

(1-Aza-4-aziniobicyclo[2.2.2]octane-*N*¹)trichloro-zinc(II)**Mingyi Wei and Roger D. Willett***

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Key indicators

Single-crystal X-ray study

 $T = 293$ KMean $\sigma(\text{C}-\text{C}) = 0.009$ Å R factor = 0.031 wR factor = 0.077

Data-to-parameter ratio = 9.4

For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.

$\text{Zn}(\text{dabcoH})\text{Cl}_3$ (dabco is 1,4-bicyclo[2.2.2]octane), $[\text{ZnCl}_3(\text{C}_6\text{H}_{13}\text{N}_2)]$, crystallizes as a neutral monomeric species with approximate C_3 symmetry. The tetrahedrally coordinated Zn^{II} ion has $\text{Zn}-\text{Cl}$ distances ranging from 2.239 (2) to 2.250 (2) Å and a $\text{Zn}-\text{N}$ distance of 2.094 (4) Å. The $\text{Cl}-\text{Zn}-\text{Cl}$ angles are slightly larger than tetrahedral [112.44 (6)–114.38 (6)°]. The protonated aza group forms a weak interaction with a Cl atom of an adjacent molecule.

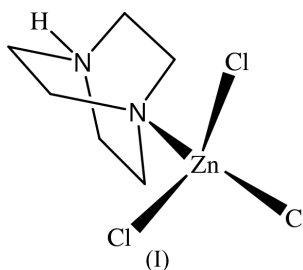
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Comment

The title compound, (I), was prepared in an attempt to make non-Jahn-Teller analogs to $(\text{dabcoH}_2)_2\text{Cl}_3[\text{CuCl}_3(\text{H}_2\text{O})_2]\cdot\text{H}_2\text{O}$ (Wei & Willett, 1996) and to $(\text{dabcoH}_2)\text{CuCl}_4$ and $(\text{dabco}_2)\text{CuCl}_4\cdot\text{H}_2\text{O}$ (Wei & Willett, 2001).

**Experimental**

A procedure similar to that employed in the synthesis of $(\text{dabcoH}_2)_2\text{Cl}_3[\text{CuCl}_3(\text{H}_2\text{O})_2]\cdot\text{H}_2\text{O}$ (Wei & Willett, 1996) was followed. Equimolar mixtures of the dabco ligand and ZnCl_2 were dissolved in a dilute HCl solution. Colorless crystals separated out of the solution after several days of slow evaporation at room temperature. These were filtered off and air-dried.

Crystal data

$[\text{ZnCl}_3(\text{C}_6\text{H}_{13}\text{N}_2)]$
 $M_r = 284.94$
 Monoclinic, $P2_1$
 $a = 6.7475$ (10) Å
 $b = 12.5446$ (16) Å
 $c = 6.9788$ (17) Å
 $\beta = 116.430$ (14)°
 $V = 528.98$ (17) Å³
 $Z = 2$

$D_x = 1.789$ Mg m⁻³
 Mo $K\alpha$ radiation
 Cell parameters from 25 reflections
 $\theta = 13.5$ – 14°
 $\mu = 3.03$ mm⁻¹
 $T = 293$ (2) K
 Rhomboid, colorless
 $0.30 \times 0.30 \times 0.25$ mm

Data collection

Bruker P4 diffractometer
 ω scans
 1317 measured reflections
 1038 independent reflections
 1005 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.030$
 $\theta_{\text{max}} = 25.0^\circ$

$h = -1 \rightarrow 7$
 $k = -1 \rightarrow 14$
 $l = -8 \rightarrow 7$
 3 standard reflections
 every 97 reflections
 intensity decay: <1%

Refinement

Refinement on F^2

$R[F^2 > 2\sigma(F^2)] = 0.031$

$wR(F^2) = 0.077$

$S = 1.08$

1038 reflections

110 parameters

H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0523P)^2]$

where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} < 0.001$

$\Delta\rho_{\max} = 0.80 \text{ e } \text{\AA}^{-3}$

$\Delta\rho_{\min} = -0.56 \text{ e } \text{\AA}^{-3}$

Absolute structure: Flack (1983)

Flack parameter = 0.00 (2)

Data collection: *XSCANS* (Siemens, 1992); cell refinement: *XSCANS*; data reduction: *XSCANS*; program(s) used to solve structure: *SHELXS90* (Sheldrick, 1990); program(s) used to refine

structure: *SHELXL92* (Sheldrick, 1992); molecular graphics: *SHELXTL* (Bruker, 1997); software used to prepare material for publication: *SHELXL97* (Sheldrick, 1997).

References

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