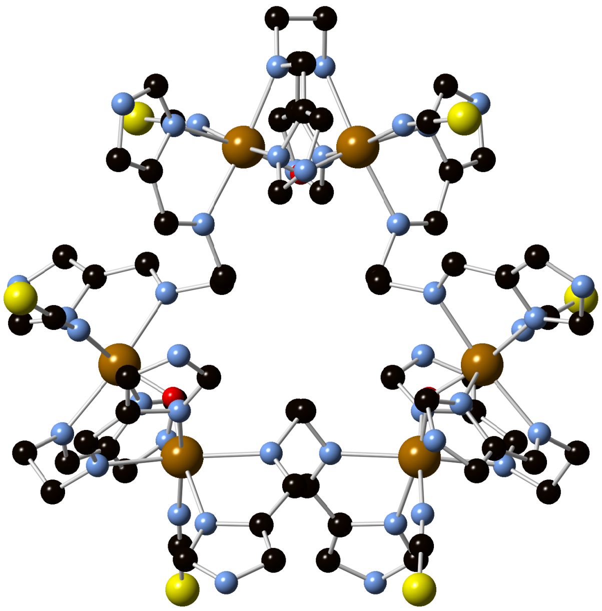


Fig. S1 Molecular structure of an hexanuclear $[Fe_6(\mu\text{-}L_1)_6(\mu\text{-}O)_3(NCS)_6]^{6+}$ complex of $[Fe_6(\mu\text{-}L_1)_6(\mu\text{-}O)_3(NCS)_6](NO_3)_6(CH_3OH)_3(H_2O)$ (**4**) (iron (brown), sulfur (yellow), oxygen (red), nitrogen (blue), carbon (black)). The complex is generated from half of the molecule, which is crystallographically independent, through a 2-fold axis.

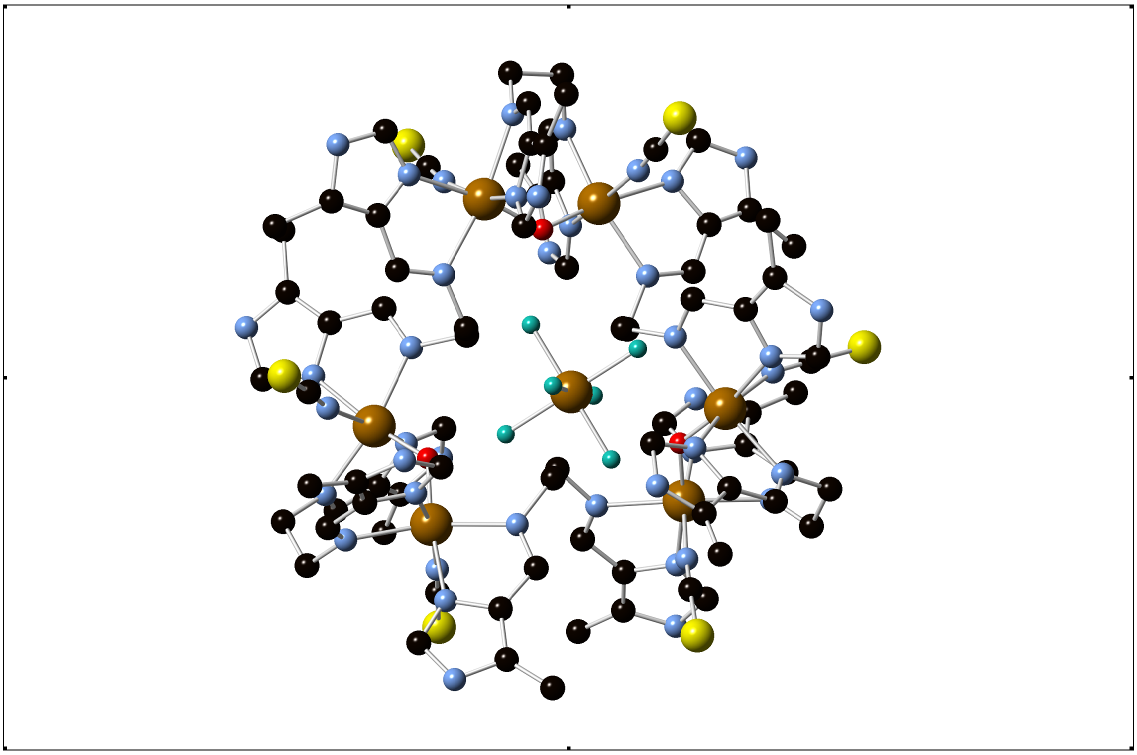
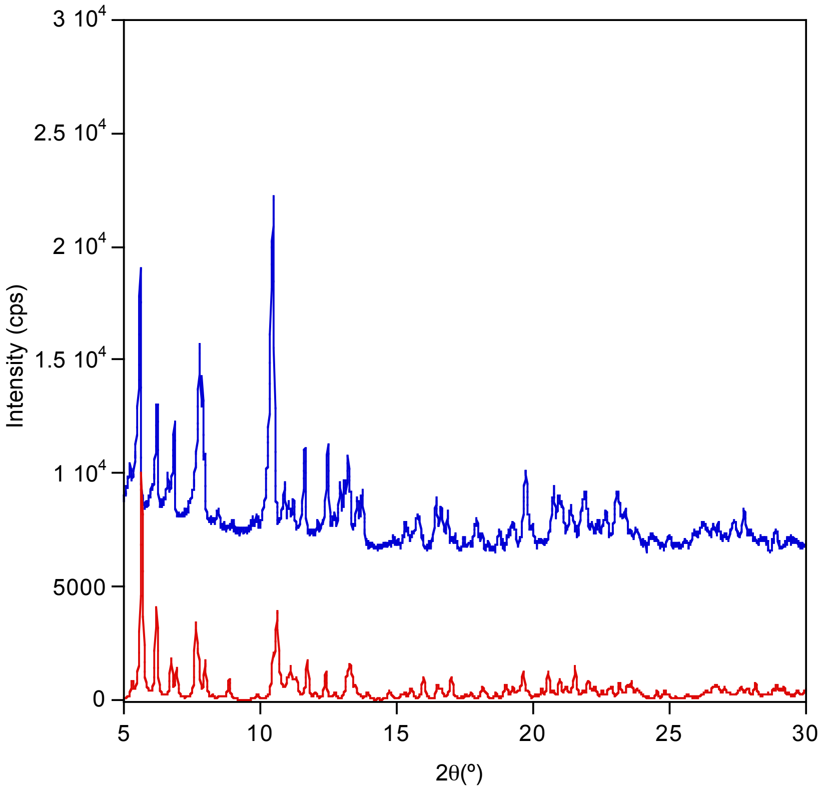


Fig. S2 Molecular structure of an hexanuclear $[Fe_6(\mu\text{-}L_2)_6(\mu\text{-}O)_3(NCS)_6]^{6+}$ complex with a $[FeF_6]^-$ anion close to the center of the ring of $[Fe_6(\mu\text{-}L_2)_6(\mu\text{-}O)_3(NCS)_6](FeF_6)_{0.5}(NCS)_{4.5}(CH_3OH)_2$_(solvate) (**5**) (iron (brown), sulfur (yellow), oxygen (red), nitrogen (blue), carbon (black), fluoride (green)). The FeF_6^{3-} complex is generated from half of the molecule, which is crystallographically independent, through a 2-fold axis linking F3, F4 and Fe7 atoms.



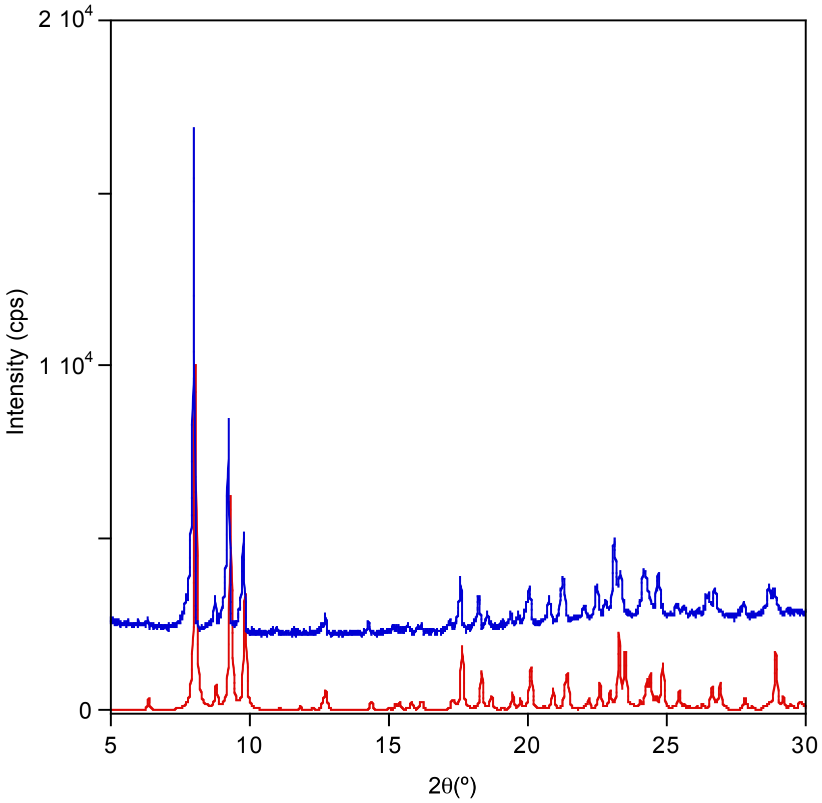


Fig. S3 Powder X-ray diffraction patterns (blue) and simulated patterns (red) of **2** (top) and **3** (bottom).

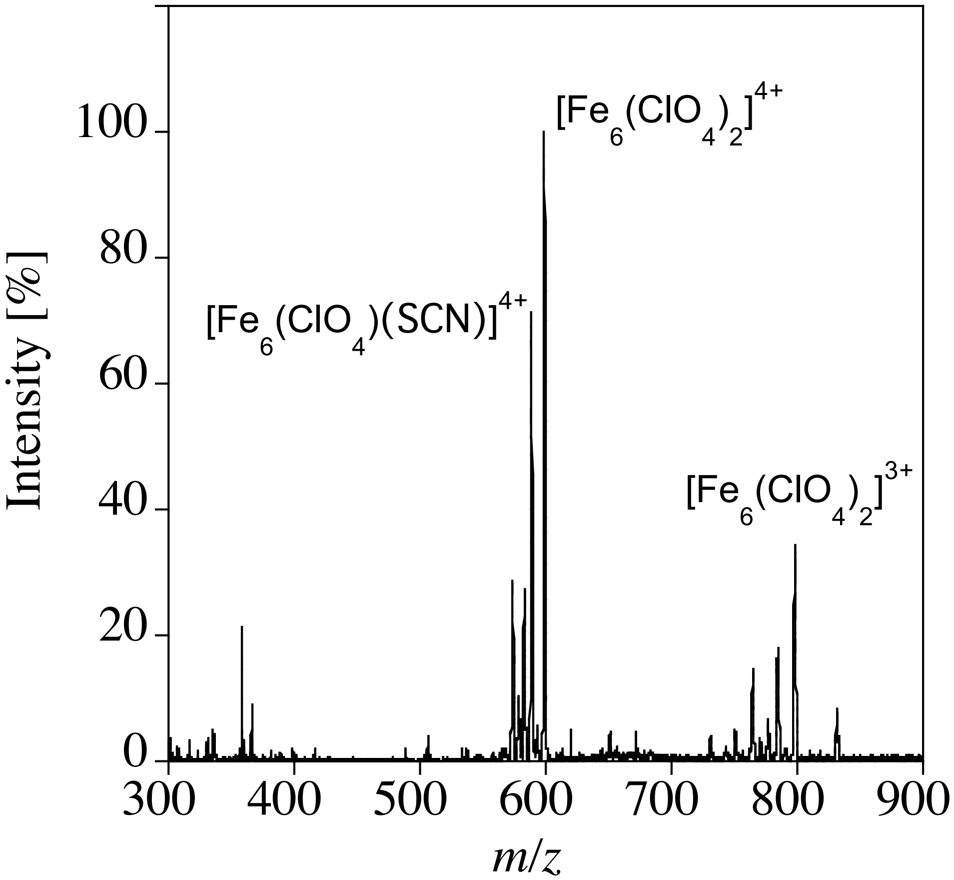


Fig. S4 ES-MS of **2** in methanol.

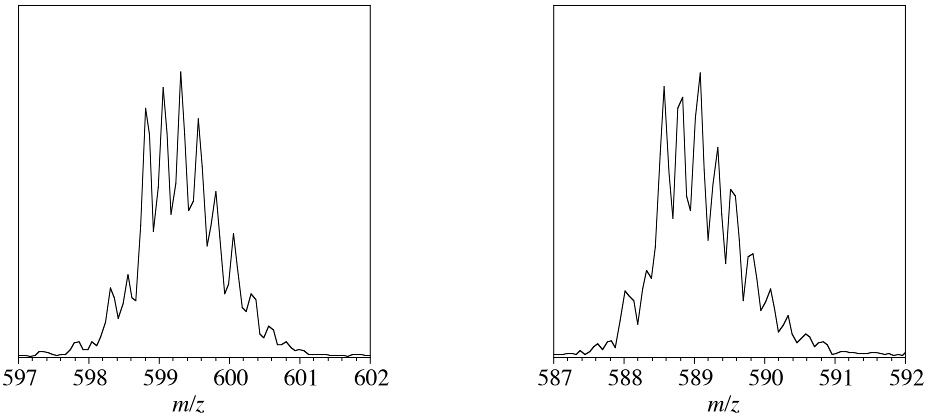


Fig. S5 ES-MS of **2** in methanol (isotopic resolution of the most intense peaks).