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tert-Butylammonium 2,3,4,5-tetrachloro-6-methoxycarbonylbenzoate

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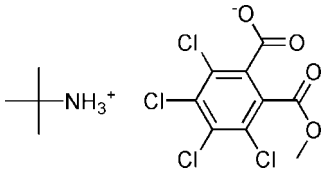
Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.005 Å; R factor = 0.044; wR factor = 0.116; data-to-parameter ratio = 13.6.

In the title compound, $C_4H_{12}N^+\cdot C_9H_3Cl_4O_4^-$, the benzene ring forms dihedral angles of 62.4 (2) and 64.0 (3)°, respectively, with the essentially planar methoxycarbonyl and carboxylate groups. In the crystal structure, intermolecular N—H···O hydrogen bonds connect anions and cations, forming one-dimensional chains along [010].

Related literature

For background information, see: Ungwitayatorn *et al.* (2001). For bond-length data, see: Allen *et al.* (1987).



Experimental

Crystal data

$C_4H_{12}N^+\cdot C_9H_3Cl_4O_4^-$
M_r = 391.06
Monoclinic, $P2_1$
a = 9.0193 (14) Å
b = 6.5084 (11) Å
c = 14.5965 (15) Å
β = 91.7570 (10)°
V = 856.4 (2) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.70 mm^{-1}
T = 298 (2) K
0.53 × 0.48 × 0.44 mm

Data collection

Bruker SMART CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.706, T_{max} = 0.747
4281 measured reflections
2790 independent reflections
2364 reflections with $I > 2\sigma(I)$
R_{int} = 0.043

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.044
$wR(F^2)$ = 0.116
S = 1.04
2790 reflections
205 parameters
1 restraint
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.21 e Å^{-3}
$\Delta\rho_{min}$ = −0.28 e Å^{-3}
Absolute structure: Flack (1983), 1147 Friedel pairs
Flack parameter: 0.00 (9)

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1A···O4^{i}	0.89	1.97	2.838 (4)	165
N1—H1B···O4^{ii}	0.89	1.97	2.850 (4)	168
N1—H1C···O3^{iii}	0.89	1.94	2.818 (4)	169

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

Data collection: *SMART* (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); software used to prepare material for publication: *SHELXTL*.

This work was supported by the Natural Science Foundation of Shandong Province (No. Y2007B61).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: LH2693).

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Ungwitayatorn, J., Matayatsuk, C. & Sothipatcharasai, P. (2001). *Sci. Asia*, **27**, 245–250.

supporting information

Acta Cryst. (2008). E**64**, o2318 [doi:10.1107/S1600536808036398]

tert-Butylammonium 2,3,4,5-tetrachloro-6-methoxycarbonylbenzoate

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S1. Comment

Phthalimides are compounds which can posses biological activity (see: e.g. Ungwitayatorn *et al.*, 2001). 2-(Methoxycarbonyl)-3,4,5,6-tetrachlorobenzoic acid is an intermediate in the sysnthesis of tetrachlorophthalimides and their derivatives. In this paper, the structure of the title compound (I) is reported. The asymmetric unit contains one *tert*-butylammonium cation and one 2-(methoxycarbonyl)-3,4,5,6-tetrachlorobenzene-1-carboxylate anion (Fig. 1). The bond lengths in (I) are normal (Allen *et al.*, 1987). In the crystal structure, intermolecular N-H···O hydrogen bonds connect anions and cations to form one-dimensional chains along [O10].

S2. Experimental

A mixture of tetrachlorophthalic anhydride (2.86 g, 0.01 mol) and methanol (20 ml) was refluxed for 0.5 h and then *tert*-butylamine (0.73 g, 0.01 mol) was added and the mixture stirred for 4 h at room temperature. After filtration, the filtrate was kept at room temperature for 5 d. Natural evaporation gave colourless single crystals of the title compound, suitable for X-ray analysis.

S3. Refinement

H atoms were initially located from difference maps and then refined in a riding-model approximation with C—H = 0.96 Å, N—H = 0.89 Å and U_{iso}(H) = 1.5U_{eq}(N, C).

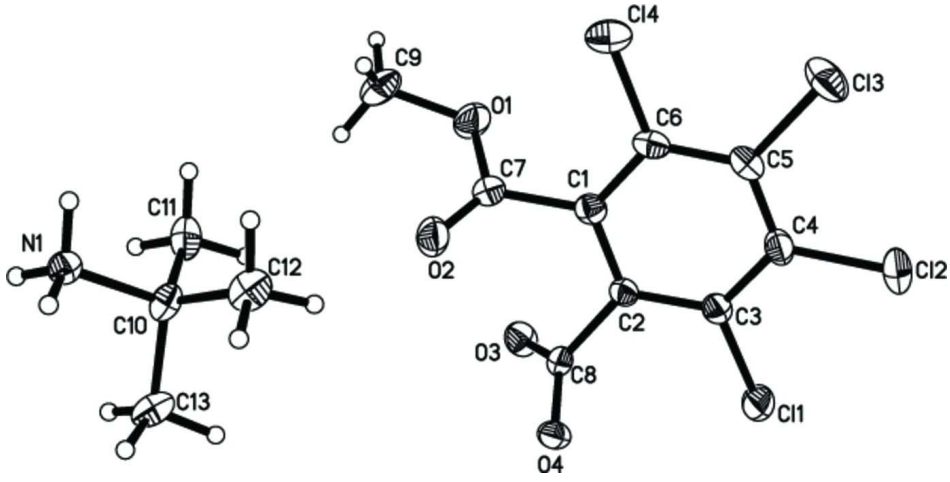

Figure 1

The molecular structure of (I), drawn with 30% probability ellipsoids.

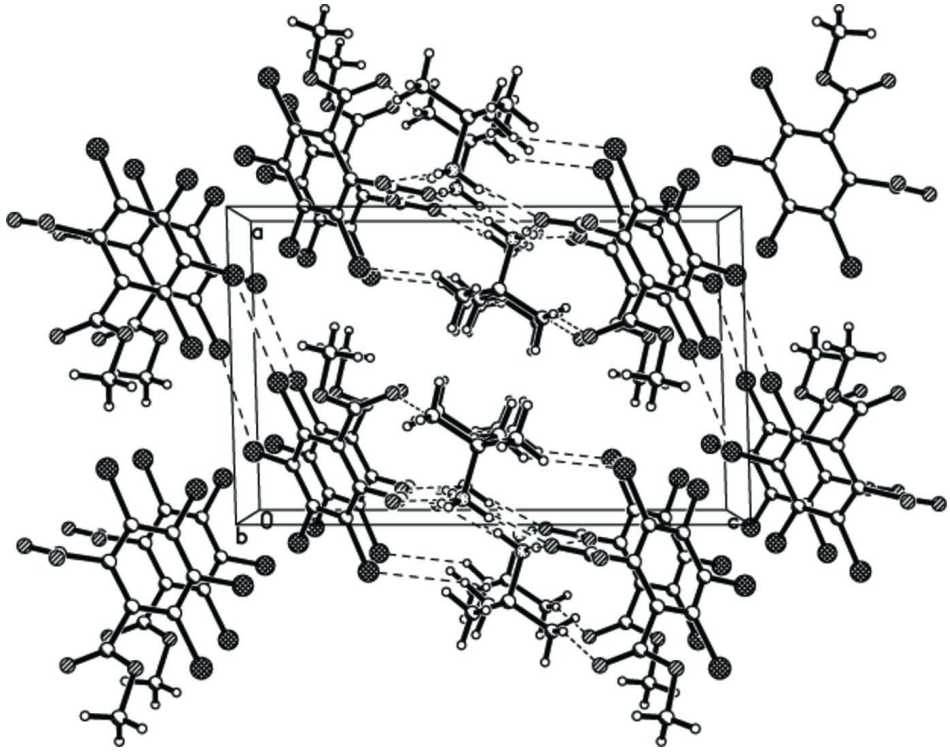

Figure 2

Part of the crystal structure of (I) with hydrogen bonds indicated by dashed lines.

tert-Butylammonium 2,3,4,5-tetrachloro-6-methoxycarbonylbenzoate

Crystal data

$C_4H_{12}N^+·C_9H_3Cl_4O_4^-$
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Monoclinic, $P2_1$
Hall symbol: P 2yb
a = 9.0193 (14) Å
b = 6.5084 (11) Å
c = 14.5965 (15) Å
β = 91.757 (1)°
V = 856.4 (2) Å^3
Z = 2
$F(000)$ = 400
D_x = 1.516 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 2177 reflections
θ = 2.3–27.0°
μ = 0.71 mm^{-1}
T = 298 K
Block, colorless
0.53 × 0.48 × 0.44 mm

Data collection

Bruker SMART CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.706, T_{max} = 0.747
4281 measured reflections
2790 independent reflections
2364 reflections with $I > 2\sigma(I)$
R_{int} = 0.043
θ_{max} = 25.0°, θ_{min} = 2.3°
h = −10→10
k = −7→7
l = −15→17

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.044$
$wR(F^2) = 0.116$
$S = 1.04$
2790 reflections
205 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-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.048P)^2 + 0.2322P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.21$ e Å^{-3}
$\Delta\rho_{min} = -0.28$ e Å^{-3}
Extinction correction: *SHELXL97* (Sheldrick, 2008), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.075 (5)
Absolute structure: Flack (1983), 1147 Friedel pairs
Absolute structure parameter: 0.00 (9)

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 (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
Cl1	−0.17713 (11)	0.5505 (2)	0.24305 (8)	0.0650 (4)
Cl2	−0.10033 (15)	0.2412 (2)	0.09021 (8)	0.0730 (4)
Cl3	0.20894 (14)	0.2526 (2)	0.00306 (8)	0.0670 (4)
Cl4	0.44428 (12)	0.5681 (2)	0.07197 (8)	0.0645 (4)
N1	0.9054 (3)	0.8116 (5)	0.5564 (2)	0.0332 (7)
H1A	0.9244	0.9328	0.5825	0.050*
H1B	0.9627	0.7954	0.5084	0.050*
H1C	0.9241	0.7119	0.5969	0.050*
O1	0.4059 (3)	0.9677 (5)	0.1703 (2)	0.0561 (8)
O2	0.4110 (3)	0.8206 (5)	0.30813 (19)	0.0555 (8)
O3	0.0729 (3)	1.0097 (4)	0.30549 (18)	0.0462 (7)
O4	0.0532 (3)	0.7277 (4)	0.39073 (15)	0.0375 (6)
C1	0.2425 (4)	0.6942 (6)	0.1932 (2)	0.0347 (9)
C2	0.1041 (4)	0.6869 (6)	0.2345 (2)	0.0323 (8)
C3	−0.0013 (4)	0.5489 (7)	0.1999 (2)	0.0375 (9)
C4	0.0307 (5)	0.4133 (7)	0.1295 (3)	0.0431 (10)
C5	0.1686 (5)	0.4186 (7)	0.0903 (2)	0.0408 (9)
C6	0.2727 (4)	0.5608 (7)	0.1215 (2)	0.0380 (9)
C7	0.3626 (4)	0.8340 (6)	0.2319 (3)	0.0378 (9)
C8	0.0731 (4)	0.8226 (6)	0.3175 (2)	0.0305 (8)
C9	0.5402 (5)	1.0814 (9)	0.1945 (4)	0.0701 (15)
H9A	0.6216	0.9874	0.2027	0.105*

H9B	0.5265	1.1554	0.2505	0.105*
H9C	0.5614	1.1766	0.1464	0.105*
C10	0.7440 (4)	0.8028 (6)	0.5251 (3)	0.0367 (9)
C11	0.7195 (5)	0.9690 (7)	0.4533 (3)	0.0511 (11)
H11A	0.7391	1.1011	0.4804	0.077*
H11B	0.6186	0.9642	0.4305	0.077*
H11C	0.7852	0.9469	0.4038	0.077*
C12	0.7164 (4)	0.5891 (7)	0.4845 (3)	0.0469 (10)
H12A	0.7874	0.5622	0.4383	0.070*
H12B	0.6180	0.5831	0.4576	0.070*
H12C	0.7264	0.4878	0.5321	0.070*
C13	0.6520 (4)	0.8364 (7)	0.6089 (3)	0.0487 (11)
H13A	0.5497	0.8082	0.5938	0.073*
H13B	0.6622	0.9763	0.6290	0.073*
H13C	0.6859	0.7459	0.6571	0.073*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.0399 (6)	0.0959 (10)	0.0596 (7)	−0.0201 (6)	0.0101 (5)	−0.0272 (7)
Cl2	0.0733 (8)	0.0769 (10)	0.0685 (8)	−0.0297 (7)	−0.0010 (6)	−0.0322 (7)
Cl3	0.0782 (8)	0.0697 (9)	0.0532 (6)	0.0129 (7)	0.0033 (5)	−0.0277 (7)
Cl4	0.0511 (6)	0.0811 (9)	0.0627 (7)	0.0029 (6)	0.0236 (5)	−0.0087 (7)
N1	0.0333 (15)	0.0302 (17)	0.0364 (16)	0.0004 (13)	0.0033 (12)	0.0002 (14)
O1	0.0603 (19)	0.059 (2)	0.0495 (17)	−0.0197 (15)	0.0029 (14)	0.0158 (15)
O2	0.0606 (18)	0.061 (2)	0.0437 (16)	−0.0225 (16)	−0.0119 (14)	0.0098 (15)
O3	0.0623 (18)	0.0323 (18)	0.0439 (15)	−0.0008 (13)	0.0001 (13)	−0.0013 (12)
O4	0.0446 (14)	0.0374 (15)	0.0308 (13)	0.0003 (13)	0.0076 (11)	−0.0008 (12)
C1	0.039 (2)	0.035 (2)	0.0301 (18)	0.0012 (16)	−0.0010 (15)	0.0053 (16)
C2	0.0373 (19)	0.034 (2)	0.0259 (17)	−0.0030 (16)	0.0004 (15)	0.0015 (15)
C3	0.0349 (19)	0.045 (2)	0.0321 (18)	−0.0021 (19)	0.0002 (14)	−0.0037 (19)
C4	0.050 (2)	0.042 (2)	0.036 (2)	−0.0076 (19)	−0.0054 (18)	−0.0059 (18)
C5	0.050 (2)	0.042 (2)	0.0309 (19)	0.0048 (19)	0.0001 (17)	−0.0057 (17)
C6	0.0403 (19)	0.045 (2)	0.0286 (17)	0.007 (2)	0.0061 (15)	0.0042 (19)
C7	0.036 (2)	0.040 (2)	0.038 (2)	−0.0019 (17)	0.0050 (17)	0.0023 (18)
C8	0.0290 (18)	0.028 (2)	0.0340 (19)	−0.0036 (16)	−0.0012 (15)	0.0000 (16)
C9	0.064 (3)	0.070 (4)	0.078 (3)	−0.032 (3)	0.022 (2)	0.003 (3)
C10	0.0268 (17)	0.033 (2)	0.050 (2)	0.0016 (16)	−0.0027 (15)	−0.0039 (18)
C11	0.045 (2)	0.050 (3)	0.057 (3)	0.007 (2)	−0.011 (2)	0.010 (2)
C12	0.039 (2)	0.042 (3)	0.059 (3)	−0.0065 (19)	−0.0001 (18)	−0.013 (2)
C13	0.040 (2)	0.045 (3)	0.062 (3)	0.006 (2)	0.0158 (19)	−0.007 (2)

Geometric parameters (Å, º)

Cl1—C3	1.724 (4)	C3—C4	1.392 (6)
Cl2—C4	1.714 (4)	C4—C5	1.385 (6)
Cl3—C5	1.718 (4)	C5—C6	1.385 (6)
Cl4—C6	1.729 (4)	C9—H9A	0.9600

N1—C10	1.513 (5)	C9—H9B	0.9600
N1—H1A	0.8900	C9—H9C	0.9600
N1—H1B	0.8900	C10—C13	1.515 (5)
N1—H1C	0.8900	C10—C11	1.517 (6)
O1—C7	1.319 (5)	C10—C12	1.529 (6)
O1—C9	1.455 (5)	C11—H11A	0.9600
O2—C7	1.186 (4)	C11—H11B	0.9600
O3—C8	1.230 (5)	C11—H11C	0.9600
O4—C8	1.252 (4)	C12—H12A	0.9600
C1—C6	1.394 (5)	C12—H12B	0.9600
C1—C2	1.404 (5)	C12—H12C	0.9600
C1—C7	1.511 (5)	C13—H13A	0.9600
C2—C3	1.391 (5)	C13—H13B	0.9600
C2—C8	1.532 (5)	C13—H13C	0.9600
C10—N1—H1A	109.5	O1—C9—H9A	109.5
C10—N1—H1B	109.5	O1—C9—H9B	109.5
H1A—N1—H1B	109.5	H9A—C9—H9B	109.5
C10—N1—H1C	109.5	O1—C9—H9C	109.5
H1A—N1—H1C	109.5	H9A—C9—H9C	109.5
H1B—N1—H1C	109.5	H9B—C9—H9C	109.5
C7—O1—C9	115.6 (3)	N1—C10—C13	107.2 (3)
C6—C1—C2	119.9 (3)	N1—C10—C11	107.5 (3)
C6—C1—C7	120.1 (3)	C13—C10—C11	112.5 (3)
C2—C1—C7	119.7 (3)	N1—C10—C12	107.2 (3)
C3—C2—C1	118.2 (3)	C13—C10—C12	110.9 (4)
C3—C2—C8	121.3 (3)	C11—C10—C12	111.2 (3)
C1—C2—C8	120.5 (3)	C10—C11—H11A	109.5
C2—C3—C4	121.5 (3)	C10—C11—H11B	109.5
C2—C3—Cl1	119.3 (3)	H11A—C11—H11B	109.5
C4—C3—Cl1	119.1 (3)	C10—C11—H11C	109.5
C5—C4—C3	119.8 (4)	H11A—C11—H11C	109.5
C5—C4—Cl2	119.8 (3)	H11B—C11—H11C	109.5
C3—C4—Cl2	120.3 (3)	C10—C12—H12A	109.5
C6—C5—C4	119.3 (4)	C10—C12—H12B	109.5
C6—C5—Cl3	120.4 (3)	H12A—C12—H12B	109.5
C4—C5—Cl3	120.2 (3)	C10—C12—H12C	109.5
C5—C6—C1	121.1 (3)	H12A—C12—H12C	109.5
C5—C6—Cl4	119.2 (3)	H12B—C12—H12C	109.5
C1—C6—Cl4	119.7 (3)	C10—C13—H13A	109.5
O2—C7—O1	125.4 (4)	C10—C13—H13B	109.5
O2—C7—C1	123.1 (4)	H13A—C13—H13B	109.5
O1—C7—C1	111.5 (3)	C10—C13—H13C	109.5
O3—C8—O4	127.6 (3)	H13A—C13—H13C	109.5
O3—C8—C2	117.2 (3)	H13B—C13—H13C	109.5
O4—C8—C2	115.2 (3)		
C6—C1—C2—C3	−2.0 (5)	Cl3—C5—C6—C1	−179.0 (3)

C7—C1—C2—C3	−176.8 (3)	C4—C5—C6—Cl4	−179.6 (3)
C6—C1—C2—C8	175.9 (3)	Cl3—C5—C6—Cl4	−0.5 (5)
C7—C1—C2—C8	1.2 (5)	C2—C1—C6—C5	−0.5 (5)
C1—C2—C3—C4	3.3 (6)	C7—C1—C6—C5	174.2 (4)
C8—C2—C3—C4	−174.6 (4)	C2—C1—C6—Cl4	−179.1 (3)
C1—C2—C3—Cl1	−174.8 (3)	C7—C1—C6—Cl4	−4.3 (5)
C8—C2—C3—Cl1	7.3 (5)	C9—O1—C7—O2	11.0 (6)
C2—C3—C4—C5	−2.0 (6)	C9—O1—C7—C1	−168.3 (4)
Cl1—C3—C4—C5	176.1 (3)	C6—C1—C7—O2	−115.3 (4)
C2—C3—C4—Cl2	178.5 (3)	C2—C1—C7—O2	59.4 (5)
Cl1—C3—C4—Cl2	−3.4 (5)	C6—C1—C7—O1	64.0 (5)
C3—C4—C5—C6	−0.6 (6)	C2—C1—C7—O1	−121.3 (4)
Cl2—C4—C5—C6	178.8 (3)	C3—C2—C8—O3	−117.6 (4)
C3—C4—C5—Cl3	−179.8 (3)	C1—C2—C8—O3	64.5 (5)
Cl2—C4—C5—Cl3	−0.3 (5)	C3—C2—C8—O4	63.6 (5)
C4—C5—C6—C1	1.9 (6)	C1—C2—C8—O4	−114.3 (4)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1*A*···O4^{i}	0.89	1.97	2.838 (4)	165
N1—H1*B*···O4^{ii}	0.89	1.97	2.850 (4)	168
N1—H1*C*···O3^{iii}	0.89	1.94	2.818 (4)	169

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