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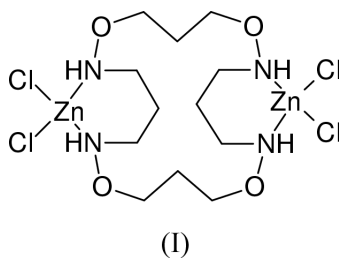
Key indicators

Single-crystal X-ray study
T = 150 K
Mean $\sigma(\text{C}-\text{C}) = 0.004 \text{ \AA}$
R factor = 0.031
wR factor = 0.084
Data-to-parameter ratio = 20.8For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.Rerefinement of (μ -1,7,11,17-tetraoxa-2,6,12,16-tetraazacycloeicosane- $\kappa^4\text{N}^1, \text{N}^{17}:\text{N}^7, \text{N}^{11}$)-bis[dichlorozinc(II)]

This rerefinement of the title compound, $[\text{Zn}_2\text{Cl}_4(\text{C}_{12}\text{H}_{28}\text{N}_4\text{O}_4)]$, based on the original intensity data, differs from the original only in the exchange of the NH and O moieties of the oxaza-macrocycle, resulting in a dramatic improvement in the *R* factors and the anisotropic displacement parameters associated with the N and O atoms. The tetrahedral coordination of Zn involves Cl at 2.2144 (6) and 2.2279 (6) Å, but now N rather than O at 2.0563 (18) and 2.0934 (17) Å, in keeping with the usual arrangement for zinc complexes of this type. The revised model permits satisfactory N—H \cdots Cl hydrogen bonds which the original did not. A crystallographic twofold axis passes through the centroid of the molecule perpendicular to its least-squares plane.

Comment

The *checkCIF* facility of the IUCr, applied to the CIF data corresponding to the structure of the title compound, (I), as reported previously by Kuksa *et al.* (2000), reported significantly disparate displacements of the N and O atoms as designated in the original model. This was sufficient to induce us to rerefine the structure with these species interchanged. The new refinement was immediately and dramatically successful, with the reduction of the previous $R[F^2 > 2\sigma(F^2)]$ and *wR2* (all F^2 data) of 0.049 and 0.151, respectively, to the corresponding values given below.



Further benefits of the model used in the rerefinement are to be seen in the coordination of Zn (Table 1) and the presence of N—H \cdots Cl hydrogen bonds (Table 2). Thus, the still somewhat distorted coordination of Zn now comprises 2N rather than 2O in addition to 2Cl, and is consistent with the binding of Zn only to N donors, as reported in complexes with 17- to 19-membered ring O₂N₂-donor ligands by Lindoy *et al.* (1980) and as mentioned by Kuksa *et al.* (2000). The remaining bond distances and angles are as expected for a molecule of this type and in no way remarkable. The N—H \cdots Cl hydrogen bonds given in Table 2 provide complete three-dimensional connectivity among the molecules and are in stark contrast to the total absence of equivalent contacts in the model prior to

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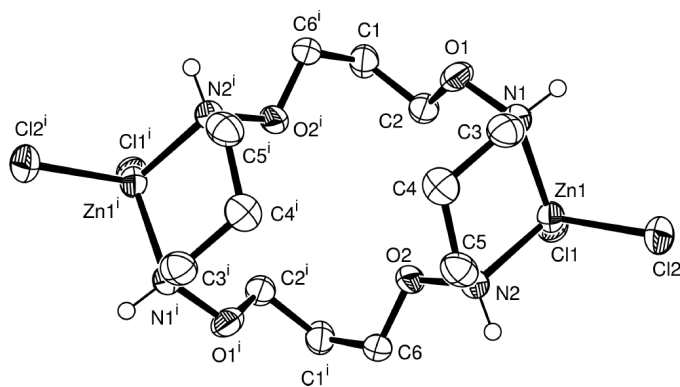


Figure 1

The molecule of (I), showing the labelling scheme. Non-H atoms are shown as 50% ellipsoids and H of NH as small circles, while methylene H atoms have been omitted for clarity. [Symmetry code: (i) $1/2-x, y, 1-z$.]

rerefinement. A feature of both models, not remarked upon previously, is the chair conformation of the six-membered chelate rings.

Experimental

The synthesis and growth of crystals of (I) has been described by Kuksa *et al.* (2000).

Crystal data

$[\text{Zn}_2\text{Cl}_4(\text{C}_{12}\text{H}_{28}\text{N}_4\text{O}_4)]$
 $M_r = 564.92$
 Monoclinic, $I2/a$
 $a = 16.4925$ (4) Å
 $b = 7.2697$ (2) Å
 $c = 19.2129$ (6) Å
 $\beta = 109.4120$ (18)°
 $V = 2172.59$ (10) Å³
 $Z = 4$

$D_x = 1.727$ Mg m⁻³
 Mo $K\alpha$ radiation
 Cell parameters from 12531 reflections
 $\theta = 2.6$ – 27.5°
 $\mu = 2.72$ mm⁻¹
 $T = 150$ (2) K
 Block, colourless
 $0.20 \times 0.15 \times 0.15$ mm

Data collection

Delft Instruments FAST diffractometer
 φ scans
 Absorption correction: refined from ΔF (DIFABS; Walker & Stuart, 1983)
 $T_{\min} = 0.589$, $T_{\max} = 0.665$
 12531 measured reflections

2470 independent reflections
 2096 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.043$
 $\theta_{\max} = 27.5^\circ$
 $h = -20 \rightarrow 21$
 $k = -8 \rightarrow 9$
 $l = -24 \rightarrow 20$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.031$
 $wR(F^2) = 0.084$
 $S = 1.06$
 2470 reflections
 119 parameters
 H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0469P)^2 + 1.0443P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} = 0.001$
 $\Delta\rho_{\max} = 0.47$ e Å⁻³
 $\Delta\rho_{\min} = -0.48$ e Å⁻³
 Extinction correction: SHELXL97
 Extinction coefficient: 0.0027 (3)

Table 1

Selected geometric parameters (Å, °).

Zn1–N2	2.0563 (18)	Zn1–Cl1	2.2144 (6)
Zn1–N1	2.0934 (17)	Zn1–Cl2	2.2279 (6)
N2–Zn1–N1	97.14 (7)	N2–Zn1–Cl2	107.32 (5)
N2–Zn1–Cl1	113.58 (5)	N1–Zn1–Cl2	100.83 (5)
N1–Zn1–Cl1	120.18 (5)	Cl1–Zn1–Cl2	115.45 (3)

Table 2

Hydrogen-bonding geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
N1–H1 ⁱ ⋯Cl1 ⁱ	0.93	2.48	3.3926 (19)	166
N2–H2 ⁱⁱ ⋯Cl1 ⁱⁱ	0.93	2.50	3.3914 (18)	161

Symmetry codes: (i) $-x, \frac{1}{2} + y, \frac{1}{2} - z$; (ii) $\frac{1}{2} - x, \frac{3}{2} - y, \frac{1}{2} - z$.

A more complete account of the data collection process is given by Darr *et al.* (1993). In this particular case, a primitive triclinic cell was used at the time of data collection. As a consequence of the adoption of the I -centred monoclinic cell, the original intensity data were reindexed prior to solution and refinement of the structure. H atoms were placed in calculated positions, with $X-H = 0.99$ and 0.93 Å for C–H and N–H, respectively, and refined as riding atoms with $U_{\text{iso}} = 1.2U_{\text{eq}}$ of X in all cases.

Data collection: MADNES (Pflugrath & Messerschmidt, 1989); cell refinement: MADNES; data reduction: ABSMAD (Karaulov, 1992); program(s) used to solve structure: SHELXS86 (Sheldrick, 1990); program(s) used to refine structure: SHELXL97 (Sheldrick, 1997); molecular graphics: ORTEP-3 (Farrugia, 1997); software used to prepare material for publication: SHELXL97.

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