Interconversion of cages and coordination networks *via* conformational change of a semi-rigid ligand

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Supplementary information

Synthesis of 1,4-bis((3,5-dimethylisoxazol-4-yl)methyl)benzene (bisox)

Hydroxylamine hydrochloride (2.11 g, 30 mmol), dissolved in 1 N NaOH solution (20 cm^3, 20 mmol), was added to 3,3'-(1,4-phenylenebis(methylene))bis(4-hydroxypent-3-en-2-one)[S1] (4.08 g, 13.5 mmol) suspended in EtOH (50 cm^3) at room temperature. The mixture was heated at reflux for 3 h and the solvents removed under reduced pressure. The residue was suspended in H_2O (100 cm^3) and the pH adjusted to 7. The solid material was collected by filtration, washed with H_2O (3 × 20 cm^3) and crystallised from hot EtOH/H_2O. Yield 3.59 g (89 %). Found: C, 72.9; H, 6.78; N, 9.31. $C_{18}H_{20}N_2O_2$ requires C, 72.9; H, 6.81; N, 9.46. δ_H (300 MHz; $CDCl_3$) 2.06 (6 H, s), 2.28 (6 H, s), 3.63 (4 H, s), 7.06 (4 H, s). δ_C (75.5 MHz, $CDCl_3$) 10.27, 10.96, 27.67, 112.26, 128.25, 136.95, 159.86, 165.32. *m/z* (ESI) 297.1586 ($[M + H]^+$. $[C_{18}H_{20}N_2O_2 + H]^+$ requires 297.1603).

Crystal structure of bisox

$C_{18}H_{20}N_2O_2$, M = 296.36, monoclinic, $P2_1/n$, a = 7.0310(3)Å, b = 14.2330(6) Å c = 8.3650(5)Å, b = 100.882(2)°, U = 822.05(7) $Å^3$, T = 150 K, Z = 2, 7770 reflections collected of which 1877 are independent [R(int) = 0.0791]. $R1$ = 0.0546, w$R2$ = 0.1310 for 885 data with $I > 2\sigma(I)$. GOF = 1.015 based on F^2.

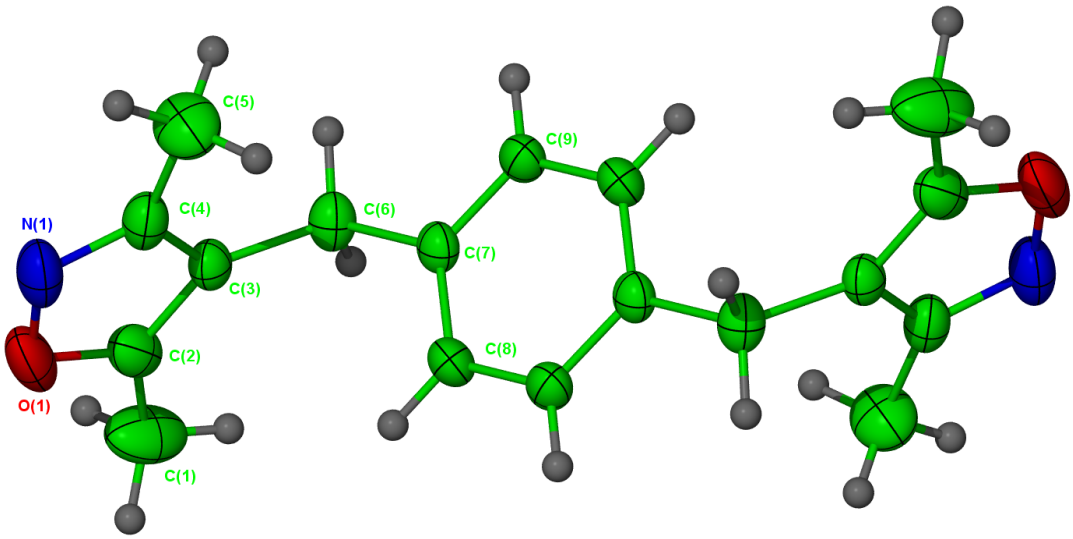

Fig S1. The molecular structure of bisox.

Syntheses of 1 and 2.

Details of the syntheses are reported in the main text. CAUTION – Although no problems were encountered during this study, metal perchlorate salts are potentially explosive and should be handled with great care.

Conversion of 2 to 1

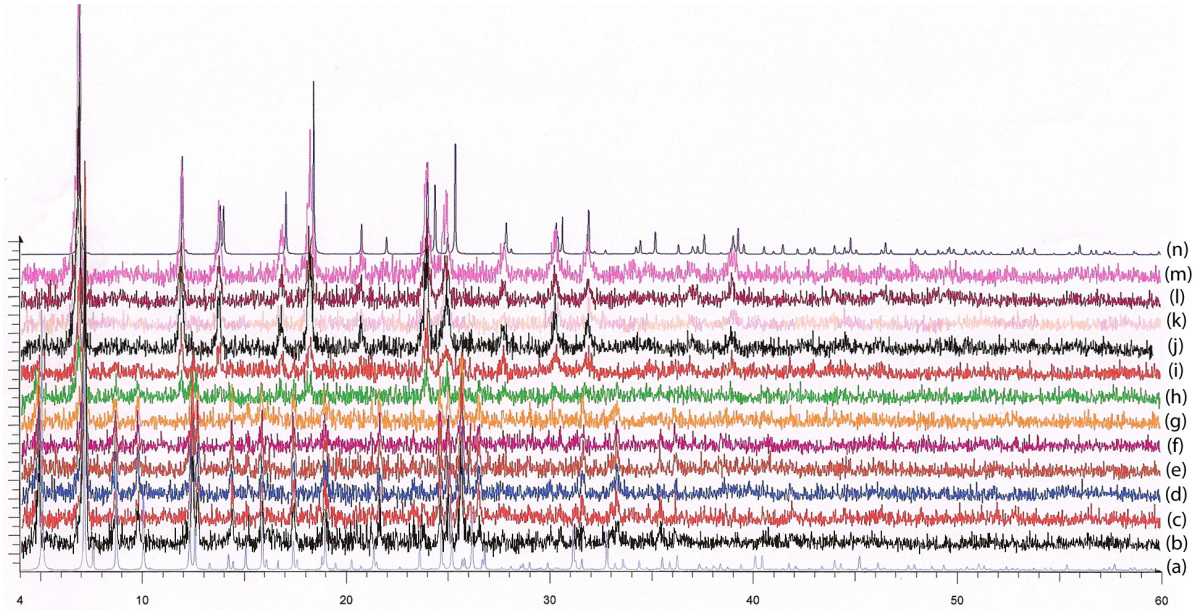

Fig. S2. Powder X-ray diffraction patterns for the conversion of **2** to **1**: (a) Simulated pattern for **2**, (b) **2**, (c) **2** under vacuum for 4 h, (d) **2** heated at 50°C under vacuum for 1 h, (e) 2 h, (f) 3 h, (g), 4 h, (h) heated at 75 °C under vacuum for 1 h, (i) 2 h, (j) 3 h, (k) 4 h, (l) heated at 100 °C under vacuum for 1 h, (m) 2 h, (n) simulated pattern for **1**.

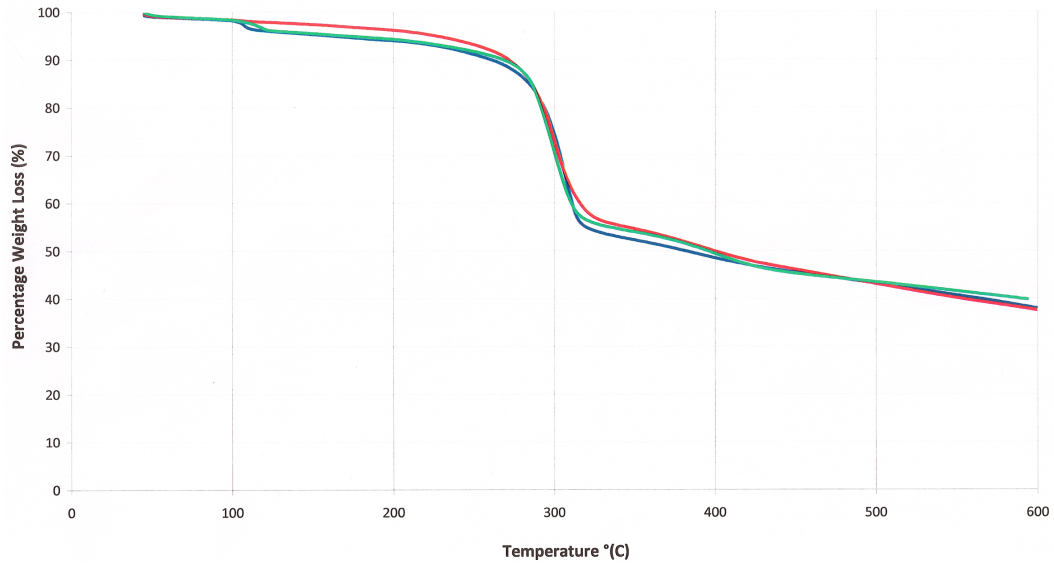

Fig. S3. Thermogravimetric analysis for **1** and **2**. The blue and green traces are for different samples of **2**, whereas the red trace is for a sample of **1**, formed by heating **2**.

Reference

S1. D. F. Martin, W. C. Fernelius and M. Shamma, *J. Am. Chem. Soc.*, 1959, **81**, 130.