Crystal structure of chlorobis(1,10-phenanthroline)zinc(II) perchlorate monohydrate, $[\text{ZnCl}(\text{C}_{12}\text{H}_8\text{N}_2)_2]\text{ClO}_4 \cdot \text{H}_2\text{O}$

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The crystal structure of $[\text{Zn}(\text{phen})_2\text{Cl}](\text{ClO}_4) \cdot \text{H}_2\text{O}$ shows that the two molecules of the 1,10-phenanthroline ligand are coordinated as bidentate. The Zn atom has an unsymmetrical five-coordinated environment formed by four nitrogen atoms of 1,10-phenanthroline and one chloride anion. The coordination polyhedron around the Zn(II) ion is a distorted trigonal bipyramid $[\text{ZnN}_4\text{Cl}]$. The N3 and N2 atoms are axial with a N3-Zn1-N2 angle of $172.5(1)^\circ$ and the atoms N4, N1 and Cl1 are distorted equatorial with angles N4-Zn1-N1 of $119.2(1)^\circ$, N4-Zn1-Cl1 of $119.55(9)^\circ$, and N3-Zn1-Cl1 of $121.24(9)^\circ$.

Table 1. Data collection and handling.

Crystal:	light-rose prism, size $0.20 \times 0.20 \times 0.30$ mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ :	13.64 cm^{-1}
Diffraction, scan mode:	Bruker SMART 1000 CCD, ϕ/ω
$2\theta_{\text{max}}$:	55.12°
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$:	12592, 5193
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2\sigma(I_{\text{obs}})$, 3301
$N(\text{param})_{\text{refined}}$:	325
Programs:	SHELXL-97 [3], SHELXTL [4]

Abstract

$\text{C}_{24}\text{H}_{18}\text{Cl}_2\text{N}_4\text{O}_5\text{Zn}$, monoclinic, $P12_1/n1$ (no. 14), $a = 11.375(2)$ Å, $b = 15.383(2)$ Å, $c = 13.026(2)$ Å, $\beta = 94.718(2)^\circ$, $V = 2271.4$ Å³, $Z = 4$, $R_{\text{gt}}(F) = 0.048$, $wR_{\text{ref}}(F^2) = 0.103$, $T = 120$ K.

Source of material

A methanolic solution of 1,10-phenanthroline (0.400 g, 2 mmol) and sodium perchlorate (0.123 g, 1 mmol) and sodium trichloroacetate (0.230 g, 1 mmol) was added to a solution of the zinc(II) acetate (0.183 g, 1 mmol) in methanol. The resulting solution was concentrated on water bath and cooled to room temperature, when white crystalline compounds were separated. The crystals were filtered off, washed with cold acetone and dried over silica gel (yield 0.207 g, 50 %). Elemental analysis: found – C, 49.30 %; H, 3.20 %; N, 9.45; calc. for $\text{C}_{24}\text{H}_{18}\text{Cl}_2\text{N}_4\text{O}_5\text{Zn}$ – C, 49.69 %; H, 3.11 %; N, 9.67 %.

Discussion

Numerous zinc(II) complexes with nitrogen or oxygen donor ligands have been synthesized and studied [1]. Some of these complexes are considered as structural models for the active site in enzymes. A major reason for the importance of zinc in enzyme chemistry is its ability to adopt coordination numbers of 4, 5 or 6 in its complexes and to change its coordination geometry comparatively easily [2].

Table 2. Atomic coordinates and displacement parameters (in Å²).

Atom	Site	x	y	z	U_{iso}
H(1)	4e	0.3650	−0.0333	0.0825	0.037
H(2)	4e	0.4684	−0.1609	0.1063	0.041
H(3)	4e	0.5094	−0.2150	0.2703	0.038
H(8)	4e	0.3046	0.0061	0.6548	0.045
H(9)	4e	0.1966	0.1287	0.6127	0.040
H(10)	4e	0.1727	0.1739	0.4427	0.039
H(11)	4e	0.4893	−0.1995	0.4624	0.040
H(12)	4e	0.4214	−0.1247	0.5936	0.040
H(13)	4e	0.1439	0.0144	−0.0049	0.037
H(14)	4e	0.1500	0.0512	−0.1761	0.039
H(15)	4e	0.2375	0.1771	−0.2206	0.039
H(20)	4e	0.4570	0.4311	0.1610	0.035
H(21)	4e	0.4266	0.3874	0.3263	0.037
H(22)	4e	0.3386	0.2567	0.3529	0.034
H(23)	4e	0.3537	0.3134	−0.1621	0.038
H(24)	4e	0.4320	0.3988	−0.0320	0.037
H(5A)	4e	0.8333	0.1649	0.0517	0.072
H(5B)	4e	0.9726	0.1568	0.0364	0.072

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Table 3. Atomic coordinates and displacement parameters (in Å²).

Atom	Site	x	y	z	U ₁₁	U ₂₂	U ₃₃	U ₁₂	U ₁₃	U ₂₃
Zn(1)	4e	0.23204(4)	0.09983(3)	0.21794(3)	0.0363(2)	0.0261(2)	0.0162(2)	0.0002(2)	−0.0014(2)	0.0002(2)
Cl(1)	4e	0.03258(8)	0.08901(6)	0.22450(6)	0.0341(5)	0.0330(6)	0.0252(4)	0.0008(4)	−0.0006(4)	0.0019(4)
N(1)	4e	0.3394(3)	−0.0101(2)	0.2266(2)	0.032(2)	0.028(2)	0.018(2)	−0.004(1)	0.001(1)	0.001(1)
N(2)	4e	0.2618(3)	0.0815(2)	0.3837(2)	0.038(2)	0.032(2)	0.016(1)	−0.001(1)	−0.002(1)	−0.002(1)
C(1)	4e	0.3798(3)	−0.0548(3)	0.1490(3)	0.038(2)	0.034(2)	0.021(2)	0.000(2)	0.005(2)	−0.002(2)
C(2)	4e	0.4428(3)	−0.1315(3)	0.1627(3)	0.039(2)	0.035(2)	0.030(2)	0.003(2)	0.006(2)	−0.004(2)
C(3)	4e	0.4669(3)	−0.1637(3)	0.2601(3)	0.033(2)	0.030(2)	0.033(2)	0.004(2)	0.001(2)	0.004(2)
C(4)	4e	0.4274(3)	−0.1189(2)	0.3440(3)	0.030(2)	0.030(2)	0.025(2)	−0.006(2)	−0.001(2)	0.003(1)
C(5)	4e	0.3652(3)	−0.0419(2)	0.3240(3)	0.028(2)	0.030(2)	0.020(2)	−0.005(2)	−0.002(1)	0.003(1)
C(6)	4e	0.3221(3)	0.0071(2)	0.4075(3)	0.028(2)	0.027(2)	0.020(2)	−0.006(2)	−0.001(1)	−0.001(1)
C(7)	4e	0.3420(3)	−0.0246(2)	0.5092(3)	0.034(2)	0.031(2)	0.020(2)	−0.009(2)	−0.004(2)	0.003(1)
C(8)	4e	0.2934(4)	0.0239(3)	0.5865(3)	0.051(3)	0.045(3)	0.016(2)	−0.012(2)	−0.003(2)	0.001(2)
C(9)	4e	0.2302(3)	0.0970(3)	0.5618(3)	0.042(2)	0.040(2)	0.018(2)	−0.005(2)	0.003(2)	−0.006(2)
C(10)	4e	0.2162(3)	0.1239(3)	0.4588(3)	0.038(2)	0.037(2)	0.022(2)	−0.000(2)	0.003(2)	−0.004(2)
C(11)	4e	0.4473(3)	−0.1483(3)	0.4486(3)	0.038(2)	0.030(2)	0.031(2)	−0.002(2)	−0.006(2)	0.008(2)
C(12)	4e	0.4070(3)	−0.1038(3)	0.5268(3)	0.035(2)	0.038(2)	0.024(2)	−0.005(2)	−0.006(2)	0.011(2)
N(1')	4e	0.2272(3)	0.1162(2)	0.0540(2)	0.035(2)	0.027(2)	0.018(1)	0.005(1)	−0.002(1)	−0.004(1)
N(2')	4e	0.3087(3)	0.2211(2)	0.2087(2)	0.033(2)	0.030(2)	0.015(1)	0.001(1)	−0.002(1)	−0.001(1)
C(1')	4e	0.1803(3)	0.0661(3)	−0.0216(3)	0.037(2)	0.030(2)	0.023(2)	−0.002(2)	−0.003(2)	−0.006(2)
C(2')	4e	0.1836(3)	0.0880(3)	−0.1252(3)	0.039(2)	0.036(3)	0.021(2)	0.007(2)	−0.003(2)	−0.008(2)
C(3')	4e	0.2353(3)	0.1624(3)	−0.1515(3)	0.044(2)	0.039(3)	0.016(2)	0.007(2)	−0.001(2)	−0.004(2)
C(4')	4e	0.2867(3)	0.2185(2)	−0.0738(2)	0.033(2)	0.031(2)	0.014(2)	0.008(2)	0.003(1)	−0.002(1)
C(5')	4e	0.2782(3)	0.1917(2)	0.0282(2)	0.027(2)	0.025(2)	0.019(2)	0.006(2)	0.000(1)	−0.003(1)
C(6')	4e	0.3242(3)	0.2480(2)	0.1113(2)	0.030(2)	0.023(2)	0.018(2)	0.003(2)	0.001(1)	0.001(1)
C(7')	4e	0.3803(3)	0.3250(2)	0.0883(3)	0.030(2)	0.029(2)	0.020(2)	0.005(2)	0.001(2)	−0.002(1)
C(8')	4e	0.4196(3)	0.3785(2)	0.1723(3)	0.032(2)	0.028(2)	0.029(2)	−0.000(2)	0.001(2)	−0.003(1)
C(9')	4e	0.4022(3)	0.3523(3)	0.2703(3)	0.036(2)	0.033(2)	0.023(2)	0.002(2)	−0.003(2)	−0.008(2)
C(10')	4e	0.3482(3)	0.2739(2)	0.2856(3)	0.037(2)	0.031(2)	0.017(2)	0.005(2)	−0.004(2)	−0.004(1)
C(11')	4e	0.3462(3)	0.2970(3)	−0.0942(3)	0.041(2)	0.035(2)	0.020(2)	0.008(2)	0.007(2)	0.004(2)
C(12')	4e	0.3923(3)	0.3481(3)	−0.0169(3)	0.035(2)	0.031(2)	0.028(2)	0.001(2)	0.005(2)	0.005(2)
Cl(2)	4e	0.61854(9)	0.14230(6)	0.14604(7)	0.0428(6)	0.0329(6)	0.0239(4)	0.0036(4)	−0.0013(4)	−0.0003(4)
O(1)	4e	0.7080(3)	0.0846(2)	0.1832(2)	0.083(3)	0.071(3)	0.050(2)	0.039(2)	0.015(2)	0.021(2)
O(2)	4e	0.6677(3)	0.2056(2)	0.0831(3)	0.075(3)	0.051(2)	0.090(3)	−0.008(2)	0.011(2)	0.034(2)
O(3)	4e	0.5706(3)	0.1843(3)	0.2311(2)	0.083(3)	0.103(3)	0.035(2)	0.047(2)	−0.006(2)	−0.023(2)
O(4)	4e	0.5260(3)	0.1003(2)	0.0856(3)	0.071(2)	0.084(3)	0.049(2)	−0.032(2)	−0.004(2)	−0.012(2)
O(5)	4e	0.8961(3)	0.1580(2)	0.0162(2)	0.059(2)	0.061(2)	0.024(2)	−0.009(2)	0.003(2)	0.009(1)

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