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Crystal structure of bis(2-propyl-1*H*-benzo[*d*]imidazol-3-ium) tetrachloridozincate(II), $C_{10}H_{13}Cl_4N_2Zn$

Table 1: Data collection and handling.

Crystal:	Colorless block
Size:	0.25 × 0.15 × 0.10 mm
Wavelength:	Mo K α radiation (0.71073 Å)
μ :	1.49 mm ⁻¹
Diffractometer, scan mode:	Bruker APEX-II, φ and ω
$\theta_{\max}$, completeness:	25.1°, 99%
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	30175, 4219, 0.118
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 2983
$N(\text{param})_{\text{refined}}$:	264
Programs:	Bruker [1], SHELX [2], Olex2 [3, 4]

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

Atom	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
C1	0.8917(3)	0.3322(4)	0.1158(2)	0.0230(10)
C2	0.9052(4)	0.2252(4)	0.1718(2)	0.0309(11)
H2a	0.9775(4)	0.2182(4)	0.1875(2)	0.0371(13)*
H2b	0.8699(4)	0.2512(4)	0.2164(2)	0.0371(13)*
C3	0.8679(4)	0.0888(5)	0.1464(3)	0.0387(13)
H3a	0.7959(4)	0.0944(5)	0.1297(3)	0.0465(15)*
H3b	0.9049(4)	0.0594(5)	0.1033(3)	0.0465(15)*
C4	0.8823(4)	−0.0132(5)	0.2092(3)	0.0468(14)
H4a	0.9540(5)	−0.024(3)	0.2228(13)	0.070(2)*
H4b	0.849(2)	0.0181(17)	0.2528(7)	0.070(2)*
H4c	0.853(2)	−0.0987(11)	0.1923(7)	0.070(2)*
C5	0.8778(3)	0.5350(4)	0.0644(2)	0.0215(9)
C6	0.8783(3)	0.4418(4)	0.0067(2)	0.0222(10)
C7	0.8714(3)	0.4792(5)	−0.0682(2)	0.0249(10)
H7	0.8705(3)	0.4154(5)	−0.1075(2)	0.0298(12)*
C8	0.8658(3)	0.6151(5)	−0.0824(3)	0.0319(11)
H8	0.8617(3)	0.6456(5)	−0.1327(3)	0.0383(14)*
C9	0.8660(3)	0.7088(5)	−0.0241(3)	0.0333(12)
H9	0.8613(3)	0.8009(5)	−0.0362(3)	0.0399(14)*
C10	0.8727(3)	0.6717(4)	0.0498(3)	0.0301(11)
H10	0.8738(3)	0.7356(4)	0.0890(3)	0.0361(13)*
C11	0.5941(4)	0.9159(6)	0.1348(3)	0.0475(15)
H11a	0.6603(9)	0.876(3)	0.1305(8)	0.071(2)*
H11b	0.5426(18)	0.846(2)	0.1297(8)	0.071(2)*
H11c	0.582(3)	0.9826(8)	0.0954(3)	0.071(2)*
C12	0.5908(4)	0.9823(5)	0.2108(2)	0.0372(12)
H12a	0.6435(4)	1.0516(5)	0.2163(2)	0.0446(15)*
H12b	0.5251(4)	1.0266(5)	0.2141(2)	0.0446(15)*

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Abstract

$C_{10}H_{13}Cl_4N_2Zn$, monoclinic, $P2_1/c$ (no. 14), $a = 13.370(2)$ Å, $b = 10.0304(14)$ Å, $c = 17.913(2)$ Å, $\beta = 93.950(4)^\circ$, $V = 2396.5(6)$ Å³, $Z = 4$, $R_{\text{gt}}(F) = 0.0461$, $wR_{\text{ref}}(F^2) = 0.1038$, $T = 150(2)$ 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.

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Table 2 (continued)

Atom	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{iso} [*] / <i>U</i> _{eq}
C13	0.6064(4)	0.8826(5)	0.2735(3)	0.0340(12)
H13a	0.5506(4)	0.8176(5)	0.2693(3)	0.0408(14)*
H13b	0.6691(4)	0.8330(5)	0.2666(3)	0.0408(14)*
C14	0.6125(3)	0.9395(4)	0.3505(2)	0.0242(10)
C15	0.6220(3)	1.0832(4)	0.4460(2)	0.0216(10)
C16	0.6221(3)	0.9538(4)	0.4742(2)	0.0240(10)
C17	0.6276(3)	0.9296(5)	0.5509(3)	0.0307(11)
H17	0.6279(3)	0.8415(5)	0.5703(3)	0.0369(13)*
C18	0.6325(3)	1.0387(5)	0.5972(2)	0.0306(11)
H18	0.6363(3)	1.0260(5)	0.6499(2)	0.0368(13)*
C19	0.6319(3)	1.1693(5)	0.5680(2)	0.0295(11)
H19	0.6347(3)	1.2425(5)	0.6018(2)	0.0354(13)*
C20	0.6273(3)	1.1948(4)	0.4917(2)	0.0253(10)
H20	0.6278(3)	1.2828(4)	0.4721(2)	0.0303(12)*
Cl1	0.68072(11)	0.31483(12)	0.27659(6)	0.0407(3)
Cl2	0.89870(10)	0.39616(14)	0.41347(7)	0.0476(4)
Cl3	0.64906(10)	0.55929(11)	0.42570(7)	0.0380(3)
Cl4	0.81548(9)	0.63129(10)	0.26699(6)	0.0267(3)
N1	0.8875(3)	0.3181(3)	0.04121(18)	0.0221(8)
H1	0.8901(3)	0.2414(3)	0.01755(18)	0.0265(10)*
N2	0.8858(3)	0.4627(3)	0.13044(18)	0.0248(8)
H2	0.8867(3)	0.4976(3)	0.17556(18)	0.0298(10)*
N3	0.6160(3)	1.0688(3)	0.36827(19)	0.0236(8)
H3	0.6147(3)	1.1349(3)	0.33592(19)	0.0283(10)*
N4	0.6155(3)	0.8684(4)	0.4131(2)	0.0283(9)
H4	0.6136(3)	0.7808(4)	0.4155(2)	0.0339(11)*
Zn1	0.76085(4)	0.47621(5)	0.34865(3)	0.02339(15)

Source of material

The starting materials, 2-propyl-1*H*-benzimidazole and zinc chloride, are commercial and used without any purification. The title compound was synthesized as follows: 0.320 g 2-propyl-1*H*-benzo[*d*]imidazole (2 mmol), was added to 20 mL 6 M hydro chloric acid at room temperature, then 0.136 g zinc chloride was added. The mixture was stirred until the zinc chloride completely dissolved. The solution was filtered and let evaporate. Three days later, colorless crystals were obtained, washed with 1 mL cold 6 M hydrochloric acid and air-dried, yield 62.3% (based on 2-propyl-1*H*-benzimidazole).

Experimental details

All H-atoms from C, N and O atoms were positioned with idealized geometry and refined isotropically ($U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$, $1.2U_{\text{eq}}(\text{N})$), for all H atoms) using a riding model.

Comment

Known as organic bases, benzimidazole and their derivatives are readily to be protonated. There are some records of 1- or 2-substituted benzimidazoles used as cations to counter

the negative charges of tetrachloridozincate anions [5–9]. As shown in the figure, the asymmetric unit consists of two 2-propyl-1*H*-benzimidazol-3-ium cations and one tetrachloridozincate anion. There is only one kind of hydrogen bonds: N—H···Cl, which link the cations and anions to generate a two-dimensional supramolecular structure. All bond lengths and angles of the title compound are comparable with its analogues [5–9]. In detail, the bonding parameter of the title cations are in perfect accord with literature known 1*H*-benzo[*d*]imidazol-3-ium salts [10, 11].

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References

1. Bruker. SAINT v8.37A. Bruker AXS Inc, Madison, WI, USA (2015).
2. Sheldrick, G. M.: A short history of SHELX. *Acta Crystallogr. A* **64** (2008) 112–122.
3. Bourhis, L. J.; Dolomanov, O. V.; Gildea, R. J.; Howard, J. A. K.; Puschmann, H.: The anatomy of a comprehensive constrained, restrained refinement program for the modern computing environment – Olex2 dissected. *Acta Crystallogr. A* **71** (2015) 59–75.
4. Dolomanov, O. V.; Bourhis, L. J.; Gildea, R. J.; Howard, J. A. K.; Puschmann, H.: OLEX2: a complete structure solution, refinement and analysis program. *J. Appl. Crystallogr.* **42** (2009) 339–341.
5. Tapia-Benavides, A. R.; Tlahuextl, M.; Tlahuext, H.; Galan-Vidal, C.: Synthesis of Zn compounds derived from 1*H*-benzimidazol-2-ylmethanamine. *Arkivoc* **9** (2008) 172–185.
6. Avila-Montiel, C.; Tapia-Benavides, A. R.; Falcon-Leon, M.; Ariza-Castolo, A.; Tlahuext, H.; Tlahuextl, M.: Synthesis and structural studies of amino amide salts derived from 2-(aminomethyl)benzimidazole and α -amino acids. *J. Mol. Struct.* **1100** (2015) 338–347.
7. Cai, C.-X.; Xu, L.; Tian, Y.-Q.; You, X.-Z.: The complex salt benzoimidazolium tetrachlorozincate. *Acta Crystallogr. E* **58** (2002) m399–m401.
8. Pan, J.; Wei, Q.; Ju, J.; Liu, B.; Jin, S.; Lin, Z.; Wang, D.: Syntheses and structural characterization of six ionic salts based on bis(benzimidazole) bis(imidazole) and perchlorometallates of Zn and Cu. *J. Coord. Chem.* **67** (2014) 3578–3597.
9. Lu, H.-J.; Fan, Y.-T.; Gao, J.; Wang, Q.: Self-assembly of 1,4-bis(1-benzimidazolylmethyl)benzene with Cd(II) and Zn(II) cations: synthesis and crystal structures of a 2-D coordination

- polymer and a 3-D supramolecular complex. *Synth. React. Inorg. Met.-Org. Nano-Met. Chem.* **35** (2005) 305–309.
10. Cameron, T. S.; Clyburne, J. A. C.; Dubey, P. K.; Grossert, J. S.; Ramaiah, K.; Ramanatham, J.; Sereda, S. V.: Compounds of chromium(VI): the pyridine – chromic anhydride complex, benzimidazolinium dichromate, and three 2-alkyl-1*H*-benzimidazolinium dichromates. *Can. J. Chem.* **81** (2003) 612–619.
11. Ojha, B.; Das, G.: Molecular recognition of 2-alkylbenzimidazole: photophysical and structural studies. *J. Photochem. Photobiol. A* **211** (2010) 176–184.