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Crystal structure of dichloro-tetrakis[(*E*)-1-(4-chlorophenyl)-4,4-dimethyl-2-(1*H*-1,2,4-triazol-1-yl)pent-1-en-3-ol- κ N]cadmium (II), $C_{60}H_{74}CdCl_6N_{12}O_4$

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Abstract

$C_{60}H_{74}CdCl_6N_{12}O_4$, triclinic, $P\bar{1}$ (no. 2), $a = 9.1036(18)$ Å, $b = 13.197(3)$ Å, $c = 15.035(3)$ Å, $\alpha = 93.716(2)^\circ$, $\beta = 99.904(2)^\circ$, $\gamma = 104.928(2)^\circ$, $V = 1708.0(6)$ Å³, $Z = 2$, $R_{gt}(F) = 0.0258$, $wR_{ref}(F^2) = 0.0684$, $T = 296(2)$ K.

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The crystal structure is shown in the figure. Tables 1 and 2 contain details on crystal structure and measurement conditions and a list of the atoms including atomic coordinates and displacement parameters.

Table 1: Data collection and handling.

Crystal:	Colorless block
Size:	0.22 × 0.15 × 0.12 mm
Wavelength:	Mo $K\alpha$ radiation (0.71073 Å)
μ :	0.61 mm ⁻¹
Diffractometer, scan mode:	Bruker APEX-II, φ and ω -scans
θ_{max} , completeness:	25.5°, >99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int} :	13223, 6307, 0.017
Criterion for I_{obs} , $N(hkl)_{gt}$:	$I_{obs} > 2 \sigma(I_{obs})$, 5840
$N(param)_{refined}$:	383
Programs:	Bruker programs [1], SHELX [2]

Source of materials

The title compound was synthesized by a hydrothermal method. A mixture of (*RS*)-uniconazole (0.0582 g, 0.2 mmol), $CdCl_2 \cdot H_2O$ (0.029 g, 0.1 mmol), ethanol (5 mL) and distilled water (5 mL) was sealed in a Teflon-lined stainless reactor (25 mL) and heated at 90 °C for 72 h, and then cooled to room temperature. The colorless block crystals were filtered and washed with water and ethanol. Yield 80% (based on $CdCl_2$). Elemental Anal. Calcd. (%) for $C_{60}H_{72}Cl_6CdN_6O_4$ (1350.41): C, 53.29; H, 5.52; N, 12.43. Found (%): C, 52.93; H, 5.55; N, 12.35. IR(KBr, cm⁻¹): 3414.61, 3165.77, 3141.55, 2956.36, 2872.20, 1593.48, 1514.70, 1491.70, 1428.50, 1399.96, 1361.07, 1235.70, 1217.83, 1198.95, 1188.90, 1131.48, 1091.88, 1070.57, 1056.01, 1014.64, 988.28, 976.80, 885.51, 875.02, 845.0, 823.68, 786.30, 705.28, 695.16, 671.12, 648.54, 522.55.

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Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²).

Atom	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{iso} */ <i>U</i> _{eq}
Cd1	0.5000	1.0000	0.0000	0.03639(7)
Cl1	−0.23120(9)	1.35273(6)	0.52676(5)	0.0929(2)
Cl2	−0.57033(10)	0.52341(6)	0.35450(7)	0.1165(3)
Cl5	0.25305(5)	1.01651(4)	−0.11319(3)	0.05589(13)
O1	0.05634(15)	0.96609(10)	0.23303(9)	0.0548(3)
H1A	−0.0121	0.9790	0.2021	0.066*
O2	−0.11748(17)	0.82013(11)	0.01346(10)	0.0579(3)
H2A	−0.1516	0.8594	0.0436	0.087*
N1	0.43798(17)	1.09239(13)	0.11890(10)	0.0481(4)
N2	0.47618(18)	1.19795(15)	0.24845(11)	0.0584(4)
N3	0.32609(16)	1.13723(12)	0.22568(9)	0.0429(3)
N4	0.34688(17)	0.84504(12)	0.04255(11)	0.0498(4)
N5	0.27551(18)	0.72730(14)	0.13827(12)	0.0584(4)
N6	0.14943(16)	0.75439(11)	0.09364(10)	0.0431(3)
C1	0.5363(2)	1.16666(18)	0.18243(14)	0.0599(5)
H1	0.6401	1.1939	0.1796	0.072*
C2	0.3067(2)	1.07656(15)	0.14813(12)	0.0452(4)
H2	0.2134	1.0296	0.1187	0.054*
C3	0.21368(19)	1.14558(14)	0.28094(11)	0.0402(4)
C4	0.2046(2)	1.24157(15)	0.30571(12)	0.0467(4)
H4	0.2756	1.3028	0.2852	0.056*
C5	0.0963(2)	1.26796(14)	0.36061(12)	0.0456(4)
C6	0.1521(3)	1.33489(16)	0.44079(14)	0.0595(5)
H6	0.2585	1.3625	0.4607	0.071*
C7	0.0520(3)	1.36130(17)	0.49184(15)	0.0668(6)
H7	0.0908	1.4060	0.5459	0.080*
C8	−0.1042(3)	1.32136(16)	0.46234(14)	0.0579(5)
C9	−0.1632(3)	1.25565(19)	0.38311(15)	0.0665(6)
H9	−0.2698	1.2291	0.3633	0.080*
C10	−0.0622(2)	1.22912(19)	0.33266(14)	0.0619(6)
H10	−0.1020	1.1842	0.2788	0.074*
C11	0.1265(2)	1.04381(14)	0.30922(12)	0.0438(4)
H11	0.0429	1.0584	0.3366	0.053*
C12	0.2236(3)	0.99458(17)	0.37980(14)	0.0612(5)
C13	0.3097(4)	1.0798(2)	0.45876(17)	0.0992(10)
H13A	0.3593	1.0484	0.5069	0.149*
H13B	0.2374	1.1118	0.4807	0.149*
H13C	0.3866	1.1326	0.4382	0.149*
C14	0.1105(4)	0.9065(2)	0.4146(2)	0.0940(9)
H14A	0.0588	0.8517	0.3656	0.141*
H14B	0.0352	0.9344	0.4377	0.141*
H14C	0.1663	0.8780	0.4622	0.141*
C15	0.3381(3)	0.9481(2)	0.33860(19)	0.0858(8)
H15A	0.4219	1.0043	0.3282	0.129*
H15B	0.2858	0.9061	0.2820	0.129*
H15C	0.3783	0.9047	0.3798	0.129*
C16	0.1960(2)	0.82465(15)	0.03734(13)	0.0480(4)
H16	0.1311	0.8550	−0.0003	0.058*
C17	0.3884(2)	0.78405(16)	0.10498(15)	0.0581(5)
H17	0.4907	0.7825	0.1232	0.070*
C18	−0.00033(19)	0.71639(13)	0.11715(12)	0.0416(4)
C19	−0.0050(2)	0.67757(16)	0.19598(13)	0.0537(5)
H19	0.0884	0.6740	0.2303	0.064*
C20	−0.1462(2)	0.63927(16)	0.23444(12)	0.0517(5)
C21	−0.2344(3)	0.70590(18)	0.25371(15)	0.0646(6)

Table 2 (continued)

Atom	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{iso} */ <i>U</i> _{eq}
H21	−0.2055	0.7757	0.2415	0.078*
C22	−0.3642(3)	0.67068(19)	0.29060(17)	0.0718(6)
H22	−0.4227	0.7161	0.3034	0.086*
C23	−0.4060(3)	0.56759(18)	0.30822(15)	0.0671(6)
C24	−0.3207(3)	0.49961(18)	0.29139(15)	0.0659(6)
H24	−0.3503	0.4301	0.3042	0.079*
C25	−0.1896(3)	0.53608(17)	0.25499(14)	0.0594(5)
H25	−0.1299	0.4908	0.2442	0.071*
C26	−0.1404(2)	0.72063(14)	0.04851(13)	0.0451(4)
H26	−0.2260	0.7141	0.0813	0.054*
C27	−0.1956(2)	0.63130(16)	−0.03128(14)	0.0548(5)
C28	−0.3516(3)	0.6396(2)	−0.08298(19)	0.0879(8)
H28A	−0.3373	0.7060	−0.1076	0.132*
H28B	−0.4229	0.6350	−0.0422	0.132*
H28C	−0.3924	0.5831	−0.1316	0.132*
C29	−0.2193(4)	0.52498(18)	0.00686(18)	0.0874(8)
H29A	−0.2582	0.4690	−0.0423	0.131*
H29B	−0.2925	0.5194	0.0465	0.131*
H29C	−0.1222	0.5198	0.0403	0.131*
C30	−0.0832(3)	0.6410(2)	−0.09681(15)	0.0739(6)
H30A	0.0136	0.6325	−0.0660	0.111*
H30B	−0.0664	0.7093	−0.1181	0.111*
H30C	−0.1263	0.5873	−0.1475	0.111*

Experimental details

All H atoms were included in calculated positions and refined as riding atoms, with C—H = 0.90–0.97 Å with *U*_{iso}(H) = 1.5 *U*_{eq}(C) for methyl H atoms and 1.2 *U*_{eq}(C) for all other H atoms.

Comment

Uniconazole, [(*E*)-(*RS*)-1-(4-chlorophenyl)-4,4-dimethyl-2-(1*H*-1,2,4-triazol-1-yl)pent-1-en-3-ol] is a highly active fungicide and plant growth regulator with low toxicity. It inhibits the biosynthesis of gibberellin and shows excellent efficacy biological activity and is widely used to protect crops, such as rice, wheat, soybean, peanut, rapeseed, apple [3–10]. Because of its strong antimicrobial activities and its wide applications, the synthesis and applications of triazole pesticides and its derivatives have attracted much attention, but the complexes of triazole pesticides are still rare [11]. We still focused on the synthesis and antibacterial activities of complexes assembled by triazole fungicides. We have already reported a series of crystal structures and antibacterial activities of diniconazole complexes [12–18]. In order to study the antimicrobial activities of triazole fungicides complexes more systematically and comprehensively, to find new antibactericidal complexes with the efficiency, broad-spectrum and low toxicity. Herein we report the synthesis and crystal structure of a new cadmium(II) complex incorporating uniconazole.

The asymmetric unit of the title compound, $[Cd(C_{15}H_{18}ClN_3O)_4Cl_2]$, consists of one half of the title complex. The Cd atom lies on an inversion center and has a slightly distorted octahedral geometry. The equatorial positions are occupied by four N atoms from four uniconazole ligands. The axial sites are occupied by two chlorido ligands (*cf.* the figure). The Cd—N distances are 2.3281(15) and 2.3541(15) Å and Cd—Cl is 2.6424(6) Å. In the crystal packing, intermolecular O—H...Cl hydrogen bonds link the molecules into chains along the *a* axis. The structure of the title compound is isostructural to previously reported zinc(II) complex [18].

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