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ISSN 2414-3146

4-Chloronaphthalen-1-yl 4-methylbenzenesulfonate

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Received 12 June 2018
Accepted 18 June 2018

Edited by J. Simpson, University of Otago, New Zealand

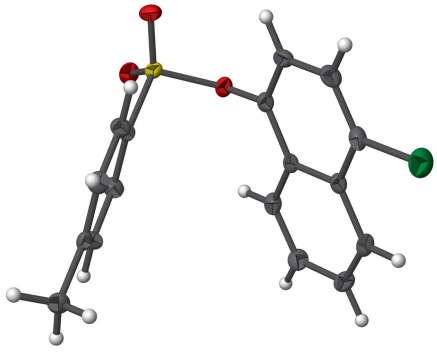
Keywords: tosylates; crystal structure; cross-coupling reactions.

CCDC reference: 1849905

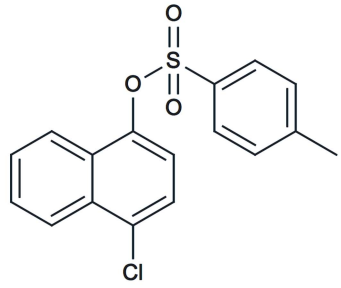
Structural data: full structural data are available from iucrdata.iucr.org

In the title compound, $C_{17}H_{13}ClO_3S$, the naphthalene ring system and the benzene ring of the tosylate substituent are inclined to one another by 55.32 (5)°. The crystal structure features weak intermolecular C—H···O hydrogen bonds, one of which forms inversion dimers. Additional C—H···O hydrogen bonds and weak Cl···Cl halogen bonds stack the molecules along the *b*-axis direction.

3D view



Chemical scheme



Structure description

Aryl tosylates are important intermediates in organic synthesis (Hugo *et al.*, 2014; Xu & Zhang, 2011; Grandane *et al.*, 2012). These compounds are easily prepared from cheap, readily available starting materials and are convenient to handle, stable crystalline solids (Bisz & Szostak, 2017*a*,*b*). These advantages make them ideal substrates in cross-coupling reactions (Piontek *et al.*, 2017; Ackermann *et al.*, 2006; Bisz *et al.*, 2018).

There is one independent molecule in the asymmetric unit of the title compound. The molecular structure is shown in Fig. 1. The molecule consists of a naphthalene ring system substituted at C1 by a 4-methylbenzenesulfonate group and with a chlorine substituent at C4. The dihedral angle between the naphthalene ring system (r.m.s. deviation = 0.0104 Å) and the benzene ring is 55.32 (5)°.

In the crystal, C2—H2···O2 hydrogen bonds, Table 1, form inversion dimers that enclose $R_2^2(14)$ rings. Additional C12—H12···O3 and C16—H16···O1 hydrogen bonds link adjacent dimers, forming double rows of molecules along the *bc* diagonal, Fig. 2. Weak Cl1···Cl1($1-x$, $1-y$, $-z$) halogen bonds [3.6817 (8) Å] also occur. These contacts combine to stack the molecules along the *b*-axis direction, Fig. 3.

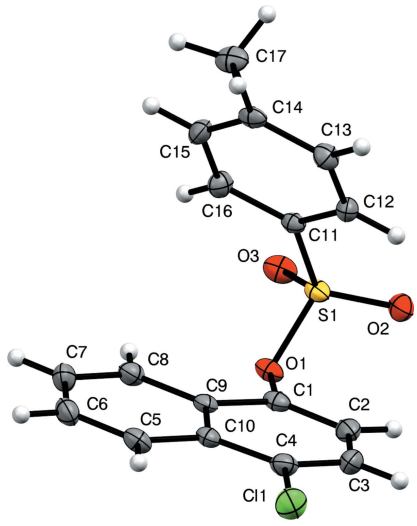


Figure 1
The molecular structure of the title compound with displacement ellipsoids drawn at the 50% probability level.

Synthesis and crystallization

The compound was synthesized according to a previously described procedure (Murai *et al.*, 2012). The crystallization was carried out in diethyl ether, previously stored over sodium. Diethyl ether (0.8 ml) was placed in a storage reaction vial (8 ml) provided with a silicone septum. The title compound was added to diethyl ether until a saturated solution was obtained. The solution was then heated to the boiling point of the solvent and the vial was screwed off with a silicone

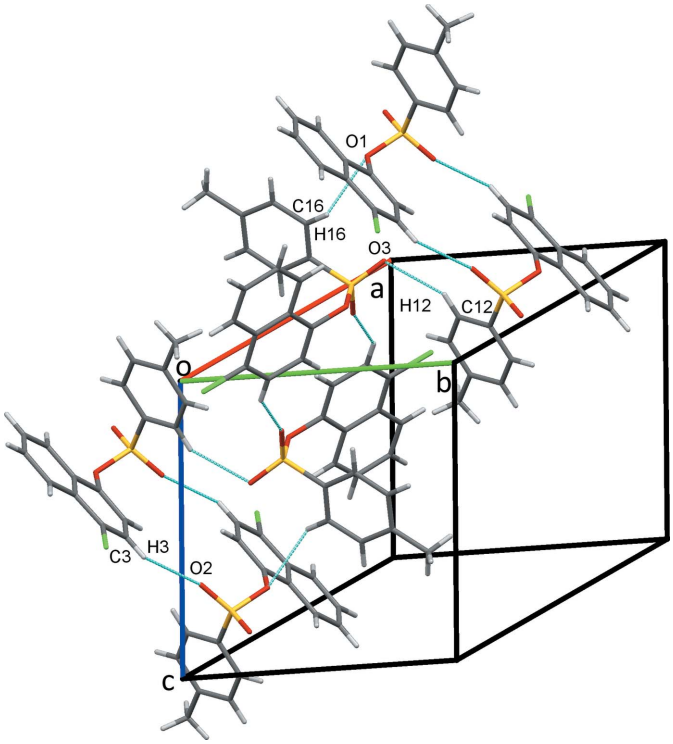


Figure 2
Rows of molecules along the *bc* diagonal. Hydrogen bonds are shown as blue dashed lines.

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

D—H···A	D—H	H···A	D···A	D—H···A
C3—H3···O2^{i}	0.93	2.53	3.2651 (19)	136
C12—H12···O3^{ii}	0.93	2.53	3.3050 (18)	141
C16—H16···O1^{iii}	0.93	2.53	3.3192 (18)	143

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

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{17}H_{13}ClO_3S$
M_r	332.78
Crystal system, space group	Monoclinic, $P2_1/c$
Temperature (K)	100
a, b, c (Å)	13.1301 (3), 11.9592 (2), 10.3738 (3)
β (°)	112.041 (3)
V (Å^3)	1509.90 (7)
Z	4
Radiation type	Mo $K\alpha$
μ (mm^{-1})	0.40
Crystal size (mm)	0.5 × 0.45 × 0.4
Data collection	
Diffractometer	Oxford Diffraction Xcalibur
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	10121, 2950, 2515
R_{int}	0.017
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.617
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.026, 0.072, 1.06
No. of reflections	2950
No. of parameters	200
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.40, −0.34

Computer programs: *CrysAlis CCD* (Oxford Diffraction, 2008), *SHELXT* (Sheldrick, 2015*a*), *SHELXL2014* (Sheldrick, 2015*b*) and *Mercury* (Macrae *et al.*, 2008).

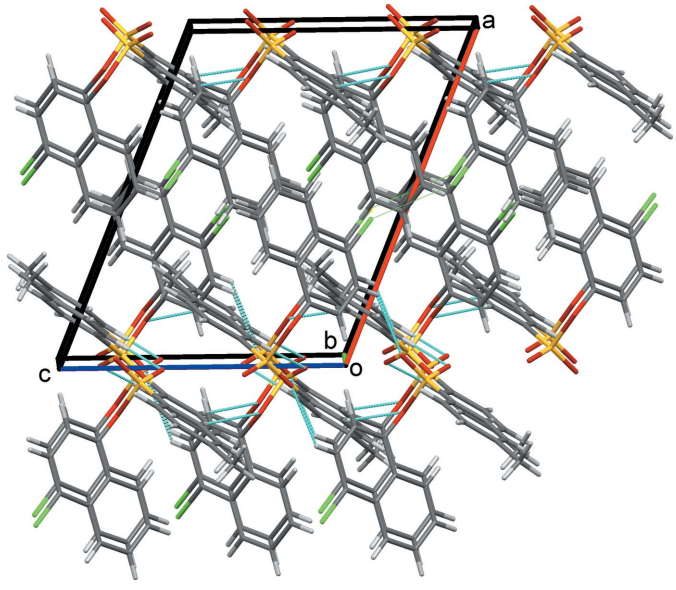


Figure 3
Overall packing viewed along the *b*-axis direction. Representative Cl···Cl contacts are shown as green dotted lines.

septum stopper. The resulting solution was then heated and left to stand in a refrigerator (−20°C), yielding colourless crystals suitable for data collection.

Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2.

Funding information

We gratefully acknowledge Narodowe Centrum Nauki (grant No. 2014/15/D/ST5/02731).

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full crystallographic data

IUCrData (2018). **3**, x180889 [https://doi.org/10.1107/S2414314618008891]

4-Chloronaphthalen-1-yl 4-methylbenzenesulfonate

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4-Chloronaphthalen-1-yl 4-methylbenzenesulfonate

Crystal data

$C_{17}H_{13}ClO_3S$
M_r = 332.78
Monoclinic, $P2_1/c$
a = 13.1301 (3) Å
b = 11.9592 (2) Å
c = 10.3738 (3) Å
β = 112.041 (3)°
V = 1509.90 (7) Å^3
Z = 4
$F(000)$ = 688
D_x = 1.464 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 10121 reflections
θ = 3.4–26.0°
μ = 0.40 mm^{-1}
T = 100 K
Irregular, colourless
0.5 × 0.45 × 0.4 mm

Data collection

Oxford Diffraction Xcalibur diffractometer
Radiation source: fine-focus sealed tube
Detector resolution: 1024 x 1024 with blocks 2 x 2 pixels mm^{-1}
ω scan
10121 measured reflections
2950 independent reflections
2515 reflections with $I > 2\sigma(I)$
R_{int} = 0.017
θ_{max} = 26.0°, θ_{min} = 3.4°
h = −16→16
k = −14→8
l = −12→12

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.026
$wR(F^2)$ = 0.072
S = 1.06
2950 reflections
200 parameters
0 restraints
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0373P)^2 + 0.4347P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.40 e Å^{-3}
$\Delta\rho_{min}$ = −0.34 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.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	y	z	U_{iso}*/U_{eq}
S1	0.03474 (3)	0.34571 (3)	0.29668 (4)	0.01694 (11)

Cl1	0.44738 (3)	0.56968 (3)	0.12070 (4)	0.02886 (12)
O1	0.13270 (8)	0.29057 (8)	0.26065 (11)	0.0190 (2)
O2	−0.04014 (9)	0.39892 (9)	0.17485 (11)	0.0238 (3)
O3	−0.00103 (9)	0.25441 (9)	0.35695 (11)	0.0234 (3)
C1	0.20522 (12)	0.36011 (12)	0.22530 (15)	0.0175 (3)
C2	0.16998 (13)	0.42354 (13)	0.10778 (15)	0.0213 (3)
H2	0.0963	0.4236	0.0490	0.026*
C3	0.24673 (13)	0.48882 (13)	0.07683 (15)	0.0226 (3)
H3	0.2236	0.5340	−0.0019	0.027*
C4	0.35509 (13)	0.48629 (13)	0.16182 (16)	0.0204 (3)
C5	0.50724 (12)	0.41223 (12)	0.37265 (16)	0.0199 (3)
H5	0.5592	0.4534	0.3518	0.024*
C6	0.53989 (13)	0.34678 (13)	0.48863 (17)	0.0227 (3)
H6	0.6138	0.3436	0.5461	0.027*
C7	0.46217 (13)	0.28373 (13)	0.52221 (17)	0.0232 (3)
H7	0.4852	0.2399	0.6021	0.028*
C8	0.35365 (13)	0.28673 (12)	0.43820 (16)	0.0199 (3)
H8	0.3033	0.2444	0.4608	0.024*
C9	0.31700 (12)	0.35393 (12)	0.31654 (15)	0.0162 (3)
C10	0.39536 (12)	0.41840 (12)	0.28356 (15)	0.0169 (3)
C11	0.10147 (11)	0.44694 (12)	0.42155 (14)	0.0144 (3)
C12	0.09851 (12)	0.55841 (12)	0.38264 (15)	0.0178 (3)
H12	0.0603	0.5802	0.2910	0.021*
C13	0.15371 (12)	0.63630 (12)	0.48313 (16)	0.0195 (3)
H13	0.1521	0.7112	0.4584	0.023*
C14	0.21162 (11)	0.60499 (13)	0.62050 (15)	0.0174 (3)
C15	0.21289 (12)	0.49281 (13)	0.65634 (15)	0.0202 (3)
H15	0.2511	0.4709	0.7479	0.024*
C16	0.15807 (13)	0.41343 (13)	0.55780 (15)	0.0196 (3)
H16	0.1592	0.3386	0.5826	0.023*
C17	0.26993 (13)	0.69201 (14)	0.72763 (17)	0.0246 (4)
H17A	0.3185	0.7342	0.6965	0.037*
H17B	0.3116	0.6560	0.8141	0.037*
H17C	0.2169	0.7413	0.7406	0.037*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.01306 (18)	0.01679 (19)	0.0198 (2)	−0.00117 (14)	0.00481 (15)	−0.00584 (14)
Cl1	0.0303 (2)	0.0307 (2)	0.0308 (2)	0.00058 (17)	0.01738 (19)	0.00840 (17)
O1	0.0171 (5)	0.0170 (5)	0.0246 (6)	−0.0006 (4)	0.0096 (5)	−0.0057 (4)
O2	0.0168 (5)	0.0287 (6)	0.0202 (6)	0.0022 (5)	0.0003 (5)	−0.0063 (5)
O3	0.0215 (6)	0.0195 (6)	0.0312 (6)	−0.0065 (4)	0.0123 (5)	−0.0071 (5)
C1	0.0184 (7)	0.0157 (7)	0.0203 (8)	−0.0004 (6)	0.0093 (6)	−0.0057 (6)
C2	0.0192 (8)	0.0254 (8)	0.0166 (7)	0.0034 (6)	0.0037 (6)	−0.0050 (6)
C3	0.0273 (8)	0.0244 (8)	0.0159 (7)	0.0077 (7)	0.0079 (7)	0.0026 (6)
C4	0.0242 (8)	0.0200 (8)	0.0213 (8)	0.0022 (6)	0.0136 (7)	−0.0002 (6)
C5	0.0181 (7)	0.0181 (8)	0.0264 (8)	0.0017 (6)	0.0116 (7)	−0.0015 (6)

C6	0.0181 (8)	0.0228 (8)	0.0251 (8)	0.0056 (6)	0.0057 (7)	0.0002 (7)
C7	0.0240 (8)	0.0229 (8)	0.0226 (8)	0.0075 (7)	0.0087 (7)	0.0062 (7)
C8	0.0223 (8)	0.0174 (7)	0.0237 (8)	0.0028 (6)	0.0126 (7)	0.0010 (6)
C9	0.0178 (7)	0.0148 (7)	0.0175 (7)	0.0033 (6)	0.0084 (6)	−0.0031 (6)
C10	0.0192 (7)	0.0152 (7)	0.0181 (7)	0.0031 (6)	0.0090 (6)	−0.0025 (6)
C11	0.0126 (7)	0.0146 (7)	0.0163 (7)	−0.0010 (5)	0.0056 (6)	−0.0038 (6)
C12	0.0189 (7)	0.0181 (7)	0.0150 (7)	0.0033 (6)	0.0047 (6)	0.0013 (6)
C13	0.0218 (8)	0.0142 (7)	0.0230 (8)	−0.0003 (6)	0.0088 (7)	0.0002 (6)
C14	0.0123 (7)	0.0215 (8)	0.0205 (8)	−0.0026 (6)	0.0085 (6)	−0.0057 (6)
C15	0.0209 (8)	0.0248 (8)	0.0133 (7)	0.0008 (6)	0.0047 (6)	0.0013 (6)
C16	0.0243 (8)	0.0161 (7)	0.0190 (7)	−0.0001 (6)	0.0089 (7)	0.0023 (6)
C17	0.0193 (8)	0.0290 (9)	0.0248 (8)	−0.0070 (7)	0.0075 (7)	−0.0108 (7)

Geometric parameters (Å, º)

S1—O3	1.4226 (11)	C7—H7	0.9300
S1—O2	1.4270 (11)	C8—C9	1.419 (2)
S1—O1	1.6093 (10)	C8—H8	0.9300
S1—C11	1.7491 (14)	C9—C10	1.426 (2)
Cl1—C4	1.7415 (16)	C11—C16	1.386 (2)
O1—C1	1.4124 (17)	C11—C12	1.389 (2)
C1—C2	1.361 (2)	C12—C13	1.383 (2)
C1—C9	1.419 (2)	C12—H12	0.9300
C2—C3	1.404 (2)	C13—C14	1.391 (2)
C2—H2	0.9300	C13—H13	0.9300
C3—C4	1.364 (2)	C14—C15	1.391 (2)
C3—H3	0.9300	C14—C17	1.505 (2)
C4—C10	1.426 (2)	C15—C16	1.383 (2)
C5—C6	1.363 (2)	C15—H15	0.9300
C5—C10	1.414 (2)	C16—H16	0.9300
C5—H5	0.9300	C17—H17A	0.9600
C6—C7	1.414 (2)	C17—H17B	0.9600
C6—H6	0.9300	C17—H17C	0.9600
C7—C8	1.363 (2)		
O3—S1—O2	120.01 (7)	C1—C9—C8	122.92 (14)
O3—S1—O1	102.24 (6)	C1—C9—C10	118.07 (13)
O2—S1—O1	108.79 (6)	C8—C9—C10	119.01 (13)
O3—S1—C11	111.22 (7)	C5—C10—C4	123.91 (14)
O2—S1—C11	109.54 (7)	C5—C10—C9	118.76 (13)
O1—S1—C11	103.51 (6)	C4—C10—C9	117.33 (13)
C1—O1—S1	119.63 (9)	C16—C11—C12	121.29 (13)
C2—C1—O1	121.67 (13)	C16—C11—S1	118.87 (11)
C2—C1—C9	123.06 (14)	C12—C11—S1	119.83 (11)
O1—C1—C9	115.18 (13)	C13—C12—C11	118.54 (13)
C1—C2—C3	118.87 (14)	C13—C12—H12	120.7
C1—C2—H2	120.6	C11—C12—H12	120.7
C3—C2—H2	120.6	C12—C13—C14	121.41 (14)

C4—C3—C2	120.26 (14)	C12—C13—H13	119.3
C4—C3—H3	119.9	C14—C13—H13	119.3
C2—C3—H3	119.9	C15—C14—C13	118.72 (13)
C3—C4—C10	122.37 (14)	C15—C14—C17	121.19 (14)
C3—C4—Cl1	118.80 (12)	C13—C14—C17	120.08 (14)
C10—C4—Cl1	118.83 (12)	C16—C15—C14	120.93 (14)
C6—C5—C10	120.84 (14)	C16—C15—H15	119.5
C6—C5—H5	119.6	C14—C15—H15	119.5
C10—C5—H5	119.6	C15—C16—C11	119.10 (14)
C5—C6—C7	120.46 (14)	C15—C16—H16	120.4
C5—C6—H6	119.8	C11—C16—H16	120.4
C7—C6—H6	119.8	C14—C17—H17A	109.5
C8—C7—C6	120.43 (14)	C14—C17—H17B	109.5
C8—C7—H7	119.8	H17A—C17—H17B	109.5
C6—C7—H7	119.8	C14—C17—H17C	109.5
C7—C8—C9	120.50 (14)	H17A—C17—H17C	109.5
C7—C8—H8	119.8	H17B—C17—H17C	109.5
C9—C8—H8	119.8		

O3—S1—O1—C1	−166.82 (10)	C3—C4—C10—C9	−1.6 (2)
O2—S1—O1—C1	65.27 (11)	Cl1—C4—C10—C9	178.03 (10)
C11—S1—O1—C1	−51.16 (11)	C1—C9—C10—C5	−179.34 (13)
S1—O1—C1—C2	−65.00 (16)	C8—C9—C10—C5	0.6 (2)
S1—O1—C1—C9	118.30 (12)	C1—C9—C10—C4	1.07 (19)
O1—C1—C2—C3	−178.46 (12)	C8—C9—C10—C4	−178.95 (13)
C9—C1—C2—C3	−2.0 (2)	O3—S1—C11—C16	30.20 (14)
C1—C2—C3—C4	1.5 (2)	O2—S1—C11—C16	165.20 (12)
C2—C3—C4—C10	0.3 (2)	O1—S1—C11—C16	−78.90 (13)
C2—C3—C4—Cl1	−179.32 (11)	O3—S1—C11—C12	−150.85 (12)
C10—C5—C6—C7	−0.1 (2)	O2—S1—C11—C12	−15.85 (14)
C5—C6—C7—C8	0.7 (2)	O1—S1—C11—C12	100.05 (12)
C6—C7—C8—C9	−0.6 (2)	C16—C11—C12—C13	0.1 (2)
C2—C1—C9—C8	−179.28 (13)	S1—C11—C12—C13	−178.83 (11)
O1—C1—C9—C8	−2.6 (2)	C11—C12—C13—C14	0.2 (2)
C2—C1—C9—C10	0.7 (2)	C12—C13—C14—C15	−0.4 (2)
O1—C1—C9—C10	177.35 (11)	C12—C13—C14—C17	−179.41 (14)
C7—C8—C9—C1	179.90 (14)	C13—C14—C15—C16	0.2 (2)
C7—C8—C9—C10	−0.1 (2)	C17—C14—C15—C16	179.22 (14)
C6—C5—C10—C4	179.00 (14)	C14—C15—C16—C11	0.1 (2)
C6—C5—C10—C9	−0.6 (2)	C12—C11—C16—C15	−0.3 (2)
C3—C4—C10—C5	178.87 (15)	S1—C11—C16—C15	178.66 (11)
Cl1—C4—C10—C5	−1.5 (2)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C3—H3···O2^{i}	0.93	2.53	3.2651 (19)	136

C12—H12···O3^{ii}	0.93	2.53	3.3050 (18)	141
C16—H16···O1^{iii}	0.93	2.53	3.3192 (18)	143

Symmetry codes: (i) $-x$, $-y+1$, $-z$; (ii) $-x$, $y+1/2$, $-z+1/2$; (iii) x, $-y+1/2$, $z+1/2$.