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trans-Diaquabis[5-(pyridine-3-carboxamido)tetrazolido-κ^2O,N^1]zinc dihydrate

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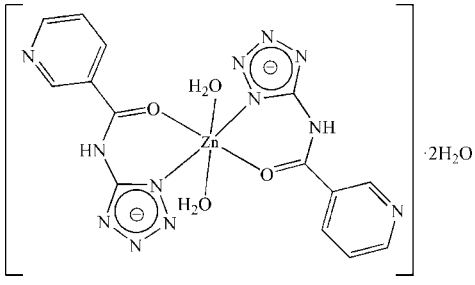
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.003 Å; R factor = 0.045; wR factor = 0.100; data-to-parameter ratio = 14.4.

The title compound, $[Zn(C_7H_5N_6O)_2(H_2O)_2]\cdot 2H_2O$, consists of one Zn^{II} ion located on the crystallographic inversion centre, two 5-(pyridine-3-carboxamido)tetrazolide ligands, two coordinated water molecules and two free water molecules. The Zn^{II} ion adopts a slightly distorted octahedral coordination geometry formed by the *N*,*O*-chelating ligands and two O water atoms. The pyridine N atoms are not coordinated. In the crystal, complex molecules are connected by N—H···O, O—H···N and O—H···O hydrogen bonds, forming a three-dimensional network.

Related literature

For pharmaceutical applications of amide derivatives, see: Foster *et al.* (1999); Rauko *et al.* (2001); Rowland *et al.* (2001, 2002). For our recent work on the design and synthesis of amide complexes, see: Wang *et al.* (2010). For the use of nicotinoylamino in building novel complexes, see: Aakeröy *et al.* (2001); Li *et al.* (2008); Moncol *et al.* (2007); Kumar *et al.* (2005). For Zn—N and Zn—O bond lengths in related structures, see: Armstrong *et al.* (2003); Liu *et al.* (2009).



Experimental

Crystal data

$[Zn(C_7H_5N_6O)_2(H_2O)_2]\cdot 2H_2O$
M_r = 515.79
Monoclinic, $P2_1/n$
a = 7.2576 (15) Å
b = 12.008 (2) Å
c = 11.917 (2) Å
β = 97.76 (3)°
V = 1029.1 (3) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 1.26 mm^{-1}
T = 293 K
0.22 × 0.15 × 0.1 mm

Data collection

Rigaku Saturn 724 CCD area-detector diffractometer
Absorption correction: multi-scan (*CrystalClear*; Rigaku, 2002)
T_{min} = 0.819, T_{max} = 1.000
8464 measured reflections
2374 independent reflections
2188 reflections with $I > 2\sigma(I)$
R_{int} = 0.043

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.045
$wR(F^2)$ = 0.100
S = 1.19
2374 reflections
165 parameters
6 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.24 e Å^{-3}
$\Delta\rho_{min}$ = −0.33 e Å^{-3}

Table 1
Selected bond lengths (Å).

Zn1—N2	2.058 (2)	Zn1—O1	2.1470 (17)
Zn1—O2	2.131 (2)		

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H6···O3	0.86	2.04	2.829 (3)	153
O2—H2A···N5^{i}	0.84 (1)	1.97 (1)	2.795 (3)	165 (3)
O2—H2B···N6^{ii}	0.84 (1)	1.89 (1)	2.727 (3)	178 (3)
O3—H3A···O2^{i}	0.84 (1)	2.08 (2)	2.843 (3)	152 (4)
O3—H3B···N4^{iii}	0.84 (1)	2.09 (1)	2.907 (3)	164 (4)

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

Data collection: *CrystalClear* (Rigaku, 2002); cell refinement: *CrystalClear*; data reduction: *CrystalClear*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 2008); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HG5200).

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trans-Diaquabis[5-(pyridine-3-carboxamido)tetrazolido-κ^2O,N^1]zinc dihydrate

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S1. Comment

In recent years, many metal compounds derived from amides, have been prepared and characterized, and have been found to possess a wide variety of pharmic applications (Foster *et al.*, 1999; Rauko *et al.*, 2001; Rowland *et al.*, 2001; Rowland *et al.*, 2002). Amides were used to construct extended frameworks sustained both by hydrogen bonds and coordination bonds owing to the inherent coordination and hydrogenbonding donor/acceptor functionalities (Aakeröy *et al.*, 2001; Li *et al.*, 2008; Moncol *et al.*, 2007; Kumar *et al.*, 2005). In this paper, we report the crystal structure of the zinc-amide complex.

The asymmetric unit of complex, (I), contains one Zn^{II} ion located on an inversion centre, one independent *N*-(tetrazol-5-yl)-nicotinamide ligand, one coordination water molecule and one free water molecule. The central Zn^{II} ion adopts a slightly distorted octahedral coordination geometry by two ligands and two coordination water molecules. The equatorial plane is formed by two tetrazole N atoms and two O atoms in bis-N, O-chelating coordination from two *N*-(tetrazol-5-yl)-nicotinamide ligands, while the axial positions are occupied by two O atoms from two coordination water molecules. The Zn–N bond length is 2.058 (2) Å. The bond lengths of Zn–O are in the range 2.131 (2)–2.147 (1) Å. All the Zn–N or Zn–O bond lengths are comparable to those reported previously for zinc compounds (Armstrong *et al.*, 2003; Liu *et al.*, 2009). The dihedral angle between tetrazole and pyridine groups is 45.988 (1)°.

In the crystal, the complex molecules are connected to a two dimensional layer by intermolecular N–H···O_{free} (O atoms from free water molecules) hydrogen bonds (Fig. 2, Table 2). The layered strcture form a three-dimensional network *via* O_{free}–H···O_{coord} (O atoms from coordination water molecules), O_{free}–H···N and O_{coord}–H···N hydrogen bonds.

S2. Experimental

The title compound was synthesized by reacting the ligand (*N*-(1*H*-tetrazol-5-yl)-nicotinamide) (0.019 g,0.01 mmol) with $Zn(CH_3COO)_2{\cdot}2H_2O$ (0.011 g, 0.05 mmol) in 5.0 ml of dimethyl sulfoxide followed by the addition of 4 ml of ethanol. The muddy solution obtained was stirred at room temperature for three hours, filtered and set aside to slowly crystallize at room temperature. The block-like crystals were obtained after about three weeks.

S3. Refinement

Four reflections, -2 1 1, -5 5 2, -5 2 3, -1 0 1, shaded by beamstop were omitted. All H atoms bonded to C and N atoms were refined in idealized positions using the riding-model approximation, with C–H = 0.93 Å, U_{iso}(H) = 1.2 U_{eq}(C) and N–H = 0.93 Å, U_{iso}(H) = 1.2 U_{eq}(N). In water molecule the O–H distances were restrained to 0.84 (5) Å, and the distance H···H to 1.32 (2) Å, with U_{iso}(H) = 1.5 U_{eq}(O).

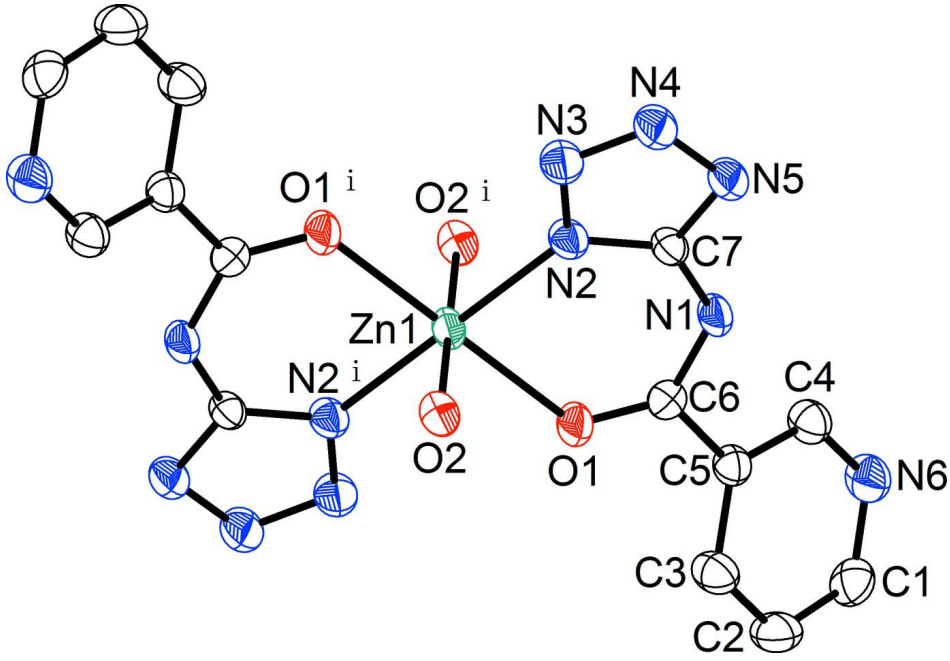

Figure 1

View of the coordination environment of Zn^{2+} in title compound at 50%. H atoms have been omitted for clarity. [Symmetry codes: (i) -*x*, -*y*, -*z*.]

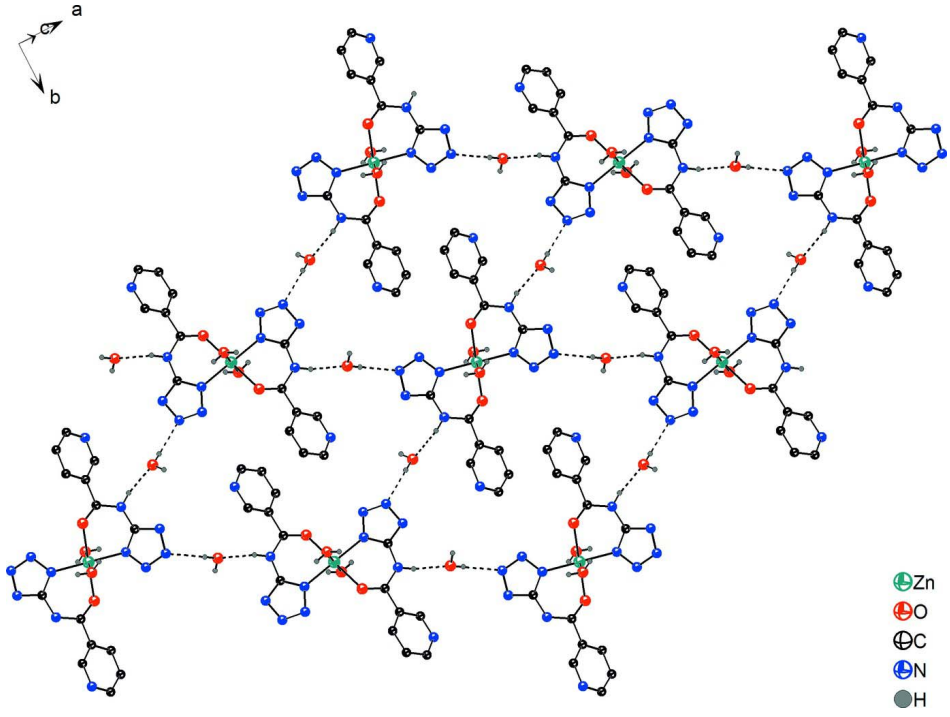

Figure 2

A view of the two-dimensional structure formed *via* hydrogen bonds.

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Crystal data

$[Zn(C_7H_5N_6O)_2(H_2O)_2]\cdot 2H_2O$
$M_r = 515.79$
Monoclinic, $P2_1/n$
Hall symbol: -P 2yn
$a = 7.2576$ (15) Å
$b = 12.008$ (2) Å
$c = 11.917$ (2) Å
$\beta = 97.76$ (3)°
$V = 1029.1$ (3) Å^3
$Z = 2$
$F(000) = 528$
$D_x = 1.665$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 3552 reflections
$\theta = 3.1$–27.5°
$\mu = 1.26$ mm^{-1}
$T = 293$ K
Prism, colorless
0.22 × 0.15 × 0.1 mm

Data collection

Rigaku Saturn 724 CCD area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
scintillation counter scans
Absorption correction: multi-scan (*CrystalClear*; Rigaku, 2002)
$T_{min} = 0.819$, $T_{max} = 1.000$
8464 measured reflections
2374 independent reflections
2188 reflections with $I > 2\sigma(I)$
$R_{int} = 0.043$
$\theta_{max} = 27.6°$, $\theta_{min} = 3.3°$
$h = -9\rightarrow 7$
$k = -15\rightarrow 14$
$l = -15\rightarrow 15$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.045$
$wR(F^2) = 0.100$
$S = 1.19$
2374 reflections
165 parameters
6 restraints
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
$w = 1/[\sigma^2(F_o^2) + (0.0354P)^2 + 0.6596P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.042$
$\Delta\rho_{max} = 0.24$ e Å^{-3}
$\Delta\rho_{min} = -0.33$ e Å^{-3}

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}
Zn1	0.0000	0.0000	0.0000	0.02817 (14)
O1	0.1128 (2)	0.16405 (14)	−0.01319 (16)	0.0348 (4)
C5	0.2633 (3)	0.3319 (2)	0.0481 (2)	0.0274 (5)

C7	0.3943 (3)	0.0364 (2)	0.1181 (2)	0.0259 (5)
C6	0.2446 (3)	0.2086 (2)	0.0460 (2)	0.0281 (5)
N4	0.5071 (3)	−0.12357 (19)	0.1550 (2)	0.0364 (5)
N5	0.5487 (3)	−0.01344 (18)	0.1645 (2)	0.0342 (5)
N6	0.3527 (3)	0.49961 (18)	0.1502 (2)	0.0370 (5)
C4	0.3380 (4)	0.3890 (2)	0.1451 (2)	0.0333 (6)
H12	0.3799	0.3480	0.2097	0.040*
C3	0.1991 (4)	0.3936 (2)	−0.0474 (2)	0.0349 (6)
H13	0.1448	0.3584	−0.1132	0.042*
C1	0.2944 (4)	0.5576 (2)	0.0566 (2)	0.0377 (6)
H15	0.3064	0.6347	0.0586	0.045*
C2	0.2168 (4)	0.5077 (2)	−0.0436 (3)	0.0415 (7)
H16	0.1772	0.5506	−0.1072	0.050*
N1	0.3798 (3)	0.15158 (17)	0.11143 (18)	0.0309 (5)
H6	0.4637	0.1899	0.1523	0.037*
N3	0.3383 (3)	−0.13789 (18)	0.1054 (2)	0.0344 (5)
N2	0.2617 (3)	−0.03656 (18)	0.08075 (18)	0.0279 (4)
O2	0.0833 (3)	−0.05113 (17)	−0.15682 (16)	0.0340 (4)
H2A	0.186 (2)	−0.025 (3)	−0.169 (3)	0.066 (12)*
H2B	0.011 (3)	−0.037 (3)	−0.2160 (16)	0.061 (11)*
O3	0.7255 (3)	0.25284 (18)	0.1891 (2)	0.0517 (6)
H3A	0.801 (4)	0.208 (3)	0.166 (3)	0.078*
H3B	0.787 (4)	0.287 (3)	0.243 (2)	0.078*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Zn1	0.0224 (2)	0.0244 (2)	0.0352 (2)	−0.00194 (15)	−0.00520 (16)	−0.00004 (16)
O1	0.0314 (10)	0.0247 (9)	0.0444 (11)	−0.0060 (7)	−0.0087 (8)	0.0033 (8)
C5	0.0270 (12)	0.0251 (12)	0.0292 (12)	−0.0025 (9)	0.0006 (10)	−0.0005 (10)
C7	0.0250 (12)	0.0264 (11)	0.0248 (11)	−0.0005 (9)	−0.0015 (9)	0.0007 (9)
C6	0.0298 (13)	0.0257 (12)	0.0284 (12)	−0.0027 (10)	0.0020 (10)	0.0018 (10)
N4	0.0299 (12)	0.0346 (12)	0.0426 (13)	0.0043 (9)	−0.0025 (10)	0.0052 (10)
N5	0.0297 (12)	0.0320 (12)	0.0382 (13)	−0.0006 (9)	−0.0055 (9)	0.0048 (9)
N6	0.0414 (14)	0.0302 (12)	0.0374 (12)	−0.0022 (10)	−0.0019 (10)	−0.0053 (9)
C4	0.0377 (15)	0.0295 (13)	0.0313 (13)	−0.0005 (11)	−0.0007 (11)	−0.0019 (10)
C3	0.0400 (15)	0.0312 (13)	0.0312 (13)	−0.0004 (11)	−0.0030 (11)	−0.0018 (10)
C1	0.0411 (16)	0.0248 (13)	0.0466 (17)	−0.0026 (11)	0.0042 (13)	−0.0005 (11)
C2	0.0529 (18)	0.0323 (15)	0.0372 (15)	0.0014 (12)	−0.0014 (13)	0.0083 (11)
N1	0.0290 (11)	0.0254 (10)	0.0348 (11)	−0.0046 (9)	−0.0083 (9)	−0.0016 (9)
N3	0.0317 (12)	0.0257 (11)	0.0437 (13)	0.0020 (9)	−0.0026 (10)	0.0036 (9)
N2	0.0253 (11)	0.0243 (10)	0.0325 (11)	−0.0001 (8)	−0.0023 (9)	0.0008 (8)
O2	0.0280 (10)	0.0396 (11)	0.0332 (10)	−0.0005 (8)	−0.0005 (8)	−0.0006 (8)
O3	0.0387 (12)	0.0347 (12)	0.0765 (17)	−0.0038 (9)	−0.0109 (11)	−0.0120 (10)

Geometric parameters (Å, º)

Zn1—N2	2.058 (2)	N4—N5	1.358 (3)
Zn1—N2^{i}	2.058 (2)	N6—C4	1.333 (3)
Zn1—O2^{i}	2.131 (2)	N6—C1	1.334 (4)
Zn1—O2	2.131 (2)	C4—H12	0.9300
Zn1—O1	2.1470 (17)	C3—C2	1.376 (4)
Zn1—O1^{i}	2.1470 (17)	C3—H13	0.9300
O1—C6	1.232 (3)	C1—C2	1.386 (4)
C5—C3	1.385 (4)	C1—H15	0.9300
C5—C4	1.389 (3)	C2—H16	0.9300
C5—C6	1.487 (3)	N1—H6	0.8600
C7—N5	1.323 (3)	N3—N2	1.353 (3)
C7—N2	1.332 (3)	O2—H2A	0.840 (5)
C7—N1	1.389 (3)	O2—H2B	0.839 (5)
C6—N1	1.354 (3)	O3—H3A	0.837 (5)
N4—N3	1.298 (3)	O3—H3B	0.838 (5)
N2—Zn1—N2^{i}	180.00 (16)	C7—N5—N4	103.8 (2)
N2—Zn1—O2^{i}	90.26 (8)	C4—N6—C1	117.9 (2)
N2^{i}—Zn1—O2^{i}	89.74 (8)	N6—C4—C5	123.3 (2)
N2—Zn1—O2	89.74 (8)	N6—C4—H12	118.3
N2^{i}—Zn1—O2	90.26 (8)	C5—C4—H12	118.3
O2^{i}—Zn1—O2	180.0	C2—C3—C5	119.0 (3)
N2—Zn1—O1	83.86 (8)	C2—C3—H13	120.5
N2^{i}—Zn1—O1	96.14 (8)	C5—C3—H13	120.5
O2^{i}—Zn1—O1	87.47 (8)	N6—C1—C2	122.7 (2)
O2—Zn1—O1	92.53 (8)	N6—C1—H15	118.6
N2—Zn1—O1^{i}	96.14 (8)	C2—C1—H15	118.6
N2^{i}—Zn1—O1^{i}	83.86 (8)	C3—C2—C1	119.0 (3)
O2^{i}—Zn1—O1^{i}	92.53 (8)	C3—C2—H16	120.5
O2—Zn1—O1^{i}	87.47 (8)	C1—C2—H16	120.5
O1—Zn1—O1^{i}	180.00 (11)	C6—N1—C7	125.5 (2)
C6—O1—Zn1	129.12 (16)	C6—N1—H6	117.3
C3—C5—C4	118.1 (2)	C7—N1—H6	117.3
C3—C5—C6	120.0 (2)	N4—N3—N2	108.3 (2)
C4—C5—C6	122.0 (2)	C7—N2—N3	105.2 (2)
N5—C7—N2	112.0 (2)	C7—N2—Zn1	126.55 (18)
N5—C7—N1	121.8 (2)	N3—N2—Zn1	128.27 (16)
N2—C7—N1	126.1 (2)	Zn1—O2—H2A	114 (2)
O1—C6—N1	123.8 (2)	Zn1—O2—H2B	117 (2)
O1—C6—C5	120.3 (2)	H2A—O2—H2B	104.1 (12)
N1—C6—C5	115.9 (2)	H3A—O3—H3B	104.7 (13)
N3—N4—N5	110.7 (2)		

Symmetry code: (i) $-x, -y, -z$.

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H6···O3	0.86	2.04	2.829 (3)	153
O2—H2*A*···N5^{ii}	0.84 (1)	1.97 (1)	2.795 (3)	165 (3)
O2—H2*B*···N6^{iii}	0.84 (1)	1.89 (1)	2.727 (3)	178 (3)
O3—H3*A*···O2^{ii}	0.84 (1)	2.08 (2)	2.843 (3)	152 (4)
O3—H3*B*···N4^{iv}	0.84 (1)	2.09 (1)	2.907 (3)	164 (4)

Symmetry codes: (ii) −*x*+1, −*y*, −*z*; (iii) *x*−1/2, −*y*+1/2, *z*−1/2; (iv) −*x*+3/2, *y*+1/2, −*z*+1/2.