Tetraaquabis{5-[2-(1*H*-tetrazol-5-yl)-ethenyl]pyrazolato-κN^2}manganese(II) dihydrate. Corrigendum

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The chemical name in the title of the paper by Hu [*Acta Cryst.* (2008), E**64**, m772] is corrected.

In the paper by Hu [*Acta Cryst.* (2008), E**64**, m772], the chemical name in the title is incorrect. The correct title should be 'Tetraaquabis{5-[2-(1*H*-tetrazol-5-yl)ethenyl]tetrazolato-κN^2}manganese(II) dihydrate'.

metal-organic compounds

Tetraaquabis{5-[2-(1*H*-tetrazol-5-yl)-ethenyl]pyrazolato-κN^2}manganese(II) dihydrate

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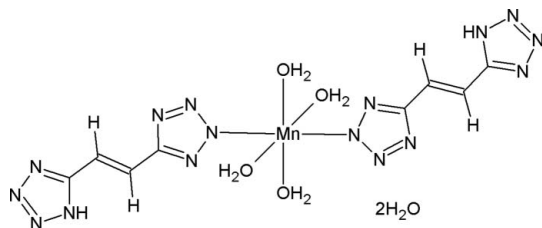Correspondence e-mail: hutuopingsx@yahoo.com.cn

The title compound, $[Mn(C_4H_3N_8)_2(H_2O)_4]\cdot2H_2O$, represents the first structurally characterized transition metal complex of the 1,2-bis(tetrazol-5-yl)ethene ligand. The complex molecule occupies a special position on an inversion centre and the Mn atom has a tetragonally distorted octahedral coordination. The bis(tetrazolyl)ethene ligand is planar within 0.0366 (7) Å. All 'active' H atoms participate in hydrogen bonds, which link molecules of the complex and the uncoordinated water molecules into an infinite three-dimensional framework.

Related literature

For related literature, see: Huang *et al.* (2005); Demko & Sharpless (2001).

Experimental

Crystal data

$[Mn(C_4H_3N_8)_2(H_2O)_4]\cdot2H_2O$	γ = 67.016 (1)°
M_r = 489.32	V = 484.70 (2) Å³
Triclinic, $P\bar{1}$	Z = 1
a = 6.2296 (2) Å	Mo $K\alpha$ radiation
b = 7.0093 (2) Å	μ = 0.75 mm⁻¹
c = 12.1212 (3) Å	T = 273 (2) K
α = 84.405 (1)°	0.36 × 0.28 × 0.16 mm
β = 89.457 (1)°	

Data collection

Bruker SMART CCD area-detector diffractometer	9107 measured reflections
	3246 independent reflections
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)	3149 reflections with $I > 2\sigma(I)$
T_{min} = 0.717, T_{max} = 0.887	R_{int} = 0.015

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.022	179 parameters
$wR(F^2)$ = 0.068	All H-atom parameters refined
S = 1.04	$\Delta\rho_{max}$ = 0.37 e Å⁻³
3246 reflections	$\Delta\rho_{min}$ = −0.22 e Å⁻³

Table 1
Selected geometric parameters (Å, °).

Mn1—O1	2.1923 (7)	Mn1—N2	2.2538 (7)
Mn1—O2	2.1835 (8)		
O2—Mn1—O1	84.57 (3)	O1—Mn1—N2	90.24 (3)
O2—Mn1—N2	91.07 (3)		

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

D—H$\cdots A$	D—H	H$\cdots A$	$D\cdots A$	D—H$\cdots A$
O1—H1$B\cdots$N1^i	0.833 (18)	2.021 (18)	2.8419 (10)	168.8 (17)
O1—H1$A\cdots$O3^{ii}	0.823 (18)	1.940 (18)	2.7599 (11)	174.0 (16)
O2—H2$B\cdots$O3^{iii}	0.790 (18)	1.996 (19)	2.7797 (11)	171.4 (18)
O2—H2$A\cdots$N6^{iv}	0.81 (2)	2.04 (2)	2.8472 (10)	173.7 (18)
O3—H3$A\cdots$N5^v	0.82 (2)	2.09 (2)	2.8922 (11)	164.2 (19)
O3—H3$B\cdots$O1	0.80 (2)	2.30 (2)	3.0693 (12)	160 (2)
N8—H8$\cdots$N4^{vi}	0.926 (18)	1.792 (18)	2.7171 (10)	176.6 (16)

Symmetry codes: (i) $x - 1, y, z$; (ii) $-x, -y + 1, -z + 1$; (iii) $-x + 1, -y + 1, -z + 1$; (iv) $x - 1, y, z + 1$; (v) $-x + 1, -y + 1, -z$; (vi) $x + 1, y, z$.

Data collection: *SMART* (Bruker, 2007); cell refinement: *SAINT-Plus* (Bruker, 2007); data reduction: *SAINT-Plus*; 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*.

References

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Demko, Z. P. & Sharpless, K. B. (2001). *J. Org. Chem.* **66**, 7945–7950.
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Sheldrick, G. M. (1996). *SADABS*. University of Göttingen, Germany.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.

supporting information

Tetraaquabis{5-[2-(1*H*-tetrazol-5-yl)ethenyl]pyrazolato-*κN²*}manganese(II) dihydrate

Tuoping Hu

S1. Comment

The 1,2-bis(1,2,3,4-tetrazol-5-yl)ethene ligand (H₂BTAE) was previously reported in its twice deprotonated form in the cystal structure of its sodium salt pentahydrate (Huang *et al.*, 2005). The present paper provides the fist example of its structurally characterized complex with a transition metal; the ligand in this complex is monodeprotonated.

The molecule of Mn(H-BTAE)₂(H₂O)₄ occupies a special position in the inversion centre (Fig. 1); the Mn1 atom has a tetragonally distorted octahedral coordination (Table 1). The H-BTAE ligand has essentailly planar conformation, the maximum deviation of the N6 atom from its mean plane being 0.0366 (7) Å. The geometry of the ligand is similar to the one observed in Huang *et al.* (2005).

All "active" hydrogen atoms in the structure participate in the H-bonding (Table 2); the extensive H-bond system links molecules of the complex and non-coordinated water molecules into three-dimensional infinite network (Fig. 2).

S2. Experimental

MnCl₂.4H₂O (0.5 mmol, 99 mg) and 1,2-bis(1,2,3,4-tetrazol-5-yl)ethene (1 mmol, 192 mg) (Demko & Sharpless, 2001) were added to 30 ml of water:MeOH (1:1) mixture. After stirring for 30 min at room temperature, the pH value was adjusted to 7 by 1*M* NaOH, and clear solution was allowed to evaporate in the air. Nice prism-shaped crystals of the title compound were obtained after 3 days. The crystals were filtered, washed by EtOH and dried in the air.

S3. Refinement

All H atoms were located in the difference map and refined isotropically [O—H 0.79 (2)–0.83 (2) Å; C—H 0.91 (1) and 0.96 (1) Å; N—H 0.93 (2) Å].
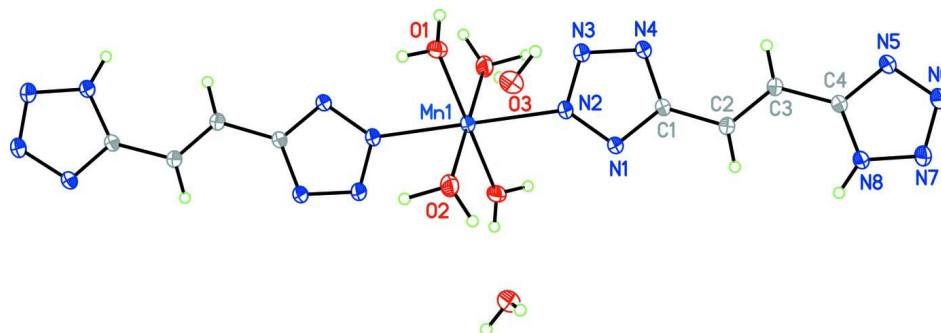

Figure 1

Molecular structure showing 50% probability displacement ellipsoids. The unlabeled atoms are derived from the reference atoms by means of the (1 - *x*, -*y*, 1 - *z*) symmetry transformation..
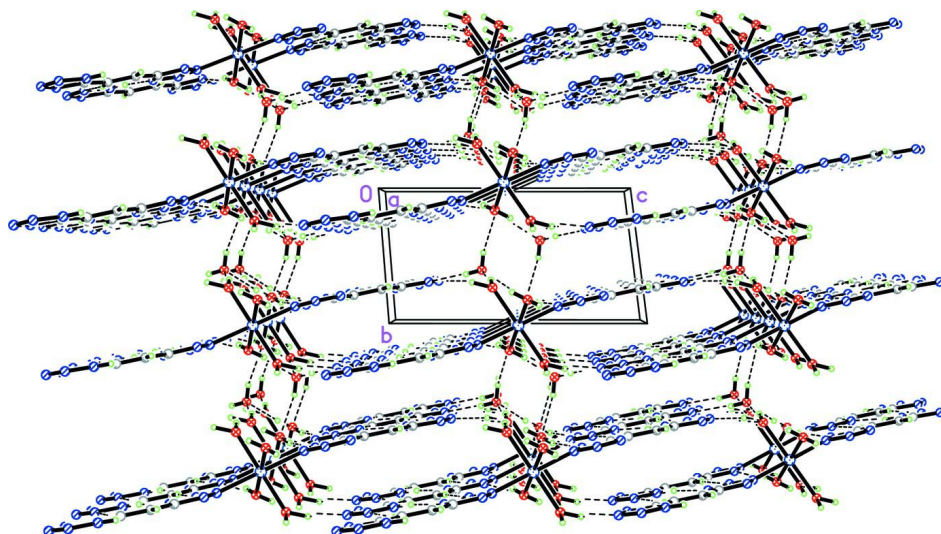
Figure 2

Packing diagram viewed down the *a* axis, The hydrogen bonds are shown as dotted lines.

Tetraaquabis{5-[2-(1H-tetrazol-5-yl)ethenyl]pyrazolato-*κ*N^2}manganese(II) dihydrate

Crystal data

[Mn(C$_4$H$_3$N$_8$)$_2$(H$_2$O)$_4$]·2H$_2$O
M_r = 489.32
Triclinic, $P\bar{1}$
Hall symbol: -P 1
a = 6.2296 (2) Å
b = 7.0093 (2) Å
c = 12.1212 (3) Å
α = 84.405 (1)°
β = 89.457 (1)°
γ = 67.016 (1)°
V = 484.70 (2) Å^3

Z = 1
$F(000)$ = 251
D_x = 1.676 Mg m^{-3}
Mo *Kα* radiation, λ = 0.71073 Å
Cell parameters from 7983 reflections
θ = 3.2–33.5°
μ = 0.75 mm^{-1}
T = 273 K
Prism, brown
0.36 × 0.28 × 0.16 mm

Data collection

Bruker SMART CCD area-detector
  diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
φ and *ω* scans
Absorption correction: multi-scan
  (*SADABS*; Sheldrick, 1996)
T_{min} = 0.717, T_{max} = 0.887

9107 measured reflections
3246 independent reflections
3149 reflections with $I > 2\sigma(I)$
R_{int} = 0.015
θ_{max} = 33.5°, θ_{min} = 3.2°
$h = -9 \rightarrow 9$
$k = -10 \rightarrow 10$
$l = -16 \rightarrow 18$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.022
$wR(F^2)$ = 0.068
S = 1.05
3246 reflections
179 parameters
0 restraints

Primary atom site location: structure-invariant
  direct methods
Secondary atom site location: difference Fourier
  map
Hydrogen site location: difference Fourier map
All H-atom parameters refined
$w = 1/[\sigma^2(F_o^2) + (0.039P)^2 + 0.093P]$
  where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.37$ e Å^{-3}
$\Delta\rho_{min} = -0.22$ e Å^{-3}

Extinction correction: *SHELXL97* (Sheldrick, 2008), Fc*=kFc[1+0.001xFc$^2\lambda^3$/sin(2θ)]$^{-1/4}$
Extinction coefficient: 0.022 (3)

Special details

Experimental. H atoms were located on intermediate difference Fourier map

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}
Mn1	0.5000	0.0000	0.5000	0.02323 (6)
O1	0.13139 (12)	0.18659 (13)	0.45633 (6)	0.03342 (15)
O2	0.48182 (15)	0.26842 (14)	0.58308 (6)	0.03732 (17)
O3	0.15295 (14)	0.60925 (13)	0.38023 (6)	0.03452 (15)
H3A	0.108 (4)	0.639 (3)	0.3151 (17)	0.063 (5)*
H3B	0.180 (4)	0.488 (3)	0.3935 (17)	0.068 (6)*
N1	0.80858 (13)	0.13919 (13)	0.30839 (6)	0.02546 (14)
N2	0.60467 (13)	0.13162 (13)	0.34123 (6)	0.02647 (14)
N3	0.46087 (14)	0.17201 (14)	0.25572 (6)	0.02953 (16)
N4	0.56698 (13)	0.20725 (13)	0.16459 (6)	0.02663 (15)
N5	1.08450 (14)	0.30165 (13)	−0.16586 (6)	0.02657 (14)
N6	1.28887 (14)	0.30586 (14)	−0.20234 (6)	0.02983 (16)
N7	1.43373 (14)	0.27581 (14)	−0.12043 (6)	0.03067 (16)
N8	1.32424 (13)	0.25048 (12)	−0.02771 (6)	0.02534 (14)
C1	0.78023 (14)	0.18638 (13)	0.19858 (6)	0.02208 (14)
C2	0.96111 (15)	0.20610 (14)	0.12755 (7)	0.02469 (15)
C3	0.93208 (15)	0.24900 (14)	0.01755 (7)	0.02401 (15)
C4	1.11010 (14)	0.26607 (13)	−0.05597 (6)	0.02185 (14)
H3	0.794 (2)	0.271 (2)	−0.0175 (12)	0.035 (3)*
H2	1.104 (3)	0.186 (2)	0.1658 (12)	0.039 (4)*
H8	1.405 (3)	0.232 (3)	0.0389 (15)	0.057 (5)*
H1A	0.047 (3)	0.239 (3)	0.5070 (15)	0.046 (4)*
H1B	0.050 (3)	0.155 (3)	0.4134 (15)	0.055 (5)*
H2B	0.587 (3)	0.304 (3)	0.5865 (15)	0.052 (4)*
H2A	0.417 (3)	0.280 (3)	0.6422 (17)	0.057 (5)*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Mn1	0.02135 (10)	0.03467 (10)	0.01477 (8)	−0.01276 (7)	0.00178 (6)	−0.00004 (6)
O1	0.0225 (3)	0.0535 (4)	0.0236 (3)	−0.0133 (3)	−0.0009 (2)	−0.0078 (3)

O2	0.0443 (4)	0.0551 (5)	0.0264 (3)	−0.0324 (4)	0.0122 (3)	−0.0141 (3)
O3	0.0370 (4)	0.0392 (4)	0.0279 (3)	−0.0154 (3)	−0.0016 (3)	−0.0036 (3)
N1	0.0241 (3)	0.0396 (4)	0.0164 (3)	−0.0169 (3)	0.0007 (2)	−0.0007 (2)
N2	0.0249 (3)	0.0414 (4)	0.0161 (3)	−0.0170 (3)	0.0019 (2)	0.0003 (3)
N3	0.0255 (3)	0.0484 (4)	0.0179 (3)	−0.0190 (3)	0.0011 (2)	0.0012 (3)
N4	0.0250 (3)	0.0419 (4)	0.0161 (3)	−0.0174 (3)	0.0002 (2)	0.0011 (3)
N5	0.0283 (3)	0.0380 (4)	0.0167 (3)	−0.0167 (3)	0.0015 (2)	−0.0020 (3)
N6	0.0302 (4)	0.0437 (4)	0.0185 (3)	−0.0178 (3)	0.0051 (3)	−0.0027 (3)
N7	0.0268 (4)	0.0464 (4)	0.0213 (3)	−0.0174 (3)	0.0052 (3)	−0.0024 (3)
N8	0.0230 (3)	0.0373 (4)	0.0172 (3)	−0.0138 (3)	0.0015 (2)	−0.0006 (3)
C1	0.0232 (3)	0.0296 (3)	0.0159 (3)	−0.0133 (3)	0.0014 (2)	−0.0012 (2)
C2	0.0236 (4)	0.0351 (4)	0.0190 (3)	−0.0158 (3)	0.0024 (3)	−0.0014 (3)
C3	0.0234 (4)	0.0332 (4)	0.0189 (3)	−0.0150 (3)	0.0025 (3)	−0.0019 (3)
C4	0.0234 (3)	0.0277 (3)	0.0164 (3)	−0.0122 (3)	0.0017 (2)	−0.0021 (2)

Geometric parameters (Å, º)

Mn1—O2^i	2.1835 (8)	N2—N3	1.3112 (10)
Mn1—O1	2.1923 (7)	N3—N4	1.3336 (9)
Mn1—O2	2.1835 (8)	N4—C1	1.3427 (11)
Mn1—O1^i	2.1923 (7)	N5—C4	1.3304 (10)
Mn1—N2^i	2.2538 (7)	N5—N6	1.3541 (10)
Mn1—N2	2.2538 (7)	N6—N7	1.2931 (11)
O1—H1A	0.823 (18)	N7—N8	1.3420 (10)
O1—H1B	0.833 (18)	N8—C4	1.3403 (11)
O2—H2B	0.790 (18)	N8—H8	0.926 (18)
O2—H2A	0.81 (2)	C1—C2	1.4526 (11)
O3—H3A	0.82 (2)	C2—C3	1.3360 (11)
O3—H3B	0.80 (2)	C2—H2	0.963 (15)
N1—C1	1.3361 (10)	C3—C4	1.4499 (11)
N1—N2	1.3468 (10)	C3—H3	0.914 (14)
O2^i—Mn1—O2	180.0	N3—N2—N1	110.34 (6)
O2^i—Mn1—O1	95.43 (3)	N3—N2—Mn1	115.77 (5)
O2—Mn1—O1	84.57 (3)	N1—N2—Mn1	132.31 (6)
O2^i—Mn1—O1^i	84.57 (3)	N2—N3—N4	108.57 (7)
O2—Mn1—O1^i	95.43 (3)	N3—N4—C1	105.93 (7)
O1—Mn1—O1^i	180.0	C4—N5—N6	105.63 (7)
O2^i—Mn1—N2^i	91.07 (3)	N7—N6—N5	111.07 (7)
O2—Mn1—N2^i	88.93 (3)	N6—N7—N8	106.55 (7)
O1—Mn1—N2^i	89.76 (3)	C4—N8—N7	108.63 (7)
O1^i—Mn1—N2^i	90.24 (3)	C4—N8—H8	134.5 (11)
O2^i—Mn1—N2	88.93 (3)	N7—N8—H8	116.8 (11)
O2—Mn1—N2	91.07 (3)	N1—C1—N4	110.73 (7)
O1—Mn1—N2	90.24 (3)	N1—C1—C2	123.47 (7)
O1^i—Mn1—N2	89.76 (3)	N4—C1—C2	125.79 (7)
N2^i—Mn1—N2	179.999 (2)	C3—C2—C1	122.84 (8)
Mn1—O1—H1A	116.9 (11)	C3—C2—H2	122.3 (9)

Mn1—O1—H1B	125.3 (12)	C1—C2—H2	114.9 (9)
H1A—O1—H1B	106.3 (16)	C2—C3—C4	124.29 (8)
Mn1—O2—H2B	123.3 (13)	C2—C3—H3	121.3 (9)
Mn1—O2—H2A	114.9 (13)	C4—C3—H3	114.4 (9)
H2B—O2—H2A	109.2 (17)	N5—C4—N8	108.11 (7)
H3A—O3—H3B	105.6 (18)	N5—C4—C3	124.43 (8)
C1—N1—N2	104.43 (7)	N8—C4—C3	127.46 (7)
C1—N1—N2—N3	0.04 (10)	N6—N7—N8—C4	−0.01 (10)
C1—N1—N2—Mn1	164.68 (7)	N2—N1—C1—N4	−0.02 (10)
O2^{i}—Mn1—N2—N3	61.58 (7)	N2—N1—C1—C2	−178.57 (8)
O2—Mn1—N2—N3	−118.42 (7)	N3—N4—C1—N1	−0.01 (10)
O1—Mn1—N2—N3	−33.84 (7)	N3—N4—C1—C2	178.50 (8)
O1^{i}—Mn1—N2—N3	146.16 (7)	N1—C1—C2—C3	178.52 (9)
O2^{i}—Mn1—N2—N1	−102.42 (8)	N4—C1—C2—C3	0.19 (14)
O2—Mn1—N2—N1	77.58 (8)	C1—C2—C3—C4	−178.84 (8)
O1—Mn1—N2—N1	162.16 (8)	N6—N5—C4—N8	0.29 (10)
O1^{i}—Mn1—N2—N1	−17.84 (8)	N6—N5—C4—C3	−179.93 (8)
N1—N2—N3—N4	−0.05 (11)	N7—N8—C4—N5	−0.18 (10)
Mn1—N2—N3—N4	−167.49 (6)	N7—N8—C4—C3	−179.96 (8)
N2—N3—N4—C1	0.03 (10)	C2—C3—C4—N5	177.57 (9)
C4—N5—N6—N7	−0.30 (10)	C2—C3—C4—N8	−2.68 (15)
N5—N6—N7—N8	0.19 (11)		

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

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1*B*···N1^{ii}	0.833 (18)	2.021 (18)	2.8419 (10)	168.8 (17)
O1—H1*A*···O3^{iii}	0.823 (18)	1.940 (18)	2.7599 (11)	174.0 (16)
O2—H2*B*···O3^{iv}	0.790 (18)	1.996 (19)	2.7797 (11)	171.4 (18)
O2—H2*A*···N6^{v}	0.81 (2)	2.04 (2)	2.8472 (10)	173.7 (18)
O3—H3*A*···N5^{vi}	0.82 (2)	2.09 (2)	2.8922 (11)	164.2 (19)
O3—H3*B*···O1	0.80 (2)	2.30 (2)	3.0693 (12)	160 (2)
N8—H8···N4^{vii}	0.926 (18)	1.792 (18)	2.7171 (10)	176.6 (16)

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