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 Zinc Complexes [t-BuC(Ni-Pr)₂]₂ZnX**

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Keywords:	Zinc, Amidinate, X-Ray Structure
<p>Note: The following files were submitted by the author for peer review, but cannot be converted to PDF. You must view these files (e.g. movies) online.</p> <p>Scheme 01.cdx Scheme 02.cdx</p>	



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[*t*-BuC(Ni-Pr)₂]₂Zn as Starting Reagent for Monoamidinate Zinc Complexes [*t*-BuC(Ni-Pr)₂]ZnX

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Abstract. Reaction of the bisamidinate zinc complex [*t*-BuC(Ni-Pr)₂]₂Zn **1** with ZnMe₂ yielded the corresponding monoamidinate zinc complex {[*t*-BuC(Ni-Pr)₂]ZnMe}₂ **2**. **2** reacts with iodine with formation of {[*t*-BuC(Ni-Pr)₂]ZnI}₂ **3**. **2** and **3** both adopt dimeric structures in the solid state as was shown by single crystal X-ray analyses. **2** and **3** were characterized by elemental analyses, mass and multinuclear (¹H, ¹³C{¹H}) NMR spectroscopy (¹H, ¹³C). In addition, X-ray crystal structures of **1** – **3** are reported.

Keywords: Zinc; Amidinate; X-Ray Structure

Introduction

N,N' chelating amidinate-ligands [RC(NR')₂] have a long tradition in s-, p-, d- and f-block metal chemistry.[1] Their steric and electronic properties can easily be modified by variation of the organic substituents R and R' and they have been found to coordinate very flexible to the metal center either as monodentate (η^1), chelating (η^2) or bridging monodentate ($\mu-\eta^1-\eta^1$) four-electron donor.[2] These prominent properties render amidinate substituents very effective for the stabilization of a variety of metal complexes, including unusual low-valent metal complexes.[3] In addition, amidinate metal complexes have found widespread technical applications as *tailor-made* catalysts for olefin polymerization[4], *rac*-lactide polymerization[5] and ring opening polymerization reactions (ROP) of ϵ -caprolactone.[6] Moreover, thin films of metals and metal nitrides were deposited by CVD processes (*chemical vapor deposition*) in recent years using metal amidinate complexes.[7] Considering these interesting applications, it came as a surprise to us, that only a very few zinc amidinate[8] as well as boramidinate complexes[9] have been reported in the literature prior to our work.

We became only recently interested in zinc amidinate complexes and reported on the reactions of ZnR₂ (R = Me, Et) with *iso*-propyl carbodiimide *i*-PrN=C=N*i*-Pr, which either proceed with formation of the expected amidinate complex [EtC(N*i*-Pr)₂ZnEt]₂ [10] or with formation of novel cluster-type complexes such as {C[C(N*i*-Pr)₂ZnMe]}₄.[11] Moreover, mono- and bis-amidinate zinc complexes of the type [*t*-BuC(NR)₂]₂Zn and [*t*-BuC(NR)₂]₂ZnX (R = *i*-Pr, Cy, X = Cl, Br) have been synthesized by salt elimination reactions.[12] In these reactions it became evident, that the synthesis of monoamidinate complexes is often complicated due to the formation of the corresponding bisamidinates. We became therefore interested in a more straightforward route to monoamidinate complexes [RC(NR')]₂ZnX (X = halide, alkyl), which are expected to be valuable starting reagents for ligand exchange and reductive coupling reactions as well as halide abstraction reactions.

Herein, we report on the reaction of [*t*-BuC(N*i*-Pr)₂]₂Zn **1** with ZnMe₂ and subsequent reaction of as-formed monoamidinate complex {[*t*-BuC(N*i*-Pr)₂]₂ZnMe}₂ **2** with iodine yielding {[*t*-BuC(N*i*-Pr)₂]₂ZnI}₂ **3**.

Experimental Section

General Procedure

Manipulations were performed in a glovebox under an Ar-atmosphere or with standard Schlenk techniques. Dry solvents were obtained from a solvent purification system (MBraun) and degassed prior to use. [*t*-BuC(Ni-Pr)₂]₂Zn **1** was prepared according to literature method,^[10] ZnMe₂ and I₂ were obtained from Acros and used as received. A Bruker Avance 500 spectrometer was used for NMR spectroscopy. ¹H and ¹³C{¹H} NMR spectra were referenced to internal C₆D₅H (¹H: δ = 7.154; ¹³C: δ = 128.0). IR spectra were recorded on a Bruker Alpha-T FT-IR spectrometer. Melting points were measured in sealed capillaries and were not corrected. Elemental analyses were performed at the *Elementaranalyse Labor* of the University of Essen.

[[*t*-BuC(Ni-Pr)₂]₂ZnMe]₂ 2: 0.48 g (5 mmol) ZnMe₂ was added to a solution of 2.16 g (5 mmol) **1** in 20 mL of toluol at ambient temperature and stirred for 3 h. The solution was concentrated in vacuum and stored at -30 °C. Colorless crystals were isolated by filtration.

Yield 1.2 g (91%), Mp: > 220 °C. Elemental Analysis C₂₄H₅₂N₄Zn₂ (527.44 g/mol): found (calcd): H, 9.72 (9.94); C, 54.17 (54.65); N, 10.48 (10.62) %.

¹H-NMR (300 MHz, C₆D₆, 25 °C): δ -0.30 (s, 3H, ZnCH₃), 1.05 (d, 6H, CH(CH₃)₂), 1.24 (s, 9H, *t*-Bu), 3.95 (sep, 2H, CH(CH₃)₂). ¹³C-NMR (125 MHz, C₆D₆, 25 °C): δ -10.0 (ZnCH₃), 26.9 (CHMe₂), 30.2 (CCMe₃), 39.8 (CCMe₃), 46.5 (CHMe₂). IR (Nujol): ν 2924, 2856, 1460, 1376, 1318, 1261, 1184, 1121, 1017, 819, 722, 656, 446 cm⁻¹.

[[*t*-BuC(Ni-Pr)₂]₂ZnI]₂ 3: A solution of 1.27 g (5 mmol) I₂ in 10 mL hexane was added dropwise to 1.32 g (5 mmol) **2** dissolved in 30 ml hexane at -30 °C. The resulting mixture was warmed to ambient temperature and stirred for additional 12 h. The solution was concentrated in vacuum and stored at -30 °C. Colorless crystals were isolated by filtration.

Yield 1.60 g (85%), Mp: > 220°C. Elemental Analysis C₁₁H₂₃IN₂Zn (751.18 g/mol): found (calcd): H, 6.08 (6.17); C, 34.92 (35.18); N, 7.33 (7.46) %.

¹H-NMR (300 MHz, [D₈]THF, 25 °C): δ 1.03 (d, ³J_{HH} = 5.9 Hz, 6H, CH(CH₃)₂), 1.38 (s, 9H, *t*-Bu), 4.24 (s, 2H, ³J_{HH} = 5.9 Hz, CH(CH₃)₂), ¹³C-NMR (125 MHz, [D₈]THF, 25 °C): δ 27.8 (CHMe₂), 30.6 (CCMe₃), 40.1 (CCMe₃), 46.8 (CHMe₂). IR (nujol): ν 2956, 2958, 2925, 2855, 1555, 1461, 1377, 1261, 1151, 1099, 1018, 801, 722, 468 cm⁻¹.

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X-ray Structure Solution and Refinement of 1-3. Crystallographic data of **1-3** are given in Table 2. Figure 1 - 3 show ORTEP diagrams of the solid state structures of **1 - 3**. Data were collected on a STOE IPDS-II diffractometer. The structures were solved by Direct Methods (SHELXS-97)[13] and refined by full-matrix least-squares on F² (SHELXL-97).[14] All non-hydrogen atoms were refined anisotropically and hydrogen atoms by a riding model. Multi-scan absorption corrections were applied. Crystallographic data of the structures (excluding structure factors) have been deposited with the Cambridge Crystallographic Data Centre as supplementary publication no. CCDC-726835 (**1**), CCDC-726836 (**2**), and CCDC-726837 (**3**). Copies of the data can be obtained free of charge on application to The Director, CCDC, 12 Union Road, Cambridge CB2 1EZ, UK (Fax: int.code_(1223)336-033; e-mail for inquiry: fileserv@ccdc.cam.ac.uk; e-mail for deposition: deposit@ccdc.cam.ac.uk).

Results and Discussion

Equimolar amounts of [*t*-BuC(Ni-Pr)₂]₂Zn **1** and ZnMe₂ react at ambient temperature with subsequent formation of {[*t*-BuC(Ni-Pr)₂]ZnMe}₂ **2** in quantitative yield. **2** can be easily converted into {[*t*-BuC(Ni-Pr)₂]ZnI}₂ **3** by reaction with an equimolar amount of I₂.

((Scheme 1 here))

2 and **3** were obtained as colorless crystalline solids after re-crystallization from solutions in toluene (**2**) and *n*-hexane (**3**), respectively, at -30 °C. ¹H and ¹³C NMR spectra of **2** show the expected resonances due to the amidinate ligand and the Me group in the expected 1:1 intensities, whereas **3** only shows the resonances of the amidinate ligand.

Single crystals of **1** were obtained by sublimation of freshly prepared **1**, whereas single crystals of **2** and **3** suitable for X-ray structure determinations were obtained from solutions in toluene (**2**) and *n*-hexane (**3**) at -30 °C.

((Figures 1 – 3 here))

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5 **1** crystallizes in the orthorhombic space group Pbcn, **2** in the monoclinic space group
6 $P2_1/n$ and **3** in the triclinic space group P-1 with two half independent molecules in the
7 asymmetric unit. **2** and **3** form centrosymmetric dimers in the solid state with bridging
8 N-centers and the center of symmetry in the Zn_2N_2 ring, whereas **1** is monomeric in the
9 solid state as was expected. The structures of **1** and **3** are comparable to those previously
10 observed for monoamidinate zinc halides $\{[t\text{-BuRC}(\text{Ni-Pr})_2]\text{ZnX}\}_2$ ($X = \text{Cl, Br}$) and
11 bisamidinate complexes $[t\text{-BuC}(\text{NCy})_2]_2\text{Zn}$, [12] whereas the structure of $[t\text{-BuC}(\text{Ni-Pr})_2\text{ZnMe}]_2$ **2** significantly differs from that reported for $[\text{EtC}(\text{Ni-Pr})_2\text{ZnEt}]_2$ **4**. **4** forms
12 an eight-membered $[\text{Zn}_2(\mu\text{-NCN})_2]$ heterocycle, which adopts a *boat-type* conformation
13 and shows short Zn-N bonds within the ring (1.991(2) - 2.014(2) Å) and only weak
14 transannular Zn-N bonds (2.669(1), 2.653(1) Å). [10] In contrast, the Zn-N bond lengths
15 within the eight-membered ring in **2** are significantly elongated (Zn1-N1 2.0974(11),
16 Zn1-N2a 2.1184(10) Å) compared to **4** whereas the transannular distances (Zn1-
17 N2/Zn1a-N2a 2.1952(11) Å) are remarkably shorter. Almost identical bond lengths are
18 observed in **3** (Zn1-N1 2.083(3), Zn1-N2a 2.053(3), Zn1-N2/Zn1a-N2a 2.136(3) Å). As
19 a consequence, the amidinate group in **2** and **3** rather serves as chelating (η^2) unit
20 whereas in **4** it serves as bridging monodentate ($\mu\text{-}\eta^1\text{-}\eta^1$) four-electron donor.
21 Moreover, the Zn atoms in **2** adopt distorted tetrahedral coordination spheres whereas in
22 **4** trigonal-planar coordination geometry was observed.

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44 Another structural difference between **1** on the one hand and **2** and **3** on the other hand
45 is reflected by the different coordination modes of the amidinate moiety. The N atoms
46 in the bis-amidinate complex **1** are threefold coordinated and the Zn-N bond lengths are
47 almost identical. The sum of the bond angles of the central carbon atom (C_{Amid}) and the
48 nitrogen atoms N1 and N2 of the planar amidinate ligand are close to 360° , indicating
49 sp^2 -hybridized C and N atoms. In addition, the almost equal C-N bond lengths observed
50 in **1** (N1- C_{Amid} 1.335(2), N2- C_{Amid} 1.343(2) Å), which are in between typical values for
51 C-N single and C=N double bonds, prove an almost perfect delocalization of the π -
52 electrons within the CN_2 backbones of both four-membered rings. In contrast, the N1/2-
53 C_{Amid} bond lengths in **2** (1.3110(16), 1.3929(15) Å) and particularly **3** (1.298(5),
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4 1.435(4) Å) differ significantly, with the distances of the four-coordinated N atoms
5 being elongated compared to those of the three-coordinated N atoms. Moreover, the
6 three-coordinate carbon and nitrogen atoms in **2** and **3** are almost planar whereas
7 significantly smaller sum of bond angles are observed for the four-coordinate nitrogen
8 atoms. These findings clearly demonstrate a rather strong disturbance of the π -electrons.
9 Consequently, the coordination mode of the amidinate ligand in **2** and **3** is better
10 described by resonance structure **A** whereas resonance structure **B** reflects the
11 coordination properties of the amidinate moiety in **1**.
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21 ((Table 2 here))
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26 **Conclusion.** Bisamidinate complexes $[\text{RC}(\text{NR}')_2]_2\text{Zn}$ can be converted into
27 monoamidinate complexes $[\text{RC}(\text{NR}')_2]\text{ZnX}$ (X = Me, I), which are promising starting
28 reagents for further derivatization reactions as well as reductive coupling and
29 halide/methyl abstraction reactions.
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33 **Acknowledgements.** We gratefully acknowledge financial support by the Deutsche
34 Forschungsgemeinschaft (DFG).
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5 **Figure / Scheme Captions**
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8 **Figure 1.** Molecular structure and atom numbering scheme of **1**; displacement
9 ellipsoids are drawn at the 50% probability level. H atoms have been omitted for clarity.
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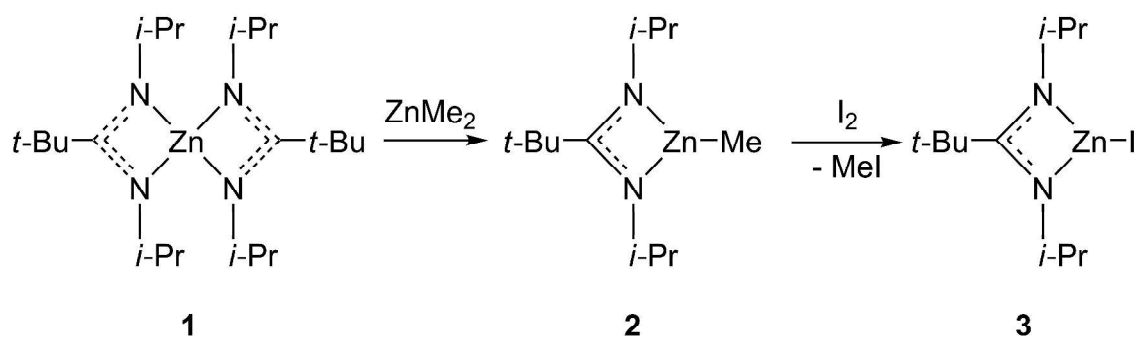
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13 **Figure 2.** Molecular structure and atom numbering scheme of **2**; displacement
14 ellipsoids are drawn at the 50% probability level. H atoms have been omitted for clarity.
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19 **Figure 3.** Molecular structure and atom numbering scheme of **3**; displacement
20 ellipsoids are drawn at the 50% probability level. H atoms and the second independent
21 molecule have been omitted for clarity.
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29 **Scheme 1.** Synthesis of **2** and **3**.
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33 **Scheme 2.** Different resonance structures of the amidinate ligand in complexes **1 – 3**.
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Scheme 1



Scheme 2

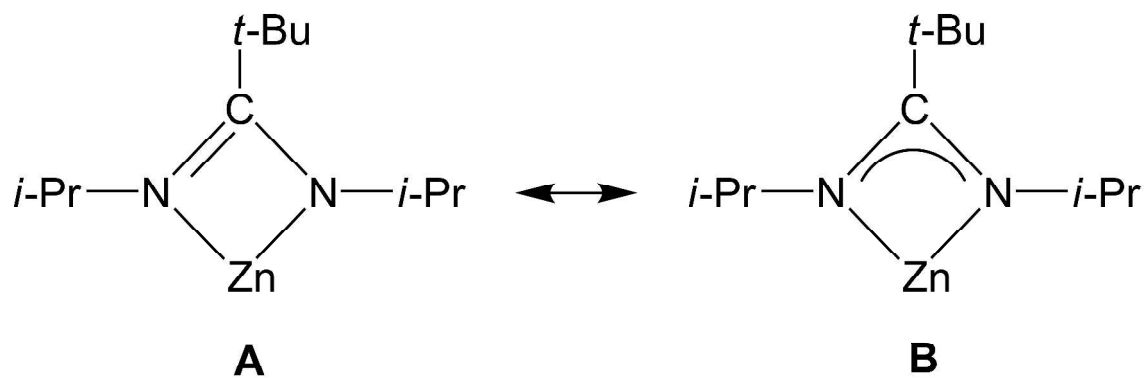


Table 1. Selected bond lengths [\AA] and angles [$^\circ$] of **1** – **3**.

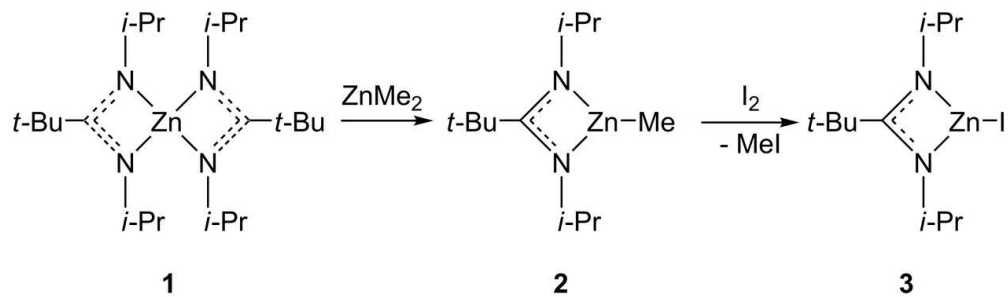
	1	2	3^a
Zn1-N1	2.0175(13)	2.0974(11)	2.083(3)
Zn1-N2	2.0297(14)	2.1952(11)	2.136(3)
Zn1-N2a	2.0297(14)	2.1184(10)	2.053(3)
Zn1-C _{Me} /I1		1.9812(16)	2.5440(4)
N1-C _{Amid}	1.335(2)	1.3110(16)	1.298(5)
N1-C _{i-Pr}	1.457(2)	1.4765(16)	1.488(5)
N2-C _{Amid}	1.343(2)	1.3929(15)	1.435(4)
N2-C _{i-Pr}	1.463(2)	1.4902(16)	1.512(4)
C _{Amid} -C _{t-Bu}	1.555(2)	1.5695(18)	1.556(5)
N1-Zn1-N2	65.34(6)	62.87(4)	64.42(11)
N1-Zn1-N1a	134.35(9)		
N1-Zn1-N2a		109.15(4)	117.11(10)
N2-Zn1-N2a	134.90(6)	94.29(4)	91.32(10)
N1-Zn1-C _{Me} /I1		124.63(7)	112.35(8)
N2-Zn1-C _{Me} /I1		132.72(7)	131.25(7)
N2a-Zn1-C _{Me} /I1		119.20(6)	125.40(7)
Zn1-N1-C _{Amid}	93.07(10)	95.52(8)	95.5(2)
Zn1-N1-C _{i-Pr}	135.24(12)	134.62(9)	136.3(2)
Zn1-N2-C _{Amid}	92.29(10)	88.94(7)	89.3(2)
Zn1-N2-C _{i-Pr}	133.28(12)	123.69(8)	118.6(2)

^a Geometric parameters are only given for one of the two independent molecules.

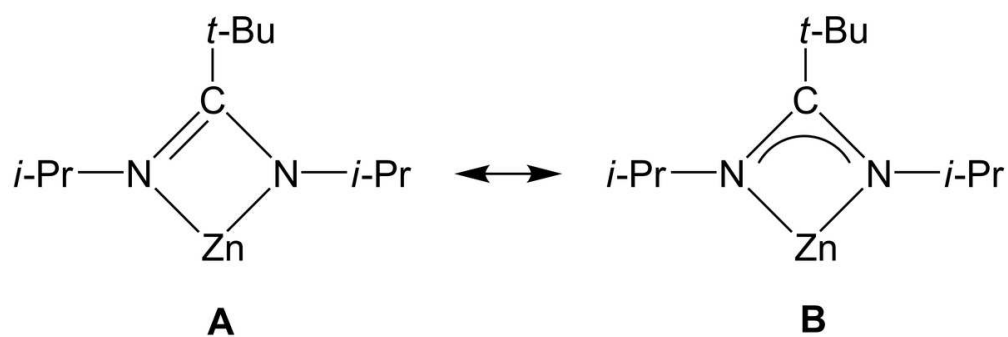
Table 2. Crystallographic details of **1** – **3**.

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emp.form.	C ₂₂ H ₄₆ N ₄ Zn	C ₂₄ H ₅₂ N ₄ Zn ₂	C ₂₂ H ₄₆ I ₂ N ₄ Zn ₂
mol. mass	432.00	527.44	751.17
cryst syst.	orthorhombic	monoclinic	triclinic
space group	Pbcn	P2 ₁ /n	P-1
a [Å]	16.7038(10)	10.5974(5)	9.9516(8)
b [Å]	9.4860(7)	12.9710(7)	10.7946(8)
c [Å]	16.7124(15)	10.6565(4)	14.6318(12)
α [°]	90	90	91.415(6)
β [°]	90	102.954(3)	107.994(6)
γ [°]	90	90	97.242(6)
V [Å ³]	2648.1(3)	1427.55(12)	1479.6(2)
Z	4	2	2
T [K]	173(2)	173(2)	173(2)
radiation (λ [Å])	Mo-K _α (0.71073)	Mo-K _α (0.71073)	Mo-K _α (0.71073)
μ [mm ⁻¹]	0.940	1.696	3.723
D _{calc.} [g cm ⁻³]	1.084	1.227	1.686
2θ _{max} [°]	51.4	59.4	51.2
cryst dim. [mm]	0.47 x 0.42 x 0.39	0.41 x 0.37 x 0.34	0.21 x 0.17 x 0.12
no. reflns	19562	36353	20535
unique	2491	3999	5518
R _{merg}	0.0559	0.0451	0.0650
param./restraints	124 / 0	137 / 0	271 / 0
R1 ^a	0.0299	0.0257	0.0324
wR2 ^b	0.0745	0.0666	0.0826
goodness of fit ^c	1.038	1.040	1.006
final max/min. Δρ, e Å ⁻³	0.221 / -0.169	0.336 / -0.372	0.975 / -1.286

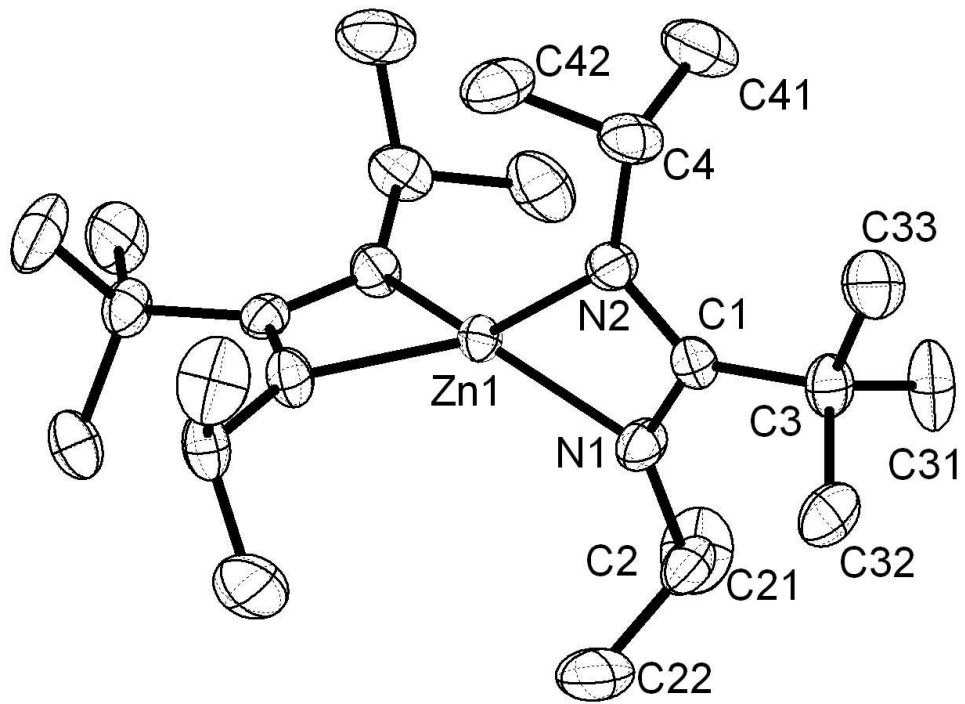
[a] $R1 = \frac{\sum (|F_o| - |F_c|)}{\sum |F_o|}$ (for $I > 2\sigma(I)$). - [b] $wR2 = \frac{\{\sum [w(F_o^2 - F_c^2)^2]\}}{\sum [w(F_o^2)^2]}^{1/2}$. - [c] Goodness of fit = $\frac{\{\sum [w(|F_o^2| - |F_c^2|)^2]\}}{(N_{\text{observns}} - N_{\text{params}})}$



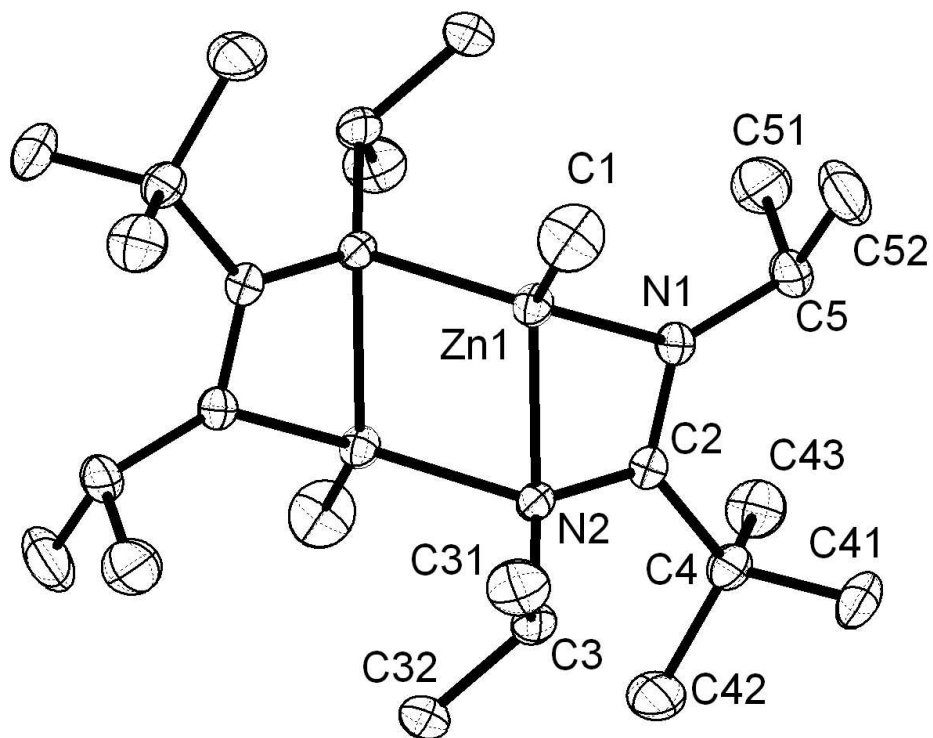
53x16mm (600 x 600 DPI)



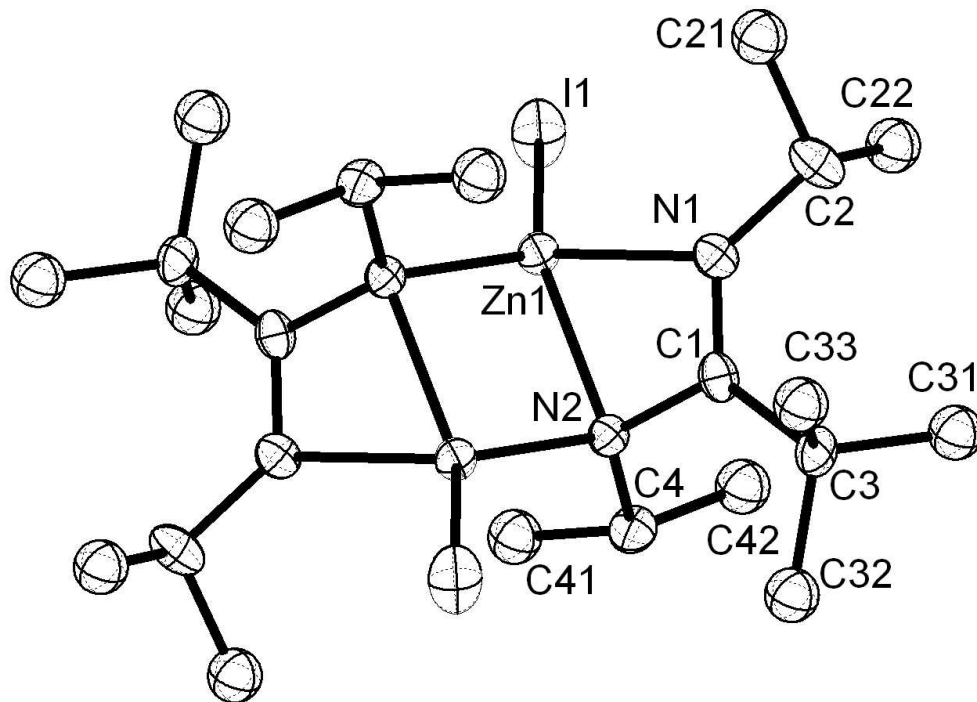
50x17mm (600 x 600 DPI)



338x270mm (96 x 96 DPI)



338x270mm (96 x 96 DPI)



338x270mm (96 x 96 DPI)