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4-{2-[(*E*)-Cyclopentylidene]hydrazin-1-yl}benzenesulfonamide

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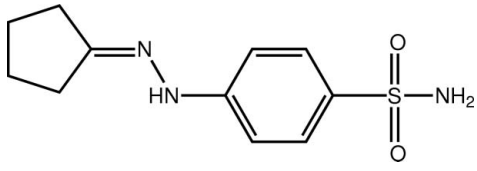
Key indicators: single-crystal X-ray study; T = 100 K; mean σ(C–C) = 0.004 Å; R factor = 0.056; wR factor = 0.123; data-to-parameter ratio = 16.7.

The title molecule, $C_{11}H_{15}N_3O_2S$, features a five-membered ring which is twisted about the middle CH_2—CH_2 bond. The benzene ring is inclined with respect to the imine residue [C—N—N—C torsion angle = 165.4 (2)°]. Supramolecular layers in the *bc* plane are formed by hydrogen bonds between the amine H atoms and sulfonamide O and imine N atoms, as well as by a weak hydrazine H-atom intermolecular interaction with the second sulfonamide O atom.

Related literature

For background to the biological applications of related sulfonamides, see: Al-Saadi *et al.* (2008). For related structures, see: Asiri *et al.* (2011, 2012).



Experimental

Crystal data

$C_{11}H_{15}N_3O_2S$
M_r = 253.32
Monoclinic, $P2_1/c$
a = 14.1173 (14) Å
b = 5.2038 (5) Å
c = 16.4239 (19) Å
β = 94.019 (10)°
V = 1203.6 (2) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.26 mm^{-1}
T = 100 K
0.35 × 0.05 × 0.02 mm

Data collection

Agilent SuperNova Dual diffractometer with an Atlas detector
Absorption correction: multi-scan (*CrysAlis PRO*; Agilent, 2011)
T_{min} = 0.914, T_{max} = 0.995
4782 measured reflections
2768 independent reflections
2007 reflections with $I > 2\sigma(I)$
R_{int} = 0.044

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.056
$wR(F^2)$ = 0.123
S = 1.03
2768 reflections
166 parameters
3 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.32 e Å^{-3}
$\Delta\rho_{min}$ = −0.38 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···O1^{i}	0.88 (1)	2.02 (1)	2.869 (3)	164 (3)
N1—H2···N3^{ii}	0.88 (1)	2.13 (1)	2.993 (3)	166 (3)
N2—H3···O2^{iii}	0.88 (1)	2.36 (1)	3.220 (3)	166 (3)

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

Data collection: *CrysAlis PRO* (Agilent, 2011); cell refinement: *CrysAlis PRO*; data reduction: *CrysAlis PRO*; 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, 2006); software used to prepare material for publication: *publCIF* (Westrip, 2010).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: JJ2126).

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supplementary materials

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4-{2-[(*E*)-Cyclopentylidene]hydrazin-1-yl}benzenesulfonamide

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Comment

Sulfonamides related to the title compound, 4-(*N*-cyclopentylidenehydrazino)benzensulfonamide (I), have been shown to display biological activity (Al-Saadi *et al.*, 2008). In continuation of structural studies of these sulfonamides (Asiri *et al.*, 2011; Asiri *et al.*, 2012), the crystal and molecular structure of (I) is reported herein.

In (I), Fig. 1, the five-membered ring is twisted about the C9—C10 bond. The imine residue is not co-planar with the benzene ring with a twist apparent about the N2—C4 bond; the C4—N2—N3—C7 torsion angle is 165.4 (2)°.

The crystal packing is dominated by N—H···O and N—H···N hydrogen bonds. The amino-H atoms form hydrogen bonds with a sulfonamide-O and imine-N atoms. The hydrazine-H atom forms a weak intermolecular interaction with the second sulfonamide-O atom, Table 1. The result is the formation of supramolecular layers in the *bc* plane, Fig. 2. Layers stack along the *a* axis with no specific intermolecular interactions between them.

Experimental

Cyclopentanone (0.84 g, 10 mmol) in ethanol was refluxed with *p*-sulfamylphenylhydrazine (2.2 g, 10 mmol) for 1 h. The reaction mixture was cooled and the precipitated product was collected by filtration, washed with ethanol, dried and recrystallized from its ethanol solution. Yield: 78%. *M*. pt: 475–477 K.

Refinement

Carbon-bound H-atoms were placed in calculated positions [C—H = 0.95 to 0.99 Å, U_{iso}(H) = 1.2U_{eq}(C)] and were included in the refinement in the riding model approximation. The N-bound H-atoms were located in a difference Fourier map and were refined with a distance restraint of N—H = 0.88±0.01 Å; their U_{iso} values were refined.

Computing details

Data collection: *CrysAlis PRO* (Agilent, 2011); cell refinement: *CrysAlis PRO* (Agilent, 2011); data reduction: *CrysAlis PRO* (Agilent, 2011); 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, 2006); software used to prepare material for publication: *publCIF* (Westrip, 2010).

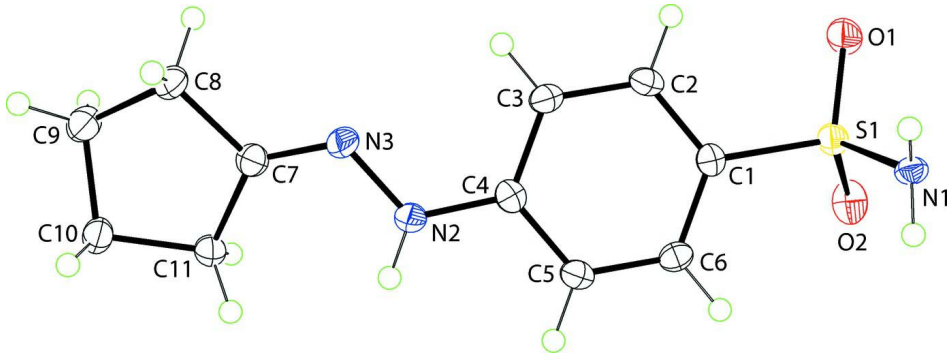

Figure 1

The molecular structure of (I) showing the atom-labelling scheme and displacement ellipsoids at the 50% probability level.

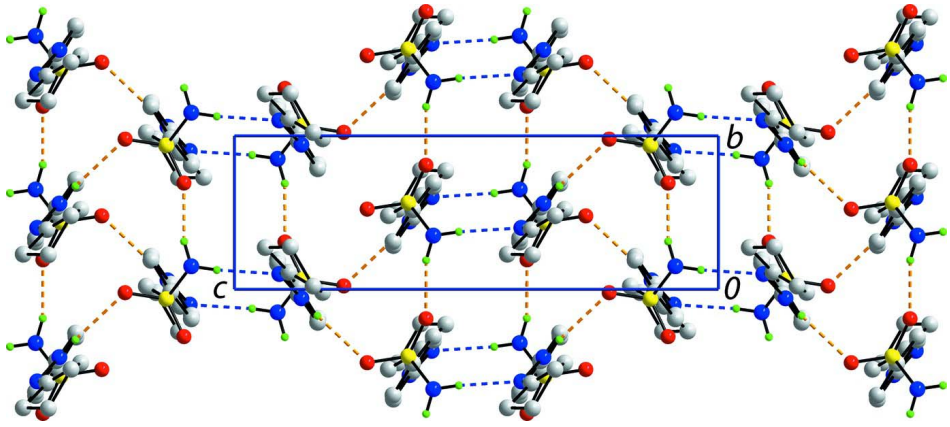

Figure 2

A view of the supramolecular layer in the *bc* plane in (I). The N—H···O and N—H···N hydrogen bonds are shown as orange and blue dashed lines, respectively.

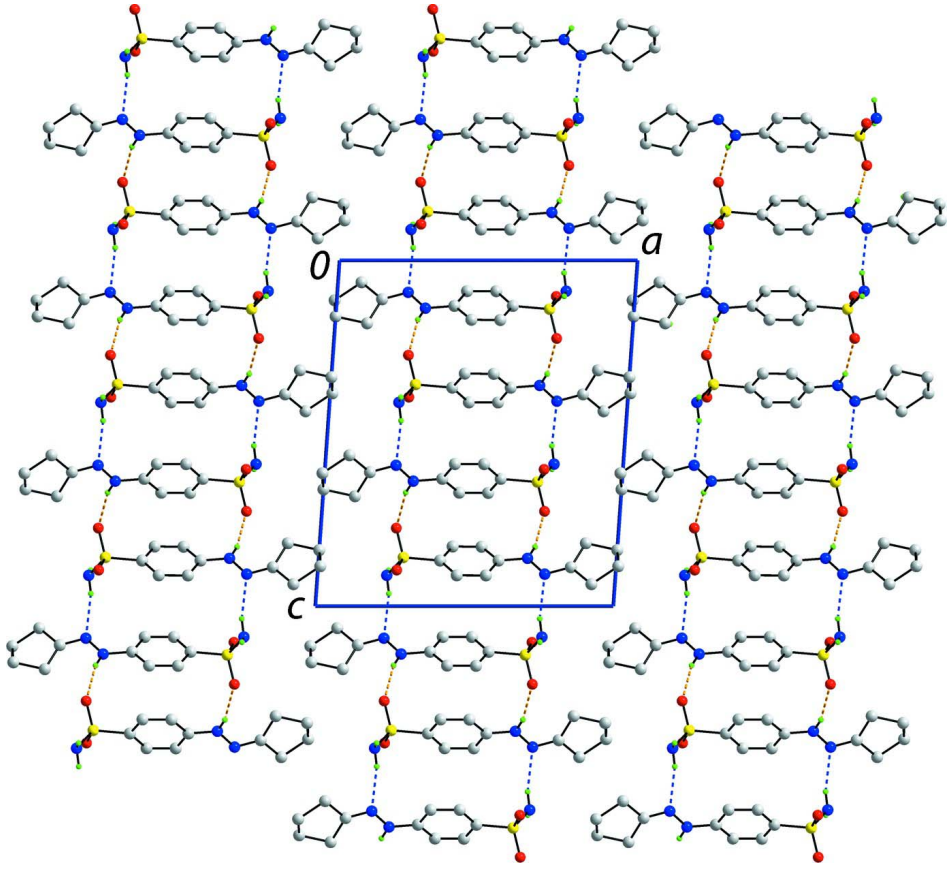

Figure 3

A view in projection down the *b* axis of the unit-cell contents of (I) showing the stacking of layers along the *a* axis. The N—H···O and N—H···N interactions are shown as orange and blue dashed lines, respectively.

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Crystal data

$C_{11}H_{15}N_3O_2S$
$M_r = 253.32$
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
$a = 14.1173\ (14)$ Å
$b = 5.2038\ (5)$ Å
$c = 16.4239\ (19)$ Å
$\beta = 94.019\ (10)°$
$V = 1203.6\ (2)$ Å^3
$Z = 4$
$F(000) = 536$
$D_x = 1.398$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 1078 reflections
$\theta = 2.5–27.5°$
$\mu = 0.26$ mm^{-1}
$T = 100$ K
Plate, colourless
0.35 × 0.05 × 0.02 mm

Data collection

Agilent SuperNova Dual diffractometer with an Atlas detector
Radiation source: SuperNova (Mo) X-ray Source
Mirror monochromator
Detector resolution: 10.4041 pixels mm^{-1}
ω scan
Absorption correction: multi-scan (*CrysAlis PRO*; Agilent, 2011)
$T_{min} = 0.914$, $T_{max} = 0.995$
4782 measured reflections
2768 independent reflections
2007 reflections with $I > 2\sigma(I)$
$R_{int} = 0.044$

θ_{max} = 27.6°, θ_{min} = 2.5°
h = −18→11
k = −4→6
l = −21→21

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.056
$wR(F^2)$ = 0.123
S = 1.03
2768 reflections
166 parameters
3 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0372P)^2 + 0.8414P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.32 e Å^{-3}
$\Delta\rho_{min}$ = −0.38 e Å^{-3}

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

	x	y	z	U_{iso}*/U_{eq}
S1	0.28398 (5)	0.43647 (12)	0.35890 (4)	0.01924 (18)
O1	0.26446 (13)	0.1919 (3)	0.39559 (13)	0.0304 (5)
O2	0.25331 (13)	0.4780 (4)	0.27417 (11)	0.0276 (5)
N1	0.23242 (16)	0.6501 (4)	0.41081 (14)	0.0185 (5)
H1	0.238 (2)	0.811 (2)	0.3960 (17)	0.028 (8)*
H2	0.244 (2)	0.626 (6)	0.4636 (7)	0.042 (10)*
N2	0.70084 (16)	0.5585 (5)	0.36168 (14)	0.0238 (5)
H3	0.7234 (19)	0.672 (4)	0.3286 (14)	0.025 (8)*
N3	0.76230 (15)	0.4003 (4)	0.40739 (13)	0.0210 (5)
C1	0.40782 (17)	0.4817 (5)	0.36814 (15)	0.0172 (5)
C2	0.46722 (18)	0.3075 (5)	0.41081 (15)	0.0192 (6)
H2A	0.4406	0.1709	0.4401	0.023*
C3	0.56513 (18)	0.3314 (5)	0.41102 (16)	0.0199 (6)
H3A	0.6054	0.2114	0.4400	0.024*
C4	0.60411 (18)	0.5343 (5)	0.36816 (15)	0.0182 (5)
C5	0.54389 (18)	0.7151 (5)	0.32815 (16)	0.0193 (6)
H5	0.5703	0.8571	0.3012	0.023*
C6	0.44676 (18)	0.6888 (5)	0.32749 (15)	0.0187 (6)
H6	0.4063	0.8108	0.2996	0.022*
C7	0.84829 (18)	0.3891 (5)	0.38745 (16)	0.0202 (6)
C8	0.92115 (18)	0.2283 (6)	0.43640 (17)	0.0247 (6)
H8A	0.8971	0.0522	0.4445	0.030*
H8B	0.9378	0.3070	0.4904	0.030*
C9	1.00726 (19)	0.2246 (5)	0.38442 (17)	0.0252 (6)
H9A	1.0673	0.2150	0.4193	0.030*
H9B	1.0041	0.0767	0.3464	0.030*
C10	1.00007 (18)	0.4790 (5)	0.33771 (17)	0.0248 (6)
H10A	1.0339	0.4684	0.2870	0.030*
H10B	1.0275	0.6213	0.3717	0.030*
C11	0.89299 (18)	0.5202 (5)	0.31788 (16)	0.0220 (6)
H11A	0.8771	0.7055	0.3160	0.026*
H11B	0.8718	0.4405	0.2650	0.026*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0185 (3)	0.0147 (3)	0.0246 (4)	−0.0019 (3)	0.0025 (3)	−0.0034 (3)
O1	0.0247 (11)	0.0141 (9)	0.0531 (15)	−0.0038 (8)	0.0079 (10)	−0.0002 (9)
O2	0.0233 (10)	0.0373 (12)	0.0215 (10)	−0.0024 (9)	−0.0031 (8)	−0.0091 (9)
N1	0.0220 (12)	0.0134 (11)	0.0203 (13)	0.0014 (9)	0.0032 (10)	0.0016 (10)
N2	0.0204 (12)	0.0261 (13)	0.0255 (13)	0.0024 (11)	0.0054 (9)	0.0109 (11)
N3	0.0200 (12)	0.0238 (12)	0.0191 (12)	0.0026 (10)	0.0003 (9)	0.0055 (10)
C1	0.0169 (13)	0.0186 (13)	0.0162 (13)	−0.0002 (10)	0.0021 (10)	−0.0031 (10)
C2	0.0242 (14)	0.0160 (13)	0.0180 (14)	−0.0013 (11)	0.0063 (11)	0.0008 (11)
C3	0.0232 (14)	0.0176 (13)	0.0189 (14)	0.0024 (11)	0.0014 (11)	0.0027 (11)
C4	0.0187 (13)	0.0201 (13)	0.0162 (13)	−0.0010 (11)	0.0029 (10)	−0.0026 (11)
C5	0.0229 (14)	0.0166 (13)	0.0189 (14)	−0.0014 (11)	0.0050 (11)	0.0004 (11)
C6	0.0230 (14)	0.0154 (12)	0.0175 (13)	0.0016 (11)	0.0001 (10)	−0.0009 (11)
C7	0.0201 (14)	0.0214 (14)	0.0190 (14)	−0.0013 (11)	−0.0006 (10)	0.0003 (11)
C8	0.0210 (14)	0.0310 (15)	0.0218 (15)	0.0034 (12)	−0.0003 (11)	0.0083 (12)
C9	0.0239 (15)	0.0278 (15)	0.0237 (15)	0.0043 (12)	0.0009 (11)	0.0027 (12)
C10	0.0179 (14)	0.0315 (16)	0.0248 (15)	0.0021 (12)	0.0003 (11)	0.0053 (12)
C11	0.0191 (14)	0.0257 (15)	0.0211 (14)	−0.0011 (11)	0.0009 (11)	0.0058 (12)

Geometric parameters (Å, º)

S1—O2	1.4444 (19)	C5—C6	1.377 (4)
S1—O1	1.4430 (19)	C5—H5	0.9500
S1—N1	1.606 (2)	C6—H6	0.9500
S1—C1	1.760 (3)	C7—C8	1.513 (4)
N1—H1	0.878 (10)	C7—C11	1.507 (3)
N1—H2	0.879 (10)	C8—C9	1.534 (4)
N2—N3	1.379 (3)	C8—H8A	0.9900
N2—C4	1.383 (3)	C8—H8B	0.9900
N2—H3	0.875 (10)	C9—C10	1.530 (4)
N3—C7	1.281 (3)	C9—H9A	0.9900
C1—C2	1.391 (4)	C9—H9B	0.9900
C1—C6	1.400 (4)	C10—C11	1.539 (3)
C2—C3	1.388 (4)	C10—H10A	0.9900
C2—H2A	0.9500	C10—H10B	0.9900
C3—C4	1.403 (4)	C11—H11A	0.9900
C3—H3A	0.9500	C11—H11B	0.9900
C4—C5	1.401 (4)		
O2—S1—O1	118.77 (12)	C5—C6—H6	120.1
O2—S1—N1	106.95 (12)	C1—C6—H6	120.1
O1—S1—N1	106.37 (12)	N3—C7—C8	120.7 (2)
O2—S1—C1	106.97 (12)	N3—C7—C11	128.9 (2)
O1—S1—C1	107.43 (12)	C8—C7—C11	110.4 (2)
N1—S1—C1	110.25 (12)	C7—C8—C9	104.3 (2)
S1—N1—H1	117.4 (19)	C7—C8—H8A	110.9
S1—N1—H2	111 (2)	C9—C8—H8A	110.9
H1—N1—H2	113 (3)	C7—C8—H8B	110.9

N3—N2—C4	119.4 (2)	C9—C8—H8B	110.9
N3—N2—H3	119.8 (19)	H8A—C8—H8B	108.9
C4—N2—H3	120.8 (19)	C10—C9—C8	103.9 (2)
C7—N3—N2	117.3 (2)	C10—C9—H9A	111.0
C2—C1—C6	119.9 (2)	C8—C9—H9A	111.0
C2—C1—S1	121.1 (2)	C10—C9—H9B	111.0
C6—C1—S1	118.9 (2)	C8—C9—H9B	111.0
C3—C2—C1	120.5 (2)	H9A—C9—H9B	109.0
C3—C2—H2A	119.7	C9—C10—C11	104.9 (2)
C1—C2—H2A	119.7	C9—C10—H10A	110.8
C2—C3—C4	119.5 (2)	C11—C10—H10A	110.8
C2—C3—H3A	120.3	C9—C10—H10B	110.8
C4—C3—H3A	120.3	C11—C10—H10B	110.8
N2—C4—C5	118.2 (2)	H10A—C10—H10B	108.9
N2—C4—C3	122.1 (2)	C7—C11—C10	103.5 (2)
C5—C4—C3	119.6 (2)	C7—C11—H11A	111.1
C6—C5—C4	120.6 (2)	C10—C11—H11A	111.1
C6—C5—H5	119.7	C7—C11—H11B	111.1
C4—C5—H5	119.7	C10—C11—H11B	111.1
C5—C6—C1	119.8 (2)	H11A—C11—H11B	109.0
C4—N2—N3—C7	165.4 (2)	N2—C4—C5—C6	175.1 (2)
O2—S1—C1—C2	−133.3 (2)	C3—C4—C5—C6	−2.9 (4)
O1—S1—C1—C2	−4.8 (2)	C4—C5—C6—C1	0.9 (4)
N1—S1—C1—C2	110.7 (2)	C2—C1—C6—C5	1.7 (4)
O2—S1—C1—C6	42.8 (2)	S1—C1—C6—C5	−174.46 (19)
O1—S1—C1—C6	171.4 (2)	N2—N3—C7—C8	177.6 (2)
N1—S1—C1—C6	−73.1 (2)	N2—N3—C7—C11	−2.2 (4)
C6—C1—C2—C3	−2.4 (4)	N3—C7—C8—C9	169.5 (3)
S1—C1—C2—C3	173.71 (19)	C11—C7—C8—C9	−10.6 (3)
C1—C2—C3—C4	0.4 (4)	C7—C8—C9—C10	28.9 (3)
N3—N2—C4—C5	173.1 (2)	C8—C9—C10—C11	−36.8 (3)
N3—N2—C4—C3	−8.9 (4)	N3—C7—C11—C10	168.0 (3)
C2—C3—C4—N2	−175.7 (2)	C8—C7—C11—C10	−11.8 (3)
C2—C3—C4—C5	2.3 (4)	C9—C10—C11—C7	29.8 (3)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···O1^{i}	0.88 (1)	2.02 (1)	2.869 (3)	164 (3)
N1—H2···N3^{ii}	0.88 (1)	2.13 (1)	2.993 (3)	166 (3)
N2—H3···O2^{iii}	0.88 (1)	2.36 (1)	3.220 (3)	166 (3)

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