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(*S*)-2-[(2,4-Dichlorophenyl)(hydroxy)methyl]-5,5-dimethyl-1,3,2-dioxaphosphinane 2-oxide

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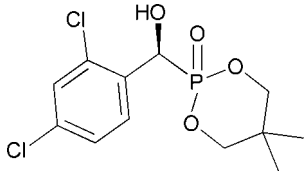
Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.005 Å; R factor = 0.042; wR factor = 0.105; data-to-parameter ratio = 14.7.

In the title molecule, $C_{12}H_{15}Cl_2O_4P$, the cyclic dioxaphosphinane ring adopts a chair conformation. In the crystal, intermolecular O—H···O hydrogen bonds link the molecules into chains propagating along the *b* axis.

Related literature

For the synthesis and biological activity of hydroxydioxaphosphinane derivatives, see: Peng *et al.* (2007); Liu *et al.* (2006). For the synthesis of chiral cyclic hydroxydioxaphosphinanes, see: Zhou *et al.* (2008).



Experimental

Crystal data

$C_{12}H_{15}Cl_2O_4P$
M_r = 325.11
Monoclinic, $P2_1$
a = 7.0263 (9) Å
b = 9.9443 (13) Å
c = 10.6462 (14) Å
β = 93.975 (2)°
V = 742.08 (17) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.55 mm^{-1}
T = 298 K
0.16 × 0.12 × 0.10 mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer
4069 measured reflections
2597 independent reflections
2478 reflections with $I > 2\sigma(I)$
R_{int} = 0.067

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.042
$wR(F^2)$ = 0.105
S = 1.01
2597 reflections
177 parameters
1 restraint
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.39 e Å^{-3}
$\Delta\rho_{min}$ = −0.25 e Å^{-3}
Absolute structure: Flack (1983), 1140 Friedel pairs
Flack parameter: −0.15 (8)

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

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1···O4^{i}	0.80 (5)	1.89 (5)	2.686 (3)	173 (4)

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

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT-Plus* (Bruker, 2001); data reduction: *SAINT-Plus*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *PLATON* (Spek, 2009); software used to prepare material for publication: *PLATON*.

We gratefully acknowledge financial support of this work by the National Basic Research Program of China (grant No. 2010CB126100) and the National Natural Science Foundation of China (grant Nos. 20772042 and 21002037). This work was supported in part by the PCSIRT (grant No. IRT0953).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: CV5059).

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supporting information

Acta Cryst. (2011). E**67**, o920 [doi:10.1107/S1600536811009585]

(*S*)-2-[(2,4-Dichlorophenyl)(hydroxy)methyl]-5,5-dimethyl-1,3,2-dioxaphosphinane 2-oxide

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S1. Comment

The cyclic alpha-hydroxydioxaphosphinanes exhibit various biological activities (Peng *et al.*, 2007; Liu *et al.*, 2006). The title compound, (I), is a chiral cyclic hydroxydioxaphosphinane derivative. Herewith we present its crystal structure.

In (I) (Fig. 1), the cyclic dioxaphosphinane ring adopts a chair conformation. In the crystal structure, intermolecular O—H···O hydrogen bonds (Table 1) link the molecules into chains propagated along *b* axis (Fig. 2).

S2. Experimental

The title compound was prepared according to the known procedure (Zhou *et al.*, 2008). Diethylaluminum chloride (1 mmol) was added to a solution of (*S*,*E*)-2-(adamantan-1-yl)-4- (*tert*-butyl)-6(((1-hydroxy-3-methylbutan-2-yl)imino)-methyl)phenol (1 mmol) in dichloromethane, The mixture was stirred at room temperature for 1 h. The aldehyde and cyclic phosphite was added and the mixture was stirred for another 2 h. The reaction was quenched by diluted hydrochloride acid. The pure title compound was afforded by column chromatography on silica gel (acetone/petroleum ether 1:2). Recrystallization from ethyl acetate over a period of one week gave colourless crystals of the title compound.

S3. Refinement

C-bound H atoms were geometrically positioned (C—H 0.93–0.98 Å) and refined as riding, with U_{iso}(H) = 1.2-1.5 U_{eq}(C). O-bound H atom was located on a difference map and refined as riding (U_{iso}(H) = 1.5U_{eq}(O)) with O—H bond length restrained to 0.80 (4) Å.

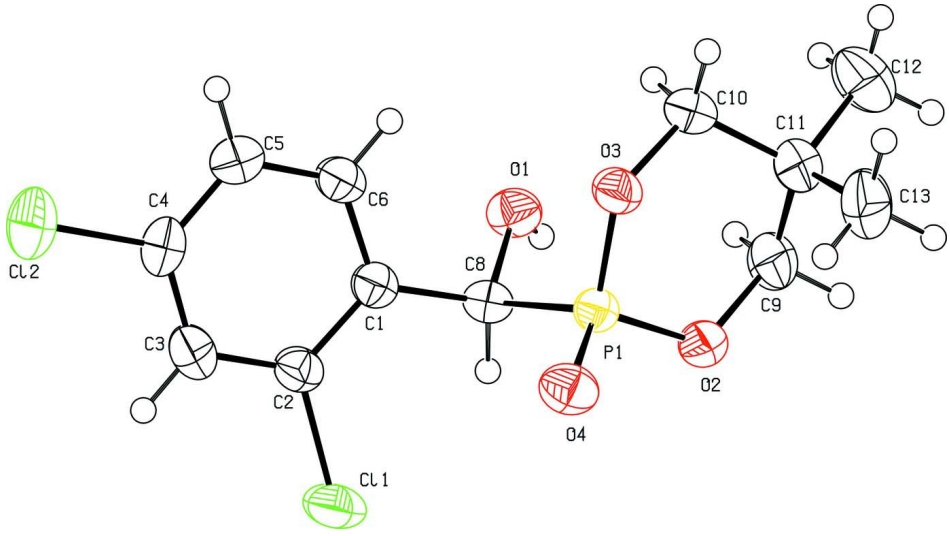

Figure 1

Molecular structure of (I), with displacement ellipsoids drawn at the 50% probability level. Hydrogen atoms are shown as spheres of arbitrary radius.

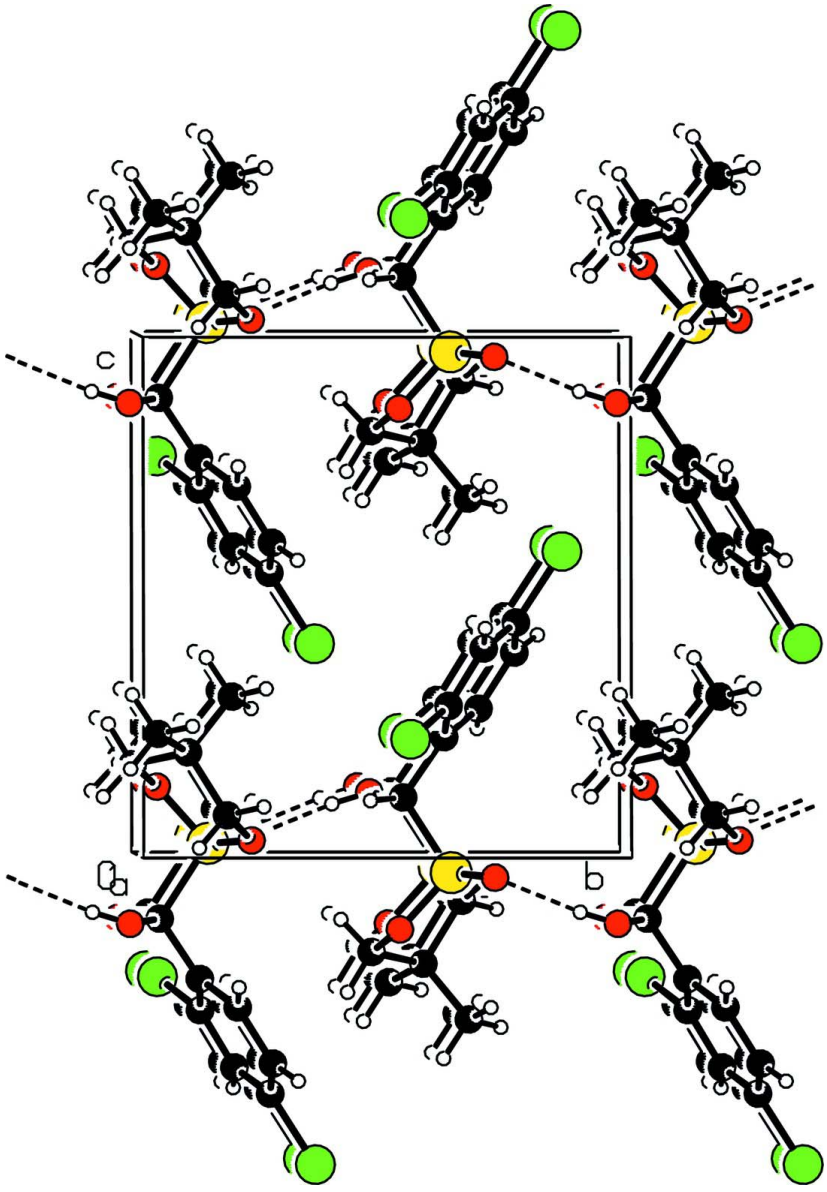

Figure 2

Part of the crystal packing, showing the intermolecular O—H···O hydrogen bonds as dashed lines.

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Crystal data

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$c = 10.6462$ (14) Å
$\beta = 93.975$ (2)°
$V = 742.08$ (17) Å^3
$Z = 2$
$F(000) = 336$
$D_x = 1.455$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 2185 reflections
$\theta = 2.8$–$28.1°$
$\mu = 0.55$ mm^{-1}
$T = 298$ K
Block, colourless
0.16 × 0.12 × 0.10 mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
φ and ω scans
4069 measured reflections
2597 independent reflections
2478 reflections with $I > 2\sigma(I)$
$R_{int} = 0.067$
$\theta_{max} = 25.5°$, $\theta_{min} = 1.9°$
$h = -8 \rightarrow 8$
$k = -11 \rightarrow 12$
$l = -12 \rightarrow 11$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.042$
$wR(F^2) = 0.105$
$S = 1.01$
2597 reflections
177 parameters
1 restraint
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
$w = 1/[\sigma^2(F_o^2) + (0.0604P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.39$ e Å^{-3}
$\Delta\rho_{min} = -0.25$ e Å^{-3}
Absolute structure: Flack (1983), 1140 Friedel pairs
Absolute structure parameter: −0.15 (8)

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}
C1	1.0374 (4)	0.6151 (3)	0.2326 (3)	0.0326 (6)
C2	1.2166 (4)	0.6199 (3)	0.2946 (3)	0.0353 (6)
C3	1.2552 (5)	0.6911 (4)	0.4050 (3)	0.0426 (7)
H3	1.3771	0.6916	0.4451	0.051*
C4	1.1093 (5)	0.7608 (3)	0.4537 (3)	0.0432 (8)
C5	0.9289 (5)	0.7620 (4)	0.3937 (3)	0.0454 (8)
H5	0.8316	0.8117	0.4264	0.054*
C6	0.8951 (4)	0.6891 (3)	0.2856 (3)	0.0395 (7)
H6	0.7728	0.6890	0.2461	0.047*
C8	0.9910 (4)	0.5319 (3)	0.1163 (3)	0.0330 (6)
H8	1.0894	0.4627	0.1112	0.040*
C9	0.7747 (5)	0.4722 (4)	−0.1830 (3)	0.0461 (8)
H9A	0.7300	0.4104	−0.1209	0.055*
H9B	0.7880	0.4222	−0.2601	0.055*
C10	0.6092 (4)	0.6611 (4)	−0.0866 (3)	0.0439 (8)

H10A	0.5200	0.7344	−0.1037	0.053*
H10B	0.5575	0.6030	−0.0242	0.053*
C11	0.6301 (5)	0.5816 (4)	−0.2073 (3)	0.0456 (8)
C12	0.4368 (6)	0.5156 (6)	−0.2449 (5)	0.0766 (14)
H12A	0.4470	0.4632	−0.3199	0.115*
H12B	0.3418	0.5841	−0.2605	0.115*
H12C	0.4008	0.4583	−0.1780	0.115*
C13	0.6874 (6)	0.6734 (5)	−0.3136 (3)	0.0617 (11)
H13A	0.8071	0.7157	−0.2893	0.093*
H13B	0.5913	0.7411	−0.3297	0.093*
H13C	0.6996	0.6211	−0.3884	0.093*
Cl1	1.40915 (11)	0.53739 (10)	0.23277 (9)	0.0563 (3)
Cl2	1.15586 (17)	0.85446 (12)	0.59048 (10)	0.0703 (3)
O1	0.8112 (3)	0.4671 (2)	0.1240 (2)	0.0404 (5)
H1	0.818 (6)	0.390 (5)	0.102 (4)	0.061*
O2	0.9609 (3)	0.5257 (2)	−0.1376 (2)	0.0425 (5)
O3	0.7922 (3)	0.7156 (2)	−0.0361 (2)	0.0390 (5)
O4	1.1503 (3)	0.7162 (2)	−0.0359 (2)	0.0450 (6)
P1	0.98124 (10)	0.63058 (8)	−0.02830 (7)	0.0317 (2)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0314 (14)	0.0335 (17)	0.0328 (14)	−0.0005 (13)	0.0022 (11)	0.0054 (13)
C2	0.0315 (14)	0.0363 (16)	0.0380 (15)	0.0030 (14)	0.0008 (11)	0.0036 (14)
C3	0.0403 (18)	0.0474 (18)	0.0386 (17)	−0.0026 (15)	−0.0067 (13)	0.0031 (15)
C4	0.055 (2)	0.0433 (19)	0.0312 (16)	−0.0031 (16)	0.0021 (14)	−0.0047 (14)
C5	0.0400 (19)	0.051 (2)	0.0456 (19)	0.0057 (16)	0.0073 (15)	−0.0045 (16)
C6	0.0305 (16)	0.0443 (18)	0.0433 (18)	0.0024 (14)	−0.0014 (13)	−0.0034 (15)
C8	0.0277 (13)	0.0318 (15)	0.0397 (16)	0.0010 (13)	0.0031 (12)	0.0010 (13)
C9	0.0453 (18)	0.0470 (19)	0.0452 (19)	−0.0084 (16)	−0.0034 (14)	−0.0094 (16)
C10	0.0304 (15)	0.055 (2)	0.0464 (18)	0.0036 (14)	0.0009 (13)	−0.0034 (16)
C11	0.0402 (18)	0.056 (2)	0.0404 (17)	−0.0018 (16)	−0.0029 (13)	−0.0051 (16)
C12	0.048 (2)	0.099 (4)	0.080 (3)	−0.014 (3)	−0.0139 (19)	−0.019 (3)
C13	0.071 (3)	0.073 (3)	0.040 (2)	−0.001 (2)	−0.0067 (17)	0.0043 (18)
Cl1	0.0313 (4)	0.0680 (6)	0.0690 (6)	0.0119 (4)	−0.0002 (4)	−0.0118 (5)
Cl2	0.0797 (7)	0.0820 (7)	0.0480 (5)	−0.0019 (6)	−0.0050 (5)	−0.0268 (5)
O1	0.0354 (12)	0.0373 (12)	0.0489 (13)	−0.0063 (10)	0.0063 (9)	−0.0050 (11)
O2	0.0359 (11)	0.0502 (14)	0.0411 (12)	0.0067 (11)	0.0015 (9)	−0.0107 (11)
O3	0.0330 (12)	0.0408 (13)	0.0425 (12)	0.0059 (9)	−0.0020 (9)	−0.0058 (10)
O4	0.0364 (12)	0.0445 (13)	0.0544 (14)	−0.0067 (10)	0.0055 (10)	0.0060 (11)
P1	0.0292 (4)	0.0331 (4)	0.0327 (4)	0.0000 (3)	0.0020 (3)	−0.0015 (3)

Geometric parameters (Å, º)

C1—C2	1.381 (4)	C9—H9B	0.9700
C1—C6	1.393 (4)	C10—O3	1.463 (4)
C1—C8	1.506 (4)	C10—C11	1.524 (5)

C2—C3	1.382 (5)	C10—H10A	0.9700
C2—Cl1	1.750 (3)	C10—H10B	0.9700
C3—C4	1.369 (5)	C11—C13	1.530 (5)
C3—H3	0.9300	C11—C12	1.536 (5)
C4—C5	1.379 (5)	C12—H12A	0.9600
C4—Cl2	1.741 (3)	C12—H12B	0.9600
C5—C6	1.366 (5)	C12—H12C	0.9600
C5—H5	0.9300	C13—H13A	0.9600
C6—H6	0.9300	C13—H13B	0.9600
C8—O1	1.425 (3)	C13—H13C	0.9600
C8—P1	1.822 (3)	O1—H1	0.80 (5)
C8—H8	0.9800	O2—P1	1.561 (2)
C9—O2	1.463 (4)	O3—P1	1.572 (2)
C9—C11	1.499 (5)	O4—P1	1.468 (2)
C9—H9A	0.9700		

C2—C1—C6	116.4 (3)	C11—C10—H10A	109.3
C2—C1—C8	123.4 (3)	O3—C10—H10B	109.3
C6—C1—C8	120.2 (2)	C11—C10—H10B	109.3
C1—C2—C3	122.9 (3)	H10A—C10—H10B	108.0
C1—C2—Cl1	120.4 (2)	C9—C11—C10	109.6 (3)
C3—C2—Cl1	116.7 (2)	C9—C11—C13	110.6 (3)
C4—C3—C2	118.2 (3)	C10—C11—C13	111.1 (3)
C4—C3—H3	120.9	C9—C11—C12	108.1 (3)
C2—C3—H3	120.9	C10—C11—C12	107.8 (3)
C3—C4—C5	121.2 (3)	C13—C11—C12	109.6 (3)
C3—C4—Cl2	119.0 (3)	C11—C12—H12A	109.5
C5—C4—Cl2	119.8 (3)	C11—C12—H12B	109.5
C6—C5—C4	119.1 (3)	H12A—C12—H12B	109.5
C6—C5—H5	120.5	C11—C12—H12C	109.5
C4—C5—H5	120.5	H12A—C12—H12C	109.5
C5—C6—C1	122.3 (3)	H12B—C12—H12C	109.5
C5—C6—H6	118.9	C11—C13—H13A	109.5
C1—C6—H6	118.9	C11—C13—H13B	109.5
O1—C8—C1	110.1 (2)	H13A—C13—H13B	109.5
O1—C8—P1	108.08 (19)	C11—C13—H13C	109.5
C1—C8—P1	113.1 (2)	H13A—C13—H13C	109.5
O1—C8—H8	108.5	H13B—C13—H13C	109.5
C1—C8—H8	108.5	C8—O1—H1	110 (3)
P1—C8—H8	108.5	C9—O2—P1	121.52 (19)
O2—C9—C11	111.9 (3)	C10—O3—P1	122.6 (2)
O2—C9—H9A	109.2	O4—P1—O2	112.27 (14)
C11—C9—H9A	109.2	O4—P1—O3	111.68 (14)
O2—C9—H9B	109.2	O2—P1—O3	106.63 (12)
C11—C9—H9B	109.2	O4—P1—C8	112.04 (13)
H9A—C9—H9B	107.9	O2—P1—C8	105.43 (14)
O3—C10—C11	111.6 (2)	O3—P1—C8	108.43 (13)
O3—C10—H10A	109.3		

C6—C1—C2—C3	1.5 (5)	O2—C9—C11—C12	−175.9 (3)
C8—C1—C2—C3	−176.6 (3)	O3—C10—C11—C9	56.3 (4)
C6—C1—C2—Cl1	−177.0 (2)	O3—C10—C11—C13	−66.1 (4)
C8—C1—C2—Cl1	5.0 (4)	O3—C10—C11—C12	173.8 (3)
C1—C2—C3—C4	−0.7 (5)	C11—C9—O2—P1	48.4 (4)
Cl1—C2—C3—C4	177.8 (3)	C11—C10—O3—P1	−44.1 (4)
C2—C3—C4—C5	−1.0 (5)	C9—O2—P1—O4	−153.1 (3)
C2—C3—C4—Cl2	−178.4 (2)	C9—O2—P1—O3	−30.4 (3)
C3—C4—C5—C6	1.8 (5)	C9—O2—P1—C8	84.7 (3)
Cl2—C4—C5—C6	179.3 (3)	C10—O3—P1—O4	151.7 (2)
C4—C5—C6—C1	−1.0 (5)	C10—O3—P1—O2	28.8 (3)
C2—C1—C6—C5	−0.6 (5)	C10—O3—P1—C8	−84.3 (3)
C8—C1—C6—C5	177.5 (3)	O1—C8—P1—O4	171.92 (19)
C2—C1—C8—O1	137.8 (3)	C1—C8—P1—O4	49.8 (2)
C6—C1—C8—O1	−40.1 (4)	O1—C8—P1—O2	−65.7 (2)
C2—C1—C8—P1	−101.1 (3)	C1—C8—P1—O2	172.18 (19)
C6—C1—C8—P1	80.9 (3)	O1—C8—P1—O3	48.2 (2)
O2—C9—C11—C10	−58.6 (4)	C1—C8—P1—O3	−73.9 (2)
O2—C9—C11—C13	64.1 (4)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1···O4^{i}	0.80 (5)	1.89 (5)	2.686 (3)	173 (4)

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