

## Tetramethylammonium hydrogen terephthalate

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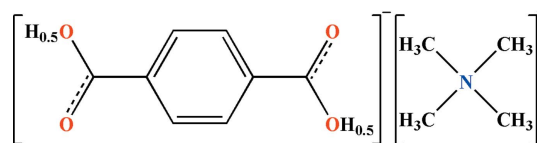
Key indicators: single-crystal X-ray study;  $T = 298$  K; mean  $\sigma(\text{C}-\text{C}) = 0.002$  Å; disorder in main residue;  $R$  factor = 0.054;  $wR$  factor = 0.147; data-to-parameter ratio = 17.0.

The asymmetric unit of the title salt,  $\text{C}_4\text{H}_{12}\text{N}^+\cdot\text{C}_8\text{H}_5\text{O}_4^-$ , contains one half of a tetramethylammonium cation and one half of a hydrogen terephthalate monoanion. The N atom of the ammonium cation lies on a twofold rotation axis and the centre of mass of the terephthalate anion is on a centre of inversion. In the crystal, the centrosymmetric terephthalate ions are linked by a very short symmetric  $\text{O}-\text{H}\cdots\text{O}$  hydrogen bond [ $\text{O}\cdots\text{O} = 2.4610(19)$  Å] into a one-dimensional polymeric chain along  $[1\bar{1}2]$ . The tetramethylammonium cations and terephthalate anions are then connected through a pair of bifurcated acceptor  $\text{C}-\text{H}\cdots\text{O}$  hydrogen bonds, generating a three-dimensional supramolecular network. The carboxylate groups at both ends of the terephthalate anion are charge-shared with an equal probability of 0.5.

### Related literature

For a review of very short  $\text{O}-\text{H}\cdots\text{O}$  hydrogen bonds, see: Speakman (1972). For recent reports of acidic salts of dicarboxylic acids with short intra- and intermolecular  $\text{O}-\text{H}\cdots\text{O}$  hydrogen bonds, see: Starosta & Leciejewicz (2010); Hemamalini & Fun (2010); Sun *et al.* (2002); Sharma *et al.* (2006); Wang *et al.* (2004); Taka *et al.* (1998). For examples of diphosphonates with strong  $\text{O}-\text{H}\cdots\text{O}$  hydrogen bonds, see: Tsaryk *et al.* (2011); Courtney *et al.* (2006); Cheng & Lin (2006). For background to symmetric and asymmetric  $\text{O}-\text{H}\cdots\text{O}$  hydrogen bonds, see: Misaki *et al.* (1986); Catti & Ferraris (1976). For graph-set analysis of hydrogen bonds, see: Etter *et al.* (1990); Bernstein *et al.* (1995). For the synthesis of

the 5,5'-(*o*-phenylene)di-1*H*-tetrazole ligand, see: Demko & Sharpless (2001).



### Experimental

#### Crystal data

$\text{C}_4\text{H}_{12}\text{N}^+\cdot\text{C}_8\text{H}_5\text{O}_4^-$   
 $M_r = 239.27$   
Monoclinic,  $C2/c$   
 $a = 16.0585(4)$  Å  
 $b = 9.1527(2)$  Å  
 $c = 11.5866(3)$  Å  
 $\beta = 132.915(2)^\circ$

$V = 1247.21(7)$  Å<sup>3</sup>  
 $Z = 4$   
Mo  $K\alpha$  radiation  
 $\mu = 0.10$  mm<sup>-1</sup>  
 $T = 298$  K  
 $0.42 \times 0.37 \times 0.32$  mm

#### Data collection

Bruker APEXII CCD diffractometer  
Absorption correction: multi-scan (SADABS; Bruker, 2008)  
 $T_{\min} = 0.708$ ,  $T_{\max} = 0.746$

20680 measured reflections  
1360 independent reflections  
1269 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.022$

#### Refinement

$R[F^2 > 2\sigma(F^2)] = 0.054$   
 $wR(F^2) = 0.147$   
 $S = 1.07$   
1360 reflections

80 parameters  
H-atom parameters constrained  
 $\Delta\rho_{\text{max}} = 0.57$  e Å<sup>-3</sup>  
 $\Delta\rho_{\text{min}} = -0.43$  e Å<sup>-3</sup>

**Table 1**

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$\text{O2}-\text{H2}\cdots\text{O2}^{\text{i}}$	1.23	1.23	2.4610 (19)	180 (1)
$\text{C8}-\text{H8A}\cdots\text{O1}^{\text{ii}}$	0.96	2.39	3.267 (3)	152
$\text{C9}-\text{H9A}\cdots\text{O1}^{\text{ii}}$	0.96	2.47	3.321 (3)	148

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

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: XPW (Siemens, 1996) and ORTEP-3 for Windows (Farrugia, 1997); software used to prepare material for publication: SHELXTL (Sheldrick, 2008) and enCIFer (Allen *et al.*, 2004).

The authors are grateful to the Islamic Azad University, Zanjan Branch, for financial support.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BG2478).

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## supporting information

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## Tetramethylammonium hydrogen terephthalate

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## S1. Comment

Acid salts of dicarboxylic acids usually form very short O—H $\cdots$ O hydrogen bonds (with O $\cdots$ O distances between 2.4 and 2.5 Å) in their crystal structures (Speakman, 1972). This type of hydrogen bond is formed between the carboxyl and carboxylate groups by intramolecular (Starosta & Leciejewicz, 2010; Hemamalini & Fun, 2010; Sun *et al.*, 2002) or intermolecular (Sharma *et al.*, 2006; Wang *et al.*, 2004; Taka *et al.*, 1998) interactions. However, diphosphonates can also display such short and strong hydrogen bonds between neighbouring phosphonate groups (Tsaryk *et al.*, 2011; Courtney *et al.*, 2006; Cheng & Lin, 2006). There are two types of short O—H $\cdots$ O hydrogen bonds: symmetric, in which two O atoms are related by crystallographic symmetry and asymmetric, in which crystal symmetry does not impose the O—H $\cdots$ O hydrogen bond to be symmetric. Furthermore, symmetric hydrogen bonds typically display a shorter (2.43–2.51 Å) O $\cdots$ O distance than asymmetric ones (2.44–2.57 Å) (Misaki *et al.*, 1986). In this work, we report the crystal structure of the title compound, whose structure contains a strong symmetric O—H $\cdots$ O hydrogen bond (Catti & Ferraris, 1976): atom H2 lies on a center of symmetry, located between two crystallographic equivalent carboxyl O2 atoms. The H atom, clearly visible on the Fourier map, is involved in a symmetric O2—H2 $\cdots$ O2<sup>i</sup> [symmetry code: (i)  $-x + 1/2, -y + 3/2, -z$ ] hydrogen bond with an O—H bond distance of 1.23 Å (Table 1).

The title compound (Fig. 1), [N(CH<sub>3</sub>)<sub>4</sub>]<sup>+</sup>[4-COOH-C<sub>6</sub>H<sub>4</sub>COO]<sup>-</sup>, consists of one half tetramethylammonium cation and one half terephthalate anion in the asymmetric unit. The cation lies on a twofold rotation axis and the anion on an inversion center. In the terephthalate anions, the two carboxyl groups are twisted from the mean plane of the benzene ring by a dihedral angle of 6.57 (2)°. Carboxyl atom O2 lies slightly farther [0.083 Å] from this plane than atom O1 [0.065 Å], owing to the strong O—H $\cdots$ O hydrogen bond between the terephthalate anions.

In the crystal, the terephthalate anions are linked end-to-end to form a one-dimensional polymeric chain in which adjacent ions are interconnected by a strong symmetric O—H $\cdots$ O (O $\cdots$ O distance of 2.4610 (19) Å) hydrogen bond (Table 1). Then the weak C—H $\cdots$ O hydrogen bonds link the ammonium cations and terephthalate anions together in a three-dimensional crystal structure. The C8—H8A $\cdots$ O1 and C9—H9A $\cdots$ O1 interactions form a pair of bifurcated acceptor bonds (Fig. 2), involving two C—H donor from an ammonium ion and an acceptor O atom from the terephthalate ion, generating an *R*<sub>2</sub><sup>1</sup>(6) ring motif (Etter *et al.*, 1990; Bernstein *et al.*, 1995).

## S2. Experimental

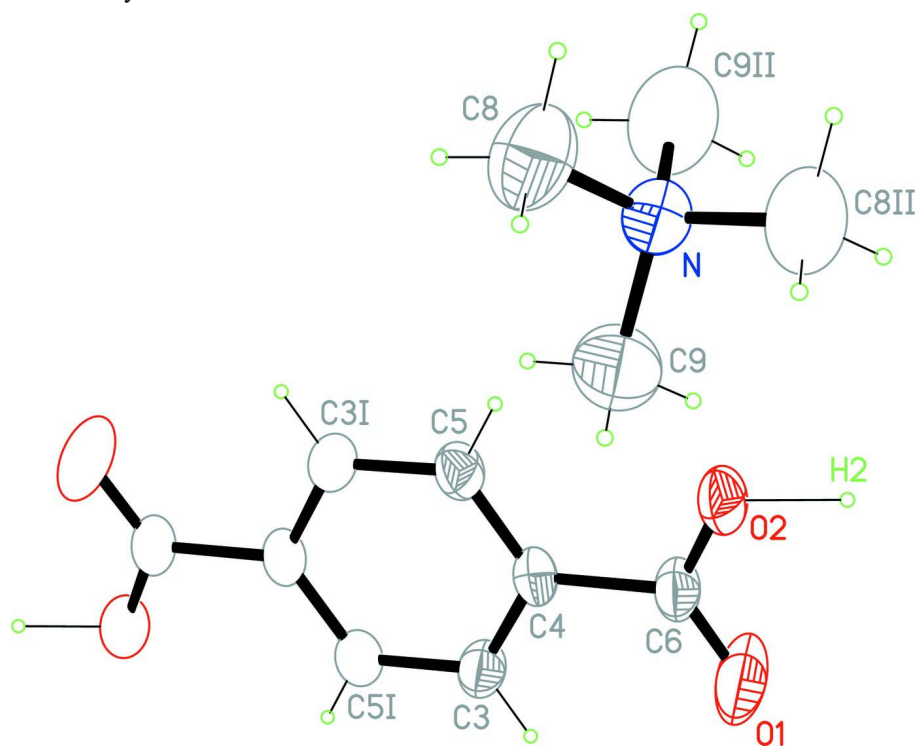
In an attempt to synthesize metal–organic framework materials, we obtained the title compound as a side-product.

The ligand H<sub>2</sub>L [5,5'-(*o*-Phenylene)di-1*H*-tetrazole] used in this work was synthesized according to literature procedures (Demko & Sharpless, 2001).

A mixture of  $\text{Zn}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$  (0.357 g, 1.2 mmol),  $\text{H}_2\text{L}$  (0.086 g, 0.4 mmol), terephthalic acid (0.066 g, 0.4 mmol), 1,4-diazabicyclo[2.2.2]octane (0.045 g, 0.4 mmol) and  $\text{CH}_3\text{OH}/\text{DMF}$  (1/2, 15 ml) were sealed in a 25 ml Teflon-lined stainless steel autoclave, heated at 433 K for 40 h, and then cooled to room temperature over a period of 90 h. The resulting solution was filtered and the filtrate was allowed to stand in air at room temperature. After several days, colorless single crystals of the title compound were isolated.

### S3. Refinement

C-H atoms were located on a  $\Delta F$  map, further idealized and finally refined in the riding model approximation  $d(\text{C}-\text{H}) = 0.93 \text{ \AA}$ ;  $U(\text{H}) = 1.2U(\text{C})_{\text{eq}}$ ;  $d(\text{C}-\text{H}_3) = 0.96 \text{ \AA}$ ;  $U(\text{H}) = 1.5U(\text{C})_{\text{eq}}$ . Atom H2 is fixed by symmetry, and its isotropic displacement factor was freely refined.



**Figure 1**

ORTEP drawing of the asymmetric unit of title compound with the atom numbering. Displacement ellipsoids are drawn at the 50% probability level. The occupancy factor for atom H2 is 0.5. Symmetry codes: (i)  $-x + 1, -y + 2, -z + 1$ ; (ii)  $-x + 1, y, -z + 1/2$ .

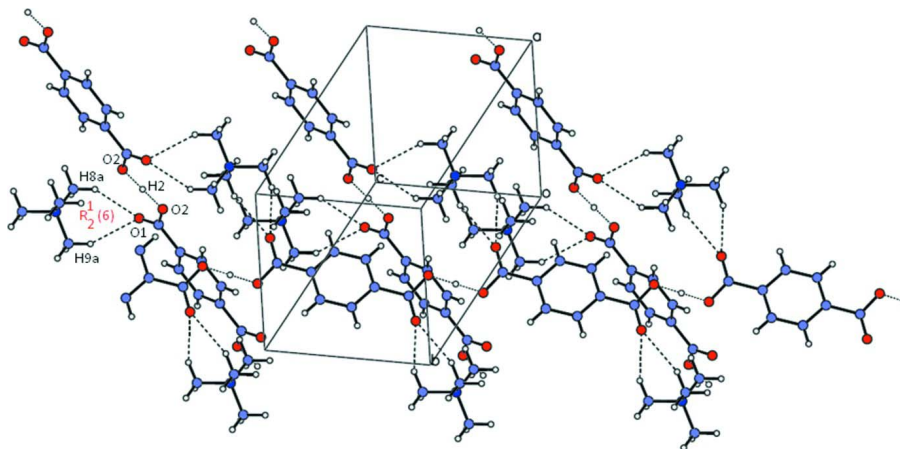


Figure 2

A view of crystal packing of the title compound, showing symmetric O—H...H hydrogen bonds (formed between terephthalate anions, dotted lines) and weak C—H...O hydrogen bonds (formed between cations and anions, dashed lines). The latter contains a pair of bifurcated acceptor hydrogen bonds, leading to an  $R_2^1(6)$  ring motif.

### Tetramethylammonium hydrogen terephthalate

#### Crystal data

$C_4H_{12}N^+ \cdot C_8H_5O_4^-$

$M_r = 239.27$

Monoclinic,  $C2/c$

Hall symbol:  $-C 2yc$

$a = 16.0585$  (4) Å

$b = 9.1527$  (2) Å

$c = 11.5866$  (3) Å

$\beta = 132.915$  (2)°

$V = 1247.21$  (7) Å<sup>3</sup>

$Z = 4$

$F(000) = 512$

$D_x = 1.274$  Mg m<sup>-3</sup>

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

Cell parameters from 9931 reflections

$\theta = 2.8$ – $30.3$ °

$\mu = 0.10$  mm<sup>-1</sup>

$T = 298$  K

Irregular, colourless

$0.42 \times 0.37 \times 0.32$  mm

#### Data collection

Bruker APEXII CCD  
diffractometer

Radiation source: fine-focus sealed tube

Graphite monochromator

$\varphi$  and  $\omega$  scans

Absorption correction: multi-scan

(*SADABS*; Bruker, 2008)

$T_{\min} = 0.708$ ,  $T_{\max} = 0.746$

20680 measured reflections

1360 independent reflections

1269 reflections with  $I > 2\sigma(I)$

$R_{\text{int}} = 0.022$

$\theta_{\max} = 27.0$ °,  $\theta_{\min} = 2.8$ °

$h = -20 \rightarrow 20$

$k = -11 \rightarrow 11$

$l = -14 \rightarrow 14$

#### Refinement

Refinement on  $F^2$

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.054$

$wR(F^2) = 0.147$

$S = 1.07$

1360 reflections

80 parameters

0 restraints

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

$$w = 1/[\sigma^2(F_o^2) + (0.0714P)^2 + 1.1167P]$$

where  $P = (F_o^2 + 2F_c^2)/3$   
 $(\Delta/\sigma)_{\max} < 0.001$

$$\Delta\rho_{\max} = 0.57 \text{ e } \text{\AA}^{-3}$$

$$\Delta\rho_{\min} = -0.43 \text{ e } \text{\AA}^{-3}$$

*Special details*

**Geometry.** All s.u.'s (except the s.u. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell s.u.'s are taken into account individually in the estimation of s.u.'s in distances, angles and torsion angles; correlations between s.u.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell s.u.'s is used for estimating s.u.'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  $F^2 > 2\sigma(F^2)$  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 ( $\text{\AA}^2$ )*

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
O2	0.32784 (9)	0.78319 (13)	0.13940 (12)	0.0451 (4)
C5	0.51647 (12)	0.89007 (16)	0.43479 (16)	0.0341 (4)
H5	0.5278	0.8165	0.3914	0.041*
C4	0.40806 (11)	0.94633 (15)	0.34990 (15)	0.0312 (3)
C6	0.30731 (12)	0.89045 (17)	0.18691 (16)	0.0363 (4)
O1	0.21412 (12)	0.94615 (19)	0.11216 (16)	0.0789 (6)
N	0.5000	0.3608 (2)	0.2500	0.0416 (5)
C9	0.4511 (2)	0.4550 (3)	0.2947 (3)	0.0794 (8)
H9A	0.5097	0.5155	0.3823	0.119*
H9B	0.4188	0.3951	0.3239	0.119*
H9C	0.3928	0.5157	0.2070	0.119*
C3	0.39205 (12)	1.05665 (16)	0.41571 (16)	0.0353 (4)
H3	0.3198	1.0950	0.3594	0.042*
C8	0.58943 (19)	0.2666 (3)	0.3849 (3)	0.0695 (6)
H8A	0.6473	0.3266	0.4737	0.104*
H8B	0.6224	0.2071	0.3565	0.104*
H8C	0.5565	0.2049	0.4120	0.104*
H2	0.2500	0.7500	0.0000	0.098 (13)*

*Atomic displacement parameters ( $\text{\AA}^2$ )*

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
O2	0.0364 (6)	0.0526 (7)	0.0300 (6)	-0.0050 (5)	0.0162 (5)	-0.0175 (5)
C5	0.0348 (7)	0.0345 (7)	0.0276 (7)	-0.0003 (5)	0.0191 (6)	-0.0076 (5)
C4	0.0315 (7)	0.0324 (7)	0.0218 (6)	-0.0030 (5)	0.0150 (6)	-0.0043 (5)
C6	0.0319 (7)	0.0405 (8)	0.0235 (6)	-0.0026 (6)	0.0138 (6)	-0.0054 (5)
O1	0.0420 (7)	0.0938 (12)	0.0411 (7)	0.0188 (7)	0.0048 (6)	-0.0263 (7)
N	0.0410 (10)	0.0367 (9)	0.0405 (10)	0.000	0.0252 (9)	0.000
C9	0.0718 (15)	0.0875 (17)	0.0706 (14)	0.0197 (12)	0.0452 (13)	-0.0130 (12)
C3	0.0292 (7)	0.0373 (8)	0.0278 (7)	0.0024 (5)	0.0149 (6)	-0.0043 (5)
C8	0.0627 (13)	0.0587 (12)	0.0605 (13)	0.0133 (10)	0.0315 (11)	0.0174 (10)

Geometric parameters ( $\text{\AA}$ ,  $^\circ$ )

O2—C6	1.2739 (19)	N—C8	1.481 (2)
C5—C4	1.390 (2)	C9—H9A	0.9600
C5—C3 <sup>i</sup>	1.3896 (19)	C9—H9B	0.9600
C5—H5	0.9300	C9—H9C	0.9600
C4—C3	1.391 (2)	C3—C5 <sup>i</sup>	1.3896 (19)
C4—C6	1.5115 (18)	C3—H3	0.9300
C6—O1	1.218 (2)	C8—H8A	0.9600
N—C9 <sup>ii</sup>	1.478 (2)	C8—H8B	0.9600
N—C9	1.478 (2)	C8—H8C	0.9600
N—C8 <sup>ii</sup>	1.481 (2)		
C4—C5—C3 <sup>i</sup>	120.25 (13)	N—C9—H9A	109.5
C4—C5—H5	119.9	N—C9—H9B	109.5
C3 <sup>i</sup> —C5—H5	119.9	H9A—C9—H9B	109.5
C5—C4—C3	119.41 (12)	N—C9—H9C	109.5
C5—C4—C6	121.22 (12)	H9A—C9—H9C	109.5
C3—C4—C6	119.37 (13)	H9B—C9—H9C	109.5
O1—C6—O2	124.70 (13)	C5 <sup>i</sup> —C3—C4	120.33 (13)
O1—C6—C4	119.89 (14)	C5 <sup>i</sup> —C3—H3	119.8
O2—C6—C4	115.39 (13)	C4—C3—H3	119.8
C9 <sup>ii</sup> —N—C9	108.6 (3)	N—C8—H8A	109.5
C9 <sup>ii</sup> —N—C8 <sup>ii</sup>	109.57 (15)	N—C8—H8B	109.5
C9—N—C8 <sup>ii</sup>	110.17 (15)	H8A—C8—H8B	109.5
C9 <sup>ii</sup> —N—C8	110.17 (15)	N—C8—H8C	109.5
C9—N—C8	109.57 (15)	H8A—C8—H8C	109.5
C8 <sup>ii</sup> —N—C8	108.7 (2)	H8B—C8—H8C	109.5
C3 <sup>i</sup> —C5—C4—C3	0.2 (3)	C5—C4—C6—O2	4.7 (2)
C3 <sup>i</sup> —C5—C4—C6	179.82 (13)	C3—C4—C6—O2	-175.69 (14)
C5—C4—C6—O1	-176.70 (17)	C5—C4—C3—C5 <sup>i</sup>	-0.2 (3)
C3—C4—C6—O1	2.9 (2)	C6—C4—C3—C5 <sup>i</sup>	-179.83 (13)

Symmetry codes: (i)  $-x+1, -y+2, -z+1$ ; (ii)  $-x+1, y, -z+1/2$ .Hydrogen-bond geometry ( $\text{\AA}$ ,  $^\circ$ )

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
O2—H2 $\cdots$ O2 <sup>iii</sup>	1.23	1.23	2.4610 (19)	180 (1)
C8—H8A $\cdots$ O1 <sup>iv</sup>	0.96	2.39	3.267 (3)	152
C9—H9A $\cdots$ O1 <sup>iv</sup>	0.96	2.47	3.321 (3)	148

Symmetry codes: (iii)  $-x+1/2, -y+3/2, -z$ ; (iv)  $x+1/2, -y+3/2, z+1/2$ .