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trans-Bis(2-acetamido-5-methylbenzoato-κO^1)tetraaquazinc

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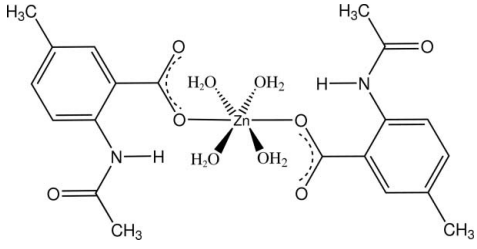
Key indicators: single-crystal X-ray study; T = 296 K; mean σ(C–C) = 0.006 Å; R factor = 0.055; wR factor = 0.179; data-to-parameter ratio = 13.0.

In the title compound, $[Zn(C_{10}H_{10}NO_3)_2(H_2O)_4]$, the Zn^{II} atom lies on a crystallographic inversion center and is six-coordinated by two monodentate *trans*-related 2-(*N*-acetylamino)-5-methylbenzoato ligands and four water molecules, giving a slightly distorted octahedral geometry. There are two intramolecular hydrogen bonds [amine N—H···$O_{carboxyl}$ and water O—H···$O_{carboxyl}$], while extensive intermolecular water O—H···O hydrogen-bonding interactions extend the complex units into a two-dimensional network structure along (100).

Related literature

The study of metal coordination polymers has enhanced our understanding of the relationship between molecular structure and material function, see: Dai *et al.* (2005); Moulton & Zaworotko (2001).



Experimental

Crystal data

$[Zn(C_{10}H_{10}NO_3)_2(H_2O)_4]$
M_r = 521.83
Monoclinic, $C2/c$
a = 19.300 (4) Å
b = 9.3000 (19) Å
c = 13.300 (3) Å
β = 107.60 (3)°
V = 2275.5 (9) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 1.14 mm^{-1}
T = 296 K
0.42 × 0.40 × 0.25 mm

Data collection

Bruker SMART CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.626, T_{max} = 0.752
10817 measured reflections
2130 independent reflections
1941 reflections with $I > 2\sigma(I)$
R_{int} = 0.029

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.055
$wR(F^2)$ = 0.179
S = 1.11
2130 reflections
164 parameters
2 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.49 e Å^{-3}
$\Delta\rho_{min}$ = −0.77 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1A···O1	0.86	1.95	2.616 (4)	133
O1W—H1WA···O2^{i}	0.81 (2)	1.90 (2)	2.704 (4)	170 (5)
O1W—H1WB···O3^{ii}	0.83 (2)	1.88 (2)	2.707 (4)	177 (4)
O2W—H2WA···O2^{i}	0.80 (4)	2.12 (4)	2.916 (5)	172 (5)
O2W—H2WB···O3^{iii}	0.83 (4)	1.84 (3)	2.627 (4)	159 (5)

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

Data collection: *SMART* (Bruker, 2004); cell refinement: *SAINT* (Bruker, 2004); 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*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: ZS2102).

References

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Dai, Y. M., Ma, E., Tang, E., Zhang, J., Li, Z. J., Huang, X. & Yao, Y. G. (2005). *Cryst. Growth Des.* **5**, 1313–1315.
Moulton, B. & Zaworotko, M. J. (2001). *Chem. Rev.* **101**, 1629–1639.
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supporting information

Acta Cryst. (2011). E**67**, m542 [doi:10.1107/S1600536811012013]

trans-Bis(2-acetamido-5-methylbenzoato-κO^1)tetraaquazinc

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S1. Comment

In recent decades, the study of metal coordination polymers has witnessed tremendous growth as an attractive interface between synthetic chemistry and materials science, which significantly boosts the understanding of the relationship between molecular structure and material function (Moulton & Zaworotko, 2001; Dai *et al.*, 2005). The crystal engineering of coordination polymers is highly influenced by the judicious choice of ligands, metal coordination geometry, template design and other subtle factors, such as counterions, solvent choice and reaction temperature. The deprotonated 2-(*N*-acetylamino)-5-methylbenzoic acid (HNB) ligands are good candidates in this respect for the construction of supramolecular architectures because in such bitopical ligands the *N*-acetyl group can act as a hydrogen-bond donor and/or acceptor, while the carboxyl function has strong coordination abilities with many metal ions. Taking these advantages into account, recently we have begun to assemble HNB and zinc ions into polymeric complexes under hydrothermal conditions. Herein, we report the synthesis and crystal structure of the title compound, $[Zn(C_{10}H_{10}NO_3)_2(H_2O)_4]$ (I).

In the structure of (I) the Zn^{II} metal center lies on a crystallographic inversion center. The local coordination environment around Zn^{II} atom is slightly distorted octahedral, comprising two monodentate *trans*-related 2-(*N*-acetyl-amino)-5-methylbenzoato ligands and four water molecules (Fig. 1). Two intramolecular hydrogen bonds [amine N—H···$O_{carboxyl}$ and water O—H···$O_{carboxyl}$] stabilize the complex units while extensive intermolecular water O—H···O_{acetyl} hydrogen-bonding interactions are observed in the structure (Table 1), giving rise to double-stranded chains. Further interactions involving the coordinated water ligands and the uncoordinated O atoms of the carboxyl group are gives a two-dimensional network structure (Fig. 2).

S2. Experimental

$ZnSO_4{\cdot}7H_2O$ (1.00 mmol, 0.28 g), 2-(*N*-acetylamino)-5-methylbenzoic acid (HNB) (1.00 mmol, 0.19 g) and NaOH (1.00 mmol, 0.04 g) were mixed in water (15 ml) and heated at 403 K for 3 days in a sealed 25 ml Teflon-lined stainless steel vessel under autogenous pressure. After cooling to room temperature at a rate of 5° C h^{-1}, yellow block crystals were isolated, washed with ethanol and then dried in air (33% yield).

S3. Refinement

H atoms attached to carbon and nitrogen were positioned geometrically and treated using a riding model, fixing the bond lengths at 0.86, 0.96 and 0.93 Å for NH, CH_2 and aromatic CH groups, respectively and $U_{iso}(H) = 1.2U_{eq}(N, C)$]. The aqua H atoms were located from difference maps and their coordinates refined but with $U_{iso}(H) = 1.5U_{eq}(O)$.

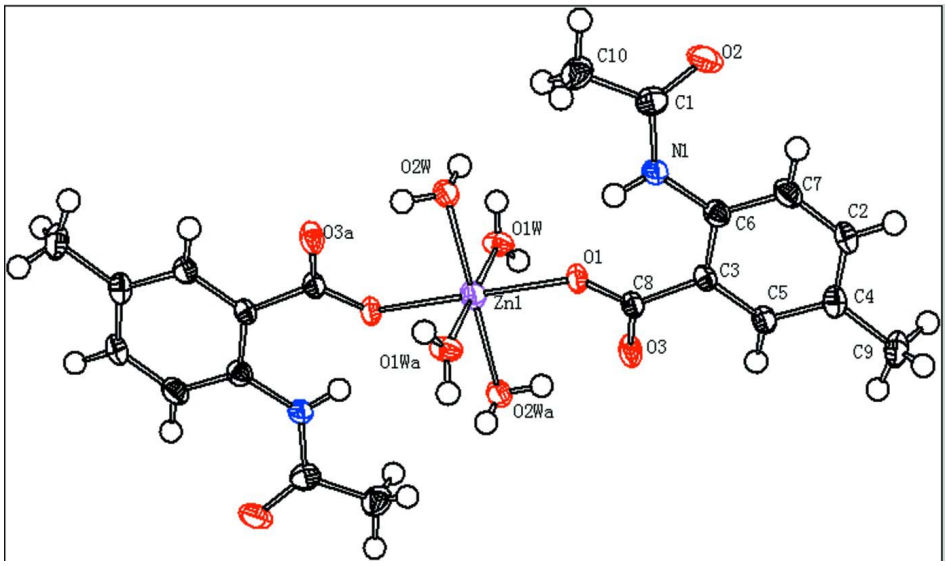

Figure 1

Local coordination around Zn ion in (I). Displacement ellipsoids are drawn at the 50% probability level. Symmetry code: (a): -*x* + 1, -*y*, -*z* + 2

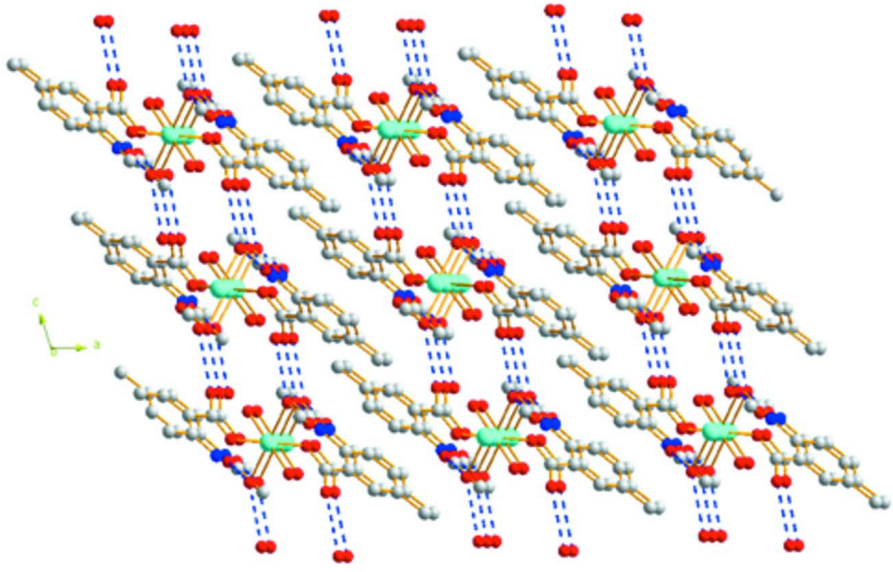

Figure 2

The crystal packing of (I) with hydrogen bonds shown as dashed lines.

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Crystal data

$[Zn(C_{10}H_{10}NO_3)_2(H_2O)_4]$
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Hall symbol: -C 2yc
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b = 9.3000 (19) Å
c = 13.300 (3) Å
β = 107.60 (3)°
V = 2275.5 (9) Å^3
Z = 4
F(000) = 1088
D_x = 1.523 Mg m^{-3}

Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 4107 reflections
θ = 3.1–25.6°
μ = 1.14 mm^{-1}
T = 296 K
Block, yellow
0.42 × 0.40 × 0.25 mm

Data collection

Bruker SMART CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.626, T_{max} = 0.752
10817 measured reflections
2130 independent reflections
1941 reflections with $I > 2\sigma(I)$
R_{int} = 0.029
θ_{max} = 25.6°, θ_{min} = 3.1°
h = −23→21
k = −10→11
l = −16→16

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.055
$wR(F^2)$ = 0.179
S = 1.11
2130 reflections
164 parameters
2 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
$w = 1/[\sigma^2(F_o^2) + (0.1075P)^2 + 9.1602P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.49 e Å^{-3}
$\Delta\rho_{min}$ = −0.77 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}
Zn1	0.5000	0.0000	1.0000	0.0283 (3)
N1	0.61735 (18)	0.4086 (4)	1.0380 (3)	0.0268 (7)
H1A	0.6034	0.3279	1.0569	0.032*
O1W	0.42661 (16)	0.0994 (3)	0.8725 (2)	0.0280 (6)
H1WA	0.410 (2)	0.173 (3)	0.890 (3)	0.034*
H1WB	0.422 (3)	0.085 (4)	0.8095 (18)	0.034*
O1	0.57941 (14)	0.1422 (3)	0.9864 (2)	0.0247 (6)
O2W	0.47378 (16)	0.1539 (3)	1.1025 (2)	0.0299 (7)
H2WA	0.449 (2)	0.211 (4)	1.063 (3)	0.036*
H2WB	0.456 (2)	0.097 (4)	1.136 (3)	0.036*
O2	0.6109 (2)	0.6481 (3)	1.0578 (3)	0.0428 (8)
O3	0.59193 (17)	0.0609 (3)	0.8358 (2)	0.0328 (7)

C1	0.5978 (2)	0.5248 (5)	1.0801 (3)	0.0296 (9)
C2	0.7488 (2)	0.4914 (4)	0.8911 (4)	0.0296 (10)
H2A	0.7806	0.5647	0.8871	0.035*
C3	0.65394 (19)	0.2748 (4)	0.9062 (3)	0.0208 (7)
C4	0.7454 (2)	0.3684 (4)	0.8315 (3)	0.0269 (8)
C5	0.6976 (2)	0.2625 (4)	0.8402 (3)	0.0244 (8)
H5A	0.6943	0.1792	0.8003	0.029*
C6	0.6584 (2)	0.3999 (4)	0.9657 (3)	0.0245 (8)
C7	0.7056 (3)	0.5073 (4)	0.9566 (4)	0.0298 (10)
H7A	0.7085	0.5916	0.9952	0.036*
C8	0.60481 (19)	0.1505 (4)	0.9089 (3)	0.0218 (8)
C9	0.7926 (2)	0.3494 (5)	0.7609 (4)	0.0374 (10)
H9A	0.7825	0.2581	0.7259	0.056*
H9B	0.8428	0.3532	0.8024	0.056*
H9C	0.7827	0.4249	0.7093	0.056*
C10	0.5580 (3)	0.4978 (5)	1.1599 (4)	0.0379 (11)
H10A	0.5459	0.5881	1.1854	0.057*
H10B	0.5884	0.4433	1.2178	0.057*
H10C	0.5142	0.4449	1.1273	0.057*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Zn1	0.0351 (5)	0.0249 (4)	0.0276 (4)	−0.0034 (2)	0.0136 (3)	−0.0013 (2)
N1	0.0366 (18)	0.0198 (16)	0.0284 (17)	−0.0013 (14)	0.0166 (14)	−0.0030 (13)
O1W	0.0396 (16)	0.0238 (14)	0.0197 (13)	0.0070 (12)	0.0077 (12)	−0.0002 (11)
O1	0.0332 (14)	0.0225 (13)	0.0230 (13)	−0.0110 (11)	0.0154 (11)	−0.0047 (10)
O2W	0.0450 (17)	0.0217 (14)	0.0295 (15)	−0.0044 (12)	0.0210 (13)	−0.0029 (11)
O2	0.066 (2)	0.0219 (16)	0.0467 (19)	0.0081 (14)	0.0265 (17)	0.0020 (13)
O3	0.0511 (18)	0.0284 (15)	0.0262 (15)	−0.0162 (14)	0.0224 (13)	−0.0083 (12)
C1	0.030 (2)	0.031 (2)	0.026 (2)	0.0014 (17)	0.0064 (17)	−0.0044 (17)
C2	0.031 (2)	0.024 (2)	0.035 (2)	−0.0084 (15)	0.0119 (18)	0.0032 (15)
C3	0.0233 (17)	0.0192 (17)	0.0194 (17)	−0.0015 (14)	0.0058 (14)	0.0028 (14)
C4	0.0258 (19)	0.032 (2)	0.0234 (19)	−0.0044 (16)	0.0076 (15)	0.0052 (16)
C5	0.0286 (19)	0.0226 (18)	0.0230 (18)	−0.0039 (15)	0.0093 (15)	−0.0005 (14)
C6	0.0295 (19)	0.0223 (19)	0.0215 (18)	−0.0003 (16)	0.0076 (15)	0.0006 (15)
C7	0.038 (2)	0.020 (2)	0.033 (2)	−0.0051 (15)	0.012 (2)	−0.0021 (15)
C8	0.0249 (18)	0.0198 (17)	0.0215 (18)	−0.0028 (14)	0.0084 (15)	0.0023 (14)
C9	0.038 (2)	0.043 (3)	0.038 (2)	−0.0123 (19)	0.021 (2)	−0.0001 (19)
C10	0.043 (3)	0.039 (3)	0.037 (3)	−0.0061 (18)	0.021 (2)	−0.0118 (18)

Geometric parameters (Å, º)

Zn1—O1W^{i}	2.070 (3)	C2—C7	1.384 (7)
Zn1—O1W	2.070 (3)	C2—C4	1.382 (6)
Zn1—O1	2.073 (2)	C2—H2A	0.9300
Zn1—O1^{i}	2.073 (2)	C3—C5	1.393 (5)
Zn1—O2W	2.140 (3)	C3—C6	1.395 (5)

Zn1—O2W^{i}	2.140 (3)	C3—C8	1.503 (5)
N1—C1	1.324 (5)	C4—C5	1.379 (5)
N1—C6	1.421 (5)	C4—C9	1.503 (6)
N1—H1A	0.8600	C5—H5A	0.9300
O1W—H1WA	0.811 (18)	C6—C7	1.382 (6)
O1W—H1WB	0.826 (18)	C7—H7A	0.9300
O1—C8	1.270 (4)	C9—H9A	0.9600
O2W—H2WA	0.80 (4)	C9—H9B	0.9600
O2W—H2WB	0.83 (4)	C9—H9C	0.9600
O2—C1	1.230 (5)	C10—H10A	0.9600
O3—C8	1.247 (5)	C10—H10B	0.9600
C1—C10	1.508 (6)	C10—H10C	0.9600

O1W^{i}—Zn1—O1W	180.00 (12)	C5—C3—C6	118.7 (3)
O1W^{i}—Zn1—O1	90.89 (11)	C5—C3—C8	117.2 (3)
O1W—Zn1—O1	89.11 (11)	C6—C3—C8	124.0 (3)
O1W^{i}—Zn1—O1^{i}	89.11 (11)	C5—C4—C2	117.4 (4)
O1W—Zn1—O1^{i}	90.89 (11)	C5—C4—C9	121.1 (4)
O1—Zn1—O1^{i}	180.0	C2—C4—C9	121.5 (4)
O1W^{i}—Zn1—O2W	90.67 (11)	C4—C5—C3	122.8 (4)
O1W—Zn1—O2W	89.33 (11)	C4—C5—H5A	118.6
O1—Zn1—O2W	87.28 (10)	C3—C5—H5A	118.6
O1^{i}—Zn1—O2W	92.72 (10)	C7—C6—C3	118.8 (4)
O1W^{i}—Zn1—O2W^{i}	89.33 (11)	C7—C6—N1	122.4 (3)
O1W—Zn1—O2W^{i}	90.67 (11)	C3—C6—N1	118.7 (3)
O1—Zn1—O2W^{i}	92.72 (10)	C6—C7—C2	121.1 (4)
O1^{i}—Zn1—O2W^{i}	87.28 (10)	C6—C7—H7A	119.4
O2W—Zn1—O2W^{i}	180.000 (1)	C2—C7—H7A	119.4
C1—N1—C6	128.4 (3)	O3—C8—O1	123.9 (3)
C1—N1—H1A	115.8	O3—C8—C3	118.3 (3)
C6—N1—H1A	115.8	O1—C8—C3	117.8 (3)
Zn1—O1W—H1WA	112 (3)	C4—C9—H9A	109.5
Zn1—O1W—H1WB	126 (3)	C4—C9—H9B	109.5
H1WA—O1W—H1WB	119 (3)	H9A—C9—H9B	109.5
C8—O1—Zn1	125.9 (2)	C4—C9—H9C	109.5
Zn1—O2W—H2WA	104 (4)	H9A—C9—H9C	109.5
Zn1—O2W—H2WB	98 (3)	H9B—C9—H9C	109.5
H2WA—O2W—H2WB	122 (3)	C1—C10—H10A	109.5
O2—C1—N1	123.5 (4)	C1—C10—H10B	109.5
O2—C1—C10	120.8 (4)	H10A—C10—H10B	109.5
N1—C1—C10	115.7 (4)	C1—C10—H10C	109.5
C7—C2—C4	121.1 (4)	H10A—C10—H10C	109.5
C7—C2—H2A	119.4	H10B—C10—H10C	109.5
C4—C2—H2A	119.4		

Symmetry code: (i) $-x+1, -y, -z+2$.

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
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