CORRECTION

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Structural and Dynamic Studies of Zinc, Gallium, and Cadmium Complexes of a Dicarboxylate Pendant-Armed Cross-Bridged Cyclen

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Inadvertently, the wrong X-ray structure was reported[1] *for the Zn-CB-DO2A complex. Instead, the structural data for a related zinc complex was actually presented. The first paragraph of the section X-ray Structural Data and Figure 5 on page 3312, Table 1 on page 3313, the last two sentences of the paragraph [Zn·**1**](NaClO$_4$)$_{1.5}$ and Table 3 on page 3314, and the paragraph X-ray Crystallography on page 3315 should be replaced as follows (in addition, a corrected X-ray CIF file was deposited with the CCDC):*

X-ray Structural Data

The core structure of the zinc complex [Zn·**1**] is shown in Figure 5. The Zn^{II} cation is fully enveloped within the *cis*-N_4O_2 donor set of the cross-bridged ligand in a distorted octahedral geometry. The axial N(3)–Zn(1)–N(4) angle of 156.97(11) [158.36(10)]° (structural data for a second independent molecule are given in brackets) is substantially away from linearity, showing that the Zn^{II} is distended from the cross-bridged cyclen cavity. Previous structural data of zinc complexes of the cross-bridged cyclen and its derivatives have also shown this distention from the ligand cavity with axial N–Zn–N angles between 148 and 159°.[16–18] Another manifestation of these distortions can also be seen in their longer axial N–Zn bonds (2.17–2.26 Å), similar to the average of 2.19 Å found in [Zn·**1**]. All Zn–N and Zn–O bond lengths are within a relatively narrow range of 2.03–2.20 Å. Selected bond lengths and angles are shown in Table 1. An X-ray structure of a DOTA complex of zinc(II) featuring a related *cis*-N_4O_2 ligand coordination mode has been reported.[19] This has an axial N–Zn–N angle of 153.9°, an axial Zn–N distance of 2.232 Å, an equatorial Zn–N distance of 2.171 Å, and a shorter Zn–O distance of 2.037 Å. However, without the cross-bridging ethylene, the equatorial N–Zn–N angle is opened up to 107.8° [compared to the 83.6(1)° for N(1)–Zn(1)–N(2) here]. Also, the coordinated carboxylate pendant arms are on the equatorial macrocyclic nitrogen atoms rather than the axial ones and this DOTA structure is thus close to C_{2v} symmetry.

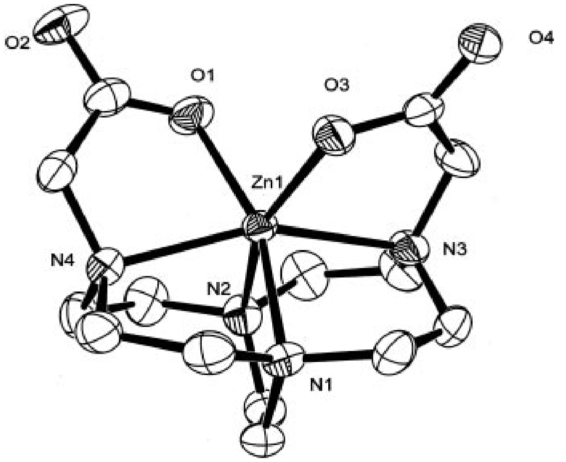

Figure 5. Molecular structure of Zn-CB-DO2A·NaClO$_4$; hydrogen atoms and sodium perchlorate have been omitted for clarity.

X-ray quality crystals were grown by ether diffusion into a sample dissolved in methanol solution. These have a composition of [Zn·**1**](NaClO$_4$)(CH$_3$OH)(H$_2$O).

X-ray Crystallography: Diffraction intensity data were collected with Bruker Smart Apex CCD ([Zn·**1**]) and Siemens P4 CCD ([Ga·**1**]NO$_3$) diffractometers. Crystal data collection and refinement parameters are given in Table 3. The structures were solved using direct methods, completed by subsequent difference Fourier syntheses, and refined by full-matrix least-squares procedures on F^2. SADABS absorption corrections were applied to [Ga·**1**]NO$_3$ data (T_{min}/T_{max} = 0.724). In both structures all non-hydrogen

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Table 1. Selected bond lengths [Å] and bond angles [°] for [Zn·**1**] (values for two symmetry-independent molecules are given).

Zn(1)–O(1)	2.032(3)	O(1)–Zn(1)–O(3)	89.58(11)
Zn(1)–O(3)	2.047(2)	O(1)–Zn(1)–N(1)	159.24(11)
Zn(1)–N(1)	2.130(3)	O(1)–Zn(1)–N(2)	98.80(12)
Zn(1)–N(2)	2.116(3)	N(1)–Zn(1)–N(2)	83.60(12)
Zn(1)–N(3)	2.193(3)	N(3)–Zn(1)–N(4)	156.97(11)
Zn(1)–N(4)	2.195(3)	N(1)–Zn(1)–N(3)	83.08(12)
Zn(2)–O(5)	2.058(2)	O(5)–Zn(2)–O(7)	89.55(9)
Zn(2)–O(7)	2.040(2)	O(5)–Zn(2)–N(5)	160.24(10)
Zn(2)–N(5)	2.123(3)	O(5)–Zn(2)–N(6)	96.84(10)
Zn(2)–N(6)	2.126(3)	N(5)–Zn(2)–N(6)	84.03(11)
Zn(2)–N(7)	2.172(3)	N(5)–Zn(2)–N(7)	83.76(10)
Zn(2)–N(8)	2.182(3)	N(7)–Zn(2)–N(8)	158.36(10)

Table 3. Crystallographic data for [Zn·**1**] and [Ga·**1**]NO$_3$.

	[Zn·**1**]	[Ga·**1**]NO$_3$
Empirical formula	$C_{30}H_{48}Cl_2N_8Na_2O_{20}Zn_2$	$C_{14}H_{24}GaN_5O_7$
Formula mass	1088.38	444.10
Crystal system, space group	monoclinic, $P2_1/c$	orthorhombic, $P2_12_12_1$
a [Å]	8.8197(6)	8.7107(7)
b [Å]	17.2311(13)	12.5119(10)
c [Å]	29.841(2)	7.5586(6)
α [°]	90.0	90.0
β [°]	96.319(2)	90.0
γ [°]	90.0	90.0
V [Å^3]	4507.4(6)	823.79(11)
Z	4	2
T [K]	218(2)	218(2)
λ [Å]	0.71073 (Mo-K_α)	0.71073 (Mo-K_α)
$\rho_{calcd.}$ [g·cm^{-3}]	1.604	1.790
μ [mm^{-1}]	1.286	1.724
Measured reflections	33251	6004
Independent reflections	10841 (R_{int}=0.0352)	1967 (R_{int}=0.0260)
$R^{[a]}$, $R^{[b]}(\omega F^2)$ (obsd. data)	0.0514, 0.1415	0.0231, 0.0553
$R^{[a]}$, $R^{[b]}(\omega F^2)$ (all data)	0.0638, 0.1495	0.0244, 0.0557
GOF	1.027	1.006

[a] $R = \Sigma||F_o| - |F_c||/\Sigma|F_o|$. [b] $R(\omega F^2) = \{\Sigma[\omega(F_o^2 - F_c^2)^2]/\Sigma[\omega(F_o^2)^2]\}^{1/2}$; $\omega = 1/[\sigma^2(F_o^2) + (aP)^2 + bP]$, $P = [2F_c^2 + \max(F_o,0)]/3$.

atoms were refined with anisotropic displacement coefficients. Hydrogen atoms were treated as idealized contributions. The Flack parameter for [Ga·**1**]NO$_3$ is 0.014(11). All software and sources of scattering factors are contained in the SHELXTL (5.10) program package (G. Sheldrick, Bruker XRD, Madison, WI, USA). CCDC-291598 ([Zn·**1**]) and -229394 ([Ga·**1**]NO$_3$) contain the supplementary crystallographic data for this paper. These data can be obtained free of charge at www.ccdc.uk/conts/retrieving.html [or from the Cambridge Crystallographic Data Centre, 12 Union Road, Cambridge CB2 1EZ, UK; Fax: +44-1223-336-033; E-mail: deposit@ccdc.cam.ac.uk].

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[1] W. Niu, E. H. Wong, G. R. Weisman, Y. Peng, C. J. Anderson, L. N. Zakharov, J. A. Golen, A. L. Rheingold, *Eur. J. Inorg. Chem.* **2004**, 3310–3315.

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