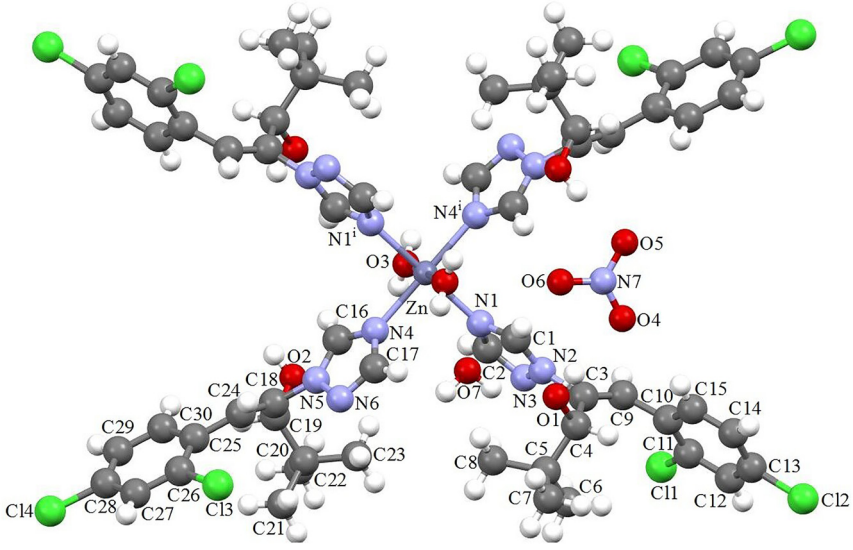
Jie Li*, Mengying Qiu, Haiwei Han, Biao Yan and Hongya Li

Crystal structure of diaqua-tetra((*E*)-(*RS*)-1-(2,4-dichlorophenyl)-4,4-dimethyl-2-(1,2,4-triazol-1-yl) pent-1-en-3-ol-κ^1N)zinc(II) dinitrate dihydrate, $C_{60}H_{76}Cl_8N_{14}O_{14}Zn$



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Abstract

$C_{60}H_{76}Cl_8N_{14}O_{14}Zn$, triclinic, $P\bar{1}$ (no. 2), a = 7.758(1) Å, b = 15.376(2) Å, c = 16.518(2) Å, α = 100.946(2)°, β = 97.977(2)°, γ = 99.581(2)°, V = 1877.8(4) Å^3, Z = 1, $R_{gt}(F)$ = 0.0456, w$R_{ref}(F^2)$ = 0.1368, T = 296(2) K.

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The title crystal structure is shown in the figure. Table 1 contains crystallographic data and Table 2 contains the list of the atoms including atomic coordinates and displacement parameters.

Table 1: Data collection and handling.

Crystal:	Colourless rodlike
Size:	0.37 × 0.35 × 0.25 mm
Wavelength:	Mo *K*α radiation (0.71073 Å)
μ:	0.68 mm^{-1}
Diffractometer, scan mode:	Bruker Apex-II, φ and ω
θ_{max}, completeness:	27.9°, 99 %
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	11,062, 8,117, 0.017
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\sigma(I_{obs})$, 6,018
N(param)$_{refined}$:	463
Programs:	Bruker,[1] Shelx,[2–4] Diamond[5]

1 Source of material

The (*E*)-(*RS*)-1-(2,4-dichlorophenyl)-4,4-dimethyl-2-(1,2,4-triazol-1-yl)pent-1-en-3-ol (0.653 g, 0.200 mmol) was dissolved in methanol (10 mL). Then, zinc nitrate hexahydrate (0.298 g, 0.100 mmol) was added. The reaction mixture was allowed to stir at room temperature for 2 h. Colorless rodlike crystal of the title compound was obtained by slow evaporation from methanol.

***Corresponding author: Jie Li**, College of Chemistry and Chemical Engineering, Xinyang Normal University, Xinyang, Henan 464000, P.R. China, E-mail: lijie@xynu.edu.cn. https://orcid.org/0000-0002-3104-1222
Mengying Qiu and Haiwei Han, College of Chemistry and Chemical Engineering, Xinyang Normal University, Xinyang, Henan 464000, P.R. China
Biao Yan and Hongya Li, School of Chemistry and Chemical Engineering, Yulin University, Yulin, Shaanxi 719000, P.R. China. https://orcid.org/0000-0002-7159-7911 (B. Yan). https://orcid.org/0000-0002-8539-8294 (H. Li)

Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2).

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
Zn	0.500000	0.500000	0.500000	0.03398 (12)
Cl1	0.25299 (10)	−0.04036 (6)	0.14618 (5)	0.0663 (2)
Cl2	0.77707 (12)	−0.13454 (5)	−0.00912 (5)	0.0642 (2)
Cl3	0.71881 (11)	0.33082 (7)	0.96400 (5)	0.0736 (3)
Cl4	0.32248 (16)	0.36455 (8)	1.20323 (5)	0.0956 (4)
N1	0.4535 (3)	0.36661 (14)	0.42132 (12)	0.0400 (5)
N2	0.4970 (3)	0.24552 (13)	0.34151 (12)	0.0366 (4)
N3	0.3249 (3)	0.22499 (18)	0.35156 (16)	0.0626 (7)
N4	0.4999 (3)	0.44148 (15)	0.60782 (12)	0.0426 (5)
N5	0.4245 (3)	0.39409 (15)	0.71656 (13)	0.0447 (5)
N6	0.5760 (4)	0.3637 (2)	0.70430 (19)	0.0802 (10)
N7	0.9145 (3)	0.40398 (17)	0.26016 (16)	0.0536 (6)
O1	0.8671 (2)	0.23930 (12)	0.37859 (11)	0.0465 (4)
H1A	0.895207	0.266972	0.343062	0.070*
O2	0.0509 (2)	0.36836 (12)	0.70416 (11)	0.0464 (4)
H2A	0.046135	0.420070	0.726576	0.070*
O3	0.2211 (2)	0.49390 (14)	0.49156 (12)	0.0461 (4)
H3A	0.147 (3)	0.4529 (15)	0.5032 (18)	0.060 (9)*
H3B	0.157 (4)	0.522 (2)	0.465 (2)	0.118 (16)*
O4	0.8797 (4)	0.32103 (15)	0.23765 (16)	0.0850 (8)
O5	0.9143 (4)	0.45208 (16)	0.20792 (14)	0.0767 (7)
O6	0.9462 (3)	0.43897 (15)	0.33658 (13)	0.0663 (6)
O7	0.9789 (3)	0.36568 (14)	0.53274 (14)	0.0594 (5)
H7D	0.956 (6)	0.3156 (14)	0.4964 (16)	0.114 (16)*
H7E	0.992 (5)	0.348 (2)	0.5793 (12)	0.103 (15)*
C1	0.5696 (3)	0.32934 (16)	0.38308 (14)	0.0370 (5)
H1	0.686141	0.357696	0.384891	0.044*
C2	0.3074 (4)	0.3004 (2)	0.3994 (2)	0.0650 (9)
H2	0.200911	0.307625	0.417053	0.078*
C3	0.5770 (3)	0.18021 (16)	0.29340 (14)	0.0365 (5)
C4	0.7401 (3)	0.15868 (17)	0.33908 (15)	0.0431 (6)
H4	0.794572	0.124360	0.296830	0.052*
C5	0.7093 (5)	0.1008 (2)	0.40445 (19)	0.0637 (9)
C6	0.5733 (6)	0.0147 (2)	0.3614 (2)	0.1003 (15)
H6A	0.458922	0.029575	0.347990	0.150*
H6B	0.566842	−0.026636	0.398248	0.150*
H6C	0.608895	−0.012991	0.310848	0.150*
C7	0.8859 (6)	0.0761 (3)	0.4348 (2)	0.0982 (15)
H7A	0.870034	0.038754	0.474630	0.147*
H7B	0.971812	0.130235	0.460751	0.147*
H7C	0.926946	0.043775	0.387938	0.147*
C8	0.6406 (6)	0.1508 (3)	0.4800 (2)	0.0852 (12)
H8A	0.523060	0.159729	0.461928	0.128*
H8B	0.718215	0.208300	0.503116	0.128*
H8C	0.637817	0.115272	0.521945	0.128*
C9	0.4973 (3)	0.14229 (17)	0.21564 (15)	0.0412 (6)
H9	0.392423	0.159115	0.195725	0.049*
C10	0.5645 (3)	0.07458 (16)	0.15812 (14)	0.0383 (5)
C11	0.4641 (3)	−0.01133 (17)	0.12337 (15)	0.0413 (6)
C12	0.5274 (4)	−0.07616 (17)	0.07205 (15)	0.0458 (6)
H12	0.458947	−0.133748	0.050314	0.055*
C13	0.6945 (4)	−0.05303 (18)	0.05409 (15)	0.0448 (6)
C14	0.7960 (4)	0.03182 (19)	0.08461 (16)	0.0503 (7)
H14	0.908144	0.046347	0.071076	0.060*
C15	0.7303 (4)	0.09552 (18)	0.13561 (16)	0.0465 (6)
H15	0.798022	0.153570	0.155343	0.056*
C16	0.3832 (3)	0.43992 (17)	0.65882 (14)	0.0381 (5)
H16	0.285068	0.467331	0.654753	0.046*
C17	0.6146 (5)	0.3937 (3)	0.6392 (2)	0.0773 (11)
H17	0.713640	0.383080	0.615839	0.093*
C18	0.3392 (3)	0.37647 (17)	0.78554 (15)	0.0420 (6)
C19	0.1468 (4)	0.32907 (17)	0.76263 (15)	0.0444 (6)
H19	0.095397	0.338215	0.813816	0.053*
C20	0.1135 (5)	0.2264 (2)	0.72690 (19)	0.0672 (9)
C21	0.2059 (7)	0.1832 (2)	0.7935 (2)	0.1120 (18)
H21A	0.169726	0.202659	0.846187	0.168*
H21B	0.332328	0.201511	0.799531	0.168*
H21C	0.173129	0.118561	0.776323	0.168*
C22	−0.0859 (6)	0.1910 (3)	0.7117 (3)	0.1072 (17)
H22A	−0.130867	0.208775	0.762307	0.161*
H22B	−0.110055	0.126213	0.694937	0.161*
H22C	−0.142541	0.215521	0.668258	0.161*
C23	0.1858 (6)	0.2034 (2)	0.6464 (2)	0.0875 (13)
H23A	0.143704	0.140406	0.621115	0.131*
H23B	0.313218	0.216055	0.658670	0.131*
H23C	0.146194	0.239085	0.608488	0.131*
C24	0.4445 (4)	0.39906 (18)	0.85968 (16)	0.0471 (6)
H24	0.560726	0.426558	0.859762	0.056*
C25	0.4047 (4)	0.38708 (18)	0.94240 (15)	0.0461 (6)
C26	0.5274 (4)	0.35912 (18)	0.99662 (17)	0.0492 (6)
C27	0.5038 (4)	0.3526 (2)	1.07694 (17)	0.0561 (8)
H27	0.588811	0.334958	1.112440	0.067*
C28	0.3523 (5)	0.3727 (2)	1.10278 (17)	0.0585 (8)
C29	0.2272 (4)	0.4007 (2)	1.05211 (17)	0.0582 (8)
H29	0.124849	0.413888	1.070810	0.070*
C30	0.2553 (4)	0.4090 (2)	0.97230 (16)	0.0525 (7)
H30	0.172347	0.429633	0.938329	0.063*

2 Experimental details

The OH-distances were restraint at 0.85(1) Å, using DFIX commands. Other hydrogen atoms were placed in their geometrically idealized positions and constrained to ride on their parent atoms, with C–H = 0.96 Å (methyl), U_{iso}(H) = 1.5 U_{eq}(C), C–H = 0.98 Å (methine), U_{iso}(H) = 1.2 U_{eq}(C), C–H = 0.93 Å (aromatic and alkenyl), U_{iso}(H) = 1.2 U_{eq}(C), and O–H = 0.82 Å (hydroxyl), U_{iso}(H) = 1.5 U_{eq}(O).

3 Comment

The complexes containing transition metal and various 1,2,4-triazole ligands have been extensively investigated because of their structures and potential applications in different fields.[6,7] The pharmacological and toxicological properties of

many drugs are improved when they form Zn(II) complexes[7–9] As a broad spectrum systemic 1,2,4-triazole fungicide, diniconazole (E)-(RS)-1- (2,4-dichlorophenyl)-4,4-dimethyl-2-(1H-1,2,4-triazol-1-yl) pent-1-en-3-ol, has been widely used to control various fungi in many kinds of crops by inhibiting steroid demethylation[10,11]

The asymmetric unit of the title structure contains a half Zn(II) cation, one coordinated water molecule, two diniconazole molecules one crystalline water molecule and one nitrate anion to construct a new mononuclear complex. The Zn(II) cation is six-coordinated by two water ligands and four triazole ligands. The Zn–N length (Zn–N1 = 2.153(2) Å, Zn–N4 = 2.142(2) Å) and Zn–O length (Zn–O3 = 2.134(2) Å), the N1–Zn–N4, N1–Zn–N4^{i}, N1–Zn–O3, N4–Zn–O3, N1–Zn–O3^{i}, and N4–Zn–O3^{i} bond angles of 89.53(8)°, 90.47(8)°, 90.82(8)°, 89.29(8)°, 89.18(8)°, and 90.71(8)°, respectively (symmetry code: (i) $-x+1, -y+1, -z+1$). The Zn(II) cation is the center of symmetry of the title compound, so the bond angle of N1–Zn–N1^{ii}, N4–Zn–N4^{i}, and O3–Zn–O3^{i} is 180°. These are in agreement with closely related Zn(II) complexes reported.[12–14] The main interaction between the zinc complex nitrate anion and water molecule is O1–H1A···O4 ($d_{O1\cdots O4}$ = 2.853(3) Å, 161.3°), O7–H7D···O1 ($d_{O7\cdots O1}$ = 2.821(3) Å, 153(3)°), O2–H2A···O5^{i} ($d_{O2\cdots O5}$ = 2.815(3) Å, 168.4°), and O3–H3B···O7^{i} ($d_{O3\cdots O7}$ = 2.916(3) Å, 148(4)°) hydrogen bond and electrostatic attraction. The title compound through O3–H3A···O7^{ii} ($d_{O3\cdots O7}$ = 2.741(3) Å, 177(3)°), O3–H3B···O6^{ii} ($d_{O3\cdots O6}$ = 2.980(3) Å, 120(3)°), (symmetry code: (ii) $x-1, y, z$) and O7–H7E···O2^{iii} ($d_{O7\cdots O2}$ = 2.800(3) Å, 153(3)°), (symmetry code: (iii) $x+1, y, z$) hydrogen bond forming a one-dimensional (1D) chain along the *a*-axis.

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Conflict of interest statement: The authors declare no conflicts of interest regarding this article.

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