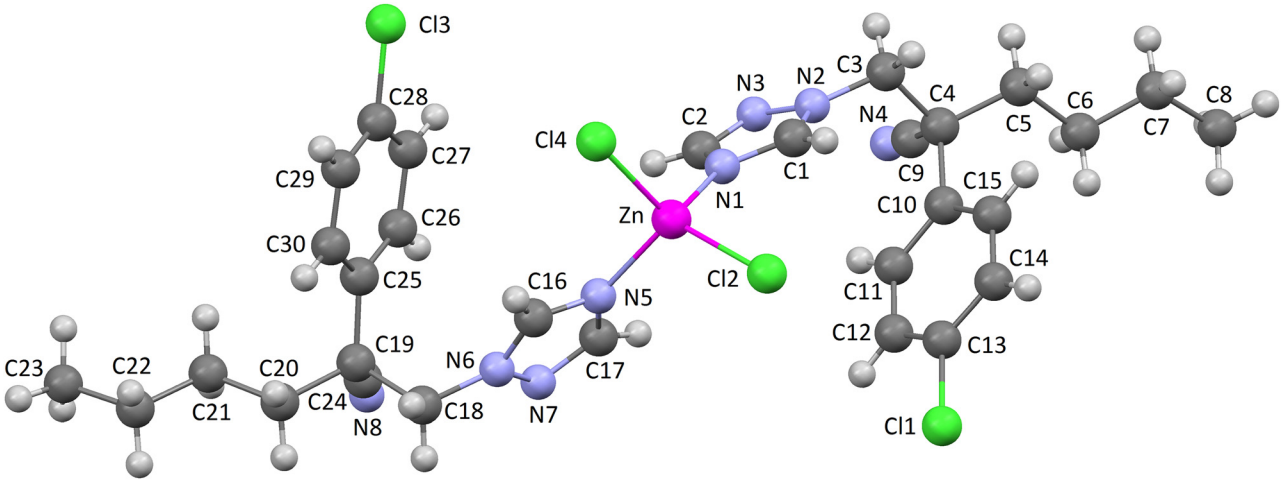
Hui-Jin Liu*

The crystal structure of dichlorido-bis((*RS*)-2-(4-chlorophenyl)-2-(1,2,4-triazol-1-ylmethyl) hexanenitrile-κ^1*N*)zinc(II), $C_{30}H_{34}Cl_4N_8Zn$



https://doi.org/10.1515/ncrs-2022-0226
Received May 1, 2022; accepted June 28, 2022;
published online July 15, 2022

Abstract

$C_{30}H_{34}Cl_4N_8Zn$, orthorhombic, $Pca2_1$ (no. 29), a = 20.235(2) Å, b = 6.5462(8) Å, c = 27.386(3) Å, V = 3627.6(7) Å^3, Z = 4, $R_{gt}(F)$ = 0.0477, $wR_{ref}(F^2)$ = 0.1128, T = 293(2) K.

CCDC No.: 1060434

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.

Source of material

The (*RS*)-2-(4-chlorophenyl)-2-(1,2,4-triazol-1-ylmethyl) hexanenitrile (0.578 g, 0.200 mmol) was dissolved in ethanol absolute (10 mL). Then, $ZnCl_2$ (0.136 g, 0.100 mmol) was added. The reaction mixture was allowed to stir at room temperature for 2 h. Colourless rod-like crystals of the title compound were obtained by slow evaporation from ethanol.

***Corresponding author: Hui-Jin Liu**, School of Chemistry and Chemical Engineering, Yulin University, Yulin, Shaanxi 719000, P. R. China, E-mail: yllhj0415@163.com. https://orcid.org/0000-0002-7173-1797

Table 1: Data collection and handling.

Crystal:	Colourless rodlike
Size:	0.36 × 0.30 × 0.20 mm
Wavelength:	Mo *K*α radiation (0.71073 Å)
μ:	1.00 mm^{-1}
Diffractometer, scan mode:	Bruker APEX-II, φ and ω
θ_{max}, completeness:	26.4°, >99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	18,750, 7301, 0.055
Criterion for I_{obs}, $N(hkl)_{gt}$:	I_{obs} > 2 σ(I_{obs}), 4734
$N(param)_{refined}$:	391
Programs:	Bruker [1], SHELX [2–4], Mercury [5]

Experimental details

Refinement as a two-component inversion twin was undertaken. All H atoms were placed geometrically and refined using a riding-model approximation, with C–H distances of 0.93 Å (aromatic), 0.97 Å (methylene), 0.96 Å (methyl), U_{iso}(H) = 1.2U_{eq} (aromatic and methylene) and 1.5U_{eq}(methyl) respectively.

Comment

Triazole fungicides, containing 1,2,4-triazole groups in the main chain, are used worldwide to protect fruits, vegetables and crops because of their excellent antifungal activity [6]. Myclobutanil (MT), (*RS*)-2-(4-chlorophenyl)-2-(1,2,4-triazol-

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

Atom	x	y	z	U_{iso}*/U_{eq}
Zn	0.87718 (3)	1.06525 (8)	0.61066 (6)	0.04490 (19)
Cl1	0.8926 (2)	0.9802 (8)	0.8258 (2)	0.158 (2)
Cl2	0.87944 (10)	1.2489 (5)	0.67869 (9)	0.0741 (8)
Cl3	0.8569 (2)	0.9981 (6)	0.39289 (19)	0.150 (2)
Cl4	0.87477 (10)	1.2484 (5)	0.54239 (9)	0.0752 (9)
N1	0.9556 (2)	0.8711 (7)	0.6152 (3)	0.0447 (13)
N2	1.0540 (2)	0.7727 (7)	0.63557 (19)	0.0355 (12)
N3	1.0278 (2)	0.6141 (7)	0.6108 (3)	0.0468 (12)
N4	1.1078 (4)	0.2591 (12)	0.6891 (3)	0.069 (2)
N5	0.7990 (2)	0.8707 (8)	0.6056 (3)	0.0448 (14)
N6	0.7001 (2)	0.7751 (8)	0.58506 (19)	0.0378 (13)
N7	0.7261 (2)	0.6137 (7)	0.6108 (3)	0.0476 (12)
N8	0.6487 (4)	0.2602 (10)	0.5325 (3)	0.0647 (19)
C1	1.0102 (3)	0.9231 (12)	0.6387 (2)	0.0407 (15)
H1	1.016823	1.046144	0.654970	0.049*
C2	0.9692 (3)	0.6798 (10)	0.5990 (3)	0.047 (2)
H2	0.939289	0.602508	0.581008	0.057*
C3	1.1183 (3)	0.7626 (9)	0.6583 (2)	0.0376 (15)
H3A	1.148817	0.697055	0.635890	0.045*
H3B	1.134068	0.900636	0.663839	0.045*
C4	1.1196 (3)	0.6464 (14)	0.7070 (3)	0.0381 (19)
C5	1.1884 (3)	0.6745 (12)	0.7302 (3)	0.051 (2)
H5A	1.221437	0.628776	0.706917	0.061*
H5B	1.195609	0.819181	0.735645	0.061*
C6	1.1997 (4)	0.5619 (16)	0.7784 (4)	0.073 (3)
H6A	1.195791	0.415904	0.773004	0.088*
H6B	1.166071	0.602090	0.801667	0.088*
C7	1.2663 (5)	0.608 (2)	0.7989 (4)	0.117 (4)
H7A	1.299345	0.570633	0.774833	0.141*
H7B	1.269547	0.754410	0.803946	0.141*
C8	1.2821 (8)	0.5063 (18)	0.8444 (6)	0.140 (7)
H8A	1.321026	0.566517	0.858348	0.210*
H8B	1.289865	0.363841	0.838356	0.210*
H8C	1.245823	0.520985	0.866762	0.210*
C9	1.1112 (4)	0.4264 (17)	0.6980 (3)	0.045 (2)
C10	1.0626 (3)	0.7230 (12)	0.7399 (2)	0.0434 (18)
C11	1.0007 (4)	0.6271 (14)	0.7396 (3)	0.067 (2)
H11	0.994541	0.509738	0.721033	0.080*
C12	0.9495 (4)	0.7038 (19)	0.7662 (4)	0.096 (3)
H12	0.908957	0.636852	0.766850	0.115*
C13	0.9586 (5)	0.8808 (19)	0.7919 (4)	0.089 (3)
C14	1.0193 (5)	0.9807 (14)	0.7941 (5)	0.070 (4)
H14	1.025263	1.097185	0.813034	0.084*
C15	1.0698 (4)	0.8986 (13)	0.7670 (3)	0.060 (2)
H15	1.110554	0.964435	0.766925	0.072*
C16	0.7443 (3)	0.9228 (11)	0.5822 (3)	0.0410 (15)
H16	0.737891	1.046100	0.565997	0.049*
C17	0.7852 (3)	0.6806 (10)	0.6224 (3)	0.049 (2)
H17	0.815021	0.604161	0.640683	0.059*
C18	0.6355 (3)	0.7635 (10)	0.5626 (2)	0.0379 (16)
H18A	0.605343	0.695430	0.584788	0.045*
H18B	0.619066	0.900832	0.557189	0.045*
C19	0.6360 (4)	0.6476 (14)	0.5134 (3)	0.0379 (19)
C20	0.5654 (3)	0.6726 (13)	0.4915 (3)	0.050 (2)
H20A	0.533445	0.627481	0.515614	0.060*
H20B	0.557553	0.816574	0.485534	0.060*
C21	0.5536 (4)	0.5577 (16)	0.4451 (4)	0.071 (3)
H21A	0.557673	0.412533	0.451543	0.085*
H21B	0.587498	0.594678	0.421653	0.085*
C22	0.4865 (5)	0.599 (2)	0.4231 (4)	0.111 (4)
H22A	0.483666	0.742823	0.414349	0.134*
H22B	0.452717	0.572187	0.447351	0.134*
C23	0.4728 (8)	0.472 (3)	0.3783 (6)	0.164 (8)
H23A	0.434054	0.522343	0.362084	0.246*
H23B	0.465746	0.332184	0.387821	0.246*
H23C	0.509875	0.479056	0.356497	0.246*
C24	0.6450 (4)	0.4289 (17)	0.5239 (3)	0.045 (2)
C25	0.6907 (3)	0.7262 (11)	0.4807 (3)	0.0430 (17)
C26	0.7515 (4)	0.6352 (14)	0.4806 (3)	0.066 (2)
H26	0.758076	0.515535	0.498249	0.080*
C27	0.8042 (4)	0.7202 (18)	0.4542 (4)	0.092 (3)
H27	0.845777	0.660050	0.454809	0.110*
C28	0.7929 (5)	0.8939 (19)	0.4276 (3)	0.083 (3)
C29	0.7342 (6)	0.9867 (15)	0.4281 (5)	0.083 (4)
H29	0.728219	1.107701	0.410837	0.100*
C30	0.6824 (4)	0.9040 (12)	0.4540 (3)	0.057 (2)
H30	0.641461	0.968343	0.453668	0.068*

1-ylmethyl) hexanenitrile, is a typical triazole fungicide used for the control of powdery mildew and scabbing of plants [7–9]. The pharmacological and toxicological properties of many drugs are improved when they form Zn(II) complexes [10–12]. In this paper, a new myclobutanil Zn(II) complex is described.

The title compound crystallizes in the orthorhombic $Pca2_1$ space group with one title complex in the asymmetric unit. The Zn(II) is four-coordinated by two chlorine atoms and two nitrogen atoms and the Zn(II) center has a distorted tetrahedral structure. The Zn–N lengths (Zn1–N1 = 2.037(4) Å; Zn–N5 = 2.036(5) Å) and the Zn–Cl lengths (Zn–Cl2 = 2.218(3) Å; Zn–Cl4 = 2.221(3) Å), the N1–Zn–N5, Cl2–Zn–Cl4 and N–Zn–Cl bond angles of 102.69(16)°, 114.51(6)° and 105.3(2)–114.3(2)°, respectively. These are in agreement with closely related Zn(II) complexes reported [13, 14].

Author contributions: The author has accepted responsibility for the entire content of this submitted manuscript and approved submission.

Research funding: The work was supported by Yulin City Industry-University-Research Cooperation Project (CXY-2020-004-02), Yulin National High-tech Industrial Development Zone Science and Technology Plan Project (CXY-2021-33).

Conflict of interest statement: The author declares no conflicts of interest regarding this article.

References

1. Bruker. *Apex2, Saint and Sadabs*; Bruker AXS Inc.: Madison, Wisconsin, USA, 2009.
2. Sheldrick G. M. *SHELXTL* – integrated space-group and crystal-structure determination. *Acta Crystallogr.* 2015, *A71*, 3–8.
3. Sheldrick G. M. Crystal structure refinement with *SHELXL*. *Acta Crystallogr.* 2015, *C71*, 3–8.
4. Sheldrick G. M. A short history of *SHELX*. *Acta Crystallogr.* 2008, *A64*, 112–122.
5. Macrae C. F., Bruno I. J., Chisholm J. A., Edgington P. R., McCabe P., Pidcock E., Rodriguez-Monge L., Taylor R., van de Streek J., Wood P. A. Mercury CSD 2.0 - new features for the visualization and investigation of crystal structures. *J. Appl. Crystallogr.* 2008, *41*, 466–470.
6. Hao W., Hu X., Zhu F., Chang J., Li J., Li W., Wang H., Guo B., Li J., Xu P., Zhang Y. Enantioselective distribution, degradation, and metabolite formation of myclobutanil and transcriptional responses of metabolic-related genes in rats. *Environ. Sci. Technol.* 2018, *52*, 8830–8837.
7. Quinn J. A., Fujimoto T. T., Egan A. R., Shaber S. H. The properties of RH 3866, a new triazole fungicide. *Pestic. Sci.* 1986, *17*, 357–362.
8. Fujimoto T. T., Shaber S. H., Chan H. F., Quinn J. A., Carlson G. R. Successful exploitation of 2-cyano arylethyltriazoles as agricultural fungicides. In *Synthesis and Chemistry of Agrochemicals*; Baker D. R., Fenyes J. G., Moberg W. K., Cross B., Eds.; American Chemical Society: Washington, DC, 1987; pp. 318–327.
9. Kemmitt G. M., Deboer G., Ouimette D., Iamauti M. Systemic properties of myclobutanil in soybean plants, affecting control of Asian soybean rust (Phakopsora pachyrhizi). *Pest Manag. Sci.* 2008, *64*, 1285–1293.
10. Li J., Xi T., Yan B., Yang M.-Y., Ma H.-X., Song J.-R. Crystal structure and antifungal activities of a new Zn(II) complex based on triadimenol. *Chin. J. Struct. Chem.* 2015, *34*, 1825–1829.
11. Ren G.-Y., Li J., Zhou J.-H., Yan B., Ren Y.-H., Sun X.-H., Ma H.-X. Enhanced antifungal activities of four Zn(II) complexes based on uniconazole. *Appl. Organomet. Chem.* 2018, *32*, e4169.
12. Ren G.-Y., Li J., Wei X.-Z., Zhou J.-H., Yan B., Guo Z.-Q., Ren Y.-H., Sun X.-H., Ma H.-X. The enhanced biological activities of three Zn(II) complexes based on different 1,2,4-triazole fungicides. *Appl. Organomet. Chem.* 2018, *32*, e4474.
13. Liu X.-J., Liu E., Jin Z.-S., Li Z.-Y., Jian F.-F., Liang T.-L. Crystal structure of dichlorido-bis(1-hexyl-1*H*-benzotriazole-k^1*N*) zinc(II), $C_{24}H_{34}N_6Cl_2Zn$. *Z. Kristallogr. N. Cryst. Struct.* 2021, *236*, 379–381.
14. Adeleke A. A., Zamisa S. J., Omondi B. Crystal structure of dichlorido-bis((*E*)-2-((pyridin-4-ylmethylene)amino)phenol) zinc(II), $C_{24}H_{20}Cl_2N_4O_2Zn$. *Z. Kristallogr. N. Cryst. Struct.* 2020, *235*, 625–628.