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4-(2,7-Dimethyl-4-oxo-1,3-thiazolo[4,5-*d*]-pyridazin-5-yl)benzenesulfonamide

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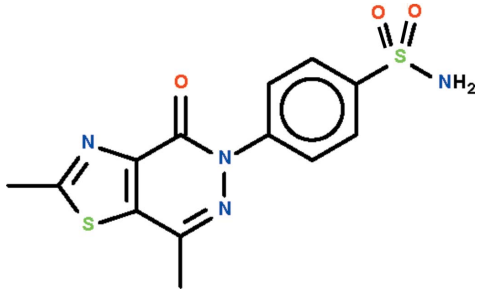
Key indicators: single-crystal X-ray study; T = 100 K; mean σ(C–C) = 0.003 Å; R factor = 0.034; wR factor = 0.097; data-to-parameter ratio = 15.9.

The thiazole–pyridazine fused-ring system of the title compound, $C_{13}H_{12}N_4O_3S_2$, is approximately planar (r.m.s. deviation = 0.037 Å); the benzene ring connected to the fused-ring system through the N atom is twisted by 39.3 (1)°. The amine group uses an H atom to form a hydrogen bond to the ketonic O atom of an inversion-related molecule to generate a dimer; adjacent dimers are linked by an N—H···O hydrogen bond to form a linear chain.

Related literature

For background to related compounds, see: Makki & Faidallah (1996).



Experimental

Crystal data

$C_{13}H_{12}N_4O_3S_2$
M_r = 336.39
Monoclinic, $P2_1/c$
a = 12.6048 (10) Å
b = 13.2273 (10) Å
c = 8.9703 (7) Å
β = 102.242 (1)°
V = 1461.6 (2) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.38 mm^{-1}
T = 100 K
0.20 × 0.15 × 0.15 mm

Data collection

Bruker SMART APEX diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.928, T_{max} = 0.945
9962 measured reflections
3333 independent reflections
2888 reflections with $I > 2\sigma(I)$
R_{int} = 0.028

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.034
$wR(F^2)$ = 0.097
S = 1.06
3333 reflections
209 parameters
2 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.39 e Å^{-3}
$\Delta\rho_{min}$ = −0.53 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N4—H1···O1^{i}	0.87 (1)	2.06 (1)	2.922 (2)	169 (2)
N4—H2···O2^{ii}	0.88 (1)	2.38 (2)	3.090 (2)	139 (2)

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

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: *X-SEED* (Barbour, 2001); software used to prepare material for publication: *publCIF* (Westrip, 2010).

We thank King Abdul Aziz University and the University of Malaya for supporting this study.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: XU5226).

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supporting information

Acta Cryst. (2011). E**67**, o1665 [doi:10.1107/S1600536811021271]

4-(2,7-Dimethyl-4-oxo-1,3-thiazolo[4,5-*d*]pyridazin-5-yl)benzenesulfonamide

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S1. Comment

This compound belongs to a class of tricyclic compounds posessing high antibacterial activity that are synthesized by reacting an aryl hydrazine with a thiazole that bears acetyl and carboxyl substituents on adjacent carbon atoms (Makki & Faidallah, 1996). A sulfonamido unit in the benzene ring of phenyl hydrazine should improved the activity. The thiazole–pyridazine fused-ring of $C_{13}H_{12}N_4O_3S_2$ (Scheme I, Fig. 1) is planar; the benzene ring that bears the sulfonamido unit is twisted by 39.3 (1)°. The amino group uses an H atom to form a hydrogen bond to the ketonic O atom of an inversion-related molecule to generate a dimer (Fig. 2); adjacent dimers are linked by a weaker N–H···O hydrogen bond to form a linear chain (Table 1).

S2. Experimental

Ethyl 5-acetyl-2-methylthiazole-4-carboxylate (0.40 g, 0.002 mol) in ethanol (25 ml) was heated with *p*-sulfonamido-phenyl hydrazine hydrochloride (0.49 g, 0.002 mol) for 2 h. The pyridazine that separated was collected and recrystallized from ethanol.

S3. Refinement

Carbon-bound H-atoms were placed in calculated positions (C–H 0.95 to 0.98 Å) and were included in the refinement in the riding model approximation, with U(H) set to 1.2 to 1.5U_{eq}(C). The amino H-atoms were located in a difference Fourier map, and were refined with a distance restraint of N–H 0.88±0.01 Å; temperature factors were refined. Omitted because of bad disagreement were (12 2 3), (1 0 0) and (-1 2 1).

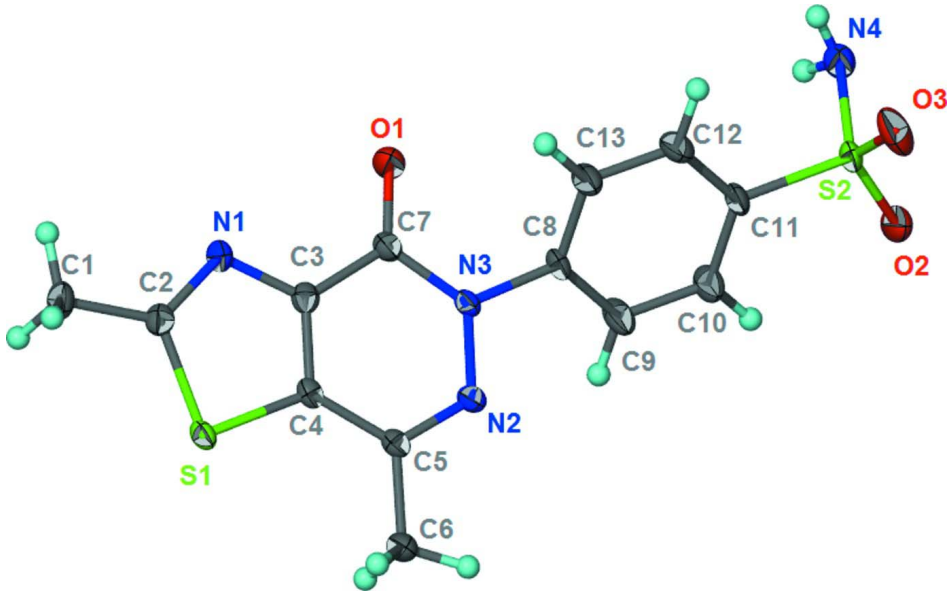

Figure 1

Thermal ellipsoid plot (Barbour, 2001) of $C_{13}H_{12}N_4O_3S_2$ at the 70% probability level; hydrogen atoms are drawn as spheres of arbitrary radius.

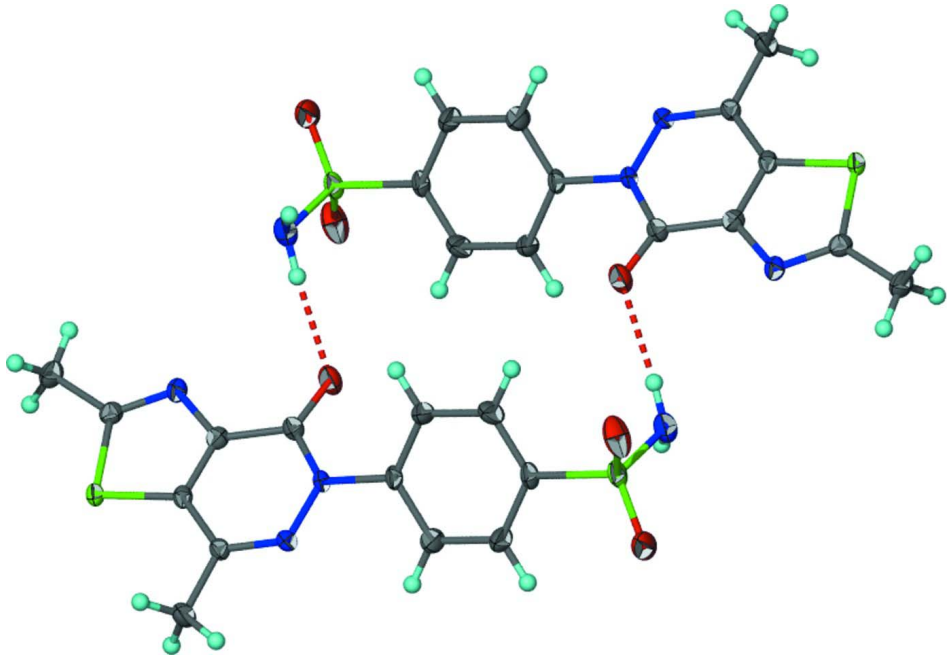

Figure 2

Hydrogen-bonded dimeric structure.

4-(2,7-dimethyl-4-oxo-1,3-thiazolo[4,5-*d*]pyridazin- 5-yl)benzenesulfonamide

Crystal data

$C_{13}H_{12}N_4O_3S_2$
M_r = 336.39

Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc

a = 12.6048 (10) Å
b = 13.2273 (10) Å
c = 8.9703 (7) Å
β = 102.242 (1)°
V = 1461.6 (2) Å^3
Z = 4
F(000) = 696
D_x = 1.529 Mg m^{-3}

Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 3931 reflections
θ = 2.3–28.2°
μ = 0.38 mm^{-1}
T = 100 K
Block, light brown
0.20 × 0.15 × 0.15 mm

Data collection

Bruker SMART APEX diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.928, T_{max} = 0.945

9962 measured reflections
3333 independent reflections
2888 reflections with $I > 2\sigma(I)$
R_{int} = 0.028
θ_{max} = 27.5°, θ_{min} = 2.3°
h = −11→16
k = −17→17
l = −11→11

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.034
$wR(F^2)$ = 0.097
S = 1.06
3333 reflections
209 parameters
2 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
$w = 1/[\sigma^2(F_o^2) + (0.0449P)^2 + 0.9855P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.39 e Å^{-3}
$\Delta\rho_{min}$ = −0.53 e Å^{-3}

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

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

	x	y	z	U_{iso}*/U_{eq}
S1	1.13140 (3)	0.42793 (3)	0.70269 (5)	0.01393 (12)
S2	0.34576 (3)	0.29976 (4)	0.11537 (5)	0.01750 (12)
O1	0.73961 (10)	0.47213 (10)	0.67068 (14)	0.0220 (3)
O2	0.33900 (11)	0.19520 (11)	0.07281 (17)	0.0305 (3)
O3	0.32382 (11)	0.37373 (12)	−0.00328 (15)	0.0302 (3)
C1	1.14993 (15)	0.57418 (14)	0.9320 (2)	0.0203 (4)
H1A	1.1070	0.6150	0.9888	0.030*
H1B	1.1924	0.6189	0.8799	0.030*
H1C	1.1991	0.5302	1.0029	0.030*
C2	1.07602 (14)	0.51116 (13)	0.81743 (19)	0.0155 (3)
N1	0.97011 (12)	0.51401 (11)	0.79149 (16)	0.0160 (3)
N2	0.86442 (11)	0.31764 (11)	0.42826 (16)	0.0142 (3)

N3	0.78999 (11)	0.37071 (11)	0.48923 (16)	0.0134 (3)
N4	0.26042 (13)	0.31912 (13)	0.22293 (18)	0.0214 (3)
H1	0.257 (2)	0.3836 (8)	0.242 (3)	0.038 (7)*
H2	0.277 (2)	0.2825 (17)	0.3059 (19)	0.041 (7)*
C3	0.92801 (14)	0.44919 (13)	0.67384 (18)	0.0142 (3)
C4	1.00186 (13)	0.39722 (13)	0.61098 (18)	0.0127 (3)
C5	0.96720 (13)	0.32902 (12)	0.48733 (18)	0.0137 (3)
C6	1.04567 (15)	0.26894 (15)	0.4208 (2)	0.0208 (4)
H6A	1.0069	0.2343	0.3286	0.031*
H6B	1.0811	0.2188	0.4953	0.031*
H6C	1.1006	0.3141	0.3946	0.031*
C7	0.81235 (14)	0.43424 (13)	0.61656 (19)	0.0149 (3)
C8	0.68056 (13)	0.35427 (13)	0.40510 (19)	0.0145 (3)
C9	0.65207 (15)	0.25831 (14)	0.3480 (2)	0.0221 (4)
H9	0.7026	0.2042	0.3701	0.027*
C10	0.54950 (15)	0.24169 (14)	0.2583 (2)	0.0219 (4)
H10	0.5293	0.1762	0.2187	0.026*
C11	0.47709 (13)	0.32119 (14)	0.22727 (19)	0.0161 (3)
C12	0.50573 (15)	0.41714 (14)	0.2838 (2)	0.0226 (4)
H12	0.4552	0.4711	0.2611	0.027*
C13	0.60796 (15)	0.43436 (14)	0.3732 (2)	0.0202 (4)
H13	0.6282	0.5000	0.4122	0.024*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0092 (2)	0.0149 (2)	0.0165 (2)	−0.00054 (15)	0.00014 (15)	−0.00010 (15)
S2	0.0100 (2)	0.0256 (3)	0.0150 (2)	0.00070 (16)	−0.00154 (15)	−0.00466 (16)
O1	0.0139 (6)	0.0306 (8)	0.0217 (6)	0.0011 (5)	0.0038 (5)	−0.0071 (5)
O2	0.0162 (7)	0.0324 (8)	0.0384 (8)	0.0005 (6)	−0.0042 (6)	−0.0187 (7)
O3	0.0172 (7)	0.0496 (10)	0.0211 (7)	0.0025 (6)	−0.0020 (5)	0.0096 (6)
C1	0.0185 (9)	0.0209 (9)	0.0190 (8)	−0.0030 (7)	−0.0015 (7)	−0.0042 (7)
C2	0.0165 (8)	0.0140 (8)	0.0150 (8)	−0.0001 (7)	0.0015 (6)	0.0000 (6)
N1	0.0130 (7)	0.0175 (7)	0.0164 (7)	−0.0013 (6)	0.0006 (5)	−0.0028 (6)
N2	0.0118 (7)	0.0138 (7)	0.0163 (7)	0.0014 (5)	0.0018 (5)	−0.0004 (5)
N3	0.0086 (7)	0.0146 (7)	0.0162 (7)	0.0002 (5)	0.0006 (5)	−0.0013 (5)
N4	0.0143 (7)	0.0297 (9)	0.0197 (8)	−0.0001 (7)	0.0027 (6)	−0.0038 (7)
C3	0.0146 (8)	0.0136 (8)	0.0136 (7)	0.0000 (6)	0.0012 (6)	0.0012 (6)
C4	0.0102 (8)	0.0137 (8)	0.0132 (7)	−0.0007 (6)	0.0002 (6)	0.0028 (6)
C5	0.0117 (8)	0.0132 (8)	0.0158 (7)	0.0007 (6)	0.0017 (6)	0.0016 (6)
C6	0.0137 (8)	0.0237 (9)	0.0234 (9)	0.0030 (7)	0.0007 (7)	−0.0072 (7)
C7	0.0122 (8)	0.0164 (8)	0.0155 (8)	0.0005 (6)	0.0015 (6)	0.0001 (6)
C8	0.0090 (8)	0.0173 (8)	0.0158 (8)	−0.0006 (6)	−0.0008 (6)	0.0009 (6)
C9	0.0165 (9)	0.0159 (9)	0.0299 (10)	0.0021 (7)	−0.0042 (7)	0.0008 (7)
C10	0.0167 (9)	0.0161 (9)	0.0293 (10)	−0.0017 (7)	−0.0032 (7)	−0.0036 (7)
C11	0.0096 (8)	0.0217 (9)	0.0154 (7)	−0.0001 (7)	−0.0011 (6)	−0.0003 (7)
C12	0.0161 (9)	0.0197 (9)	0.0289 (10)	0.0060 (7)	−0.0020 (8)	−0.0030 (7)
C13	0.0151 (9)	0.0152 (9)	0.0275 (9)	0.0011 (7)	−0.0021 (7)	−0.0047 (7)

Geometric parameters (Å, º)

S1—C4	1.7142 (17)	N4—H2	0.875 (10)
S1—C2	1.7502 (17)	C3—C4	1.371 (2)
S2—O3	1.4288 (15)	C3—C7	1.453 (2)
S2—O2	1.4326 (15)	C4—C5	1.425 (2)
S2—N4	1.6105 (16)	C5—C6	1.489 (2)
S2—C11	1.7674 (17)	C6—H6A	0.9800
O1—C7	1.231 (2)	C6—H6B	0.9800
C1—C2	1.488 (2)	C6—H6C	0.9800
C1—H1A	0.9800	C8—C9	1.387 (3)
C1—H1B	0.9800	C8—C13	1.390 (2)
C1—H1C	0.9800	C9—C10	1.388 (3)
C2—N1	1.306 (2)	C9—H9	0.9500
N1—C3	1.376 (2)	C10—C11	1.382 (3)
N2—C5	1.300 (2)	C10—H10	0.9500
N2—N3	1.3746 (19)	C11—C12	1.386 (3)
N3—C7	1.398 (2)	C12—C13	1.385 (3)
N3—C8	1.441 (2)	C12—H12	0.9500
N4—H1	0.872 (10)	C13—H13	0.9500
C4—S1—C2	88.46 (8)	N2—C5—C4	120.34 (15)
O3—S2—O2	118.12 (9)	N2—C5—C6	117.64 (15)
O3—S2—N4	106.80 (9)	C4—C5—C6	122.01 (15)
O2—S2—N4	107.68 (9)	C5—C6—H6A	109.5
O3—S2—C11	108.86 (8)	C5—C6—H6B	109.5
O2—S2—C11	107.57 (8)	H6A—C6—H6B	109.5
N4—S2—C11	107.36 (8)	C5—C6—H6C	109.5
C2—C1—H1A	109.5	H6A—C6—H6C	109.5
C2—C1—H1B	109.5	H6B—C6—H6C	109.5
H1A—C1—H1B	109.5	O1—C7—N3	121.93 (15)
C2—C1—H1C	109.5	O1—C7—C3	125.48 (16)
H1A—C1—H1C	109.5	N3—C7—C3	112.59 (14)
H1B—C1—H1C	109.5	C9—C8—C13	120.93 (16)
N1—C2—C1	125.00 (16)	C9—C8—N3	118.39 (15)
N1—C2—S1	115.69 (13)	C13—C8—N3	120.53 (15)
C1—C2—S1	119.30 (13)	C8—C9—C10	119.70 (17)
C2—N1—C3	109.40 (14)	C8—C9—H9	120.2
C5—N2—N3	118.94 (14)	C10—C9—H9	120.2
N2—N3—C7	126.57 (14)	C11—C10—C9	119.39 (17)
N2—N3—C8	111.88 (13)	C11—C10—H10	120.3
C7—N3—C8	121.55 (14)	C9—C10—H10	120.3
S2—N4—H1	109.6 (17)	C10—C11—C12	120.90 (16)
S2—N4—H2	110.8 (18)	C10—C11—S2	119.45 (14)
H1—N4—H2	113 (2)	C12—C11—S2	119.64 (14)
C4—C3—N1	116.28 (15)	C13—C12—C11	120.05 (17)
C4—C3—C7	120.28 (15)	C13—C12—H12	120.0
N1—C3—C7	123.44 (15)	C11—C12—H12	120.0

C3—C4—C5	120.99 (15)	C12—C13—C8	119.02 (17)
C3—C4—S1	110.16 (12)	C12—C13—H13	120.5
C5—C4—S1	128.86 (13)	C8—C13—H13	120.5
C4—S1—C2—N1	1.29 (14)	C4—C3—C7—O1	175.26 (17)
C4—S1—C2—C1	−177.20 (15)	N1—C3—C7—O1	−4.5 (3)
C1—C2—N1—C3	177.37 (16)	C4—C3—C7—N3	−4.9 (2)
S1—C2—N1—C3	−1.03 (19)	N1—C3—C7—N3	175.35 (15)
C5—N2—N3—C7	−3.4 (2)	N2—N3—C8—C9	37.7 (2)
C5—N2—N3—C8	176.49 (15)	C7—N3—C8—C9	−142.50 (17)
C2—N1—C3—C4	0.1 (2)	N2—N3—C8—C13	−137.94 (16)
C2—N1—C3—C7	179.88 (16)	C7—N3—C8—C13	41.9 (2)
N1—C3—C4—C5	−179.24 (15)	C13—C8—C9—C10	−0.3 (3)
C7—C3—C4—C5	1.0 (2)	N3—C8—C9—C10	−175.91 (17)
N1—C3—C4—S1	0.84 (19)	C8—C9—C10—C11	0.0 (3)
C7—C3—C4—S1	−178.93 (13)	C9—C10—C11—C12	0.3 (3)
C2—S1—C4—C3	−1.12 (13)	C9—C10—C11—S2	−179.11 (15)
C2—S1—C4—C5	178.97 (16)	O3—S2—C11—C10	−129.82 (16)
N3—N2—C5—C4	−1.4 (2)	O2—S2—C11—C10	−0.72 (18)
N3—N2—C5—C6	178.80 (15)	N4—S2—C11—C10	114.92 (16)
C3—C4—C5—N2	2.5 (2)	O3—S2—C11—C12	50.76 (17)
S1—C4—C5—N2	−177.63 (13)	O2—S2—C11—C12	179.85 (15)
C3—C4—C5—C6	−177.76 (16)	N4—S2—C11—C12	−64.51 (17)
S1—C4—C5—C6	2.1 (3)	C10—C11—C12—C13	−0.3 (3)
N2—N3—C7—O1	−173.80 (15)	S2—C11—C12—C13	179.15 (15)
C8—N3—C7—O1	6.4 (3)	C11—C12—C13—C8	−0.1 (3)
N2—N3—C7—C3	6.3 (2)	C9—C8—C13—C12	0.4 (3)
C8—N3—C7—C3	−173.48 (14)	N3—C8—C13—C12	175.85 (16)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N4—H1···O1^{i}	0.87 (1)	2.06 (1)	2.922 (2)	169 (2)
N4—H2···O2^{ii}	0.88 (1)	2.38 (2)	3.090 (2)	139 (2)

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