Supplementary Information



Fig. S1 ORTEP view of $[{Rh_2(pram)_4}_3(\mu_3-Br)_2] \cdot 6H_2O$ (**5** $\cdot 6H_2O$). Thermal ellipsoids are shown at 50% probability. Water molecules, ethyl hydrogen atoms and some disordered ethyl groups are omitted for clarity. The blue dotted lines show hydrogen bonds from the NH of pram ligands to the O atoms of pram ligands. Symmetry operations *: -x, -y, -z+1; ': -x, -y, -z; ": -x+1, -y+1, -z+1.

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Fig. S2 ORTEP view of $[\{Rh_2(pram)_4\}_3(\mu_3-I)_2]\cdot 6H_2O$ (**6**·6H₂O). Thermal ellipsoids are shown at 50% probability. Water molecules, ethyl hydrogen atoms and some disordered ethyl groups are omitted for clarity. The blue dotted lines show hydrogen bonds from the NH of pram ligands to the O atoms of pram ligands. Symmetry operations *: -x, -y, -z+1; ': -x, -y, -z; ": -x+1, -y+1, -z+1; #: -x, -y+1, -z+1.



Fig. S3 Diffuse reflectance spectra of $1.4H_2O$ (green), $2.10H_2O$ (blue), $3.4H_2O$ (brown), [Rh₂(acam)₄(H₂O)₂]·6H₂O (black) and [Rh₂(acam)₄(H₂O)₂]ClO₄ (red). The spectra were recorded for powder samples ground with MgO powder. The reflectance spectra are given in the form of Kubelka-Munk conversion {log $f(r_{\infty})$ }.^{S1}



Fig. S4 Diffuse reflectance spectra of $4.6H_2O$ (green), $5.6H_2O$ (blue), $6.6H_2O$ (brown), [Rh₂(pram)₄(py]·6H₂O (black) and [Rh₂(pram)₄(H₂O)₂]PF₆ (red). The spectra were recorded for powder samples ground with MgO powder. The reflectance spectra are given in the form of Kubelka-Munk conversion {log $f(r_{\infty})$ }.^{S1}

(S1) P. Kubelka, F. Z. Munk, Tech. Phys. 1931, 12, 593.

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Fig. S5 ORTEP view of $[\{Rh_2(acam)_4\}_3(\mu_3-I)_2]\cdot 10H_2O$ (**3**·10H₂O). Thermal ellipsoids are shown at 30% probability. Methyl hydrogen atoms are omitted for clarity. Disordered O and N atoms of acam ligands O2A, N2A and O2B, N2B are drawn in a ratio of 2:1. Symmetry operations *: 1/3–x, 2/3–y, 2/3–z; ': –x+y, –x, z; ": –y, x–y, z; '*: 1/3+x–y, –1/3+x, 2/3–z; "*: –2/3+y, –1/3–x+y, 2/3–z.



Fig. S6 (a) Weight loss of $2 \cdot 10 H_2 O$ under reduced pressure. (b) Weight increase of the dehydrated sample 2 under moisture saturated argon atmosphere.



Fig. S7 (a) Weight loss of $4.6H_2O$ under reduced pressure. (b) Weight increase of the dehydrated sample 4 under moisture saturated argon atmosphere.