Crystal structure of dichloro[3-(pyridin-2-yl)-1*H*-pyrazol-1-yl]propanamidezinc(II), $ZnCl_2(C_{11}H_{12}N_4O)$

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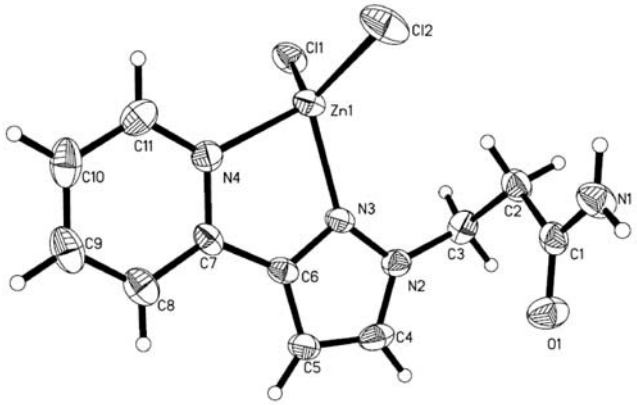

Abstract

$C_{11}H_{12}Cl_2N_4OZn$, triclinic, $P\bar{1}$ (no. 2), a = 7.487(2) Å, b = 8.604(2) Å, c = 11.952(2) Å, α = 69.13(3)°, β = 80.47(3)°, γ = 84.03(3)°, V = 708.6 Å^3, Z = 2, $R_{gt}(F)$ = 0.048, $wR_{ref}(F^2)$ = 0.167, T = 296 K.

Source of material

A solution of the 3-[3-(pyridin-2-yl)-1*H*-pyrazol-1-yl]propanamide ligand (0.0432 g, 0.2 mmol) in acetonitrile (15 ml) was added dropwise to a solution of $ZnCl_2$ (0.0272 g, 0.2 mmol) in methanol (10 ml) in a 50 ml beaker. The mixture was stirred for 30 min at 323 K, then filtered. The resulted solution was kept at room temperature. Colorless block-shaped single crystals suitable for X-ray diffraction were obtained after two weeks (yield 25 % based on Zn). Elemental analysis — found: C, 37.59 %; H, 3.21 %; Cl, 20.26 %; N, 15.97 %; Zn, 18.69 %; calculated for $C_{11}H_{12}Cl_2N_4OZn$: C, 37.84 %; H, 3.43 %; Cl, 20.11 %; N, 15.89 %; Zn, 18.55 %.

Discussion

In recent years, 3-(2-pyridyl)pyrazole-based ligands have been found for a wide range of application in coordination chemistry, because they can act as bridging or chelating ligands and exhibit a series of intriguing structures and potential applications as functional materials [1,2]. Nowadays, much attention has been focused on the synthetic approach and the structural control of coordination architectures [3,4]. The synthesis and crystallographic description of the 3-[3-(pyridin-2-yl)-1*H*-pyrazol-1-yl]propan-amide ligand were first put forward in our laboratory [5], but its coordination complexes with metals have not been yet reported.

In the title complex, the dihedral angle between pyrazole and pyridine ring is 2.8(4)°, and the torsion angle of N3–C6–C7–C8 is 176.7(6)°. The Zn(II) centre is four-coordinated by two N atoms from one 3-(3-(pyridin-2-yl)-1*H*-pyrazol-1-yl)propanamide ligand and two Cl atoms. The complexes are interconnected into a three-dimensional supermolecular network through intermolecular N–H···O and N–H···Cl hydrogen bonds. The crystal structure is also stabilized by slipped π-π stacking interactions between the N2–N3–C6–C5–C4 and the N4–C11–C10–C9–C8–C7 rings with centroid-to-centroid distance of 3.626(1) Å and interplanar distance of 3.474(1) Å.

Table 1. Data collection and handling.

Crystal:	colorless block, size 0.06 × 0.18 × 0.38 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	21.06 cm^{-1}
Diffractometer, scan mode:	Rigaku R-AXIS RAPID, ω
$2\theta_{max}$:	54.92°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	6977, 3219
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 1936
$N(param)_{refined}$:	173
Programs:	SHELXS-97, SHELXL-97 [6]

Table 2. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	x	y	z	U_{iso}
H(1A)	2*i*	–0.0132	1.3687	0.4656	0.071
H(1B)	2*i*	0.0804	1.2128	0.4535	0.071
H(2A)	2*i*	0.4361	1.2091	0.4441	0.064
H(2B)	2*i*	0.3303	1.0918	0.5637	0.064
H(3A)	2*i*	0.6153	1.1364	0.5978	0.063
H(3B)	2*i*	0.5716	1.3291	0.5539	0.063
H(4A)	2*i*	0.4816	1.4306	0.7348	0.065
H(5A)	2*i*	0.3436	1.3059	0.9488	0.059
H(8A)	2*i*	0.1825	1.0740	1.1305	0.067
H(9A)	2*i*	0.0545	0.8483	1.2852	0.079
H(10A)	2*i*	0.0335	0.6055	1.2488	0.082
H(11A)	2*i*	0.1416	0.5908	1.0599	0.068

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Table 3. Atomic coordinates and displacement parameters (in $Å^2$).

Atom	Site	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Zn(1)	2*i*	0.3441(1)	0.8251(1)	0.81470(8)	0.0468(5)	0.0458(5)	0.0492(5)	0.0009(3)	–0.0080(3)	–0.0259(4)
Cl(1)	2*i*	0.6115(3)	0.6925(3)	0.8063(2)	0.058(1)	0.067(1)	0.065(1)	0.0182(9)	–0.0137(9)	–0.039(1)
Cl(2)	2*i*	0.1527(3)	0.7886(3)	0.7065(2)	0.070(1)	0.110(2)	0.101(2)	–0.006(1)	–0.033(1)	–0.065(2)
O(1)	2*i*	0.2268(7)	1.4523(6)	0.5413(5)	0.056(3)	0.054(3)	0.093(4)	0.008(3)	–0.024(3)	–0.035(3)
N(1)	2*i*	0.0800(8)	1.3001(8)	0.4727(6)	0.053(4)	0.071(4)	0.069(4)	0.003(3)	–0.018(3)	–0.038(4)
N(2)	2*i*	0.4463(7)	1.2096(7)	0.7188(5)	0.047(3)	0.040(3)	0.048(3)	–0.005(2)	–0.009(3)	–0.016(3)
N(3)	2*i*	0.3742(7)	1.0674(6)	0.7995(5)	0.044(3)	0.039(3)	0.043(3)	0.001(2)	–0.009(2)	–0.021(2)
N(4)	2*i*	0.2298(7)	0.8175(7)	0.9863(5)	0.043(3)	0.043(3)	0.045(3)	0.001(2)	–0.007(2)	–0.015(3)
C(1)	2*i*	0.2233(9)	1.3292(9)	0.5134(6)	0.047(4)	0.052(4)	0.032(3)	0.001(3)	0.002(3)	–0.015(3)
C(2)	2*i*	0.378(1)	1.2018(9)	0.5245(7)	0.060(5)	0.060(4)	0.042(4)	0.004(4)	–0.006(3)	–0.022(3)
C(3)	2*i*	0.5190(9)	1.2216(9)	0.5956(6)	0.045(4)	0.052(4)	0.049(4)	0.003(3)	0.001(3)	–0.011(3)
C(4)	2*i*	0.441(1)	1.3233(9)	0.7720(8)	0.058(4)	0.037(4)	0.077(6)	0.001(3)	–0.026(4)	–0.023(4)
C(5)	2*i*	0.365(1)	1.2558(8)	0.8903(7)	0.057(4)	0.045(4)	0.053(4)	0.007(3)	–0.017(3)	–0.026(3)
C(6)	2*i*	0.3252(8)	1.0952(8)	0.9030(6)	0.036(3)	0.044(3)	0.046(4)	0.000(3)	–0.012(3)	–0.024(3)
C(7)	2*i*	0.2402(8)	0.9616(8)	1.0069(6)	0.035(3)	0.056(4)	0.039(4)	0.011(3)	–0.012(3)	–0.023(3)
C(8)	2*i*	0.1747(9)	0.975(1)	1.1177(7)	0.043(4)	0.076(5)	0.053(5)	0.004(4)	–0.007(3)	–0.031(4)
C(9)	2*i*	0.098(1)	0.841(1)	1.2096(7)	0.048(4)	0.099(7)	0.047(5)	0.007(4)	0.001(3)	–0.028(5)
C(10)	2*i*	0.086(1)	0.697(1)	1.1880(7)	0.053(5)	0.086(6)	0.045(5)	0.004(4)	0.007(4)	–0.006(4)
C(11)	2*i*	0.152(1)	0.6884(9)	1.0746(7)	0.048(4)	0.047(4)	0.068(5)	–0.001(3)	–0.009(4)	–0.012(4)

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