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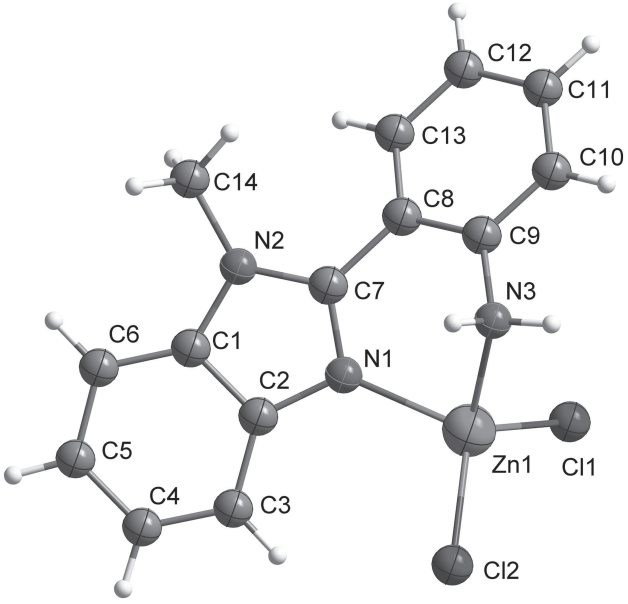
Crystal structure of dichlorido(2-(1-methyl-1*H*-benzo[*d*]imidazol-2-yl)aniline-κ²*N*,*N*′)zinc(II)



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Abstract

$C_{14}H_{13}Cl_2N_3Zn$, $M = 359.54$, orthorhombic, $Pna2_1$ (No. 33), $a = 10.1222(5)$ Å, $b = 18.6435(10)$ Å, $c = 7.6190(4)$ Å, $V = 1437.81(12)$ Å^3, $Z = 4$, $R_{gt}(F) = 0.0407$, $wR_{ref}(F^2) = 0.0780$, $T = 102$ K.

CCDC no.: 1451307

The crystal structure is shown in the figure, Tables 1–3 contain details of the measurement method and a list of the atoms including atomic coordinates and displacement parameters.

Source of material

A mixture of 2-(1-methyl-1*H*-benzo[*d*]imidazol-2-yl)aniline (0.560 g, 2.5 mmol) and zinc chloride (0.341 g, 2.5 mmol) was dissolved in acetonitrile (20 mL). The solution was cooled to room temperature and filtered. Needle shaped pale yellow single crystals were obtained from the filtrate by slow evaporation after 6 days (Yield: 0.35 g, 38.8%). IR (KBr pellet, cm^{-1}): 3297(*s*), 3236(*s*), 1616(*m*), 1573(*m*), 1483(*vs*), 1441(*m*), 1399(*s*), 1333(w), 1057(*s*), 771(*m*), 758(*m*), 750(*m*), 577(*m*). Elemental analysis: found – C, 46.53%; H, 3.86%; N, 11.61% (VarioEL); calcd. for $C_{14}H_{13}C_{l2}N_3Zn$ (359.54) – C, 46.76%; H, 3.64%; N, 11.68%.

Experimental details

All H atoms were placed in calculated positions.

Table 1: Data collection and handling.

Crystal:	Pale yellow, needle, size 0.04×0.04×0.15 mm
Wavelength:	Mo K_α radiation (0.7107 Å)
μ:	20.71 cm^{-1}
Diffractometer, scan mode:	Xcalibur, Eos, Gemini, ω scans
$2\theta_{max}$:	51.98°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	4579, 2419
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 2162
$N(param)_{refined}$:	182
Programs:	Crysalis^{PRO} [5], SHELX [6], Diamond [7]

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

Atom	Site	*x*	*y*	*z*	U_{iso}
H(10)	4*a*	0.5905	0.6345	−0.4759	0.023
H(12)	4*a*	0.8654	0.7901	−0.5276	0.021
H(14A)	4*a*	1.1806	0.7133	−0.1200	0.031
H(14B)	4*a*	1.1229	0.6631	−0.2721	0.031
H(14C)	4*a*	1.2270	0.6314	−0.1343	0.031
H(3A)	4*a*	0.7360	0.5316	−0.2089	0.023
H(3B)	4*a*	0.5997	0.5573	−0.2470	0.023
H(6)	4*a*	1.2663	0.6233	0.1851	0.022
H(3)	4*a*	0.8487	0.5549	0.4153	0.021
H(11)	4*a*	0.6741	0.7328	−0.6280	0.022
H(4)	4*a*	1.0410	0.5417	0.5778	0.024
H(13)	4*a*	0.9700	0.7493	−0.2749	0.022
H(5)	4*a*	1.2455	0.5752	0.4686	0.025

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Table 3: Fractional atomic coordinates and atomic displacement parameters (Å^2).

Atom	Site	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Zn(1)	4*a*	0.65154(5)	0.59258(2)	0.06945(9)	0.0153(3)	0.0201(3)	0.0183(3)	−0.0008(2)	0.0032(3)	0.0018(3)
Cl(1)	4*a*	0.5103(1)	0.68298(6)	0.0888(2)	0.0202(6)	0.0237(5)	0.0242(7)	0.0036(5)	0.0005(6)	0.0014(6)
Cl(2)	4*a*	0.6139(1)	0.49639(6)	0.2371(2)	0.0248(7)	0.0246(6)	0.0270(7)	−0.0050(5)	0.0059(7)	0.0066(6)
N(1)	4*a*	0.8474(3)	0.6151(2)	0.0755(8)	0.014(2)	0.017(2)	0.013(2)	−0.002(1)	0.000(2)	0.000(2)
C(1)	4*a*	1.0676(5)	0.6184(2)	0.1354(6)	0.017(3)	0.011(2)	0.012(3)	0.004(2)	−0.002(2)	0.001(2)
C(10)	4*a*	0.6689(4)	0.6572(2)	−0.4358(9)	0.016(2)	0.022(2)	0.020(2)	0.002(2)	−0.006(3)	−0.006(3)
C(12)	4*a*	0.8309(5)	0.7499(2)	−0.4660(6)	0.023(3)	0.016(2)	0.014(3)	0.000(2)	0.001(2)	0.002(2)
C(14)	4*a*	1.1526(5)	0.6645(3)	−0.1497(7)	0.017(3)	0.026(3)	0.018(3)	−0.005(2)	0.000(2)	−0.002(2)
C(7)	4*a*	0.9127(5)	0.6405(2)	−0.0637(7)	0.013(3)	0.012(2)	0.014(3)	−0.002(2)	−0.003(2)	−0.003(2)
C(8)	4*a*	0.8463(5)	0.6655(2)	−0.2256(6)	0.015(3)	0.020(3)	0.009(3)	0.003(2)	0.001(2)	−0.003(2)
N(2)	4*a*	1.0435(4)	0.6429(2)	−0.0338(5)	0.015(2)	0.015(2)	0.015(2)	−0.002(2)	0.002(2)	−0.001(2)
N(3)	4*a*	0.6792(4)	0.5698(2)	−0.1973(6)	0.015(2)	0.017(2)	0.025(3)	0.000(2)	−0.002(2)	0.000(2)
C(9)	4*a*	0.7326(5)	0.6310(2)	−0.2877(6)	0.018(3)	0.013(2)	0.013(3)	0.000(2)	0.006(2)	−0.005(2)
C(2)	4*a*	0.9428(5)	0.5996(2)	0.2020(7)	0.017(3)	0.013(2)	0.019(3)	−0.001(2)	−0.002(2)	−0.004(2)
C(6)	4*a*	1.1825(5)	0.6100(2)	0.2313(7)	0.018(3)	0.017(2)	0.020(3)	0.002(2)	0.001(2)	−0.004(2)
C(3)	4*a*	0.9317(5)	0.5693(2)	0.3691(6)	0.022(3)	0.014(2)	0.017(3)	0.001(2)	0.003(2)	0.003(2)
C(11)	4*a*	0.7178(5)	0.7159(3)	−0.5257(6)	0.020(3)	0.025(3)	0.010(3)	0.010(2)	0.000(2)	−0.006(2)
C(4)	4*a*	1.0459(5)	0.5615(2)	0.4632(7)	0.036(3)	0.011(2)	0.014(3)	0.005(2)	−0.001(3)	−0.003(2)
C(13)	4*a*	0.8934(5)	0.7252(2)	−0.3165(7)	0.012(3)	0.021(3)	0.022(3)	−0.001(2)	0.005(2)	−0.001(2)
C(5)	4*a*	1.1693(5)	0.5814(2)	0.3976(7)	0.023(3)	0.020(3)	0.018(3)	0.004(2)	−0.010(2)	−0.003(2)

Discussion

The bidentate ligand 2-(1-methyl-1*H*-benzo[*d*]imidazol-2-yl)aniline (L) with steric hindrance has been used to prepare a series of mononuclear transitional metal complex with halide anions as the active leaving group in our subsequent catalytic research. In this work, a mononuclear zinc complex $ZnLCl_2$ is reported. Zinc complexes bearing various ancillary ligands have been applied in the catalysis of the copolymerization of cyclohexene oxide and CO_2 [1–3].

The asymmetric unit of the title complex contains one independent neutral complex molecule, which consists of one center zinc ion, one bidentate ligand and two chlorido ligands. The ligand has two moieties: the benzimidazole and the aniline group. The benzimidazole and aniline planes are not coplanar and their dihedral angle is about 35° [4]. The bond lengths of C7—N1, C7—N2, C1—N2 and C2—N1 are in the range of 1.336(6) to 1.395(6) Å. The center Zn(II) ion adopts a distorted tetrahedral coordination polyhedron. The four-coordinated zinc(II) is coordinated by one imidazole nitrogen atom N1, one aniline nitrogen atom N3 and two chlorido ligands. The distances from the zinc(II) to its coordination atoms are all in the expected range. The six angles around the zinc(II) are in the range of 86.2(2) to 118.18(9)°, of which the smallest angle N1—Zn1—N3 is formed by the two nitrogen atoms from the bidentate ligand.

In the crystal structure, the adjacent molecules are linked each other through N—H$\cdots$Cl hydrogen bonds between the aniline N3 and the coordinate Cl2 anion from the neighbour molecule (N3—H3B$\cdots$Cl2($-x+1$, $-y+1$, $z-1/2$): 3.252(5) Å, 157.2°) to form an infinite zigzag chain along the *c* axis.

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