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catena-Poly[[[triaquazinc(**II**)]-μ-chloro-[aquacucurbit[5]urilpotassium(**I**)]] tetrachlorozincate(**II**) pentahydrate]

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In the crystal structure of the title compound, $[Zn\{K(C_{30}H_{30}N_{20}O_{10})(H_2O)\}Cl(H_2O)][ZnCl_4]\cdot5H_2O$, the $[(H_2O)_3ClZn]^+$ cation links adjacent $[(C_{30}H_{30}N_{20}O_{10})(H_2O)K]^+$ cations into a polycationic chain. The chain is surrounded by water molecules and disordered $[ZnCl_4]^{2-}$ anions and they interact to form a hydrogen-bonded layer structure. One water molecule is encapsulated by the cryptand. The cryptand and the encapsulated water molecule, as well as the K and Zn atoms, one coordinated water molecule and one free water molecule, lie on special positions of site symmetry *m*.

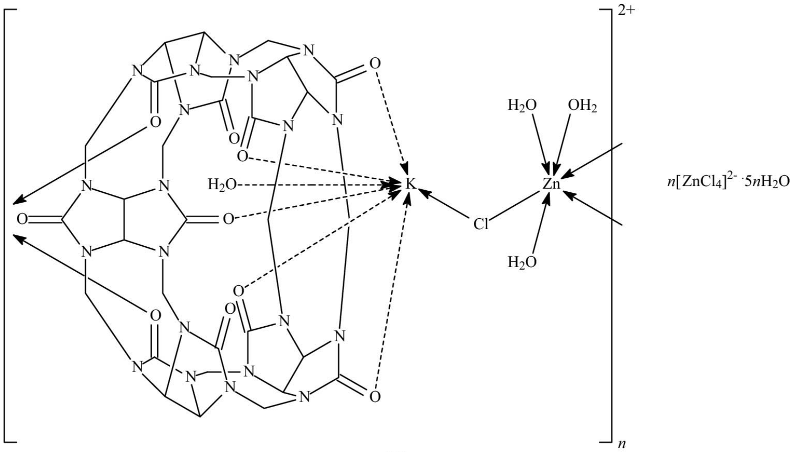
Key indicators

Single-crystal X-ray study
T = 295 K
Mean σ(C–C) = 0.005 Å
Disorder in solvent or counterion
R factor = 0.044
wR factor = 0.134
Data-to-parameter ratio = 13.5

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

Comment

The class of cucurbit[*n*]uril molecular hosts (Lee *et al.*, 2003) has been expanded to include partially substituted analogues (Day *et al.*, 2003). The guest-binding ability is exemplified by the adduct of tetraaquacopper(II) sulfate with cucurbit[5]uril (Liu *et al.*, 2004); this extensively hydrated compound has the Cu atom in a square-planar environment that is made up of four water molecules; two cryptand molecules interact with the metal atom at 2.665 Å. Cucurbit[5]uril, which exists as a hydrate (Kim *et al.*, 2000), is not particularly soluble in water (Buschmann *et al.*, 2001), but it readily dissolves in it when metal ions, such as, for example, alkali and alkaline earths, are added (Buschmann *et al.*, 2001). In the present study, potassium chloride was added to an aqueous mixture of cucurbit[5]uril and zinc acetate in the hope of facilitating the reaction in an acid medium. The potassium cation is incorporated in the resulting compound, (I), which consists of the $[(C_{30}H_{30}N_{20}O_{10})(H_2O)K]^+$ and $[(H_2O)_3ClZn]^+$ cations and the $[ZnCl_4]^{2-}$ anion, along with five water molecules (Fig. 1).



(I)

The potassium-containing cations are linked into a chain motif through the $[(H_2O)_3ClZn]^+$ component; its Cl atom

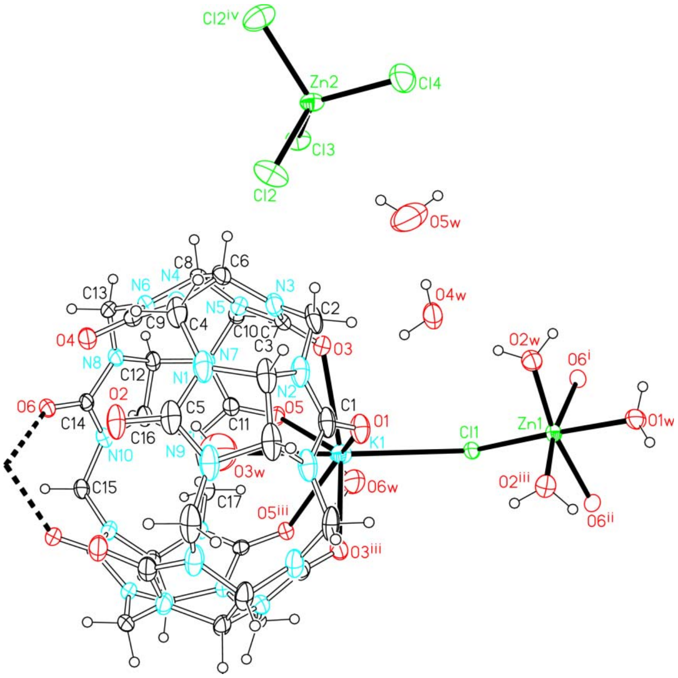


Figure 1
ORTEPII plot (Johnson, 1976) of (I); displacement ellipsoids are drawn at the 50% probability level and H atoms as spheres of arbitrary radii. Only one set of disordered atoms is shown for the $[ZnCl_4]^{2-}$ anion. [Symmetry codes: (i) $x - 1, y, z$; (ii) $x - 1, \frac{3}{2} - y, z$; (iii) $x, \frac{3}{2} - y, z$.]

interacts with the K atom. The octahedrally coordinated Zn atom behaves as a Lewis acid in accepting two donor O atoms of the cryptand of another $[(C_{30}H_{30}N_{20}O_{10})(H_2O)K]^+$ unit. The bridging behaviour gives rise to the formation of a linear polycationic chain (Fig. 2). Of the other water molecules, one is encapsulated in the cavity of the cryptand (Fig. 3); the other water molecules interact with the polycation chain and with the tetrahedral $[ZnCl_4]^{2-}$ ions through hydrogen bonds (Table 2). There are no voids between the layers, as confirmed by *PLATON* (Spek, 2003).

Experimental

Cucurbit[5]uril (2.07 g, 2.5 mmol) was first dissolved in 1.5 *M* hydrochloric acid (100 ml). To this solution (10 ml) was added zinc acetate heptahydrate (0.44 g, 2 mmol) and potassium chloride (0.019 g, 0.025 mmol). The mixture was heated to dissolve the reactants. The filtered solution was set aside for three months to allow colourless crystals to deposit. The amount of potassium chloride was substantially less than that of the zinc reagent, as more would have led to the formation of a potassium product without any zinc component.

Crystal data

$[ZnKCl(C_{30}H_{30}N_{20}O_{10})(H_2O)_4]$-$[ZnCl_4]\cdot 5H_2O$
$M_r = 1339.97$
Orthorhombic, *Pnma*
$a = 13.3944\ (5)$ Å
$b = 13.6063\ (6)$ Å
$c = 26.354\ (1)$ Å
$V = 4803.0\ (3)$ Å^3
$Z = 4$
$D_x = 1.853$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 7889 reflections
$\theta = 2.3$–$28.0°$
$\mu = 1.46$ mm^{-1}
$T = 295\ (2)$ K
Block, colourless
$0.40 \times 0.22 \times 0.15$ mm

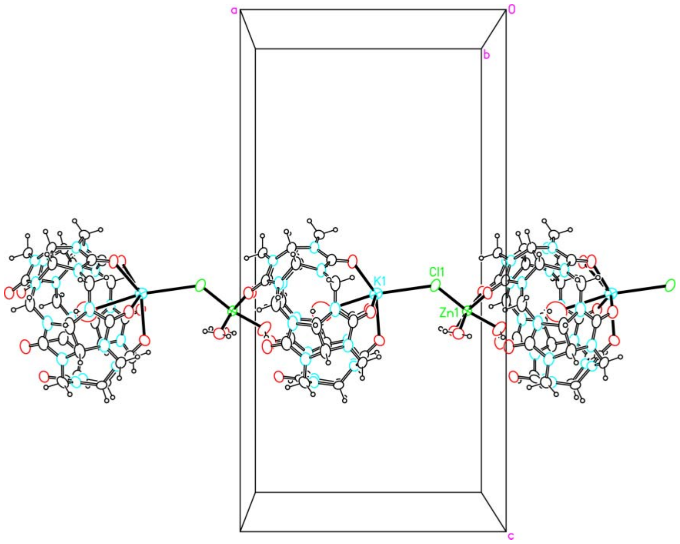


Figure 2
ORTEPII plot (Johnson, 1976) of the polycationic $[(C_{30}H_{30}N_{20}O_{10})(H_2O)K]^+[(H_2O)_3ClZn]^+$ chain.

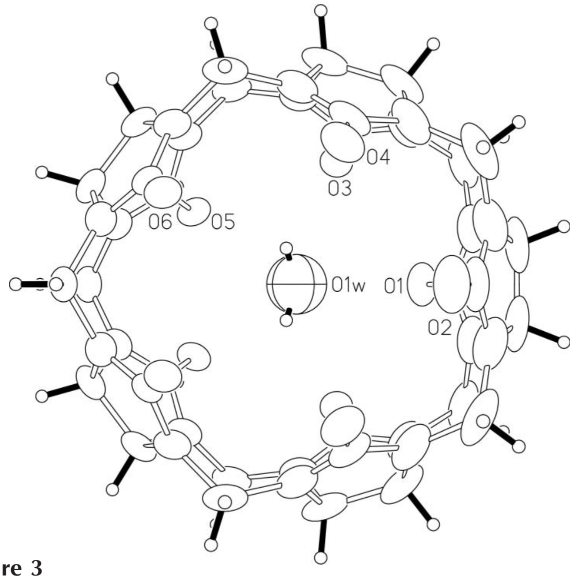


Figure 3
ORTEPII plot (Johnson, 1976) of the $C_{30}H_{30}N_{20}O_{10}$ cryptand encapsulating a water molecule.

Data collection

Bruker SMART APEX area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2002)
$T_{min} = 0.538$, $T_{max} = 0.811$
39 547 measured reflections
5703 independent reflections
4854 reflections with $I > 2\sigma(I)$
$R_{int} = 0.028$
$\theta_{max} = 27.5°$
$h = -16 \rightarrow 17$
$k = -17 \rightarrow 17$
$l = -33 \rightarrow 33$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.044$
$wR(F^2) = 0.134$
$S = 1.03$
5703 reflections
424 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0887P)^2 + 2.5416P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.80$ e Å^{-3}
$\Delta\rho_{min} = -0.38$ e Å^{-3}

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Table 1
Selected geometric parameters (Å, °).

Zn1—O6^{i}	2.178 (2)	Zn2′—Cl3′	2.239 (8)
Zn1—O1*w*	2.092 (3)	Zn2′—Cl4′	2.224 (6)
Zn1—O2*w*	2.082 (2)	K1—O1	2.669 (3)
Zn1—Cl1	2.333 (1)	K1—O3	2.752 (2)
Zn2—Cl2	2.251 (6)	K1—O5	2.740 (2)
Zn2—Cl3	2.311 (4)	K1—O3*w*	3.264 (7)
Zn2—Cl4	2.268 (5)	K1—Cl1	3.416 (1)
Zn2′—Cl2′	2.316 (6)		
O6^{i}—Zn1—O6^{ii}	95.8 (1)	Cl2′—Zn2′—Cl4′	112.8 (3)
O6^{i}—Zn1—O1*w*	79.9 (1)	Cl3′—Zn2′—Cl4′	105.9 (4)
O6^{i}—Zn1—O2*w*	82.4 (1)	O1—K1—O3	70.3 (1)
O6^{i}—Zn1—O2*w*^{iii}	168.3 (1)	O1—K1—O5	134.9 (1)
O6^{i}—Zn1—Cl1	93.6 (1)	O1—K1—O3*w*	76.0 (2)
O1*w*—Zn1—O2*w*	88.3 (1)	O1—K1—Cl1	94.0 (1)
O1*w*—Zn1—Cl1	170.3 (1)	O3—K1—O3^{iii}	136.4 (1)
O2*w*—Zn1—Cl1	98.1 (1)	O3—K1—O5	70.0 (1)
O2*w*—Zn1—O2*w*^{iii}	96.9 (1)	O3—K1—O5^{iii}	133.1 (1)
Cl2—Zn2—Cl2^{iv}	113.5 (5)	O3—K1—O3*w*	76.7 (1)
Cl2—Zn2—Cl3	102.3 (2)	O3—K1—Cl1	100.2 (1)
Cl2—Zn2—Cl4	106.8 (2)	O5—K1—O5^{iii}	67.0 (1)
Cl3—Zn2—Cl4	125.3 (3)	O5—K1—O3*w*	75.0 (1)
Cl2′—Zn2′—Cl2′^{iv}	106.6 (4)	O5—K1—Cl1	113.2 (1)
Cl2′—Zn2′—Cl3′	109.3 (3)	O3*w*—K1—Cl1	169.9 (2)

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

Table 2
Hydrogen-bonding geometry (Å, °).

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1*w*—H1*w*1···O4^{i}	0.85 (1)	1.87 (1)	2.678 (2)	159 (2)
O2*w*—H2*w*1···O4^{i}	0.85 (1)	2.25 (2)	2.963 (3)	141 (2)
O2*w*—H2*w*2···O4*w*	0.85 (1)	1.82 (1)	2.659 (3)	169 (3)
O4*w*—H4*w*1···O3	0.84 (1)	2.08 (2)	2.853 (3)	154 (4)
O4*w*—H4*w*2···O5*w*	0.84 (1)	1.83 (1)	2.672 (5)	176 (4)
O5*w*—H5*w*2···Cl2^{iv}	0.86 (1)	2.52 (5)	3.16 (1)	132 (5)
O5*w*—H5*w*2···Cl2′^{iv}	0.86 (1)	2.70 (5)	3.320 (9)	130 (5)
O5*w*—H5*w*1···Cl3	0.86 (1)	2.43 (4)	3.236 (6)	155 (7)
O5*w*—H5*w*1···Cl3′	0.86 (1)	2.29 (4)	3.083 (9)	154 (7)
O6*w*—H6*w*1···O5	0.86 (1)	2.32 (3)	3.028 (4)	139 (4)

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

The $[ZnCl_4]^{2-}$ anion, which lies on a mirror plane, is disordered over two sites; as the site-occupation factors refined to a 50:50 ratio, they were constrained to 1/2. Zn—Cl distances for this anion were restrained to be equal for both disordered parts, with an effective s.u. of 0.02 Å. The carbon-bound H atoms were placed in calculated positions, with C—H distances constrained to 0.98 Å for the methine H atoms and to 0.97 Å for the methylene H atoms, and were included in the refinement with $U_{\rm iso}$(H) values set to 1.2 times $U_{\rm eq}$ of the parent atoms in the riding-model approximation. The water H atoms were located in difference maps and refined with distances restrained to O—H = 0.85 (1) Å and H···H = 1.39 (1) Å; their isotropic displacement parameters were refined.

Data collection: *SMART* (Bruker, 2002); cell refinement: *SAINT* (Bruker, 2002); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEPII* (Johnson, 1976); software used to prepare material for publication: *SHELXL97*.

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