0.0430 (13)	0.0374 (12)	0.0434 (13)	−0.0130 (10)	−0.0041 (10)	−0.0145 (10)
C9	0.0443 (14)	0.0476 (14)	0.0509 (15)	−0.0201 (11)	−0.0050 (11)	−0.0176 (12)
C10	0.0484 (15)	0.0482 (14)	0.0384 (13)	−0.0223 (12)	−0.0030 (11)	−0.0121 (11)
C11	0.082 (2)	0.0406 (15)	0.085 (2)	−0.0242 (15)	−0.0304 (18)	−0.0055 (15)
C12	0.181 (5)	0.083 (3)	0.115 (4)	−0.086 (3)	0.035 (3)	−0.027 (3)
C13	0.159 (5)	0.056 (2)	0.121 (4)	−0.018 (3)	0.045 (3)	−0.042 (2)
C14	0.0657 (19)	0.0586 (18)	0.0640 (19)	−0.0112 (15)	−0.0194 (15)	−0.0095 (15)

Geometric parameters (Å, º)

S—O2	1.493 (2)	C6—C7	1.378 (4)
S—C1	1.761 (2)	C6—H6	0.9300
S—C14	1.790 (3)	C8—C9	1.482 (3)
Cl—C4	1.747 (3)	C9—C10	1.506 (4)
O1—C8	1.372 (3)	C9—H9A	0.9700
O1—C7	1.374 (3)	C9—H9B	0.9700
O3—C10	1.331 (3)	C11—C13	1.469 (6)
O3—C11	1.472 (3)	C11—C12	1.513 (6)
O4—C10	1.196 (3)	C11—H11	0.9800
C1—C8	1.348 (3)	C12—H12A	0.9600
C1—C2	1.445 (3)	C12—H12B	0.9600
C2—C7	1.392 (3)	C12—H12C	0.9600
C2—C3	1.396 (3)	C13—H13A	0.9600
C3—C4	1.376 (4)	C13—H13B	0.9600
C3—H3	0.9300	C13—H13C	0.9600
C4—C5	1.390 (4)	C14—H14A	0.9600
C5—C6	1.378 (4)	C14—H14B	0.9600
C5—H5	0.9300	C14—H14C	0.9600
O2—S—C1	107.34 (12)	C10—C9—H9A	109.0
O2—S—C14	106.36 (14)	C8—C9—H9B	109.0
C1—S—C14	98.16 (14)	C10—C9—H9B	109.0
C8—O1—C7	106.25 (18)	H9A—C9—H9B	107.8
C10—O3—C11	117.8 (2)	O4—C10—O3	124.9 (2)
C8—C1—C2	107.3 (2)	O4—C10—C9	125.3 (2)
C8—C1—S	123.59 (19)	O3—C10—C9	109.8 (2)
C2—C1—S	129.04 (18)	C13—C11—O3	107.5 (3)
C7—C2—C3	119.7 (2)	C13—C11—C12	111.2 (4)
C7—C2—C1	104.6 (2)	O3—C11—C12	104.8 (3)
C3—C2—C1	135.7 (2)	C13—C11—H11	111.0
C4—C3—C2	116.3 (2)	O3—C11—H11	111.0
C4—C3—H3	121.8	C12—C11—H11	111.0
C2—C3—H3	121.8	C11—C12—H12A	109.5
C3—C4—C5	123.4 (2)	C11—C12—H12B	109.5
C3—C4—Cl	118.1 (2)	H12A—C12—H12B	109.5
C5—C4—Cl	118.5 (2)	C11—C12—H12C	109.5
C6—C5—C4	120.5 (2)	H12A—C12—H12C	109.5
C6—C5—H5	119.8	H12B—C12—H12C	109.5
C4—C5—H5	119.8	C11—C13—H13A	109.5
C5—C6—C7	116.4 (2)	C11—C13—H13B	109.5
C5—C6—H6	121.8	H13A—C13—H13B	109.5
C7—C6—H6	121.8	C11—C13—H13C	109.5
O1—C7—C6	125.7 (2)	H13A—C13—H13C	109.5
O1—C7—C2	110.7 (2)	H13B—C13—H13C	109.5
C6—C7—C2	123.6 (2)	S—C14—H14A	109.5
C1—C8—O1	111.2 (2)	S—C14—H14B	109.5

C1—C8—C9	132.5 (2)	H14A—C14—H14B	109.5
O1—C8—C9	116.3 (2)	S—C14—H14C	109.5
C8—C9—C10	113.1 (2)	H14A—C14—H14C	109.5
C8—C9—H9A	109.0	H14B—C14—H14C	109.5
O2—S—C1—C8	133.8 (2)	C3—C2—C7—O1	179.7 (2)
C14—S—C1—C8	−116.2 (2)	C1—C2—C7—O1	0.9 (3)
O2—S—C1—C2	−42.4 (3)	C3—C2—C7—C6	0.1 (4)
C14—S—C1—C2	67.7 (3)	C1—C2—C7—C6	−178.6 (2)
C8—C1—C2—C7	−0.3 (3)	C2—C1—C8—O1	−0.5 (3)
S—C1—C2—C7	176.39 (19)	S—C1—C8—O1	−177.37 (16)
C8—C1—C2—C3	−178.7 (3)	C2—C1—C8—C9	−178.7 (2)
S—C1—C2—C3	−2.1 (4)	S—C1—C8—C9	4.4 (4)
C7—C2—C3—C4	−0.9 (4)	C7—O1—C8—C1	1.1 (3)
C1—C2—C3—C4	177.4 (3)	C7—O1—C8—C9	179.6 (2)
C2—C3—C4—C5	1.1 (4)	C1—C8—C9—C10	75.3 (4)
C2—C3—C4—Cl	−178.30 (19)	O1—C8—C9—C10	−102.9 (2)
C3—C4—C5—C6	−0.5 (4)	C11—O3—C10—O4	4.8 (4)
Cl—C4—C5—C6	178.9 (2)	C11—O3—C10—C9	−177.6 (2)
C4—C5—C6—C7	−0.3 (4)	C8—C9—C10—O4	−13.6 (4)
C8—O1—C7—C6	178.3 (2)	C8—C9—C10—O3	168.8 (2)
C8—O1—C7—C2	−1.2 (3)	C10—O3—C11—C13	91.0 (4)
C5—C6—C7—O1	−179.0 (2)	C10—O3—C11—C12	−150.6 (3)
C5—C6—C7—C2	0.5 (4)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C13—H13*B*···*Cg*^{i}	0.96	2.78	3.515 (3)	134

Symmetry code: (i) *x*, *y*+1, *z*.