Syntheses, Crystal Structures and Magnetic Behaviour of
[Li(12-crown-4)₂][Li(12-crown-4)(OH₂)]₂[Nb₆Cl₁₈],
[Li(15-crown-5)₂(OH₂)]₃[Nb₆Cl₁₈] and [(18-crown-6)₂(O₂H₅)]₃[Nb₆Cl₁₈]

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Niobium Cluster, Chloride, Crown Ether

The title compounds were prepared through reactions of Li₂Nb₆Cl₁₆ with the corresponding crown ethers in acetone. All three compounds were obtained as dark brown crystals. Their structures were solved with the means of single-crystal X-ray diffraction.

[Li(12-crown-4)₂][Li(12-crown-4)(OH₂)]₂[Nb₆Cl₁₈]: space group $P\bar{2}_1/n$, $Z=2$, $a=1320.4(1)$, $b=1879.1(1)$, $c=1321.7(1)$ pm, $\beta=92.515(6)^\circ$, $R_1=0.0297$ ($I > 2\sigma(I)$). The crystal structure contains Li⁺ sandwiched by two 12-crown-4-ethers plus Li⁺ coordinated by one 12-crown-4-ether and one water molecule.

[Li(15-crown-5)₂(OH₂)]₃[Nb₆Cl₁₈]: space group $R\bar{3}$, $Z=3$, $a=b=2081.7(1)$, $c=1991.7(1)$ pm, $R_1=0.0395$ ($I > 2\sigma(I)$). In the crystal structure Li⁺ and one water molecule are sandwiched by two 15-crown-5-ethers.

[(18-crown-6)₂(O₂H₅)]₃[Nb₆Cl₁₈]: space group $P\bar{1}$, $Z=1$, $a=1405.1(1)$, $b=1461.1(2)$, $c=1492.2(2)$ pm; $\alpha=98.80(1)^\circ$, $\beta=98.15(1)^\circ$, $\gamma=97.41(1)^\circ$, $R_1=0.0538$ ($I > 2\sigma(I)$). H₅O₂⁺ was found in the structure refinement sandwiched between two 18-crown-6-ethers.

All compounds reported contain [Nb₆Cl₁₈] clusters with Nb-Nb distances between 299 and 301 pm. The paramagnetic behaviour expected for [Nb₆Cl₁₈]³⁻ in all three compounds was confirmed by magnetic measurements.