Crystal structure of dichlorido[*N*-(pyridin-2-ylmethyl)aniline]zinc(II), $C_{12}H_{12}Cl_2N_2Zn$

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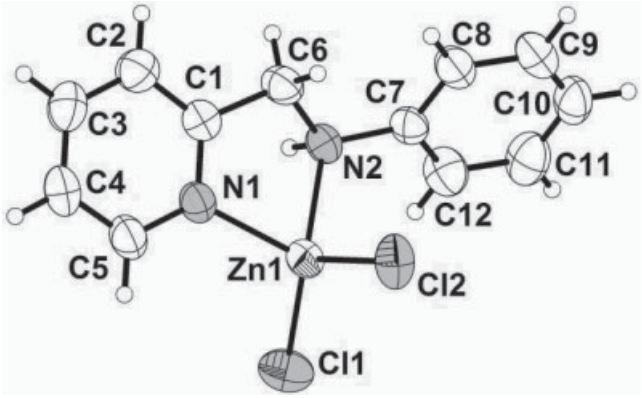

Abstract

$C_{12}H_{12}Cl_2N_2Zn$, monoclinic, $P2_1/c$ (no. 14), a = 8.615(2) Å, b = 10.589(2) Å, c = 16.525(2) Å, β = 116.598(6)°, V = 1347.9 Å^3, Z = 4, $R_{gt}(F)$ = 0.0247, $wR_{ref}(F^2)$ = 0.0697, T = 298 K.

Table 1. Data collection and handling.

Crystal:	colourless blocks, size 0.10×0.10×0.20 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	21.96 cm^{-1}
Diffractometer, scan mode:	SMART CCD area-detector, φ and ω
$2\theta_{max}$:	52°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	7012, 2636
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 2309
$N(param)_{refined}$:	154
Programs:	SADABS [5], SHELX [6]

Source of material

Picolinaldehyde (1.07 g, 10 mmol) and aniline (0.93 g, 10 mmol) in methanol (50 mL) were heated to reflux for 1 h. After the mixture was cooled to room temperature, $NaBH_4$ (0.074 g, 20 mmol) was added slowly with vigorous stirring. Then the solvent was removed in a rotary evaporator and the solid residue treated with 50 mL of water. The mixture was neutralized with glacial acetic acid and then extracted three times with 50 mL of diethyl ether. The combined extracts were washed with water (50 mL), evaporated to dryness in vacuo. Afterwards $ZnCl_2$ and one molar equivalents of *N*-(pyridin-2-ylmethyl)aniline were dissolved in methanol with stirring. After allowing the clear purple solution obtained to stand at room temperature in air for 3 days, large colourless block-shaped crystals were formed at the bottom of the vessel on slow evaporation of the solvent.

Experimental details

The hydrogen atom H2A was located in a difference Fourier map and the positional parameters were refined with U_{iso}(H) = 1.2 U_{eq}(N). All other H-atoms were placed in calculated positions and treated as riding: C–H = 0.93 or 0.97 Å, with U_{iso}(H) = 1.2 U_{eq}(C).

Discussion

α-amino-pyridines metal complexes, in particular those of gallium(III), nickel(II) and zinc(II), have a great potential to be developed into novel anticancer drugs [1-3], and its cationic palladium complexes can catalyze the copolymerization of ethylene and norbornene [4]. The title complex was prepared by the reaction of $ZnCl_2$ with the potentially bidentate ligand *N*-(pyridin-2-ylmethyl)aniline. The molecular structure of the title compound is shown in the figure with ellipsoids drawn at the 50% probability level. The Zn^{II} ion is in a distorted tetrahedral coordination environment. The angles around the Zn^{II} atom are from 81.53(7) to 120.79(5)°. The Zn–N [2.0469(2) and 2.1085(2) Å] bond lengths in the title structure are slightly shorter than those [2.149(6)-2.259(7) Å] in a similar complex [3]. The N–H···Cl hydrogen bonds link the complexes into one-dimensional chains which run along *b*.

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

Atom	Site	x	y	z	U_{iso}
H(2)	4*e*	–0.00309	0.33807	0.13805	0.0647
H(2A)	4*e*	0.44670	0.53822	0.28949	0.0685
H(3)	4*e*	–0.10381	0.32154	–0.01765	0.0709
H(4)	4*e*	0.08838	0.34930	–0.07877	0.0661
H(5)	4*e*	0.37539	0.38720	0.01589	0.0606
H(6A)	4*e*	0.35144	0.28411	0.29094	0.0574
H(6B)	4*e*	0.23883	0.40314	0.28597	0.0574
H(8)	4*e*	0.47612	0.28620	0.42940	0.0680
H(9)	4*e*	0.65858	0.27638	0.58227	0.0796
H(10)	4*e*	0.88087	0.41580	0.64899	0.0723
H(11)	4*e*	0.92244	0.57082	0.56325	0.0769
H(12)	4*e*	0.74042	0.58341	0.40955	0.0685

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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)	4*e*	0.60930(3)	0.39967(2)	0.22819(2)	0.0453(2)	0.0467(2)	0.0354(2)	0.0004(1)	0.0192(1)	–0.0003(1)
Cl(1)	4*e*	0.7570(1)	0.54057(7)	0.19479(5)	0.0759(4)	0.0667(4)	0.0783(4)	–0.0110(3)	0.0475(4)	0.0048(3)
Cl(2)	4*e*	0.72895(8)	0.21258(6)	0.28010(4)	0.0650(4)	0.0497(3)	0.0444(3)	0.0105(3)	0.0121(3)	–0.0028(2)
N(1)	4*e*	0.3583(2)	0.3803(2)	0.1309(1)	0.047(1)	0.046(1)	0.0350(9)	0.0029(8)	0.0170(8)	0.0007(7)
N(2)	4*e*	0.4824(2)	0.4483(2)	0.3073(1)	0.046(1)	0.0385(9)	0.0337(8)	–0.0003(8)	0.0175(8)	–0.0011(7)
C(1)	4*e*	0.2466(3)	0.3678(2)	0.1660(1)	0.048(1)	0.036(1)	0.040(1)	0.0016(9)	0.020(1)	–0.0008(8)
C(2)	4*e*	0.0729(3)	0.3465(2)	0.1124(2)	0.051(1)	0.059(1)	0.052(1)	–0.003(1)	0.023(1)	–0.002(1)
C(3)	4*e*	0.0128(3)	0.3378(3)	0.0197(2)	0.050(1)	0.065(2)	0.048(1)	–0.002(1)	0.009(1)	–0.001(1)
C(4)	4*e*	0.1268(3)	0.3535(3)	–0.0165(2)	0.060(2)	0.058(1)	0.036(1)	0.004(1)	0.012(1)	0.003(1)
C(5)	4*e*	0.2982(3)	0.3754(2)	0.0405(2)	0.056(1)	0.058(1)	0.036(1)	0.006(1)	0.019(1)	0.004(1)
C(6)	4*e*	0.3234(3)	0.3696(2)	0.2679(2)	0.052(1)	0.056(1)	0.040(1)	–0.009(1)	0.024(1)	–0.005(1)
C(7)	4*e*	0.5892(3)	0.4366(2)	0.4040(1)	0.046(1)	0.045(1)	0.034(1)	0.0051(9)	0.0213(9)	–0.0043(9)
C(8)	4*e*	0.5657(3)	0.3445(3)	0.4558(2)	0.055(1)	0.072(2)	0.041(1)	–0.010(1)	0.020(1)	0.005(1)
C(9)	4*e*	0.6757(4)	0.3386(3)	0.5474(2)	0.070(2)	0.086(2)	0.044(1)	–0.002(2)	0.027(1)	0.012(1)
C(10)	4*e*	0.8075(3)	0.4213(3)	0.5873(2)	0.056(2)	0.084(2)	0.036(1)	0.017(1)	0.016(1)	–0.004(1)
C(11)	4*e*	0.8322(4)	0.5132(3)	0.5362(2)	0.059(2)	0.068(2)	0.052(1)	–0.003(1)	0.013(1)	–0.015(1)
C(12)	4*e*	0.7229(3)	0.5209(2)	0.4441(2)	0.066(2)	0.051(1)	0.050(1)	–0.006(1)	0.022(1)	–0.003(1)

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