

Acta Crystallographica Section E

Structure Reports

Online

ISSN 1600-5368

(1*R,2*S**)-2-Nitro-1-(4-nitrophenyl)-propanol**

Xu Zhang, Le Yang, Jun-na Zhang and Wei He*

Department of Chemistry, School of Pharmacy, Fourth Military Medical University, Shaanxi Province, Xi'an 710032, People's Republic of China
Correspondence e-mail: weihechem@fmmu.edu.cn

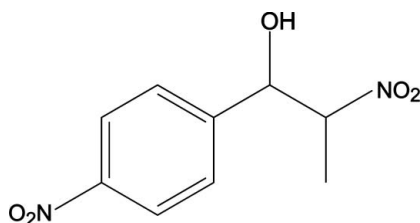
Received 16 January 2012; accepted 21 February 2012

Key indicators: single-crystal X-ray study; $T = 296$ K; mean $\sigma(\text{C}-\text{C}) = 0.003$ Å; R factor = 0.045; wR factor = 0.119; data-to-parameter ratio = 12.5.

The title compound, $\text{C}_9\text{H}_{10}\text{N}_2\text{O}_5$, presents a racemic mixture of two enantiomeric diastereomers. In the crystal, molecules assemble into zigzag chains parallel to the b axis [$C(6)$ motif] due to the formation of elongated $\text{O}-\text{H}\cdots\text{O}(\text{N})$ hydrogen bonds. Of interest is the fact that only the aliphatic nitro group is involved in hydrogen bonding and it adopts a *gauche* conformation with respect to the OH group.

Related literature

For the preparation and synthetic utilities of 2-nitroethanols, see: Palomo *et al.* (2005); Palomo (2007). For the structure of the closely related 1-(anthracen-9-yl)-2-nitroethanol, see: Niazimbetova *et al.* (1998). For spectroscopic data and chemical properties of the title compound, see: Blay *et al.* (2008). For hydrogen-bond graph-set notation, see: Etter *et al.* (1990).



Experimental

Crystal data

$\text{C}_9\text{H}_{10}\text{N}_2\text{O}_5$
 $M_r = 226.19$
Monoclinic, $P2_1/c$
 $a = 7.4013$ (15) Å
 $b = 10.504$ (2) Å
 $c = 13.681$ (3) Å
 $\beta = 100.465$ (4)°

$V = 1046.0$ (4) Å³
 $Z = 4$
Mo $K\alpha$ radiation
 $\mu = 0.12$ mm⁻¹
 $T = 296$ K
 $0.40 \times 0.28 \times 0.14$ mm

Data collection

Bruker APEXII diffractometer
Absorption correction: multi-scan
(*SADABS*; Sheldrick, 1996)
 $T_{\min} = 0.954$, $T_{\max} = 0.984$

5155 measured reflections
1868 independent reflections
1502 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.017$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.045$
 $wR(F^2) = 0.119$
 $S = 1.05$
1868 reflections
150 parameters

H atoms treated by a mixture of independent and constrained refinement
 $\Delta\rho_{\max} = 0.24$ e Å⁻³
 $\Delta\rho_{\min} = -0.16$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-\text{H}\cdots A$	$D-\text{H}$	$\text{H}\cdots A$	$D\cdots A$	$D-\text{H}\cdots A$
$\text{O3}-\text{H3}\cdots\text{O1}^i$	0.80 (3)	2.24 (3)	3.010 (2)	162 (3)

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

Data collection: *APEX2* (Bruker, 2007); cell refinement: *SAINT* (Bruker, 2007); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008) and *OLEX2* (Dolomanov *et al.*, 2009); software used to prepare material for publication: *SHELXTL* and *OLEX2*.

Financial support from the Natural Science Foundation of China (project No. 21072228) is gratefully acknowledged. We are also grateful to Dr Maxim V. Borzov (a Foreign Expert at the North-West University, Xi'an, China) for his great help in revising this contribution.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HG5165).

References

- Blay, J., Domingo, L. R., Hernández-Olmos, V. & Pedro, J. R. (2008). *Chem. Eur. J.* **14**, 4725–4730.
Bruker (2007). *APEX2* and *SAINT*. Bruker AXS Inc., Madison, Wisconsin, USA.
Dolomanov, O. V., Bourhis, L. J., Gildea, R. J., Howard, J. A. K. & Puschmann, H. (2009). *J. Appl. Cryst.* **42**, 339–341.
Etter, M. C., MacDonald, J. C. & Bernstein, J. (1990). *Acta Cryst.* **B46**, 256–262.
Niazimbetova, Z., Treimer, S. E., Evans, D. H., Guzei, I. & Rheingold, A. L. (1998). *J. Electrochem. Soc.* **145**, 2768–2774.
Palomo, C. (2007). *Eur. J. Org. Chem.* pp. 2561–2574.
Palomo, C., Oiarbide, M. & Laso, M. (2005). *Angew. Chem. Int. Ed.* **44**, 3881–3884.
Sheldrick, G. M. (1996). *SADABS*. University of Göttingen, Germany.
Sheldrick, G. M. (2008). *Acta Cryst.* **A64**, 112–122.

supplementary materials

Acta Cryst. (2012). E68, o881 [doi:10.1107/S1600536812007775]

(1*R,2*S**)-2-Nitro-1-(4-nitrophenyl)propanol**

Xu Zhang, Le Yang, Jun-na Zhang and Wei He

Comment

The title compound, C₉H₁₀N₂O₅, I, belongs to the family of β -nitroalcohols which can serve as convenient synthetic precursors for a variety of 1,2-amino alcohols, amino sugars, nitroketones, nitroalkenes, ketones, and other practically important compounds (Palomo *et al.*, 2005; Palomo, 2007).

β -Nitroalcohol I was prepared by a modified procedure described by Blay *et al.*, 2008 (see Experimental). Only one pair of diastereomers is observed among the reaction products, what could be a result of the application of an enantiomerically pure base, natural quinine (see Experimental). In the unsymmetrical unit of I, the N—C(aliphatic) bond is by 0.05 Å longer than the N—C(aromatic) one due to the evident p - π conjugation. The aromatic NO₂ group is slightly twisted in respect to the Ph-ring plane [the C6/C7/N2/O4 dihedral angle equals to 17.9 (3)^o]. In crystal lattice, the molecules of I assemble in zigzag chains parallel to the *b*-axis [a C(6) motif (Etter *et al.*, 1990)] due to formation of somewhat elongated [2.24 (3) Å] O—H \cdots O(N) hydrogen bonds. Of interest, only the aliphatic nitro-group is involved into the H-binding and adopts a *gauche*-conformation respectively to the OH-group, with the N1/C2/C3/O3 dihedral angle being rather close to 60^o [55.2 (2)^o]. The same H-binding motif was observed earlier for the case of closely related 1-(antracen-9-yl)-2-nitroethanol (Niazimbetova *et al.*, 1998).

Experimental

Quinine [(*R*)-(6-methoxyquinolin-4-yl)((2*S*,4*S*,8*R*)-8-vinylquinuclidin-2-yl)methanol, 32.4 mg, 0.1 mmol], Zn(OTf)₂ [zinc bis(trifluoromethanesulfonate), 36.3 mg, 0.1 mmol], and *p*-nitrobenzaldehyde (151.1 mg, 1 mmol) were dissolved in THF (5 ml). To this solution, excess of nitroethane (10 mmol) was added. After keeping the mixture for 1 h at 253 K, *N*-ethyl-*N,N*-diisopropylamine (17.3 μ l, 0.1 mmol) was entered and the slurry was allowed to stay for additional 12 h at the same temperature. The resultant solution was concentrated under reduced pressure and then subjected to silica gel flash column chromatography (hexane/ethyl acetate = 10/1), what gave I as a racemic mixture. Purity of the product was proved additionally by the HPLC method. Single crystal of I suitable for the X-ray diffraction analysis was grown from a CH₂Cl₂–methanol medium (volume ratio 2: 1). NMR spectral data are in consistence with reported earlier (Blay *et al.*, 2008).

Refinement

Non-H atoms were refined anisotropically. All H atoms except of the OH group one were treated as riding atoms with distances C—H = 0.96 (CH₃), 0.98 (CH), 0.93 Å (C_{Ar}H), and $U_{iso}(H) = 1.5U_{eq}(C)$, $1.2U_{eq}(C)$, and $1.2U_{eq}(C)$, respectively. The hydroxy-group H-atom was found from the difference Fourier syntheses and refined isotropically.

Computing details

Data collection: *APEX2* (Bruker, 2007); cell refinement: *SAINTE* (Bruker, 2007); data reduction: *SAINTE* (Bruker, 2007); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97*

(Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008) and *OLEX2* (Dolomanov *et al.*, 2009); software used to prepare material for publication: *SHELXTL* (Sheldrick, 2008) and *OLEX2* (Dolomanov *et al.*, 2009).

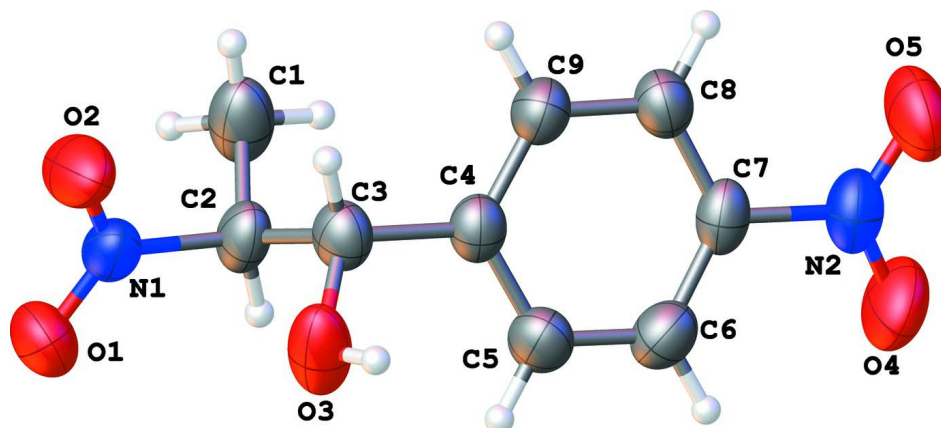


Figure 1

Unsymmetrical unit of I with labeling. Thermal displacement ellipsoids are shown at the 50% probability level.

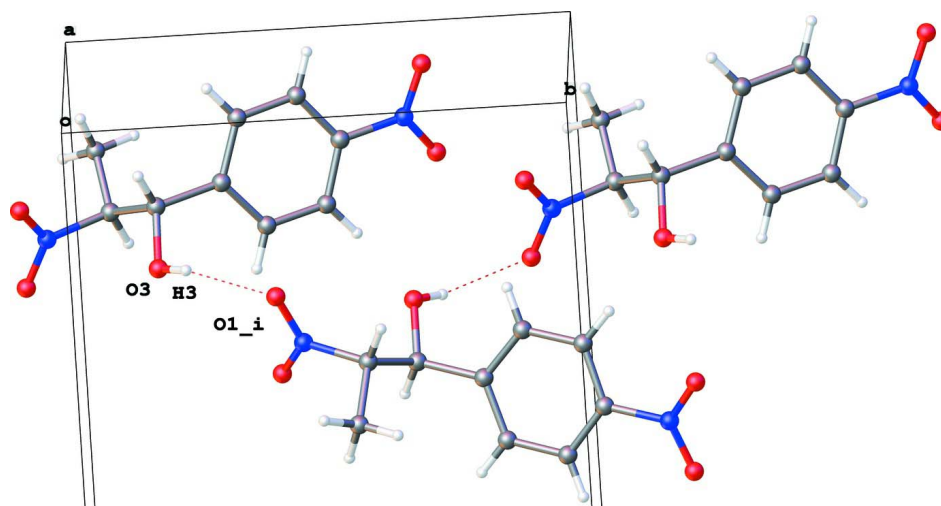


Figure 2

Chain-assembly of I in the crystal lattice (ball-and-stick drawing). Hydrogen bonds are depicted as dashed lines. Only O3, H3, and O1_i atoms are labeled. Symmetry code (i): $-x, y + 1/2, -z + 1/2$.

(1*R**,2*S**)-2-Nitro-1-(4-nitrophenyl)propanol

Crystal data

$C_9H_{10}N_2O_5$

$M_r = 226.19$

Monoclinic, $P2_1/c$

Hall symbol: $-P\ 2_1/c$

$a = 7.4013$ (15) Å

$b = 10.504$ (2) Å

$c = 13.681$ (3) Å

$\beta = 100.465$ (4)°

$V = 1046.0$ (4) Å³

$Z = 4$

$F(000) = 472$

$D_x = 1.436$ Mg m⁻³

Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å

Cell parameters from 1711 reflections

$\theta = 2.5$ – 24.3 °

$\mu = 0.12$ mm⁻¹

$T = 296$ K

Prism, colourless

$0.40 \times 0.28 \times 0.14$ mm

Data collection

Bruker APEXII diffractometer	5155 measured reflections
Radiation source: fine-focus sealed tube	1868 independent reflections
Graphite monochromator	1502 reflections with $I > 2\sigma(I)$
Detector resolution: 8.333 pixels mm^{-1}	$R_{\text{int}} = 0.017$
φ and ω scans	$\theta_{\text{max}} = 25.1^\circ$, $\theta_{\text{min}} = 2.5^\circ$
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	$h = -8 \rightarrow 5$
$T_{\text{min}} = 0.954$, $T_{\text{max}} = 0.984$	$k = -12 \rightarrow 12$
	$l = -14 \rightarrow 16$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.045$	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2) = 0.119$	$w = 1/[\sigma^2(F_o^2) + (0.0497P)^2 + 0.3436P]$
$S = 1.05$	where $P = (F_o^2 + 2F_c^2)/3$
1868 reflections	$(\Delta\sigma)_{\text{max}} < 0.001$
150 parameters	$\Delta\rho_{\text{max}} = 0.24 \text{ e } \text{\AA}^{-3}$
0 restraints	$\Delta\rho_{\text{min}} = -0.16 \text{ e } \text{\AA}^{-3}$
Primary atom site location: structure-invariant direct methods	

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 (\AA^2)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
N1	0.1671 (2)	-0.04487 (15)	0.18274 (12)	0.0528 (4)
N2	0.3598 (3)	0.67086 (16)	0.05631 (16)	0.0611 (5)
O1	0.2260 (2)	-0.09283 (15)	0.26305 (12)	0.0718 (5)
O2	0.0497 (3)	-0.09303 (15)	0.12111 (11)	0.0800 (6)
O3	0.0003 (3)	0.17439 (15)	0.21466 (14)	0.0757 (6)
O4	0.4510 (3)	0.72894 (16)	0.12539 (14)	0.0886 (6)
O5	0.3235 (3)	0.71279 (16)	-0.02787 (15)	0.0872 (6)
C1	0.3609 (4)	0.0580 (2)	0.07942 (18)	0.0722 (7)
H1A	0.4478	-0.0088	0.1005	0.108*
H1B	0.4254	0.1345	0.0684	0.108*
H1C	0.2816	0.0334	0.0188	0.108*
C2	0.2477 (3)	0.08152 (17)	0.15862 (16)	0.0522 (5)
H2	0.3297	0.1121	0.2184	0.063*
C3	0.0905 (3)	0.17561 (18)	0.13254 (15)	0.0512 (5)
H3A	0.0060	0.1456	0.0734	0.061*
C4	0.1629 (3)	0.30631 (17)	0.11196 (14)	0.0464 (5)

C5	0.2240 (3)	0.3894 (2)	0.18973 (15)	0.0551 (5)
H5	0.2211	0.3644	0.2546	0.066*
C6	0.2890 (3)	0.50863 (19)	0.17205 (15)	0.0555 (5)
H6	0.3295	0.5643	0.2243	0.067*
C7	0.2926 (3)	0.54335 (17)	0.07561 (15)	0.0489 (5)
C8	0.2320 (3)	0.46360 (18)	-0.00374 (15)	0.0522 (5)
H8	0.2347	0.4892	-0.0685	0.063*
C9	0.1676 (3)	0.34498 (18)	0.01569 (15)	0.0520 (5)
H9	0.1266	0.2898	-0.0368	0.062*
H3	-0.076 (4)	0.230 (3)	0.209 (2)	0.103 (11)*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.0716 (11)	0.0402 (9)	0.0517 (10)	-0.0058 (8)	0.0245 (9)	-0.0030 (8)
N2	0.0612 (11)	0.0396 (10)	0.0843 (14)	-0.0007 (8)	0.0185 (10)	0.0046 (10)
O1	0.0958 (12)	0.0593 (10)	0.0614 (10)	-0.0052 (8)	0.0171 (8)	0.0150 (8)
O2	0.1145 (14)	0.0619 (10)	0.0603 (10)	-0.0379 (10)	0.0069 (9)	-0.0026 (8)
O3	0.0893 (12)	0.0504 (9)	0.1043 (14)	0.0075 (9)	0.0623 (11)	0.0127 (9)
O4	0.1091 (14)	0.0488 (10)	0.1031 (14)	-0.0220 (9)	0.0061 (11)	-0.0073 (9)
O5	0.1046 (14)	0.0596 (10)	0.0953 (13)	-0.0183 (9)	0.0122 (11)	0.0244 (9)
C1	0.0846 (16)	0.0519 (13)	0.0924 (17)	0.0049 (11)	0.0487 (14)	0.0075 (12)
C2	0.0625 (12)	0.0343 (10)	0.0650 (13)	-0.0074 (9)	0.0251 (10)	-0.0013 (9)
C3	0.0542 (11)	0.0440 (11)	0.0587 (12)	-0.0049 (9)	0.0191 (9)	0.0039 (9)
C4	0.0475 (10)	0.0385 (10)	0.0560 (11)	0.0008 (8)	0.0168 (9)	0.0023 (8)
C5	0.0657 (13)	0.0511 (12)	0.0531 (12)	-0.0037 (10)	0.0233 (10)	0.0009 (9)
C6	0.0598 (12)	0.0486 (11)	0.0595 (13)	-0.0036 (9)	0.0151 (10)	-0.0098 (10)
C7	0.0461 (10)	0.0336 (10)	0.0695 (13)	0.0013 (8)	0.0176 (9)	0.0020 (9)
C8	0.0607 (12)	0.0430 (11)	0.0548 (12)	0.0012 (9)	0.0155 (9)	0.0067 (9)
C9	0.0614 (12)	0.0405 (11)	0.0548 (12)	-0.0028 (9)	0.0126 (9)	-0.0021 (9)

Geometric parameters (\AA , $^\circ$)

N1—O2	1.206 (2)	C2—H2	0.9800
N1—O1	1.215 (2)	C3—C4	1.519 (3)
N1—C2	1.516 (2)	C3—H3A	0.9800
N2—O5	1.217 (2)	C4—C9	1.385 (3)
N2—O4	1.220 (2)	C4—C5	1.387 (3)
N2—C7	1.469 (2)	C5—C6	1.378 (3)
O3—C3	1.407 (2)	C5—H5	0.9300
O3—H3	0.80 (3)	C6—C7	1.374 (3)
C1—C2	1.505 (3)	C6—H6	0.9300
C1—H1A	0.9600	C7—C8	1.380 (3)
C1—H1B	0.9600	C8—C9	1.377 (3)
C1—H1C	0.9600	C8—H8	0.9300
C2—C3	1.519 (3)	C9—H9	0.9300
O2—N1—O1	123.54 (17)	O3—C3—H3A	109.4
O2—N1—C2	118.50 (17)	C4—C3—H3A	109.4
O1—N1—C2	117.96 (17)	C2—C3—H3A	109.4

O5—N2—O4	123.24 (19)	C9—C4—C5	118.93 (17)
O5—N2—C7	118.44 (19)	C9—C4—C3	120.81 (17)
O4—N2—C7	118.31 (19)	C5—C4—C3	120.26 (18)
C3—O3—H3	110 (2)	C6—C5—C4	120.86 (19)
C2—C1—H1A	109.5	C6—C5—H5	119.6
C2—C1—H1B	109.5	C4—C5—H5	119.6
H1A—C1—H1B	109.5	C7—C6—C5	118.55 (19)
C2—C1—H1C	109.5	C7—C6—H6	120.7
H1A—C1—H1C	109.5	C5—C6—H6	120.7
H1B—C1—H1C	109.5	C6—C7—C8	122.29 (18)
C1—C2—N1	107.80 (16)	C6—C7—N2	118.74 (18)
C1—C2—C3	116.12 (18)	C8—C7—N2	118.96 (18)
N1—C2—C3	107.79 (16)	C9—C8—C7	118.15 (19)
C1—C2—H2	108.3	C9—C8—H8	120.9
N1—C2—H2	108.3	C7—C8—H8	120.9
C3—C2—H2	108.3	C8—C9—C4	121.22 (19)
O3—C3—C4	113.00 (16)	C8—C9—H9	119.4
O3—C3—C2	105.12 (16)	C4—C9—H9	119.4
C4—C3—C2	110.52 (16)		
O2—N1—C2—C1	-69.5 (2)	C3—C4—C5—C6	-179.58 (18)
O1—N1—C2—C1	109.8 (2)	C4—C5—C6—C7	-0.2 (3)
O2—N1—C2—C3	56.6 (2)	C5—C6—C7—C8	0.6 (3)
O1—N1—C2—C3	-124.2 (2)	C5—C6—C7—N2	179.34 (18)
C1—C2—C3—O3	176.23 (17)	O5—N2—C7—C6	-162.9 (2)
N1—C2—C3—O3	55.2 (2)	O4—N2—C7—C6	17.9 (3)
C1—C2—C3—C4	-61.5 (2)	O5—N2—C7—C8	15.9 (3)
N1—C2—C3—C4	177.48 (16)	O4—N2—C7—C8	-163.3 (2)
O3—C3—C4—C9	-146.5 (2)	C6—C7—C8—C9	-0.6 (3)
C2—C3—C4—C9	96.0 (2)	N2—C7—C8—C9	-179.31 (17)
O3—C3—C4—C5	32.9 (3)	C7—C8—C9—C4	0.2 (3)
C2—C3—C4—C5	-84.6 (2)	C5—C4—C9—C8	0.2 (3)
C9—C4—C5—C6	-0.1 (3)	C3—C4—C9—C8	179.61 (18)

Hydrogen-bond geometry (\AA , $^\circ$)

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
O3—H3 \cdots O1 ⁱ	0.80 (3)	2.24 (3)	3.010 (2)	162 (3)

Symmetry code: (i) $-x, y+1/2, -z+1/2$.