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The crystal structure of dichlorido-bis(3-methyl-3-imidazolium-1-ylpropionato- $\kappa^2 O, O'$)-zinc(II), $C_{14}H_{20}Cl_2N_4O_4Zn$

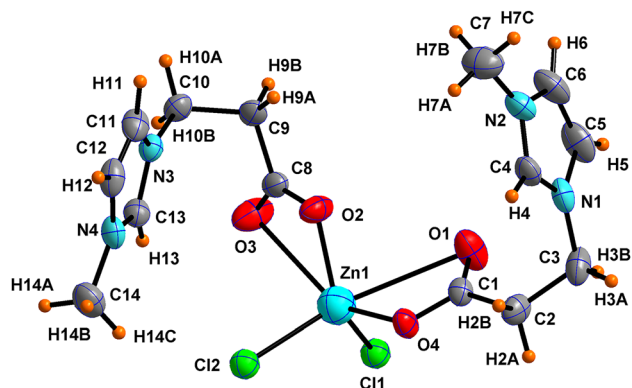


Table 1: Data collection and handling.

Crystal:	Colorless block
Size:	0.24 × 0.20 × 0.16 mm
Wavelength:	Mo K α radiation (0.71073 Å)
μ :	1.62 mm ⁻¹
Diffractometer, scan mode:	Bruker APEX-II, φ and ω
$\theta_{\max}$, completeness:	25.0°, >99 %
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	9134, 3297, 0.043
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 2875
$N(\text{param})_{\text{refined}}$:	228
Programs:	BRUKER [1], OLEX2 [2], SHELX [3, 4]

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Abstract

$C_{14}H_{20}Cl_2N_4O_4Zn$, monoclinic, $P2_1/n$ (no. 14), $a = 8.562(2)$ Å, $b = 27.953(8)$ Å, $c = 8.804(2)$ Å, $\beta = 117.092(4)^\circ$, $V = 1875.9(9)$ Å³, $Z = 4$, $R_{\text{gt}}(F) = 0.0441$, $wR_{\text{ref}}(F^2) = 0.1031$, $T = 296$ K.

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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.

1 Source of materials

1-Carboxyethyl-3-methyl imidazolium chloride was prepared according to the reported methods [5]. In a 25 mL round-bottom flask, 1-carboxyethyl-3-methyl imidazolium chloride (5 mmol) and zinc powder (10 mmol) were added to distilled water (10 mL). The mixture was stirred at room temperature for 3 days, followed by careful filtration and evaporation until only a small amount of water remained. Subsequently, the mixture

was allowed to stand at room temperature for 2 weeks, and the colorless crystals were obtained. FT-IR spectrum was measured on a Perkin-Elmer Spectrum 400F spectrophotometer using the KBr pellet technique. Powder X-ray diffraction (PXRD) patterns were collected on a BRUKER D8 Advance diffractometer at room temperature. IR(KBr): 3141w, 3113m, 3094m, 3077m, 2921m, 2853w, 1618vs, 1573s, 1450m, 1406s, 1357w, 1333w, 1300s, 1282m, 1220w, 1175s, 1151m, 1052w, 986m, 855w, 829m, 763m, 646m, 622m, 585w cm⁻¹. PXRD: $\theta = 11.6, 12.0, 12.3, 12.7, 12.9, 13.2, 13.5, 17.0, 17.2, 17.5, 19.4, 20.2, 20.6, 22.1, 22.5, 23.3, 25.2, 28.0, 21.2, 34.3^\circ$.

2 Experimental details

All hydrogen atoms in ring and methylene groups were refined using riding coordinates with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$, whereas the hydrogen atoms of methyl group were placed in the idealized location with $U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{C})$.

3 Comment

The functionalization of traditional ionic liquids can enrich the number of ionic liquids, extend their applications, and attract more and more attention as an effective approach for a special task. Carboxyl-functionalized ionic liquids not only exhibit designability and diversity of ionic liquid but also possess coordination ability derived from carboxyl groups to construct the various coordinating polymers. These unique properties make them crucial in absorption and separation, catalysis, proton conduction, and other significant fields [6–8].

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Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²).

Atom	x	y	z	<i>U</i> _{iso} */ <i>U</i> _{eq}
C1	0.5107 (5)	0.26995 (13)	−0.2318 (4)	0.0298 (8)
C2	0.4392 (5)	0.21997 (13)	−0.2369 (5)	0.0366 (9)
H2A	0.438842	0.213565	−0.128725	0.044*
H2B	0.318579	0.218797	−0.325292	0.044*
C3	0.5425 (6)	0.18110 (14)	−0.2704 (5)	0.0449 (11)
H3A	0.488373	0.150341	−0.274304	0.054*
H3B	0.539399	0.186527	−0.380603	0.054*
C4	0.7771 (5)	0.18052 (13)	0.0278 (5)	0.0357 (9)
H4	0.703361	0.179665	0.079251	0.043*
C5	0.8735 (7)	0.18143 (16)	−0.1627 (6)	0.0512 (12)
H5	0.876370	0.181509	−0.267056	0.061*
C6	1.0126 (6)	0.18264 (16)	−0.0076 (6)	0.0505 (12)
H6	1.129897	0.183485	0.015515	0.061*
C7	1.0530 (6)	0.1845 (2)	0.2951 (6)	0.0598 (13)
H7A	0.976340	0.190665	0.345401	0.090*
H7B	1.138096	0.209723	0.325227	0.090*
H7C	1.111996	0.154558	0.336428	0.090*
C8	0.8517 (5)	0.40946 (14)	−0.0353 (5)	0.0308 (8)
C9	1.0370 (5)	0.41175 (14)	0.1114 (5)	0.0337 (9)
H9A	1.060247	0.382951	0.179753	0.040*
H9B	1.120778	0.413083	0.065600	0.040*
C10	1.0612 (5)	0.45483 (14)	0.2227 (5)	0.0337 (9)
H10A	1.181980	0.455970	0.310792	0.040*
H10B	1.038953	0.483560	0.154135	0.040*
C11	0.9852 (6)	0.43862 (13)	0.4654 (5)	0.0387 (10)
H11	1.092539	0.426298	0.544322	0.046*
C12	0.8418 (6)	0.44443 (13)	0.4891 (5)	0.0393 (10)
H12	0.830878	0.436737	0.586585	0.047*
C13	0.7795 (5)	0.46922 (13)	0.2328 (5)	0.0330 (9)
H13	0.718960	0.481601	0.123151	0.040*
C14	0.5386 (6)	0.47901 (17)	0.3101 (6)	0.0497 (11)
H14A	0.536273	0.513136	0.321217	0.074*
H14B	0.509409	0.464000	0.391948	0.074*
H14C	0.454942	0.469739	0.197082	0.074*
Cl1	0.41683 (14)	0.38394 (4)	−0.48333 (12)	0.0405 (3)
Cl2	0.36959 (14)	0.41645 (3)	−0.09949 (13)	0.0419 (3)
N1	0.7255 (4)	0.18006 (11)	−0.1380 (4)	0.0359 (8)
N2	0.9502 (4)	0.18240 (12)	0.1093 (5)	0.0404 (8)
N3	0.9444 (4)	0.45420 (10)	0.3034 (4)	0.0288 (7)
N4	0.7146 (4)	0.46399 (11)	0.3409 (4)	0.0344 (7)
O1	0.6310 (4)	0.27606 (10)	−0.2677 (4)	0.0523 (8)
O2	0.7670 (3)	0.37135 (9)	−0.0436 (4)	0.0436 (7)
O3	0.7941 (4)	0.44341 (11)	−0.1327 (4)	0.0566 (9)
O4	0.4356 (3)	0.30309 (9)	−0.1886 (3)	0.0347 (6)
Zn1	0.51684 (5)	0.36879 (2)	−0.20274 (5)	0.02850 (14)

For example, Huang et al. [8] reported a novel clean method for separating zinc and cobalt using a carboxyl-functionalized ionic liquid and a higher separation effect on zinc and cobalt. Ma and co-workers [7] have synthesized a series of carboxyl-functionalized imidazolium salts, and further investigation

demonstrated that their hydrosilylation reactions exhibited higher styrene conversion and β -adduct selectivity. In this work, we used the prepared carboxyethyl-3-methylimidazolium chloride to react with zinc, and obtained a mononuclear Zn (II) coordination compound.

The title compound crystallizes in the monoclinic system, and its asymmetric unit, as shown in figure, contains one Zn²⁺ cation, two Cl[−] anions, and two 3-methyl-3-imidazolium-1-ylpropionate zwitter ions, similar to those reported in a Cd(II) complex [9]. The central Zn²⁺ cation is four-coordinated in a slightly distorted tetrahedral geometry with two Cl[−] anions and two oxygen atoms of two carboxylate groups. In this tetrahedron, the bond distance of Cl1–Zn1 and Cl2–Zn1 is 2.2556(12) Å and 2.2879(11) Å, respectively. Meanwhile, the bond lengths of Zn1–O2 (1.955(3) Å) and Zn1–O4 (1.988(2) Å) fall in the desired ranges. Interestingly, the alkyl chain of carboxyethyl-3-methylimidazolium adopts a highly bent conformation. The torsion angles of O1–C1–C2–C3 and C1–C2–C3–N1 are 6.1 (5)° and 60.2(5)°, respectively. In contrast, the carbon atoms of methyl and ethyl groups, closest to the ring, and imidazole ring are approximately coplanar, because the torsion angles of C7–N2–C6–C5 and C3–N1–C5–C6 are −178.5(4) Å and 176.4(4) Å. This conformation of cation is the same as those of the anhydrous zwitterionic compound [10] and salt with acid anion [5] in the crystal structure, but different from that in another Zn(II) complex [11]. In the crystal packing, neighboring complexes are connected by numerous weak C–H...O and C–H...Cl interactions. In addition, the C...C short contact with a distance of 3.399 Å between two adjacent imidazolium rings is present, forming a one-dimensional chain along the *a*-axis of the crystal.

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Conflict of interest statement: The authors declare no conflicts of interest regarding this article.

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