Structural Characterization of a Base-Stabilized [Zn₂]²⁺ Dication**

Stephan Schulz,* Daniella Schuchmann, Ingo Krossing,* Daniel Himmel, Dieter Bläser und Roland Boese

Dedicated to Prof. Dr. M. Jansen on the occasion of his 65th birthday

The landmark discovery of decamethyldizincocene Cp*₂Zn₂ **1** by Carmona *et al.* in 2004^[1] has led to increasing research activities on the synthesis of low-valent metal complexes of group 2 and 12 metals in recent years and several complexes containing Mg-Mg^[2], Cd-Cd^[3,4], Hg-Hg^[4] and Zn-Zn^[5-11] bonds, which are typically kinetically stabilized by use of sterically demanding or chelating organic substituents, have been synthesized. However, it should be noted that the first molecular complex with a Zn-Zn bond, Zn₂H₂, was trapped in an Ar matrix at 12 K and characterized by vibrational spectroscopy and computational calculations.^[12] With respect to the rather large number of organometallic complexes containing a direct Zn-Zn bond it is somehow surprising, that only a single study proves the existence of a [Zn₂]²⁺ dication, which was obtained in a melt of Zn in ZnCl₂ and characterized by Raman spectroscopy.^[13] This is in remarkable contrast to the well known [Hg₂]²⁺ and [Cd₂]²⁺ dications and has been subject to several computational investigations.^[14] In addition, a very few reports demonstrated the presence of [Zn]⁺ cations in microporous materials.^[15]

Recently, we became interested not only in the synthesis but also in the reactivity of low-valent organozinc complexes. Our initial studies focused on reactions of Cp*₂Zn₂ **1** due to the promising steric and electronic flexibility of the Cp* substituent.^[16] The reaction with 4-dimethylamino pyridine (dmap), which has been previously shown in our group to be a valuable Lewis base for the synthesis of base-stabilized monomeric group 13/15 compounds,^[17] resulted in the formation of Cp*₂Zn-Zn(dmap)₂Cp* **2**,^[18] in which the dmap molecules bind in a geminal coordination mode to only a single Zn atom. Disproportionation reaction into elemental zinc and the corresponding Zn(II) compound, as was previously observed in reactions of **1** with Lewis bases, did not occur.^[1,5] At the same time, low valent organomagnesium(I)

complexes were found to react with Lewis bases with subsequent formation of the *vicinal* coordinated adducts.^[19] Moreover, we reported on the reaction of **1** with [(2,4,6-Me₃C₆H₂)N(Me)C₂CH]H (MesnacnacH), which proceeded with protonation of the Cp* substituent and subsequent formation of the new Zn-Zn bonded complex Mesnacnac₂Zn₂ **3**.^[20]

Scheme 1. Reactions of Cp*₂Zn₂ **1**.

In order to elucidate, if this specific reaction might give a general access to low-valent organozinc complexes, we began to investigate the reaction of both **1** and **2** with protonating reagents. Herein, we report on the reaction of **2** with [H(OEt₂)₂][Al{OC(CF₃)₃}₄], which proceeds at relatively low temperature with elimination of Cp*H and subsequent formation of [Zn₂(dmap)₆][Al{OC(CF₃)₃}₄]₂ **4** containing the base-stabilized [Zn₂]²⁺ dication.

Scheme 2. Synthesis of [Zn₂(dmap)₆][Al{OC(CF₃)₃}₄]₂ **4**

A solution of **2** in CH₂Cl₂ reacts with two equivalents of [H(OEt₂)₂][Al{OC(CF₃)₃}₄] at -30 °C with formation of **4**, which was isolated after crystallization at -30 °C as a colorless crystalline solid. NMR spectra (¹H, ¹³C, ¹⁹F) of **4** show resonances only due to the dmap group and the aluminate anion, whereas no indication for the presence of a Cp* group was found.^[21] Single crystals of **4** were obtained from a solution in CH₂Cl₂ upon storage at -30 °C.^[22] **4** consists of a base stabilized [Zn₂(dmap)₆]²⁺ dication and two [Al{OC(CF₃)₃}₄]⁻ anions. The Zn atoms adopt tetrahedral coordination spheres with the Zn-N bond distances (2.136(5) Å) slightly elongated compared to those in **2** (2.115(2), 2.125(2) Å). The central Zn-Zn bond distance of 2.419(1) Å atom is almost identical to that reported for **2** (2.418(1) Å), whereas Zn₂R₂ complexes typically show shorter Zn-Zn bond distances, ranging from 2.29 to 2.35 Å. Only **3** (R = Mesnacnac, 2.3813(8) Å) and the doubly-reduced diimine derivative (R = [(2,6-*i*-Pr₂C₆H₃)N(Me)C]₂, 2.3994(6) Å)^[8] show comparable Zn-Zn bond lengths. The

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$[\text{Al}\{\text{OC}(\text{CF}_3)_3\}_4]^-$ anion is disordered.^[23] Theoretical calculations of **4** at BP86/def-SV(P) niveau revealed a gas phase minimum structure **4'** with S_6 symmetry.^[24] An alternative structure with the Zn atoms connected by two bridging H-atoms was unstable and split into separated $(\text{dmap})_3\text{ZnH}^+$ cations during the geometry optimization. The calculated Zn-Zn (2.399 Å) and Zn-N bond lengths (2.161 Å) of **4'** agree very well with the experimental values observed for **4** and the recently calculated base-free $[\text{Zn}_2]^{2+}$ dication (2.46 Å),^[25] whereas the calculated value of **1** (2.331 Å) is shorter.^[26]

Figure 1. Molecular structure of the $[\text{Zn}_2(\text{dmap})_6]^{2+}$ dication of **4**. H atoms and the $[\text{Al}\{\text{OC}(\text{CF}_3)_3\}_4]^-$ anions are omitted for clarity. Selected bond lengths [Å] and angles [°]: Zn1-Zn1a 2.419(2), Zn1-N1 2.136(4); N1-Zn1-N2 93.50(16), N1-Zn1-Zn1' 122.75(11).

The existence of the $[\text{Zn}_2]^{2+}$ dication in Zn/ZnCl₂ glasses was exclusively based on Raman spectra,^[13] showing a strong absorption band at $175 \pm 5 \text{ cm}^{-1}$. In contrast, no single mode but four distinctive Raman and three IR active modes were identified for Cp^*_2Zn_2 **1**.^[27]

Figure 2. Raman spectrum of **4** between 100 and 2000 cm^{-1} (50 mW, 8 cm^{-1})

The IR and Raman spectra of **4** are somewhat complex due to the presence of the Lewis base dmap and the anion $[\text{Al}\{\text{OC}(\text{CF}_3)_3\}_4]^-$. However, the anion modes were identified by

comparison with spectra of known complexes^[28] and the dmap modes by comparison with pure dmap and dmap coordinated zinc complexes.^[29] In addition, the IR and Raman spectra of **4** were simulated using DFT methods.

The IR spectrum of **4** shows characteristic strong anion vibrations at 1353, 1260, 1240, 968, 725 and 442 cm^{-1} , which correspond very well with experimental and calculated data.^[28] Vibrations at 2962, 1614, 1536, 1065, 1006 and 811 cm^{-1} agree with calculated bands of the dmap-stabilized dication and can be addressed to vibrations of the dmap group as can be seen when comparing the IR spectra of **4** with those of dmap, $(\text{dmap})_2\text{ZnCl}_2$, and $(\text{dmap})_2\text{ZnMe}_2$, respectively.^[29] Theoretical calculations of **4'** with BP86/def-SV(P) predict three vibrations with larger Zn-Zn stretch character, coupled with dmap torsions, at 249, 279, and 305 cm^{-1} , but due to their a_g symmetry they should not be visible in the IR spectrum. The calculated force constant of the Zn-Zn bond of 1.09 $\text{mdyne}/\text{\AA}$ is in between that one reported for $[\text{Zn}_2]^{2+}$ in Zn/ZnCl₂^[13] (0.6 $\text{mdyne}/\text{\AA}$) and Cp^*_2Zn_2 **1** (1.42 $\text{mdyne}/\text{\AA}$).^[27]

The experimental Raman spectrum of **4** shows characteristic anion bands at 1290, 798, 759, 539, and 323 cm^{-1} and typical dmap absorptions. In addition, a vibration observed at 174 cm^{-1} corresponds very well with the calculated a_g -vibration of **4'** (171 cm^{-1}), which exhibits some Zn-Zn character. A strong band at 175 cm^{-1} was reported for the Zn/ZnCl₂ glasses,^[12] in which the $[\text{Zn}_2]^{2+}$ dication most likely exists as Zn_2Cl_2 unit. Theoretical calculations (BP86/SVP) predict an a_{1g} -vibration for Zn_2Cl_2 at 194 cm^{-1} .

4 is the first structurally characterized complex containing the $[\text{Zn}_2]^{2+}$ dication. Coordination of the strong Lewis base dmap may have a stabilizing effect for the $[\text{Zn}_2]^{2+}$ unit as is suggested by very recent studies on the stability of base-stabilized, dimeric Mg_2Cl_2 .^[30] DFT calculations were performed in order to evaluate the stability of the base-stabilized dication. Coulomb explosion of the $[\text{Zn}_2(\text{dmap})_6]^{2+}$ dication with subsequent formation of two equivalents of $[\text{Zn}(\text{dmap})_3]^+$ was found to be strongly endergonic at 298.15 K both in the gas phase ($\Delta H^0 = 146 \text{ kJ/mol}$; $\Delta G^0 = 89 \text{ kJ/mol}$) as well as in solution ($\Delta G^0 = 183 \text{ kJ/mol}$), since the Gibbs solvation energies of $[\text{Zn}(\text{dmap})_3]^+$ (-113 kJ/mol) and $[\text{Zn}_2(\text{dmap})_6]^{2+}$ (-321 kJ/mol) are strongly exergonic. According to these calculations, the base-stabilized dication is stable in solution. According to NBO analysis, the Zn atoms in $[\text{Zn}_2(\text{dmap})_6]^{2+}$ bear a positive charge of 0.61 (Mulliken: 0.41), which is practically equal to the base-free zinc atom (0.60) in $\text{Cp}^*\text{Zn-Zn}(\text{dmap})_2\text{Cp}^*$ **2**, in which the dmap-coordinated zinc atom (1.15) carries a significantly higher electronic charge. The NBO analysis (Wiberg bond index) shows a bond order of 0.90, which is in between the values reported for **1** (0.93) and **2** (0.85), respectively. In accordance with the $3d^{10}4s^14p^0$ electron configuration of a Zn^+ monocation, a molecular orbital study revealed mainly (76 %) s-character of the Zn-Zn bond (p: 23 %, d: < 1 %). The Zn-N bond order of 0.23 shows a mainly electrostatic coordination of the dmap ligands. Interestingly, the calculated negative charge of the aromatic N atom in dmap increases by coordination to the Zn from -0.52 to -0.63 (Mulliken: -0.12 to -0.25). The loss of electron density in the electron lone pair due to coordination seems to be overcompensated mainly by the aromatic π system, as the negative charge of the N atom in the dimethylamino group remains almost unchanged upon coordination (NBO: -0.47 to -0.46; Mulliken: -0.31 to -0.30).

Experimental Section

All manipulations were performed under an Ar atmosphere. Solvents were dried and degassed prior to use. ^1H , $^{13}\text{C}\{^1\text{H}\}$ and $^{19}\text{F}\{^1\text{H}\}$ NMR

spectra were recorded on a Bruker Avance 500 spectrometer and are referenced to internal CD_2Cl_2 (^1H : $\delta = 5.32$; ^{13}C : $\delta = 53.5$). IR spectra were recorded on a ALPHA-T FT-IR spectrometer and Raman spectra on a Bruker IFS 55 EQUINOX (with FRA 106 Raman attachment). Melting points were measured in sealed capillaries and were not corrected.

4: 0.16 g $\text{Cp}^*\text{Zn-Zn}(\text{dmap})_2\text{Cp}^*$ **2** (0.25 mmol) dissolved in 10 mL of CH_2Cl_2 was added at -30°C to a solution of 0.42 g $[\text{H}(\text{OEt})_2]_2[\text{Al}(\text{OC}(\text{CF}_3)_3)_4]$ (0.5 mmol) in 10 mL of CH_2Cl_2 and stirred for 15 min at -30°C . The resulting solution was concentrated in vacuum and stored at -30°C . Colorless crystals of **4** (0.20 g, 0.07 mmol, 28% based on **2**) were obtained after 24 h. The yield of isolated crystals of **4** increased to 64% when a cold solution (-30°C) of **dmap** (0.12 g, 1 mmol) in 20 mL CH_2Cl_2 was added after the reaction of **2** and $[\text{H}(\text{OEt})_2]_2[\text{Al}(\text{OC}(\text{CF}_3)_3)_4]$ was completed. Melting point: 120°C (dec.). $^1\text{H-NMR}$ (500 MHz, CD_2Cl_2 , 25°C): $\delta = 3.08$ (s, 6 H, NMe_2), 6.61 (m (AA'XX' spin system, 4 H, C(3)-H), 7.89 (m, 2 H, C(2)-H). $^{13}\text{C}\{^1\text{H}\}\text{-NMR}$ (125 MHz, CD_2Cl_2 , 25°C): $\delta = 39.5$ (NMe_2), 107.8 (C(3)), 121.6 (m, $^1\text{J}_{\text{C,F}} = 293.51$ Hz), 147.9 (C(2)), 156.2 (C(4)). $^{19}\text{F}\{^1\text{H}\}$ (235 MHz, CD_2Cl_2 , 25°C): $\delta = -75.8$.

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First structural characterization of a base-stabilized $[\text{Zn}_2]^{2+}$ dication, which was obtained from the reaction of $\text{Cp}^*_2\text{Zn}_2(\text{dmap})_2$ with two equivalents $[\text{H}(\text{OEt}_2)_2][\text{Al}\{\text{OC}(\text{CF}_3)_3\}_4]$.

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