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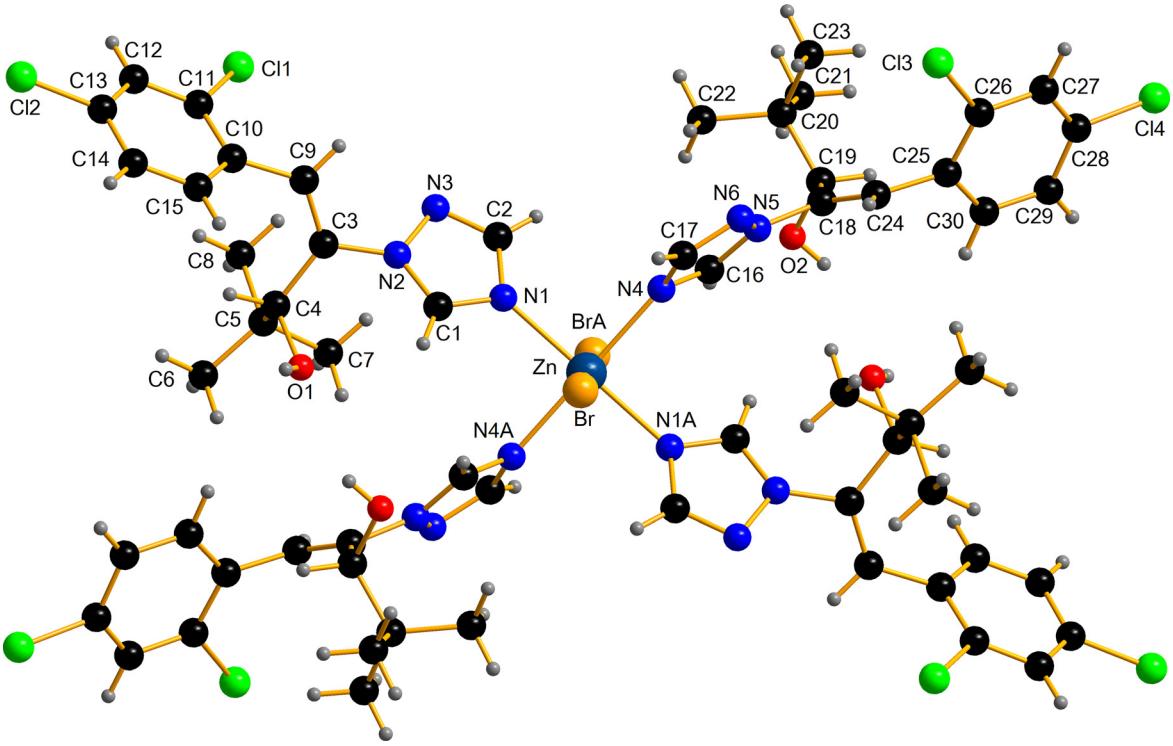
Crystal structure of dibromido-tetra((*E*)-(*RS*)-1-(2,4-dichlorophenyl)-4,4-dimethyl-2-(1,2,4-triazol-1-yl)pent-1-en-3-ol-κ^1N)zinc(II), $C_{60}H_{68}O_4N_{12}Br_2Cl_8Zn$



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Abstract

$C_{60}H_{68}O_4N_{12}Br_2Cl_8Zn$, triclinic, $P\bar{1}$ (no. 2), a = 9.0149 (13) Å, b = 13.671 (2) Å, c = 15.168 (2) Å, α = 92.280 (3)°, β = 97.441 (2)°, γ = 107.862 (2)°, V = 1758.0 (4) A^3, Z = 1, R_{gt} (F) = 0.0417, wR_{ref} (F^2) = 0.1005, T = 296 (2) K.

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The molecular 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:	Stick, colourless
Size:	0.39 × 0.30 × 0.26 mm
Wavelength:	Mo *K*α radiation (0.71073 Å)
μ:	1.84 mm^{-1}
Diffractometer, scan mode:	Bruker APEX-II, φ and ω-scans
θ_{max}, completeness:	27.4°, >99%
N (hkl)$_{measured}$, N(hkl)$_{unique}$, R_{int}:	10,124, 7338, 0.023
Criterion for I_{obs}, N(hkl)$_{gt}$:	I_{obs} > 2 σ(I_{obs}), 5015
N(*param*)$_{refined}$:	396
Programs:	Bruker programs [1], SHELX [2–4], DIAMOND [5]

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

	x	*y*	*z*	U_{iso}*/U_{eq}
Zn	0.000000	0.000000	0.500000	0.03388 (14)
Br	0.24579 (4)	−0.01731 (3)	0.61651 (2)	0.04691 (12)
Cl1	0.11145 (11)	−0.35660 (8)	0.02380 (7)	0.0680 (3)
Cl2	0.69040 (15)	−0.35700 (10)	−0.02233 (9)	0.0991 (4)
Cl3	0.62816 (12)	0.52985 (8)	0.24711 (8)	0.0705 (3)
Cl4	1.16357 (10)	0.46568 (8)	0.18232 (7)	0.0660 (3)
N1	0.0457 (3)	−0.09177 (19)	0.39593 (16)	0.0359 (6)
N2	0.1527 (3)	−0.13447 (19)	0.28551 (16)	0.0348 (6)
N3	−0.0013 (3)	−0.1960 (2)	0.26990 (18)	0.0486 (7)
N4	0.1558 (3)	0.13737 (19)	0.45635 (17)	0.0362 (6)
N5	0.3642 (3)	0.23629 (18)	0.40800 (16)	0.0333 (6)
N6	0.2424 (3)	0.2682 (2)	0.3709 (2)	0.0521 (8)
O1	0.4328 (2)	0.03415 (17)	0.27033 (15)	0.0473 (6)
H1A	0.517860	0.027479	0.290229	0.071*
O2	0.6359 (3)	0.18520 (17)	0.49308 (15)	0.0481 (6)
H2A	0.671088	0.148791	0.463030	0.072*
C1	0.1774 (3)	−0.0746 (2)	0.3600 (2)	0.0381 (7)
H1	0.273867	−0.026973	0.384089	0.046*
C2	−0.0579 (4)	−0.1667 (3)	0.3377 (2)	0.0490 (9)
H2	−0.162590	−0.195567	0.345511	0.059*
C3	0.2625 (3)	−0.1405 (2)	0.22617 (19)	0.0330 (7)
C4	0.3596 (4)	−0.0396 (2)	0.1960 (2)	0.0383 (7)
H4	0.443694	−0.053329	0.167634	0.046*
C5	0.2681 (4)	0.0103 (3)	0.1280 (2)	0.0534 (9)
C6	0.3905 (6)	0.0986 (3)	0.0929 (3)	0.0888 (15)
H6A	0.462362	0.071947	0.065944	0.133*
H6B	0.338073	0.130637	0.049214	0.133*
H6C	0.447664	0.148694	0.141408	0.133*
C7	0.1542 (5)	0.0534 (3)	0.1707 (3)	0.0782 (13)
H7A	0.077203	−0.001679	0.192823	0.117*
H7B	0.211729	0.103529	0.219125	0.117*
H7C	0.102137	0.085471	0.126931	0.117*
C8	0.1792 (6)	−0.0696 (3)	0.0501 (3)	0.0924 (15)
H8A	0.252293	−0.095928	0.023998	0.139*
H8B	0.101974	−0.125237	0.071457	0.139*
H8C	0.127618	−0.037729	0.005910	0.139*
C9	0.2634 (3)	−0.2342 (2)	0.19963 (19)	0.0380 (7)
H9	0.189647	−0.289862	0.218890	0.046*
C10	0.3712 (4)	−0.2581 (2)	0.1422 (2)	0.0365 (7)
C11	0.3129 (4)	−0.3181 (2)	0.0620 (2)	0.0432 (8)
C12	0.4092 (5)	−0.3486 (3)	0.0107 (2)	0.0536 (9)
H12	0.367502	−0.389056	−0.042874	0.064*
C13	0.5682 (5)	−0.3177 (3)	0.0409 (3)	0.0559 (10)
C14	0.6309 (4)	−0.2573 (3)	0.1189 (3)	0.0525 (9)
H14	0.739151	−0.236036	0.137626	0.063*
C15	0.5330 (4)	−0.2281 (2)	0.1697 (2)	0.0436 (8)
H15	0.575832	−0.187614	0.223133	0.052*
C16	0.3106 (3)	0.1594 (2)	0.4588 (2)	0.0356 (7)
H16	0.372532	0.126116	0.491296	0.043*
C17	0.1224 (4)	0.2070 (3)	0.4028 (2)	0.0498 (9)
H17	0.020979	0.211088	0.389451	0.060*
C18	0.5201 (3)	0.2771 (2)	0.3841 (2)	0.0323 (7)
C19	0.6561 (3)	0.2819 (2)	0.4552 (2)	0.0367 (7)
H19	0.750183	0.295626	0.425719	0.044*
C20	0.6924 (4)	0.3677 (3)	0.5315 (2)	0.0445 (8)
C21	0.8522 (4)	0.3724 (4)	0.5848 (3)	0.0746 (12)
H21A	0.843463	0.307746	0.609781	0.112*
H21B	0.930840	0.385654	0.545937	0.112*
H21C	0.881986	0.426668	0.632074	0.112*
C22	0.5674 (4)	0.3454 (3)	0.5946 (2)	0.0643 (11)
H22A	0.559419	0.280618	0.619054	0.096*
H22B	0.597466	0.399350	0.642205	0.096*
H22C	0.467230	0.342474	0.562006	0.096*
C23	0.7067 (5)	0.4700 (3)	0.4922 (3)	0.0759 (13)
H23A	0.607172	0.467282	0.458923	0.114*
H23B	0.736534	0.524256	0.539478	0.114*
H23C	0.785389	0.483242	0.453341	0.114*
C24	0.5313 (3)	0.3099 (2)	0.3038 (2)	0.0408 (8)
H24	0.439440	0.309393	0.267666	0.049*
C25	0.6826 (3)	0.3475 (2)	0.2679 (2)	0.0374 (7)
C26	0.7396 (4)	0.4471 (2)	0.2420 (2)	0.0394 (7)
C27	0.8854 (4)	0.4839 (3)	0.2134 (2)	0.0437 (8)
H27	0.921369	0.550897	0.196654	0.052*
C28	0.9750 (4)	0.4196 (3)	0.2104 (2)	0.0431 (8)
C29	0.9195 (4)	0.3183 (3)	0.2302 (2)	0.0492 (9)
H29	0.978866	0.274125	0.224627	0.059*
C30	0.7741 (4)	0.2840 (3)	0.2583 (2)	0.0468 (8)
H30	0.736135	0.215701	0.271336	0.056*

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 bromide (0.225 g, 0.100 mmol) was added. Reaction was allowed to stir at room temperature for 2 h. Colorless stick crystal of the title compound was obtained by slow evaporation from propanone.

Experimental details

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).

Comment

As a broad spectrum systemic 1,2,4-triazole fungicide, diniconazole (*E*)-(*RS*)-1-(2,4-dichlorophenyl)-4,4-dimethyl-2-(1*H*-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 [6, 7]. Complexation of pesticides with transition metals is a valuable way to improve bioactivity of original pesticides [8–16]. Zinc is a transition metal essential to the life of crops. Many triazole fungicides zinc complexes have been reported [13–19], the complexes synthesized shows better antifungal activities than the ligand [13–16]. In this paper, a new diniconazole Zn(II) complex is described.

The asymmetric unit of the title structure contains a half Zn(II) cation, one bromine anion and two diniconazole molecules to construct a mononuclear complex. The Zn(II) cation is six-coordinated by two bromine anions and nitrogen atoms of triazole cycles of four organic ligands. The Zn–N length (Zn–N1 = 2.136 (2) Å, Zn–N4 = 2.159 (2) Å) and Zn–Br length (Zn–Br = 2.7257 (4) Å), the N1–Zn–N4, N1–Zn–N4^{i}, N1–Zn–Br, N4–Zn–Br, N1–Zn–Br^{i}, and N4–Zn–Br^{i} bond angles of 90.27 (9)°, 89.73 (9)°, 92.61 (7)°, 89.49 (7)°, 87.39 (7)°, and 90.51 (7)°, respectively (symmetry code: (i) $-x, -y, -z+1$). Zn(II) cation is located in a special position at center of symmetry, so the bond angle of N1–Zn–N1^{ii}, N4–Zn–N4^{ii}, and Br–Zn–Br^{ii} is 180°. This is in agreement with closely related Zn(II) complexes reported [20]. The complexes are bound by O1–H1A···Br^{ii} ($d_{O1\cdots Br}$ = 3.248 (2) Å, 166.4°), and O2–H2A···Br^{ii} ($d_{O2\cdots Br}$ = 3.290 (2) Å, 171.5°), (symmetry code: (ii) $-x+1, -y, -z+1$) hydrogen bonds forming a one-dimensional (1D) chain along the a-axis.

Author contributions: All the authors have accepted responsibility for the entire content of this submitted manuscript and approved submission.

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

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