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Dimethylammonium tetrahydropentaborate

R. Angharad Baber, Jonathan P. H. Charmant,* Nicholas C. Norman, A. Guy Orpen and Jean Rossi

School of Chemistry, University of Bristol, Bristol BS8 1TS, England

Correspondence e-mail: jon.charmant@bris.ac.uk

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Key indicators

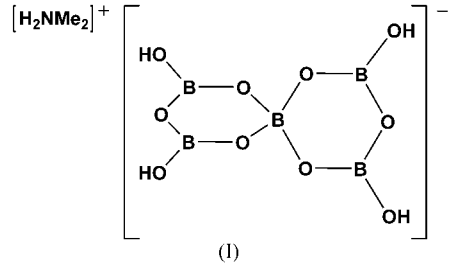
Single-crystal X-ray study
T = 100 K
Mean σ(N–C) = 0.002 Å
R factor = 0.034
wR factor = 0.088
Data-to-parameter ratio = 12.6

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

The title compound [systematic name: dimethylammonium 1,1′-spiro-bis(3,5,-dihydroxy-2,4,6-trioxa-1,3,5-triboracyclohexane)borate], $C_2H_8N^+\cdot B_5H_4O_{10}^-$, contains the $[B_5O_6(OH)_4]^-$ tetrahydropentaborate anion, which possesses typical geometrical parameters, accompanied by dimethylammonium cations. The packing of these species is influenced by cation-to-anion N—H···O and anion-to-anion O—H···O hydrogen bonds.

Comment

The tetrahydropentaborate anion, $[B_5O_6(OH)_4]^-$, has been crystallized with a variety of ammonium cations: $[NH_4]^+$ (Loboda *et al.*, 1993); $[H_2NC_5H_{10}]^+$, $[NMe_4]^+$ and $[NEt_4]^+$ (Wiebcke *et al.*, 1993); $[HNEt_3]^+$ (Loboda *et al.*, 1994); $[HNBu^n_3]^+$ (Turdybekov *et al.*, 1992) and $[NPr^n_4]^+$ (Freyhardt *et al.*, 1994). In this paper, we report the crystal structure of a dimethylammonium salt of this anion, $[H_2NMe_2]^+$-$[B_5O_6(OH)_4]^-$, (I) (Fig. 1).



The anion consists of a central BO_4 tetrahedron fused to four trigonal planar $BO_2(OH)$ units and shows normal geometrical parameters (Table 1). Hydrogen bonding (Table 2) between adjacent $[B_5O_6(OH)_4]^-$ units results in $R_2^2(8)$ (Etter, 1990) dimers (Fig. 2). This anion-to-anion hydrogen-bonding framework is supplemented by the formation of two hydrogen bonds from each dimethylammonium cation to two adjacent $[B_5O_6(OH)_4]^-$ anions.

Experimental

A large excess of $B(OH)_3$ (55.6 mmol, 3.44 g, dried by the Dean–Stark method) was added to a stirred solution of $B_2(NMe_2)_4$ (1 ml, 5.56 mmol) in tetrahydrofuran (25 ml), and the solution left to stir overnight. After removal of the solvent *in vacuo*, a white solid remained, which was shown to contain some $B_2(OH)_4$ and a majority of $B(OH)_3$ by $^{11}B\{^1H\}$ NMR spectroscopy. Dissolution of this solid in degassed water followed by slow evaporation over several days afforded a small crop of thin needle-like crystals approximately 5 mm long, a fragment of one of which was shown to be $[H_2NMe_2][B_5O_6(OH)_4]$.

Crystal data

$C_2H_8N^+\cdot B_5H_4O_{10}^-$
$M_r = 264.18$
Monoclinic, $C2/c$
$a = 13.3664$ (3) Å
$b = 11.4709$ (3) Å
$c = 17.1147$ (4) Å
$\beta = 112.160$ (1)°
$V = 2430.27$ (10) Å^3
$Z = 8$
$D_x = 1.444$ Mg m^{-3}
Cu $K\alpha$ radiation
Cell parameters from 4393 reflections
$\theta = 5.3$–70.2°
$\mu = 1.19$ mm^{-1}
$T = 100$ (2) K
Block, colourless
0.18 × 0.10 × 0.10 mm

Data collection

Bruker Proteum CCD area-detector diffractometer
ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2003)
$T_{min} = 0.792$, $T_{max} = 0.886$
9127 measured reflections
2225 independent reflections
1847 reflections with $I > 2\sigma(I)$
$R_{int} = 0.026$
$\theta_{max} = 70.2°$
$h = -15 \rightarrow 16$
$k = -13 \rightarrow 13$
$l = -20 \rightarrow 20$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.034$
$wR(F^2) = 0.088$
$S = 0.99$
2225 reflections
177 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0624P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.25$ e Å^{-3}
$\Delta\rho_{min} = -0.25$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

B1—O1	1.4623 (16)	B3—O4	1.3612 (18)
B1—O6	1.4679 (16)	B3—O3	1.3849 (17)
B1—O10	1.4680 (16)	B4—O6	1.3530 (18)
B1—O5	1.4726 (18)	B4—O7	1.3550 (17)
B2—O2	1.3506 (18)	B4—O8	1.3843 (17)
B2—O1	1.3628 (18)	B5—O9	1.3522 (17)
B2—O3	1.3860 (18)	B5—O10	1.3643 (18)
B3—O5	1.3571 (18)	B5—O8	1.3813 (17)
B3—O3—B2	118.78 (11)	B5—O8—B4	119.22 (11)
B3—O5—B1	123.11 (10)	B5—O10—B1	123.93 (10)
B4—O6—B1	123.62 (10)		

Table 2
Hydrogen-bonding geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1A···O1^{i}	0.92	1.86	2.7707 (15)	170
N1—H1B···O4^{ii}	0.92	1.96	2.8765 (15)	173
O2—H2A···O7^{iii}	0.848 (17)	1.852 (17)	2.6972 (15)	175.1 (16)
O4—H4A···O5^{iv}	0.814 (16)	1.926 (16)	2.7340 (12)	171.6 (17)
O7—H7A···O10^{v}	0.841 (19)	1.862 (18)	2.7015 (13)	175.8 (18)
O9—H9A···O6^{vi}	0.822 (18)	1.942 (18)	2.7526 (13)	168.8 (19)

Symmetry codes: (i) $1-x, y-1, \frac{1}{2}-z$; (ii) $x-1, 1-y, z-\frac{1}{2}$; (iii) $\frac{1}{2}+x, \frac{3}{2}-y, \frac{1}{2}+z$; (iv) $2-x, y, \frac{1}{2}-z$; (v) $\frac{3}{2}-x, y-\frac{1}{2}, \frac{1}{2}-z$; (vi) $\frac{3}{2}-x, \frac{1}{2}+y, \frac{1}{2}-z$.

The methyl H atoms of the cation were located using a rotating group refinement, with C—H bond lengths constrained to 0.96 Å and displacement parameters equal to 1.5 times U_{eq} of their parent C atom. The remaining H atoms of the cation were constrained to ideal geometries (Table 2) and refined with displacement parameters equal to 1.2 times U_{eq}(N). All hydroxyl H atoms were located in Fourier difference maps, assigned displacement parameters equal to 1.5U_{eq}(O) and refined with a distance restraint of 0.84 (3) Å on the O—H bonds.

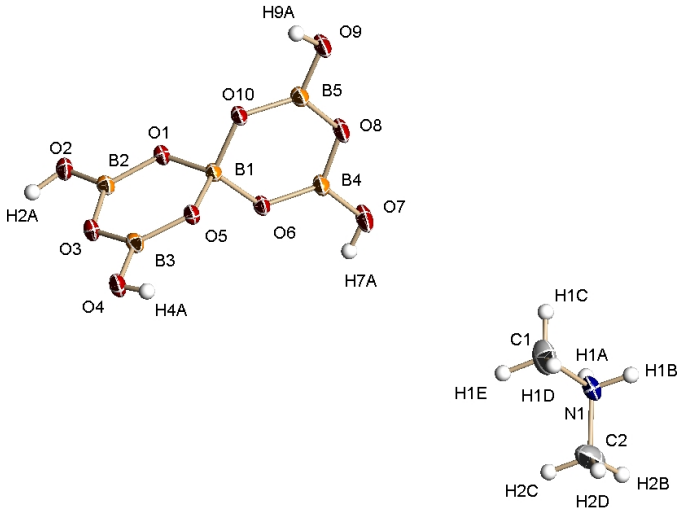


Figure 1
The molecular structure of (I), showing the atom labelling scheme (50% displacement ellipsoids).

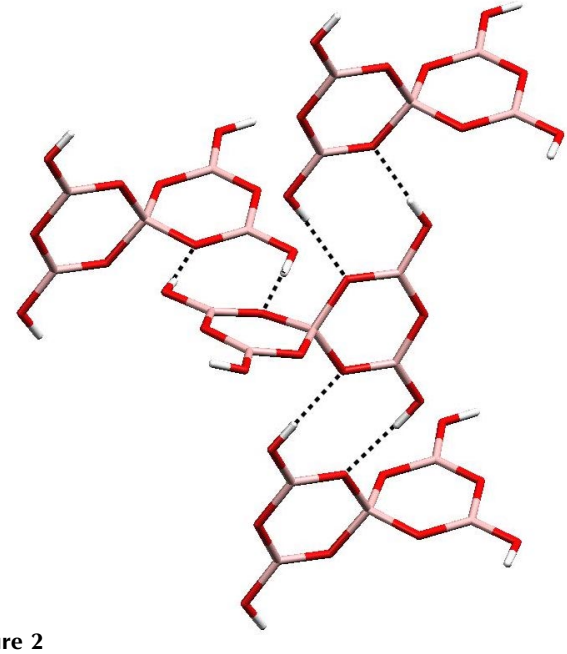


Figure 2
Detail of (I) in stick representation (key: B pink, O red and H white) illustrating the dimeric $R_2^2(8)$ hydrogen-bonding motif linking adjacent $[B_5O_6(OH)_4]^-$ anions.

Data collection: *SMART* (Bruker, 2002); cell refinement: *SAINT* (Bruker, 2002); data reduction: *SAINT* and *SHELXTL* (Bruker, 2002); program(s) used to solve structure: *SHELXTL*; program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

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supporting information

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dimethylammonium 1,1′-spiro-bis(3,5,-dihydroxy-2,4,6-trioxa-1,3,5-triboracyclohexane)borate

Crystal data

$C_2H_8N^+·B_5H_4O_{10}^-$
M_r = 264.18
Monoclinic, *C*2/*c*
Hall symbol: -C 2yc
a = 13.3664 (3) Å
b = 11.4709 (3) Å
c = 17.1147 (4) Å
β = 112.160 (1)°
V = 2430.27 (10) Å^3
Z = 8
F(000) = 1088
D_x = 1.444 Mg m^{-3}
Cu *Kα* radiation, *λ* = 1.54178 Å
Cell parameters from 4393 reflections
θ = 5.3–70.2°
μ = 1.19 mm^{-1}
T = 100 K
Block, colourless
0.18 × 0.10 × 0.10 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Radiation source: MAC Science M06X CE Rotating anode
Osmic CMF12-38Cu6 (blue) optics monochromator
Detector resolution: 5.6 pixels mm^{-1}
ω scans
Absorption correction: multi-scan (SADABS; Sheldrick, 2003)
T_{min} = 0.792, T_{max} = 0.886
9127 measured reflections
2225 independent reflections
1847 reflections with $I > 2\sigma(I)$
R_{int} = 0.026
θ_{max} = 70.2°, θ_{min} = 5.3°
h = −15→16
k = −13→13
l = −20→20

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.034
$wR(F^2)$ = 0.088
S = 0.99
2225 reflections
177 parameters
4 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: difmap (O-H) and geom (C-H and N-H)
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0624P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.25 e Å^{-3}
$\Delta\rho_{min}$ = −0.25 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.

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 $F^2 > \sigma(F^2)$ 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 (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
B1	0.81860 (12)	0.90832 (12)	0.31606 (9)	0.0166 (3)
B2	0.93003 (12)	0.87280 (12)	0.46759 (9)	0.0180 (3)
B3	1.01942 (12)	0.89925 (12)	0.37177 (9)	0.0181 (3)
B4	0.66922 (12)	0.80519 (13)	0.20378 (9)	0.0204 (3)
B5	0.65759 (12)	1.01176 (13)	0.21409 (9)	0.0197 (3)
O1	0.83319 (7)	0.89692 (7)	0.40480 (5)	0.0176 (2)
O2	0.93013 (8)	0.85132 (8)	0.54521 (6)	0.0224 (2)
H2A	0.9918 (12)	0.8304 (16)	0.5797 (10)	0.034*
O3	1.02458 (7)	0.87224 (8)	0.45206 (5)	0.0195 (2)
O4	1.11499 (7)	0.90211 (8)	0.36063 (6)	0.0221 (2)
H4A	1.1035 (15)	0.9148 (15)	0.3112 (10)	0.033*
O5	0.92372 (7)	0.92038 (7)	0.30757 (5)	0.0177 (2)
O6	0.76559 (7)	0.80361 (7)	0.26945 (5)	0.0180 (2)
O7	0.62507 (8)	0.70632 (8)	0.16148 (6)	0.0300 (3)
H7A	0.6629 (15)	0.6470 (15)	0.1818 (12)	0.045*
O8	0.61277 (8)	0.90800 (8)	0.17587 (6)	0.0261 (2)
O9	0.60298 (8)	1.11071 (8)	0.18115 (6)	0.0257 (2)
H9A	0.6414 (14)	1.1673 (15)	0.2022 (12)	0.039*
O10	0.75402 (7)	1.01298 (7)	0.28109 (5)	0.0178 (2)
N1	0.28012 (9)	0.01583 (10)	0.01395 (7)	0.0234 (3)
H1A	0.2448	−0.0171	0.0454	0.028*
H1B	0.2287	0.0366	−0.0374	0.028*
C1	0.33666 (14)	0.12213 (15)	0.05735 (11)	0.0417 (4)
H1C	0.2854	0.1734	0.0691	0.063*
H1D	0.3672	0.1630	0.0212	0.063*
H1E	0.3949	0.1004	0.1105	0.063*
C2	0.35208 (15)	−0.07212 (17)	0.00023 (11)	0.0435 (4)
H2B	0.3096	−0.1404	−0.0278	0.065*
H2C	0.4067	−0.0955	0.0547	0.065*
H2D	0.3878	−0.0390	−0.0353	0.065*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
B1	0.0141 (7)	0.0157 (7)	0.0165 (7)	−0.0001 (5)	0.0018 (6)	0.0000 (5)
B2	0.0174 (7)	0.0131 (7)	0.0195 (7)	−0.0003 (5)	0.0025 (6)	−0.0016 (5)

B3	0.0167 (7)	0.0158 (7)	0.0184 (7)	−0.0003 (5)	0.0027 (6)	−0.0027 (5)
B4	0.0177 (7)	0.0193 (8)	0.0186 (7)	−0.0003 (6)	0.0006 (6)	0.0002 (5)
B5	0.0157 (7)	0.0190 (8)	0.0209 (8)	0.0000 (6)	0.0029 (6)	0.0001 (5)
O1	0.0135 (4)	0.0203 (5)	0.0159 (5)	0.0004 (3)	0.0020 (4)	−0.0002 (3)
O2	0.0174 (5)	0.0275 (6)	0.0180 (5)	0.0019 (4)	0.0018 (4)	0.0037 (4)
O3	0.0137 (4)	0.0233 (5)	0.0173 (5)	0.0018 (4)	0.0011 (4)	0.0010 (3)
O4	0.0148 (5)	0.0319 (6)	0.0164 (5)	0.0005 (4)	0.0024 (4)	−0.0010 (4)
O5	0.0140 (4)	0.0201 (5)	0.0161 (5)	0.0001 (3)	0.0023 (4)	0.0003 (3)
O6	0.0154 (5)	0.0152 (5)	0.0189 (5)	0.0010 (3)	0.0012 (4)	−0.0004 (3)
O7	0.0257 (5)	0.0164 (5)	0.0301 (6)	0.0026 (4)	−0.0097 (4)	−0.0019 (4)
O8	0.0194 (5)	0.0176 (5)	0.0268 (5)	0.0004 (4)	−0.0077 (4)	−0.0003 (4)
O9	0.0189 (5)	0.0165 (5)	0.0290 (5)	−0.0001 (4)	−0.0055 (4)	0.0003 (4)
O10	0.0149 (4)	0.0155 (5)	0.0181 (5)	0.0001 (3)	0.0006 (4)	−0.0004 (3)
N1	0.0162 (5)	0.0291 (6)	0.0211 (6)	−0.0007 (5)	0.0026 (5)	0.0052 (4)
C1	0.0343 (9)	0.0355 (9)	0.0417 (10)	−0.0121 (7)	−0.0011 (8)	0.0030 (7)
C2	0.0373 (9)	0.0534 (12)	0.0407 (10)	0.0168 (8)	0.0158 (8)	0.0065 (7)

Geometric parameters (Å, º)

B1—O1	1.4623 (16)	B5—O8	1.3813 (17)
B1—O6	1.4679 (16)	O2—H2A	0.848 (14)
B1—O10	1.4680 (16)	O4—H4A	0.814 (14)
B1—O5	1.4726 (18)	O7—H7A	0.841 (15)
B2—O2	1.3506 (18)	O9—H9A	0.822 (15)
B2—O1	1.3628 (18)	N1—C2	1.473 (2)
B2—O3	1.3860 (18)	N1—C1	1.479 (2)
B3—O5	1.3571 (18)	N1—H1A	0.9200
B3—O4	1.3612 (18)	N1—H1B	0.9200
B3—O3	1.3849 (17)	C1—H1C	0.9800
B4—O6	1.3530 (18)	C1—H1D	0.9800
B4—O7	1.3550 (17)	C1—H1E	0.9800
B4—O8	1.3843 (17)	C2—H2B	0.9800
B5—O9	1.3522 (17)	C2—H2C	0.9800
B5—O10	1.3643 (18)	C2—H2D	0.9800
O1—B1—O6	109.83 (10)	B4—O6—B1	123.62 (10)
O1—B1—O10	108.91 (10)	B4—O7—H7A	112.5 (14)
O6—B1—O10	111.01 (10)	B5—O8—B4	119.22 (11)
O1—B1—O5	110.64 (10)	B5—O9—H9A	109.3 (14)
O6—B1—O5	107.83 (10)	B5—O10—B1	123.93 (10)
O10—B1—O5	108.62 (10)	C2—N1—C1	113.84 (13)
O2—B2—O1	117.44 (13)	C2—N1—H1A	108.8
O2—B2—O3	121.67 (12)	C1—N1—H1A	108.8
O1—B2—O3	120.89 (12)	C2—N1—H1B	108.8
O5—B3—O4	122.01 (12)	C1—N1—H1B	108.8
O5—B3—O3	121.49 (13)	H1A—N1—H1B	107.7
O4—B3—O3	116.50 (12)	N1—C1—H1C	109.5
O6—B4—O7	121.26 (12)	N1—C1—H1D	109.5

O6—B4—O8	121.48 (12)	H1C—C1—H1D	109.5
O7—B4—O8	117.24 (12)	N1—C1—H1E	109.5
O9—B5—O10	122.15 (12)	H1C—C1—H1E	109.5
O9—B5—O8	117.16 (12)	H1D—C1—H1E	109.5
O10—B5—O8	120.69 (12)	N1—C2—H2B	109.5
B2—O1—B1	123.50 (11)	N1—C2—H2C	109.5
B2—O2—H2A	112.2 (12)	H2B—C2—H2C	109.5
B3—O3—B2	118.78 (11)	N1—C2—H2D	109.5
B3—O4—H4A	109.3 (14)	H2B—C2—H2D	109.5
B3—O5—B1	123.11 (10)	H2C—C2—H2D	109.5
O2—B2—O1—B1	−172.54 (11)	O7—B4—O6—B1	178.29 (12)
O3—B2—O1—B1	8.38 (19)	O8—B4—O6—B1	−0.3 (2)
O6—B1—O1—B2	104.43 (13)	O1—B1—O6—B4	119.72 (13)
O10—B1—O1—B2	−133.81 (11)	O10—B1—O6—B4	−0.78 (17)
O5—B1—O1—B2	−14.49 (16)	O5—B1—O6—B4	−119.64 (13)
O5—B3—O3—B2	−3.31 (18)	O9—B5—O8—B4	175.72 (13)
O4—B3—O3—B2	177.43 (11)	O10—B5—O8—B4	−3.0 (2)
O2—B2—O3—B3	−177.60 (12)	O6—B4—O8—B5	2.2 (2)
O1—B2—O3—B3	1.43 (19)	O7—B4—O8—B5	−176.43 (13)
O4—B3—O5—B1	174.74 (11)	O9—B5—O10—B1	−176.76 (12)
O3—B3—O5—B1	−4.48 (19)	O8—B5—O10—B1	1.9 (2)
O1—B1—O5—B3	12.53 (16)	O1—B1—O10—B5	−121.03 (13)
O6—B1—O5—B3	−107.59 (12)	O6—B1—O10—B5	0.01 (17)
O10—B1—O5—B3	132.03 (11)	O5—B1—O10—B5	118.39 (13)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1*A*···O1^{i}	0.92	1.86	2.7707 (15)	170
N1—H1*B*···O4^{ii}	0.92	1.96	2.8765 (15)	173
O2—H2*A*···O7^{iii}	0.85 (2)	1.85 (2)	2.6972 (15)	175 (2)
O4—H4*A*···O5^{iv}	0.81 (2)	1.93 (2)	2.7340 (12)	172 (2)
O7—H7*A*···O10^{v}	0.84 (2)	1.86 (2)	2.7015 (13)	176 (2)
O9—H9*A*···O6^{vi}	0.82 (2)	1.94 (2)	2.7526 (13)	169 (2)

Symmetry codes: (i) −*x*+1, *y*−1, −*z*+1/2; (ii) *x*−1, −*y*+1, *z*−1/2; (iii) *x*+1/2, −*y*+3/2, *z*+1/2; (iv) −*x*+2, *y*, −*z*+1/2; (v) −*x*+3/2, *y*−1/2, −*z*+1/2; (vi) −*x*+3/2, *y*+1/2, −*z*+1/2.