Acta Crystallographica Section E
**Structure Reports Online**
ISSN 1600-5368

# *trans*-Tetraaquabis(isonicotinamide-$\kappa N^1$)zinc bis(3-hydroxybenzoate) tetrahydrate

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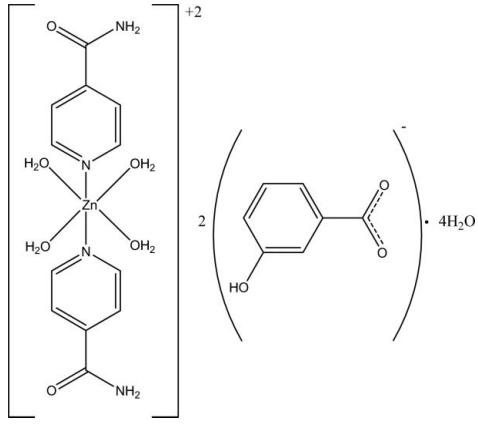
Received 4 March 2013; accepted 6 March 2013

Key indicators: single-crystal X-ray study; $T$ = 100 K; mean $\sigma$(C–C) = 0.004 Å; $R$ factor = 0.047; $wR$ factor = 0.114; data-to-parameter ratio = 14.4.

The asymmetric unit of the title compound, $[Zn(C_6H_6N_2O)_2(H_2O)_4](C_7H_5O_3)_2\cdot 4H_2O$, contains half of the complex cation with the $Zn^{II}$ ion located on an inversion center, a 3-hydroxybenzoate counter-anion and two uncoordinating water molecules. Four water O atoms in the equatorial plane around the $Zn^{II}$ ion [Zn—O = 2.089 (2) and 2.128 (2) Å] form a slightly distorted square-planar arrangement and the distorted octahedral geometry is completed by the two N atoms [Zn—N = 2.117 (2) Å] from two isonicotinamide ligands. In the anion, the carboxylate group is twisted from the attached benzene ring at 9.0 (2)°. In the crystal, a three-dimensional hydrogen-bonding network, formed by classical O—H···O and N—H···O and weak C—H···O hydrogen bonds, consolidates the crystal packing, which exhibits $\pi$–$\pi$ stacking between the benzene and pyridine rings, with centroid–centroid distances of 3.458 (2) and 3.609 (2) Å. One of the two H atoms of each uncoordinating water molecule is disordered over two orientations with an occupancy ratio of 0.60:0.40.

## Related literature

For related structures, see: Hökelek *et al.* (2009*a*,*b*,*c*,*d*,*e*); Sertçelik *et al.* (2009*a*,*b*). For isostructural Ni and Co complexes, see: Zaman *et al.* (2012*a*,*b*).



## Experimental

### Crystal data

$[Zn(C_6H_6N_2O)_2(H_2O)_4](C_7H_5O_3)_2\cdot 4H_2O$
$M_r$ = 727.99
Monoclinic, $P2_1/n$
$a$ = 6.7002 (2) Å
$b$ = 17.0005 (4) Å
$c$ = 13.6000 (3) Å
$\beta$ = 99.993 (3)°
$V$ = 1525.63 (7) Å$^3$
$Z$ = 2
Mo $K\alpha$ radiation
$\mu$ = 0.89 mm$^{-1}$
$T$ = 100 K
0.38 × 0.38 × 0.32 mm

### Data collection

Bruker Kappa APEXII CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2005)
$T_{min}$ = 0.720, $T_{max}$ = 0.752
14180 measured reflections
3808 independent reflections
3497 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.033

### Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.047
$wR(F^2)$ = 0.114
$S$ = 1.27
3808 reflections
264 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 1.41 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.47 e Å$^{-3}$

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

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| O3—H3$A$···O8$^{i}$ | 0.83 (5) | 1.88 (5) | 2.705 (3) | 172 (5) |
| N2—H21···O7$^{ii}$ | 0.83 (4) | 2.24 (4) | 3.017 (3) | 157 (3) |
| N2—H22···O2$^{i}$ | 0.82 (4) | 2.21 (4) | 3.016 (3) | 172 (3) |
| O5—H51···O2$^{iii}$ | 0.85 (5) | 1.98 (5) | 2.800 (3) | 162 (4) |
| O5—H52···O3$^{ii}$ | 0.76 (4) | 1.97 (4) | 2.719 (3) | 170 (4) |
| O6—H61···O2$^{iv}$ | 0.83 (5) | 1.89 (5) | 2.689 (3) | 161 (5) |
| O6—H62···O4$^{v}$ | 0.77 (5) | 1.92 (5) | 2.687 (3) | 179 (5) |
| O7—H71···O1 | 0.85 (5) | 1.91 (5) | 2.761 (3) | 178 (3) |
| O7—H72$A$···O8$^{vi}$ | 0.76 (9) | 2.08 (9) | 2.814 (4) | 163 (8) |
| O7—H72$B$···O7$^{vii}$ | 0.78 (9) | 2.03 (9) | 2.783 (3) | 160 (8) |
| O8—H81···O1 | 0.89 (5) | 1.85 (5) | 2.739 (3) | 177 (4) |
| O8—H82$A$···O7$^{viii}$ | 0.69 (8) | 2.13 (8) | 2.814 (4) | 167 (6) |
| O8—H82$B$···O8$^{ix}$ | 0.82 (9) | 1.96 (9) | 2.787 (3) | 178 (6) |
| C11—H11···O7$^{ii}$ | 0.93 | 2.54 | 3.455 (3) | 168 |

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

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:

*ORTEP-3 for Windows* (Farrugia, 2012); software used to prepare material for publication: *WinGX* (Farrugia, 2012) and *PLATON* (Spek, 2009).

The authors are indebted to Anadolu University and the Medicinal Plants and Medicine Research Centre of Anadolu University, Eskişehir, Turkey, for the use of the X-ray diffractometer.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: XU5683).

## References

Bruker (2005). *SADABS*. Bruker AXS Inc., Madison, Wisconsin, USA.
Bruker (2007). *APEX2* and *SAINT*. Bruker AXS Inc., Madison, Wisconsin, USA.
Farrugia, L. J. (2012). *J. Appl. Cryst.* **45**, 849–854.
Hökelek, T., Dal, H., Tercan, B., Aybirdi, Ö. & Necefoğlu, H. (2009*a*). *Acta Cryst.* E**65**, m627–m628.
Hökelek, T., Dal, H., Tercan, B., Özbek, F. E. & Necefoğlu, H. (2009*b*). *Acta Cryst.* E**65**, m1330–m1331.
Hökelek, T., Yılmaz, F., Tercan, B., Gürgen, F. & Necefoğlu, H. (2009*c*). *Acta Cryst.* E**65**, m1101–m1102.
Hökelek, T., Yılmaz, F., Tercan, B., Özbek, F. E. & Necefoğlu, H. (2009*d*). *Acta Cryst.* E**65**, m768–m769.
Hökelek, T., Yılmaz, F., Tercan, B., Sertçelik, M. & Necefoğlu, H. (2009*e*). *Acta Cryst.* E**65**, m1130–m1131.
Sertçelik, M., Tercan, B., Şahin, E., Necefoğlu, H. & Hökelek, T. (2009*a*). *Acta Cryst.* E**65**, m326–m327.
Sertçelik, M., Tercan, B., Şahin, E., Necefoğlu, H. & Hökelek, T. (2009*b*). *Acta Cryst.* E**65**, m389–m390.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Spek, A. L. (2009). *Acta Cryst.* D**65**, 148–155.
Zaman, I. G., Çaylak Delibaş, N., Necefoğlu, H. & Hökelek, T. (2012*a*). *Acta Cryst.* E**68**, m200–m201.
Zaman, İ. G., Çaylak Delibaş, N., Necefoğlu, H. & Hökelek, T. (2012*b*). *Acta Cryst.* E**68**, m249–m250.

# supplementary materials

*Acta Cryst.* (2013). E**69**, m198–m199 [doi:10.1107/S1600536813006466]

## *trans*-Tetraaquabis(isonicotinamide-$\kappa N^1$)zinc bis(3-hydroxybenzoate) tetrahydrate

**Ibrahim Göker Zaman, Nagihan Çaylak Delibaş, Hacali Necefoğlu and Tuncer Hökelek**

### Comment

As a part of our ongoing investigation on transition metal complexes of nicotinamide (NA) and/or the nicotinic acid derivative *N*,*N*-diethylnicotinamide (DENA) (Hökelek *et al.*, 2009*a*; Hökelek *et al.*, 2009*b*; Hökelek *et al.*, 2009*c*; Hökelek *et al.*, 2009*d*; Hökelek *et al.*, 2009*e*; Sertçelik *et al.*, 2009*a,b*), the title compound was synthesized and its crystal structure is reported herein.

The title compound (I) is isostructural with the related Ni (Zaman *et al.*, 2012*a*) and Co (Zaman *et al.*, 2012*b*) complexes. In (I) (Fig. 1), four O atoms (O5, O6, and the symmetry-related atoms, O5′, O6′) in the equatorial plane around the Zn atom form a slightly distorted square-planar arrangement, while the slightly distorted octahedral coordination is completed by the two pyridine N atoms (N1, N1′) of the INA ligands at 2.117 (2) Å from the Zn atom in the axial positions (Fig. 1). The average Zn—O bond length is 2.108 (2) Å. The intramolecular O—H···O hydrogen bonds (Table 1)link the uncoordinated water molecules to the HB anion. The dihedral angle between the planar carboxylate group (O1/O2/C1) and the benzene ring A (C2—C7) is 9.0 (2)°, while that between rings A and B (N1/C8—C12) is 1.26 (8)°.

In the crystal structure, intermolecular O—H···O, N—H···O and weak C—H···O hydrogen bonds (Table 1) link the molecules into a three-dimensional network, in which they may be effective in the stabilization of the structure. $\pi\cdots\pi$ Contacts between the benzene and phenyl rings, *Cg*1—*Cg*2 and *Cg*2—*Cg*1$^{i}$, [symmetry code: (i) 1 + *x*, *y*, *z*, where *Cg*1 and *Cg*2 are centroids of the rings A (C2—C7) and B (N1/C8—C12), respectively] may further stabilize the structure, with centroid-centroid distances of 3.609 (2) and 3.458 (2) Å, respectively.

### Experimental

The title compound was prepared by the reaction of $ZnSO_4.H_2O$ (0.89 g, 5 mmol) in $H_2O$ (100 ml) and INA (1.220 g, 10 mmol) in $H_2O$ (50 ml) with sodium 3-hydroxybenzoate (1.601 g, 10 mmol) in $H_2O$ (100 ml). The mixture was filtered and set aside to crystallize at ambient temperature for four weeks, giving colorless single crystals.

### Refinement

Atoms H51, H52, H61, H62, H71, H72, H81 and H82 (for $H_2O$), H21 and H22 (for $NH_2$) and H3A (for OH) were located in a difference Fourier map and refined isotropically. The C-bound H-atoms were positioned geometrically with C—H = 0.93 Å, for aromatic H-atoms, and constrained to ride on their parent atoms, with $U_{iso}$(H) = 1.2$U_{eq}$(C). During the refinement process the disordered H72A, H82A and H72B, H82B atoms were refined with occupancies ratios of 0.60:0.40. The highest residual electron density was found 1.38 Å from O6 and the deepest hole 0.83 Å from H61.

### Computing details

Data collection: *APEX2* (Bruker, 2007); cell refinement: *SAINT* (Bruker, 2007); data reduction: *SAINT* (Bruker, 2007); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 2012); software used to prepare material for publication: *WinGX* (Farrugia, 2012) and *PLATON* (Spek, 2009).

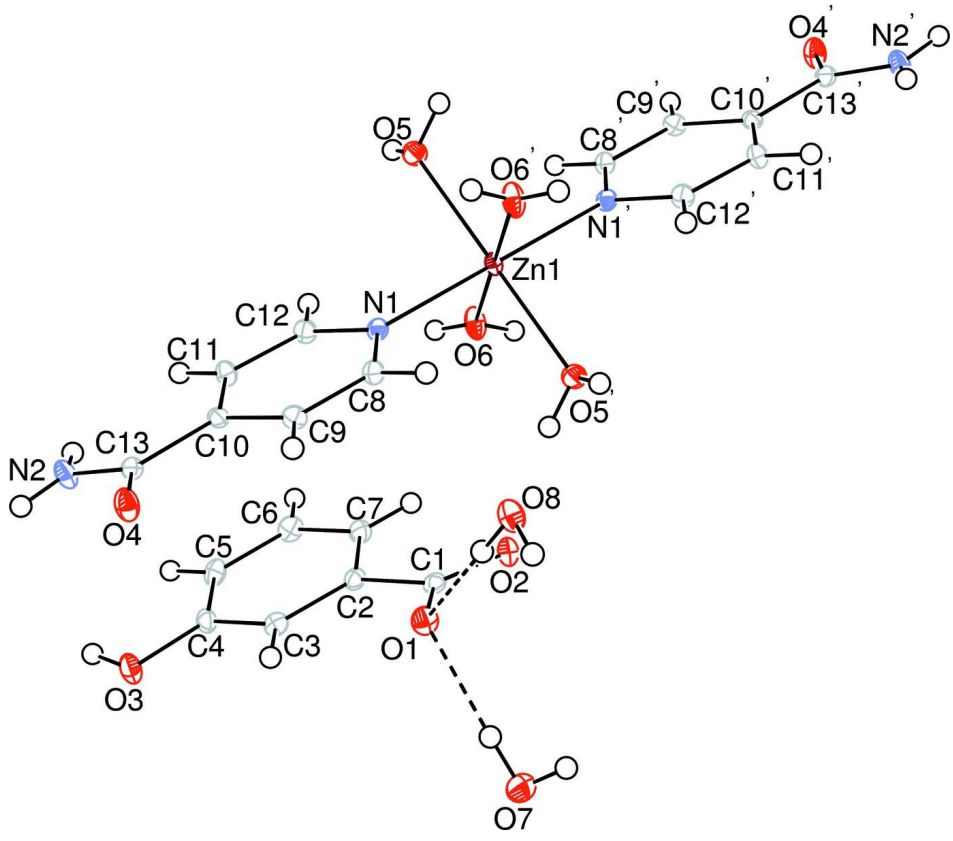



**Figure 1**

The molecular structure of the title molecule with the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level. Primed atoms are generated by the symmetry operator: (′) - *x*, - *y*, - *z*. Only one of the disordered hydrogen atoms for each of the two uncoordinated water molecules is shown for clarity. Hydrogen bonds are shown as dashed lines.

### *trans*-Tetraaquabis(isonicotinamide-$\kappa N^1$)zinc bis(3-hydroxybenzoate) tetrahydrate

*Crystal data*

$[Zn(C_6H_6N_2O)_2(H_2O)_4](C_7H_5O_3)_2\cdot 4H_2O$
$M_r = 727.99$
Monoclinic, $P2_1/n$
Hall symbol: -P 2yn
$a = 6.7002$ (2) Å
$b = 17.0005$ (4) Å
$c = 13.6000$ (3) Å
$\beta = 99.993$ (3)°
$V = 1525.63$ (7) Å$^3$
$Z = 2$
$F(000) = 760$
$D_x = 1.585$ Mg m$^{-3}$
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 7045 reflections
$\theta = 2.4$–$28.4°$
$\mu = 0.89$ mm$^{-1}$
$T = 100$ K
Block, colorless
0.38 × 0.38 × 0.32 mm

*Data collection*

Bruker Kappa APEXII CCD area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
$\varphi$ and $\omega$ scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2005)
$T_{min}$ = 0.720, $T_{max}$ = 0.752
14180 measured reflections
3808 independent reflections
3497 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.033
$\theta_{max}$ = 28.5°, $\theta_{min}$ = 2.4°
$h$ = −8→8
$k$ = −22→22
$l$ = −18→18

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.047
$wR(F^2)$ = 0.114
$S$ = 1.27
3808 reflections
264 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 atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0244P)^2 + 3.4855P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 1.41 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.47 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 (Å²)*

| | $x$ | $y$ | $z$ | $U_{iso}$*/$U_{eq}$ | Occ. (<1) |
|---|---|---|---|---|---|
| Zn1 | 0.5000 | 0.0000 | 0.5000 | 0.00963 (12) | |
| O1 | 0.1790 (3) | 0.11717 (11) | 0.83803 (14) | 0.0153 (4) | |
| O2 | 0.0781 (3) | 0.04165 (11) | 0.70518 (14) | 0.0141 (4) | |
| O3 | 0.0140 (3) | 0.39187 (11) | 0.71927 (15) | 0.0172 (4) | |
| H3A | 0.004 (7) | 0.428 (3) | 0.678 (4) | 0.051 (14)* | |
| O4 | 0.6024 (3) | 0.33081 (11) | 0.81904 (14) | 0.0168 (4) | |
| O5 | 0.6799 (3) | 0.04833 (12) | 0.40047 (15) | 0.0144 (4) | |
| H51 | 0.763 (7) | 0.017 (3) | 0.381 (3) | 0.041 (12)* | |
| H52 | 0.636 (6) | 0.070 (2) | 0.353 (3) | 0.028 (11)* | |
| O6 | 0.2262 (3) | 0.03391 (12) | 0.41227 (15) | 0.0166 (4) | |
| H61 | 0.140 (8) | 0.002 (3) | 0.385 (4) | 0.052 (14)* | |
| H62 | 0.192 (6) | 0.073 (3) | 0.386 (3) | 0.037 (12)* | |
| O7 | −0.0930 (4) | 0.06755 (13) | 0.95598 (17) | 0.0201 (4) | |
| H71 | −0.009 (7) | 0.084 (3) | 0.920 (3) | 0.040 (12)* | |
| H72A | −0.201 (14) | 0.059 (5) | 0.930 (6) | 0.05 (2)* | 0.60 |
| H72B | −0.05 (2) | 0.026 (8) | 0.967 (9) | 0.059* | 0.40 |

| | | | | | |
|---|---|---|---|---|---|
| O8 | 0.5012 (4) | 0.01950 (12) | 0.90057 (16) | 0.0171 (4) | |
| H81 | 0.398 (7) | 0.052 (3) | 0.882 (3) | 0.045 (13)* | |
| H82A | 0.596 (12) | 0.037 (4) | 0.910 (5) | 0.03 (2)* | 0.60 |
| H82B | 0.497 (16) | 0.008 (6) | 0.959 (7) | 0.030* | 0.40 |
| N1 | 0.5135 (3) | 0.10959 (12) | 0.57473 (16) | 0.0104 (4) | |
| N2 | 0.4845 (4) | 0.39638 (14) | 0.67657 (18) | 0.0148 (5) | |
| H21 | 0.440 (6) | 0.395 (2) | 0.616 (3) | 0.037 (11)* | |
| H22 | 0.474 (5) | 0.438 (2) | 0.705 (3) | 0.019 (9)* | |
| C1 | 0.1097 (4) | 0.10822 (15) | 0.74642 (19) | 0.0111 (5) | |
| C2 | 0.0586 (4) | 0.18077 (15) | 0.68340 (19) | 0.0109 (5) | |
| C3 | 0.0647 (4) | 0.25421 (15) | 0.72947 (19) | 0.0117 (5) | |
| H3 | 0.1021 | 0.2584 | 0.7984 | 0.014* | |
| C4 | 0.0147 (4) | 0.32091 (15) | 0.67183 (19) | 0.0124 (5) | |
| C5 | −0.0367 (4) | 0.31508 (15) | 0.5682 (2) | 0.0144 (5) | |
| H5 | −0.0688 | 0.3600 | 0.5298 | 0.017* | |
| C6 | −0.0395 (4) | 0.24190 (16) | 0.5227 (2) | 0.0149 (5) | |
| H6 | −0.0723 | 0.2380 | 0.4536 | 0.018* | |
| C7 | 0.0063 (4) | 0.17457 (15) | 0.57989 (19) | 0.0132 (5) | |
| H7 | 0.0022 | 0.1255 | 0.5493 | 0.016* | |
| C8 | 0.5621 (4) | 0.11492 (15) | 0.67488 (19) | 0.0120 (5) | |
| H8 | 0.5891 | 0.0689 | 0.7118 | 0.014* | |
| C9 | 0.5736 (4) | 0.18572 (15) | 0.72495 (19) | 0.0120 (5) | |
| H9 | 0.6091 | 0.1871 | 0.7941 | 0.014* | |
| C10 | 0.5317 (4) | 0.25512 (14) | 0.67106 (19) | 0.0101 (5) | |
| C11 | 0.4793 (4) | 0.24979 (15) | 0.56786 (19) | 0.0122 (5) | |
| H11 | 0.4491 | 0.2948 | 0.5293 | 0.015* | |
| C12 | 0.4728 (4) | 0.17650 (15) | 0.52330 (19) | 0.0127 (5) | |
| H12 | 0.4385 | 0.1736 | 0.4541 | 0.015* | |
| C13 | 0.5426 (4) | 0.33143 (15) | 0.72775 (19) | 0.0118 (5) | |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| Zn1 | 0.0128 (2) | 0.00708 (19) | 0.00863 (19) | 0.00011 (15) | 0.00070 (14) | −0.00048 (15) |
| O1 | 0.0184 (10) | 0.0151 (9) | 0.0118 (9) | 0.0005 (7) | 0.0012 (7) | 0.0016 (7) |
| O2 | 0.0166 (9) | 0.0098 (8) | 0.0150 (9) | −0.0010 (7) | 0.0003 (7) | −0.0002 (7) |
| O3 | 0.0250 (11) | 0.0099 (9) | 0.0162 (9) | 0.0003 (8) | 0.0025 (8) | −0.0012 (7) |
| O4 | 0.0261 (11) | 0.0121 (9) | 0.0112 (9) | −0.0017 (8) | 0.0006 (8) | −0.0013 (7) |
| O5 | 0.0175 (10) | 0.0138 (9) | 0.0125 (9) | 0.0015 (8) | 0.0048 (8) | 0.0016 (7) |
| O6 | 0.0172 (10) | 0.0104 (9) | 0.0187 (10) | 0.0010 (8) | −0.0061 (8) | 0.0019 (8) |
| O7 | 0.0234 (12) | 0.0196 (10) | 0.0189 (10) | −0.0029 (9) | 0.0078 (9) | −0.0003 (8) |
| O8 | 0.0185 (11) | 0.0133 (9) | 0.0183 (10) | 0.0019 (8) | 0.0000 (8) | −0.0001 (8) |
| N1 | 0.0094 (10) | 0.0106 (10) | 0.0110 (10) | −0.0018 (8) | 0.0010 (8) | −0.0011 (8) |
| N2 | 0.0215 (12) | 0.0100 (10) | 0.0124 (11) | 0.0012 (9) | 0.0015 (9) | −0.0027 (8) |
| C1 | 0.0079 (11) | 0.0122 (11) | 0.0135 (12) | −0.0005 (9) | 0.0028 (9) | 0.0007 (9) |
| C2 | 0.0106 (11) | 0.0105 (11) | 0.0117 (11) | 0.0000 (9) | 0.0023 (9) | 0.0023 (9) |
| C3 | 0.0095 (11) | 0.0142 (12) | 0.0107 (11) | −0.0013 (9) | 0.0001 (9) | −0.0001 (9) |
| C4 | 0.0114 (11) | 0.0098 (11) | 0.0158 (12) | −0.0014 (9) | 0.0016 (9) | −0.0018 (9) |
| C5 | 0.0150 (12) | 0.0123 (12) | 0.0152 (12) | −0.0013 (10) | 0.0008 (10) | 0.0038 (9) |
| C6 | 0.0162 (13) | 0.0169 (13) | 0.0110 (12) | −0.0017 (10) | 0.0008 (9) | 0.0004 (9) |

| | | | | | | |
|---|---|---|---|---|---|---|
| C7 | 0.0144 (12) | 0.0124 (11) | 0.0130 (12) | −0.0016 (9) | 0.0030 (9) | −0.0012 (9) |
| C8 | 0.0131 (12) | 0.0101 (11) | 0.0129 (12) | 0.0004 (9) | 0.0025 (9) | 0.0014 (9) |
| C9 | 0.0127 (12) | 0.0130 (12) | 0.0102 (11) | 0.0005 (9) | 0.0015 (9) | −0.0002 (9) |
| C10 | 0.0081 (11) | 0.0103 (11) | 0.0122 (11) | −0.0006 (9) | 0.0021 (9) | −0.0021 (9) |
| C11 | 0.0141 (12) | 0.0095 (11) | 0.0126 (12) | 0.0014 (9) | 0.0010 (9) | 0.0009 (9) |
| C13 | 0.0117 (12) | 0.0110 (11) | 0.0131 (12) | −0.0018 (9) | 0.0034 (9) | −0.0020 (9) |
| C12 | 0.0149 (12) | 0.0125 (12) | 0.0105 (11) | −0.0008 (9) | 0.0020 (9) | −0.0009 (9) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| Zn1—O5 | 2.1276 (19) | N2—C13 | 1.327 (3) |
| Zn1—O5$^{i}$ | 2.1276 (19) | N2—H21 | 0.83 (4) |
| Zn1—O6 | 2.089 (2) | N2—H22 | 0.81 (4) |
| Zn1—O6$^{i}$ | 2.089 (2) | C2—C1 | 1.507 (3) |
| Zn1—N1 | 2.117 (2) | C2—C3 | 1.394 (3) |
| Zn1—N1$^{i}$ | 2.117 (2) | C2—C7 | 1.394 (3) |
| O1—C1 | 1.261 (3) | C3—H3 | 0.9300 |
| O2—C1 | 1.264 (3) | C4—C3 | 1.386 (4) |
| O3—C4 | 1.369 (3) | C5—C4 | 1.395 (4) |
| O3—H3A | 0.83 (5) | C5—C6 | 1.388 (4) |
| O4—C13 | 1.237 (3) | C5—H5 | 0.9300 |
| O5—H51 | 0.85 (5) | C6—H6 | 0.9300 |
| O5—H52 | 0.76 (4) | C7—C6 | 1.388 (4) |
| O6—H61 | 0.83 (5) | C7—H7 | 0.9300 |
| O6—H62 | 0.77 (5) | C8—C9 | 1.379 (3) |
| O7—H71 | 0.85 (5) | C8—H8 | 0.9300 |
| O7—H72A | 0.76 (9) | C9—C10 | 1.392 (3) |
| O7—H72B | 0.78 (13) | C9—H9 | 0.9300 |
| O8—H81 | 0.88 (5) | C10—C11 | 1.389 (3) |
| O8—H82A | 0.69 (8) | C10—C13 | 1.504 (3) |
| O8—H82B | 0.82 (9) | C11—C12 | 1.383 (3) |
| N1—C8 | 1.347 (3) | C11—H11 | 0.9300 |
| N1—C12 | 1.338 (3) | C12—H12 | 0.9300 |
| | | | |
| O5—Zn1—O5$^{i}$ | 180.0 | C3—C2—C1 | 119.4 (2) |
| O6—Zn1—O5 | 93.93 (8) | C3—C2—C7 | 120.3 (2) |
| O6$^{i}$—Zn1—O5 | 86.07 (8) | C7—C2—C1 | 120.3 (2) |
| O6—Zn1—O5$^{i}$ | 86.07 (8) | C2—C3—H3 | 120.2 |
| O6$^{i}$—Zn1—O5$^{i}$ | 93.93 (8) | C4—C3—C2 | 119.5 (2) |
| O6—Zn1—O6$^{i}$ | 180.00 (10) | C4—C3—H3 | 120.2 |
| O6—Zn1—N1 | 89.51 (8) | O3—C4—C3 | 118.4 (2) |
| O6$^{i}$—Zn1—N1 | 90.49 (8) | O3—C4—C5 | 121.2 (2) |
| O6—Zn1—N1$^{i}$ | 90.49 (8) | C3—C4—C5 | 120.4 (2) |
| O6$^{i}$—Zn1—N1$^{i}$ | 89.51 (8) | C4—C5—H5 | 120.1 |
| N1—Zn1—O5 | 89.07 (8) | C6—C5—C4 | 119.7 (2) |
| N1$^{i}$—Zn1—O5 | 90.93 (8) | C6—C5—H5 | 120.1 |
| N1—Zn1—O5$^{i}$ | 90.93 (8) | C5—C6—H6 | 119.8 |
| N1$^{i}$—Zn1—O5$^{i}$ | 89.07 (8) | C7—C6—C5 | 120.3 (2) |
| N1—Zn1—N1$^{i}$ | 180.00 (5) | C7—C6—H6 | 119.8 |
| C4—O3—H3A | 110 (3) | C2—C7—H7 | 120.1 |

| | | | |
|---|---|---|---|
| Zn1—O5—H51 | 116 (3) | C6—C7—C2 | 119.7 (2) |
| Zn1—O5—H52 | 123 (3) | C6—C7—H7 | 120.1 |
| H52—O5—H51 | 102 (4) | N1—C8—C9 | 122.7 (2) |
| Zn1—O6—H61 | 123 (3) | N1—C8—H8 | 118.6 |
| Zn1—O6—H62 | 131 (3) | C9—C8—H8 | 118.6 |
| H61—O6—H62 | 103 (4) | C8—C9—C10 | 119.4 (2) |
| H71—O7—H72B | 96 (10) | C8—C9—H9 | 120.3 |
| H72A—O7—H71 | 118 (7) | C10—C9—H9 | 120.3 |
| H72A—O7—H72B | 104 (10) | C9—C10—C13 | 118.3 (2) |
| H81—O8—H82A | 116 (6) | C11—C10—C9 | 118.0 (2) |
| H81—O8—H82B | 106 (8) | C11—C10—C13 | 123.7 (2) |
| H82A—O8—H82B | 96 (9) | C10—C11—H11 | 120.5 |
| C8—N1—Zn1 | 121.81 (17) | C12—C11—C10 | 119.0 (2) |
| C12—N1—Zn1 | 120.62 (17) | C12—C11—H11 | 120.5 |
| C12—N1—C8 | 117.6 (2) | N1—C12—C11 | 123.3 (2) |
| C13—N2—H21 | 122 (3) | N1—C12—H12 | 118.4 |
| C13—N2—H22 | 121 (2) | C11—C12—H12 | 118.4 |
| H22—N2—H21 | 117 (4) | O4—C13—N2 | 123.2 (2) |
| O1—C1—O2 | 123.4 (2) | O4—C13—C10 | 119.1 (2) |
| O1—C1—C2 | 118.1 (2) | N2—C13—C10 | 117.7 (2) |
| O2—C1—C2 | 118.4 (2) | | |

| | | | |
|---|---|---|---|
| O5—Zn1—N1—C8 | −131.4 (2) | C1—C2—C7—C6 | −179.7 (2) |
| O5$^{i}$—Zn1—N1—C8 | 48.6 (2) | C3—C2—C7—C6 | 0.1 (4) |
| O5—Zn1—N1—C12 | 48.9 (2) | O3—C4—C3—C2 | 177.4 (2) |
| O5$^{i}$—Zn1—N1—C12 | −131.1 (2) | C5—C4—C3—C2 | −1.6 (4) |
| O6—Zn1—N1—C8 | 134.7 (2) | C6—C5—C4—O3 | −178.3 (2) |
| O6$^{i}$—Zn1—N1—C8 | −45.3 (2) | C6—C5—C4—C3 | 0.6 (4) |
| O6—Zn1—N1—C12 | −45.1 (2) | C4—C5—C6—C7 | 0.7 (4) |
| O6$^{i}$—Zn1—N1—C12 | 134.9 (2) | C2—C7—C6—C5 | −1.0 (4) |
| Zn1—N1—C8—C9 | 179.39 (19) | N1—C8—C9—C10 | 0.7 (4) |
| C12—N1—C8—C9 | −0.8 (4) | C8—C9—C10—C11 | 0.1 (4) |
| Zn1—N1—C12—C11 | −179.9 (2) | C8—C9—C10—C13 | 179.2 (2) |
| C8—N1—C12—C11 | 0.3 (4) | C9—C10—C11—C12 | −0.6 (4) |
| C3—C2—C1—O1 | −8.0 (4) | C13—C10—C11—C12 | −179.6 (2) |
| C3—C2—C1—O2 | 170.9 (2) | C9—C10—C13—O4 | 5.2 (4) |
| C7—C2—C1—O1 | 171.8 (2) | C9—C10—C13—N2 | −174.0 (2) |
| C7—C2—C1—O2 | −9.3 (4) | C11—C10—C13—O4 | −175.7 (2) |
| C1—C2—C3—C4 | −179.0 (2) | C11—C10—C13—N2 | 5.1 (4) |
| C7—C2—C3—C4 | 1.2 (4) | C10—C11—C12—N1 | 0.4 (4) |

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* |
|---|---|---|---|---|
| O3—H3*A*···O8$^{ii}$ | 0.83 (5) | 1.88 (5) | 2.705 (3) | 172 (5) |
| N2—H21···O7$^{iii}$ | 0.83 (4) | 2.24 (4) | 3.017 (3) | 157 (3) |
| N2—H22···O2$^{ii}$ | 0.82 (4) | 2.21 (4) | 3.016 (3) | 172 (3) |
| O5—H51···O2$^{i}$ | 0.85 (5) | 1.98 (5) | 2.800 (3) | 162 (4) |

| | | | | |
|---|---|---|---|---|
| O5—H52···O3$^{iii}$ | 0.76 (4) | 1.97 (4) | 2.719 (3) | 170 (4) |
| O6—H61···O2$^{iv}$ | 0.83 (5) | 1.89 (5) | 2.689 (3) | 161 (5) |
| O6—H62···O4$^{v}$ | 0.77 (5) | 1.92 (5) | 2.687 (3) | 179 (5) |
| O7—H71···O1 | 0.85 (5) | 1.91 (5) | 2.761 (3) | 178 (3) |
| O7—H72*A*···O8$^{vi}$ | 0.76 (9) | 2.08 (9) | 2.814 (4) | 163 (8) |
| O7—H72*B*···O7$^{vii}$ | 0.78 (9) | 2.03 (9) | 2.783 (3) | 160 (8) |
| O8—H81···O1 | 0.89 (5) | 1.85 (5) | 2.739 (3) | 177 (4) |
| O8—H82*A*···O7$^{viii}$ | 0.69 (8) | 2.13 (8) | 2.814 (4) | 167 (6) |
| O8—H82*B*···O8$^{ix}$ | 0.82 (9) | 1.96 (9) | 2.787 (3) | 178 (6) |
| C11—H11···O7$^{iii}$ | 0.93 | 2.54 | 3.455 (3) | 168 |

Symmetry codes: (i) −*x*+1, −*y*, −*z*+1; (ii) −*x*+1/2, *y*+1/2, −*z*+3/2; (iii) *x*+1/2, −*y*+1/2, *z*−1/2; (iv) −*x*, −*y*, −*z*+1; (v) *x*−1/2, −*y*+1/2, *z*−1/2; (vi) *x*−1, *y*, *z*; (vii) −*x*, −*y*, −*z*+2; (viii) *x*+1, *y*, *z*; (ix) −*x*+1, −*y*, −*z*+2.