

## Electronic Supplementary Information

### Heterometallic coordination polymers based on homo- and heteroleptic Au(III) dithiolene complexes

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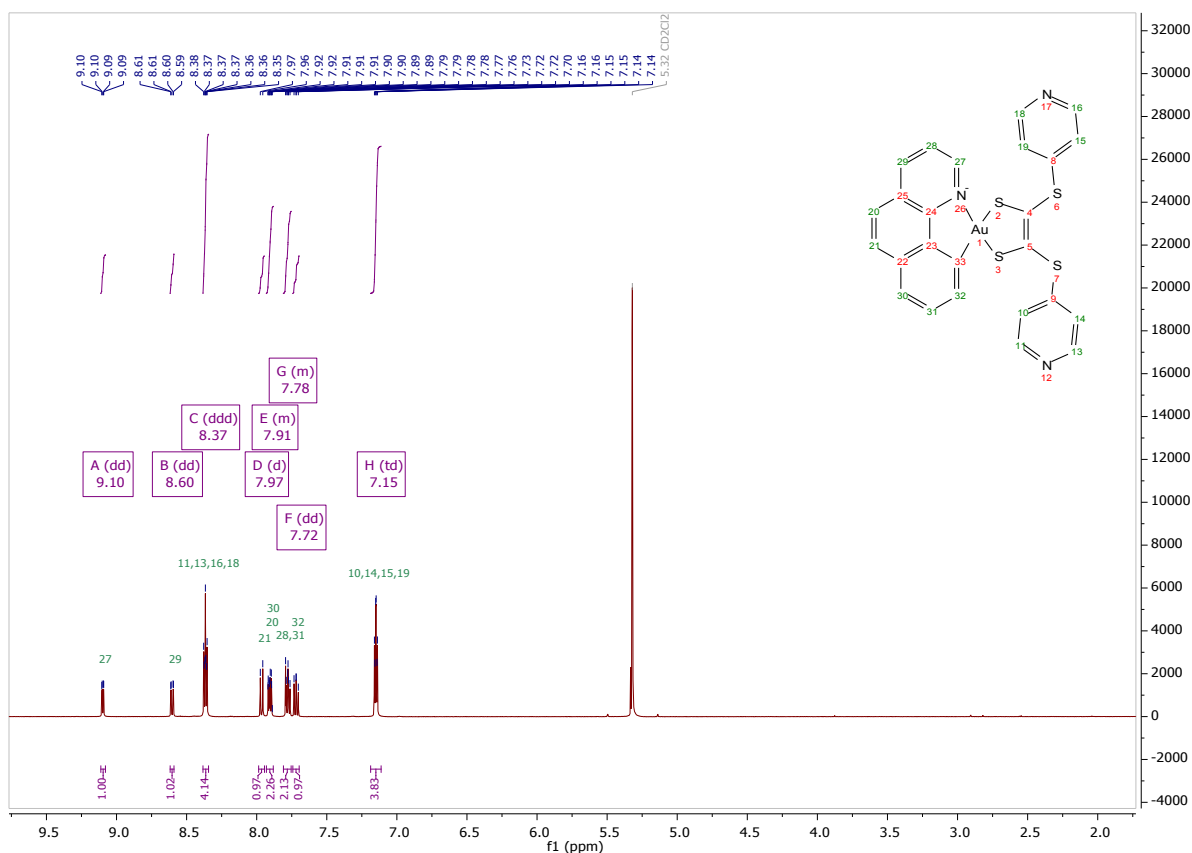
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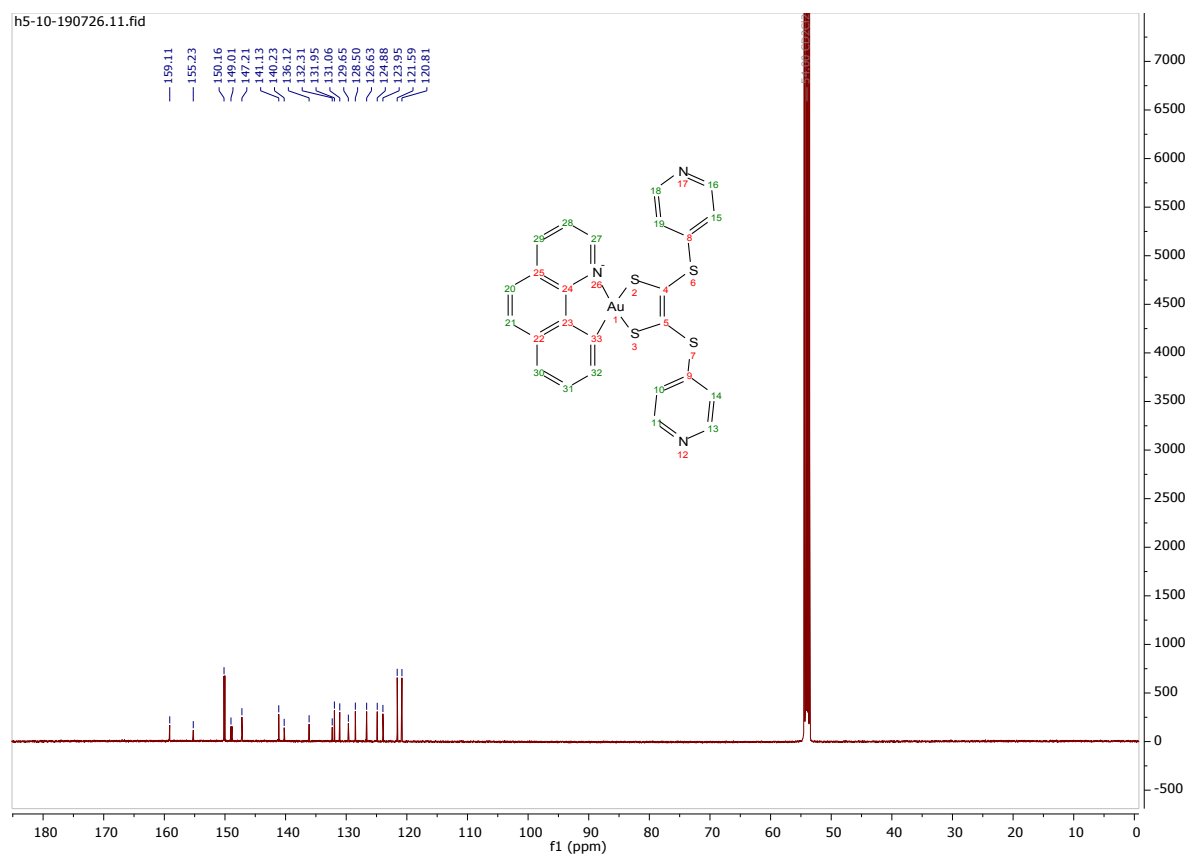
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$^1\text{H}$ - and  $^{13}\text{C}$ -NMR spectra were recorded at 25 °C on a Bruker AV500 (500 MHz) spectrometer with the deuterated solvent as the internal reference. NMR chemical shifts and  $J$  values are given in parts per million (ppm) and in Hertz, respectively.

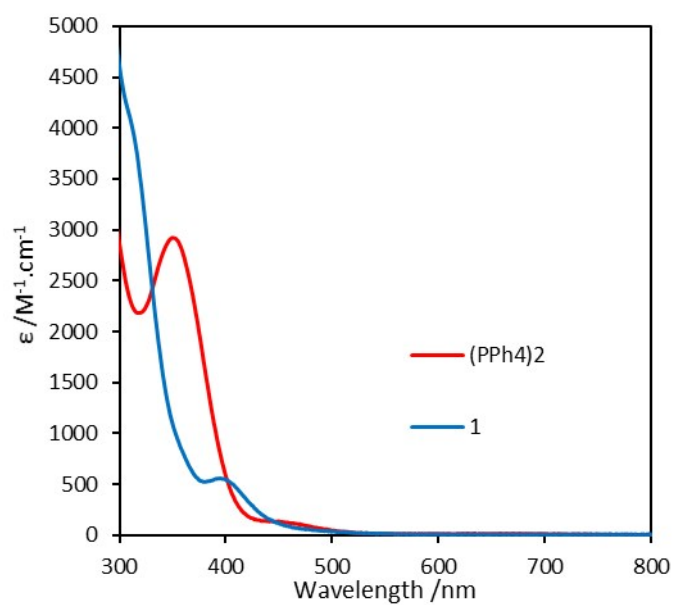


**Fig. ESI1**  $^1\text{H}$ -NMR spectrum of heteroleptic complex **1** in  $\text{CD}_2\text{Cl}_2$ .

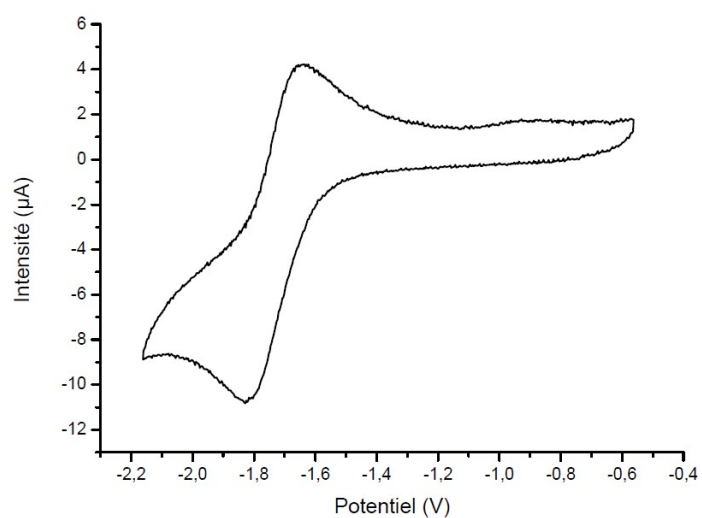


**Fig. ESI2**  $^{13}\text{C}$ -NMR spectrum of heteroleptic complex 1 in  $\text{CD}_2\text{Cl}_2$ .

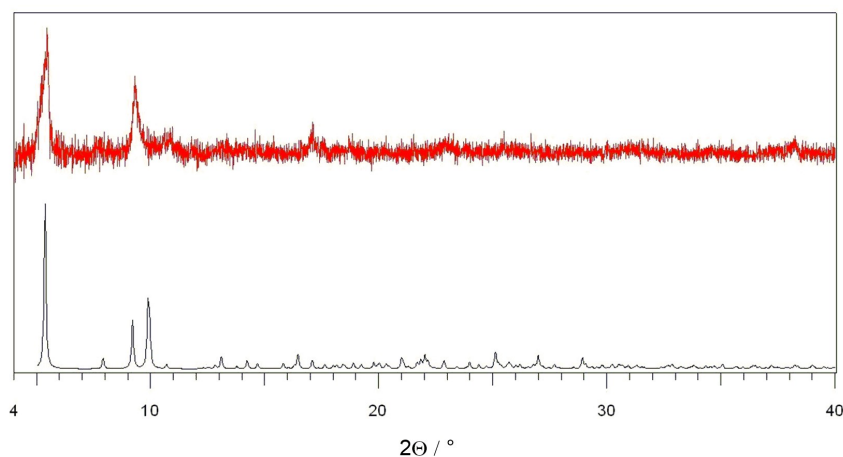




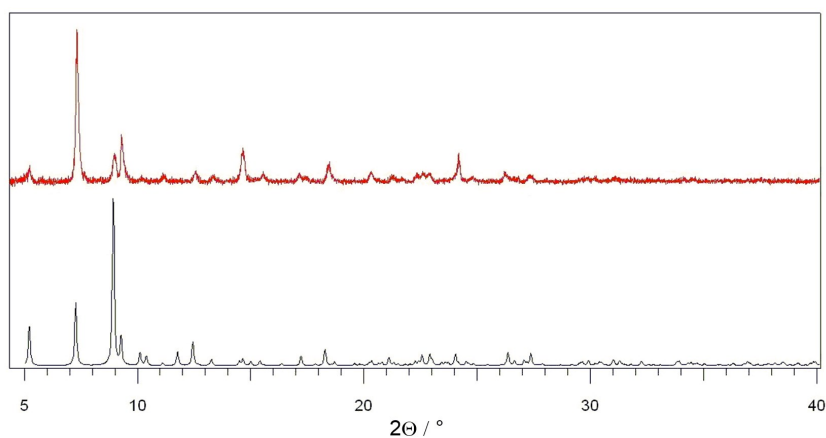
**Fig. ESI5** UV-Visible spectrum of complexes **1** and (PPh<sub>4</sub>)**2** in DMF at room temperature.



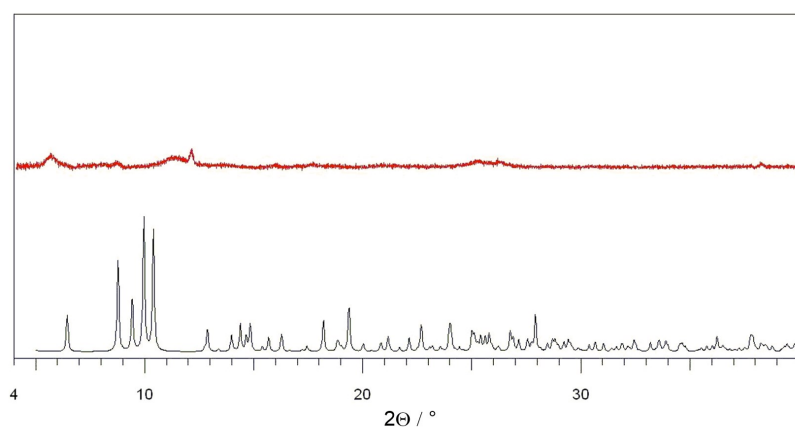
**Fig. ESI6** Cyclic voltammogram of (PPh<sub>4</sub>)**2** in MeCN (0.1 M *n*-Bu<sub>4</sub>NPF<sub>6</sub>) vs Fc/Fc<sup>+</sup>, scan rate: 200 mv.s<sup>-1</sup>.



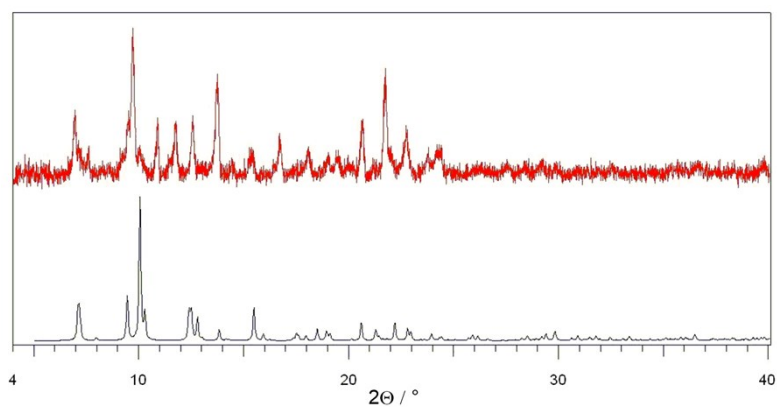
**Fig. ESI7** Simulated (black) and experimental (red) PRXD pattern for CP **5**. Broadening and weakening of the diffraction peaks result from solvent loss.



**Fig. ESI8** Simulated (black) and experimental (red) PRXD pattern for CP **6**. Difference in peak intensity result from preferential orientation.



**Fig. ESI9** Simulated PRXD pattern for CP **7** (black) and experimental pattern (red) obtained upon analyzing the batch containing a rapidly desolvating major phase and few crystals of **7**.



**Fig. ESI10** Simulated (black) and experimental (red) PRXD pattern for CP **8**. Broadening and weakening of the diffraction peaks result from solvent loss.