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Accessary Publication

Copper(I) Template Synthesis of a 2,2'-Bipyridine Derived 2-Catenane: Synthetic, Modelling and X-ray Studies

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MS-ESI Spectra

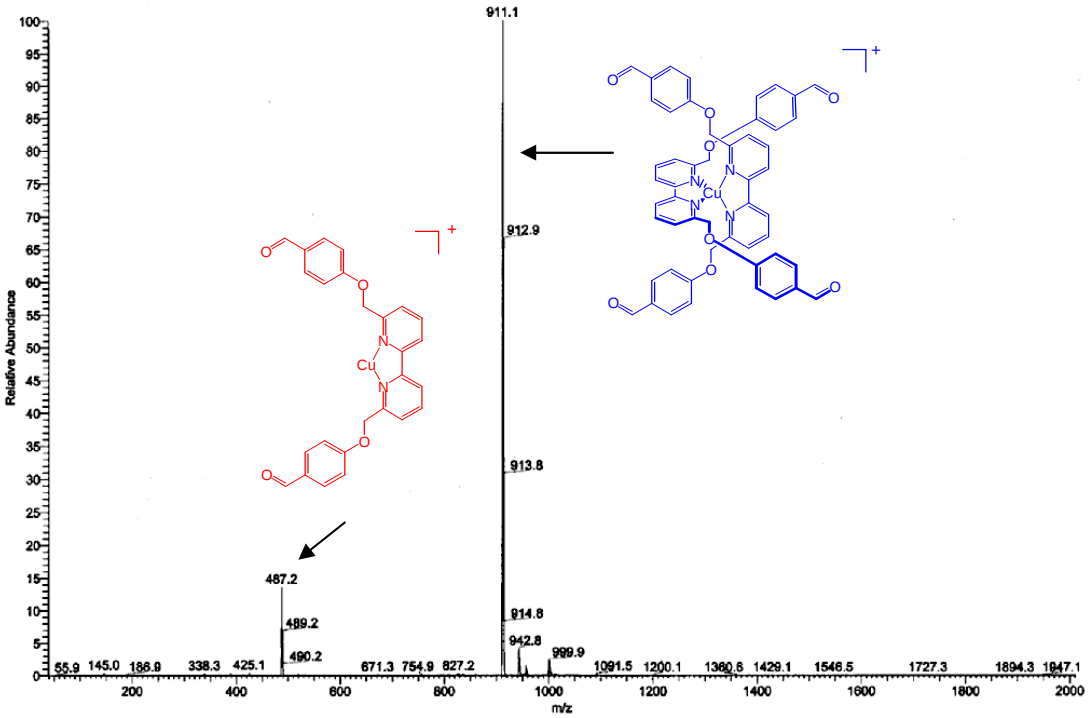
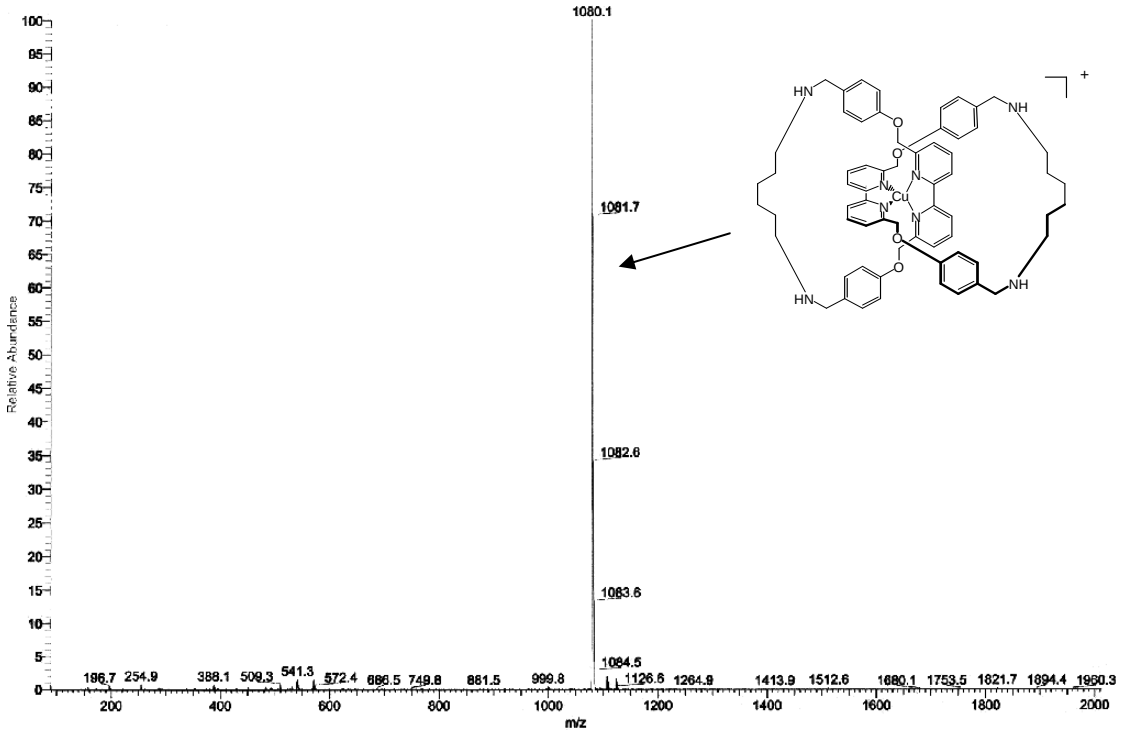
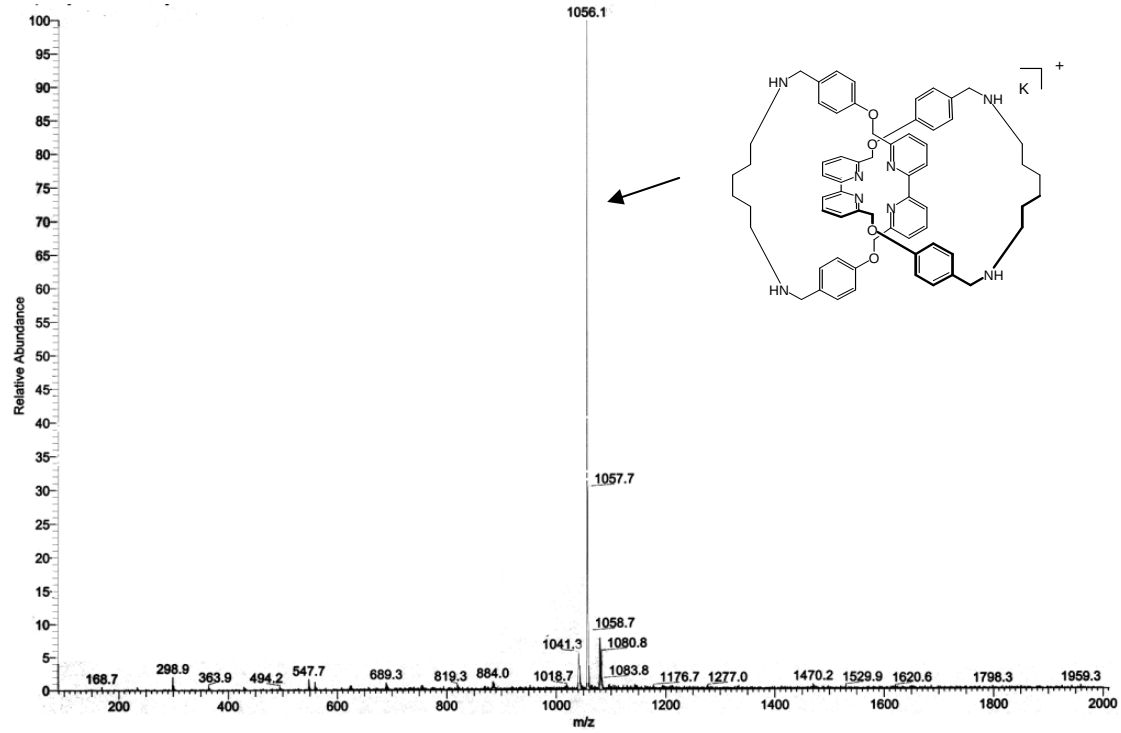


Fig. AP 1. The electrospray mass spectrum of the 2 : 1, **2** : $[Cu(CH_3CN)_4]PF_6$ reaction mixture.



(a)



(b)

Fig. AP2. The electrospray mass spectrum of the [2]-catenane **4**. **(a)** as its Cu(I) complex and **(b)** with the copper removed following reaction with KCN.

^{1}H NMR Spectra

^{1}H NMR spectra for dialdehyde **2** and [Cu(**2**)$_2$]PF_6 are given in Fig. AP3(a) and (b) respectively. There is a substantial downfield shift of the *para* coupled protons H_e and H_f, upon complexation to Cu(I) ($\Delta\delta_H$ 0.38, 0.82 ppm), in keeping with shielding of these aromatic protons due to the proximity to the bipyridine moiety, consistent with the preorganisation arguments discussed in the main text.

The ^{1}H NMR spectra of the copper complex of the catenane **4**, [Cu(**4**)]PF_6, and free **4** are given in Fig. AP3(c) and (d), respectively.

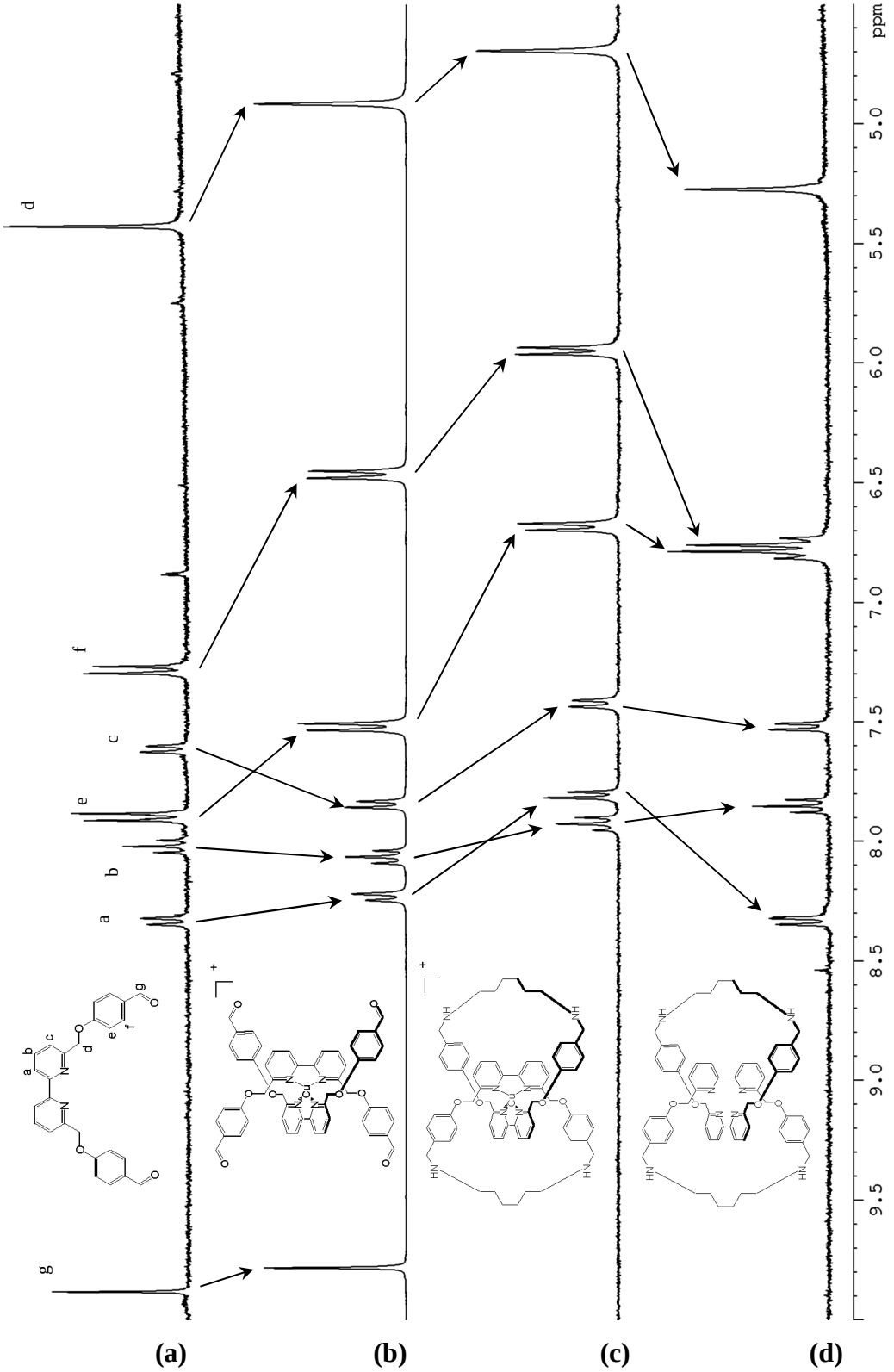


Fig. AP3. The ^{1}H NMR spectra of **(a)** the dialdehyde **2**, **(b)** the Cu(I) complex of **2**, **(c)** the copper containing [2]-catenane **4** and **(d)** the free catenane **4**.

X-ray Hydrogen Bond Network

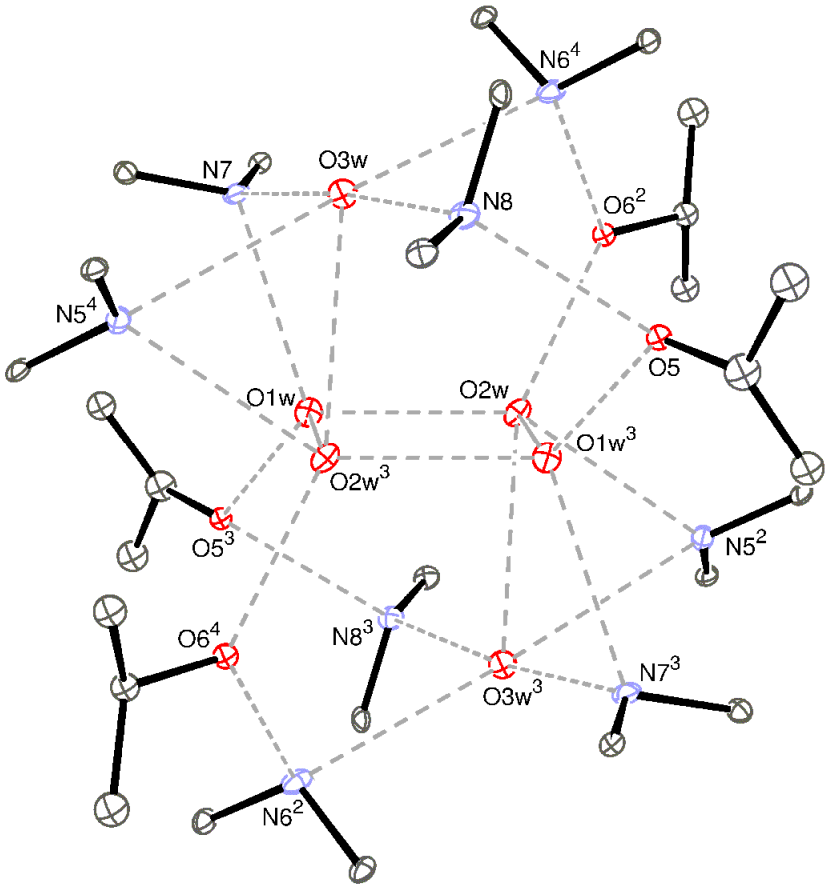


Fig. AP4. The hydrogen bond network (dashed lines) in the crystal lattice of [Cu(**4**)]PF_6)·$(iPrOH)_{1.5}$·$(H_2O)_{2.75}$ involving the water molecules, the isopropanol solvate molecules and the amino nitrogen atoms of the ligands. Superscripts indicate the symmetry operation used to generate equivalent atom positions (1: x, y, z. 2: -x + ½, y + ½, -z + ½. 3: -x, -y, -z. 4: x + ½, -y + ½, z + ½).