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# Triaqua(18-crown-6)calcium(II) tetrachlorozincate(II)

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**Key indicators**

Single-crystal X-ray study
*T* = 293 K
Mean σ(C–C) = 0.005 Å
*R* factor = 0.040
*wR* factor = 0.108
Data-to-parameter ratio = 22.4

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



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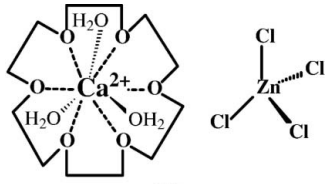
Diffusion of diethyl ether into a reaction mixture of zinc salts, crown ether and calcium chloride in tetrahydrofuran (THF) led to the title compound, $[Ca(C_{12}H_{24}O_6)(H_2O)_3][ZnCl_4]$. The cation is linked to the anion *via* O—H···Cl hydrogen bonding.

## Comment

The ability of 18-crown-6 ether (18-C-6) to form complexes with different metal ions has been widely investigated. We report here the synthesis and crystal structure of the title compound, (I).




(I)

The crystal structure of (I) consists of $[Ca(18—C-6)(H_2O)_3]^{2+}$ cations and $[ZnCl_4]^{2-}$ anions. The structure of the cation is similar to that found in $[Ca(18—C-6)(H_2O)_3][Cu_5I_7]$ (Nurtaeva & Holt, 2002). The crystal structure of the $[ZnCl_4]^{2-}$ anion is in good agreement with that in reported complexes (Jackson *et al.*, 1981; Smolenaers *et al.*, 1981; Otter & Hartshorn, 2004). The cation is linked to the anion *via* O—H···Cl hydrogen bonding (Table 1).

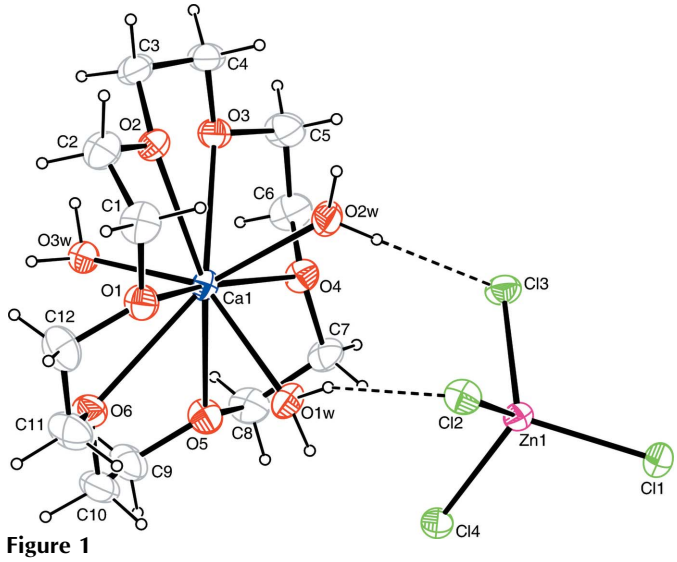



**Figure 1**
The molecular structure of (I) with 30% probability displacement ellipsoids (arbitrary spheres for H atoms). Dashed lines indicate hydrogen bonds.

## Experimental

$ZnCl_2·2H_2O$ (35 mg, 0.2 mmol), 18-C-6 (53 mg, 0.2 mmol) and $CaCl_2$ (22 mg, 0.2 mmol) were added to THF (10 ml) and the reaction mixture was stirred at 333 K for 6 h. The mixture was filtered and the filtrate was reduced to 5 ml in a small tube, which was loaded into a large vial containing 5 ml diethyl ether. The large vial was sealed and left undisturbed at room temperature; colorless crystals of (I) were obtained after 7 d (yield: 76%). Calculated for $C_{12}H_{30}CaCl_4O_9Zn$: C 25.48, H 5.35%; found: C 25.60, H 5.42%.

*Crystal data*

$[Ca(C_{12}H_{24}O_6)(H_2O)_3][ZnCl_4]$
$M_r$ = 565.61
Monoclinic, $P2_1/c$
$a$ = 12.573 (3) Å
$b$ = 9.489 (2) Å
$c$ = 20.546 (5) Å
$\beta$ = 101.262 (3)°
$V$ = 2404.0 (11) Å$^3$
$Z$ = 4
$D_x$ = 1.563 Mg m$^{-3}$
Mo $K\alpha$ radiation
$\mu$ = 1.72 mm$^{-1}$
$T$ = 293 (2) K
Prism, colorless
0.30 × 0.25 × 0.25 mm

*Data collection*

Rigaku Mercury CCD diffractometer
$\omega$ scans
Absorption correction: multi-scan (*CrystalClear*; Rigaku, 2002)
$T_{min}$ = 0.515, $T_{max}$ = 0.655
17093 measured reflections
5468 independent reflections
4955 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.035
$\theta_{max}$ = 27.5°

*Refinement*

Refinement on $F^2$
$R[F^2 > 2\sigma(F^2)]$ = 0.040
$wR(F^2)$ = 0.108
$S$ = 1.00
5468 reflections
244 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.06P)^2 + P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.002
$\Delta\rho_{max}$ = 0.34 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.39 e Å$^{-3}$

**Table 1**
Hydrogen-bond geometry (Å, °).

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| O1$W$—H1$E$···Cl4 | 0.93 | 2.34 | 3.227 (2) | 158 |
| O1$W$—H1$F$···Cl2 | 0.86 | 2.48 | 3.306 (2) | 161 |
| O2$W$—H2$E$···Cl3 | 0.92 | 2.35 | 3.236 (2) | 163 |
| O2$W$—H2$F$···Cl2$^{i}$ | 0.99 | 2.33 | 3.285 (2) | 163 |
| O3$W$—H3$E$···Cl4$^{ii}$ | 0.85 | 2.67 | 3.410 (2) | 146 |
| O3$W$—H3$F$···Cl1$^{ii}$ | 0.96 | 2.22 | 3.157 (2) | 165 |

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

Water H atoms were located in a difference Fourier map and refined as riding in their as-found relative positions, with $U_{iso}$(H) = 1.5$U_{eq}$(O). Other H atoms were placed in calculated positions, with C—H = 0.97Å, and refined in riding mode, with $U_{iso}$(H) = 1.2$U_{eq}$(C).

Data collection: *CrystalClear* (Rigaku, 2002); cell refinement: *CrystalClear*; data reduction: *CrystalClear*; program(s) used to solve structure: *SHELXTL* (Siemens, 1994); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

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