Electronic Supplementary Material (ESI) for RSC Advances.

Supporting Information

Transformations and reductions of γ-octamolybdates with their monomeric and dimeric aminopolycarboxylates

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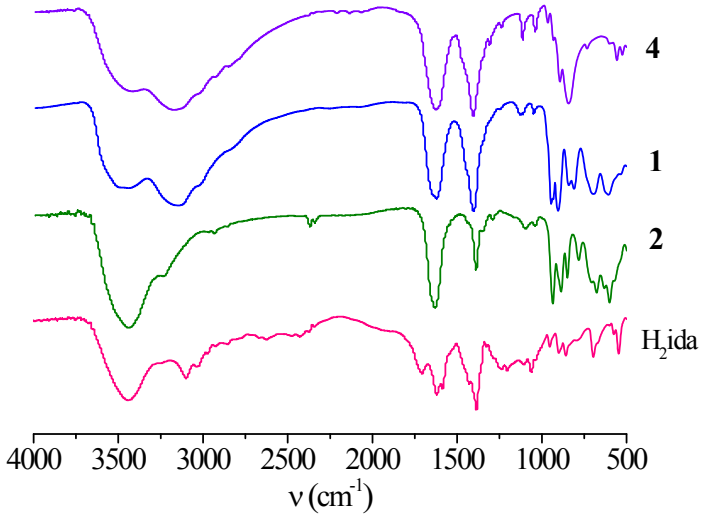
4
1
2
H2ida
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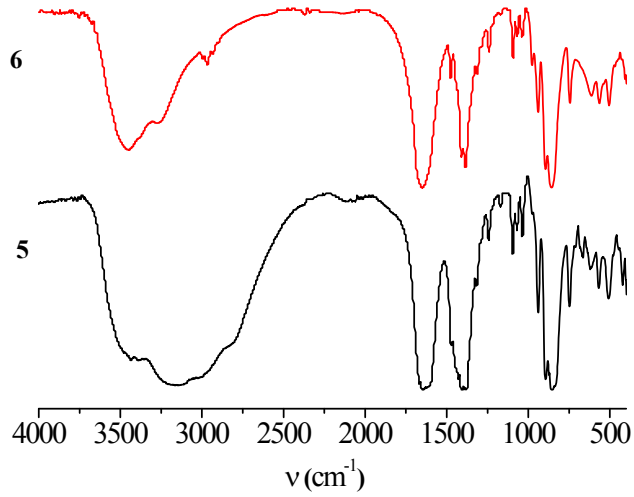
ν (cm-1)

(a)


6
5
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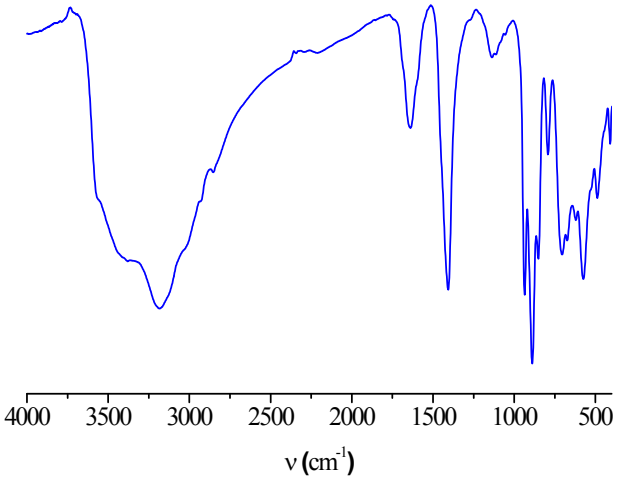
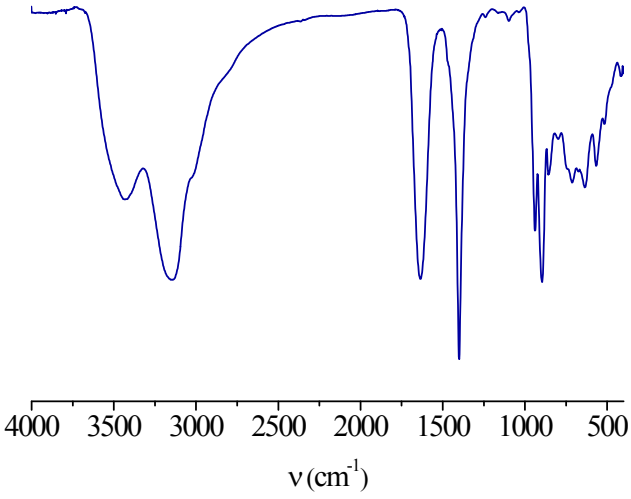
ν (cm-1)

(b)



(c)



(d)

Figure S1. (a) IR spectra of iminodiacetato molybdates: $(NH_4)_4[MoO_3(ida)]_2 \cdot 3H_2O$ (**4**) top; $(NH_4)_8\{\gamma\text{-}Mo_8O_{26}[MoO_3(ida)]_2\} \cdot 6H_2O$ (**1**) middle, and $K_8\{\gamma\text{-}Mo_8O_{24}[MoO_3(ida)]_2\} \cdot 12H_2O$ (**2**) bottom; (b) IR spectra of dimeric propanediaminetetraacetato molybdates: $(NH_4)_5[Mo_2O_6(1,3\text{-pdta})]Cl \cdot 2H_2O$ (**5**), $K_4[Mo_2O_6(1,3\text{-pdta})] \cdot 3H_2O$(**6**); (c) IR spectrum of the complex $(NH_4)_{6n}(Mo_8O_{27})_n \cdot 4nH_2O$ (**7**). (d) IR spectrum of the complex $(NH_4)_{4n}(Mo_8O_{26})_n$(**8**).

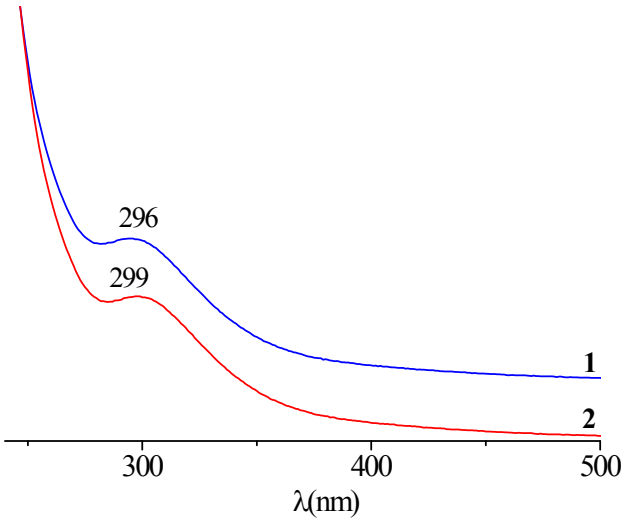


Figure S2. UV spectra of $(NH_4)_8\{\gamma\text{-}Mo_8O_{26}[MoO_3(ida)]_2\}\cdot 6H_2O$ (**1**) and $K_8\{\gamma\text{-}Mo_8O_{26}[MoO_2(ida)]_2\}\cdot 12H_2O$ (**2**).

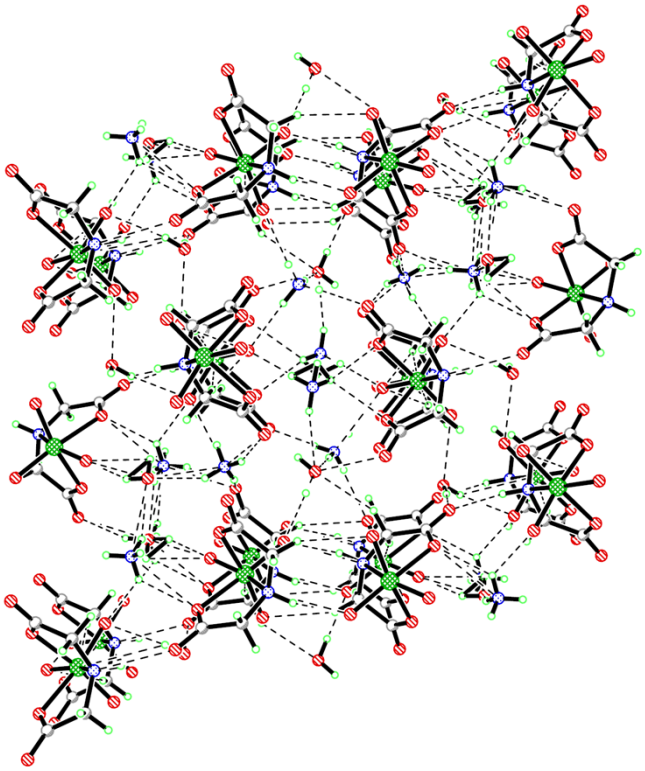


Figure S3. Perspective view of the 3D structure of $(NH_4)_4[MoO_3(ida)]_2 \cdot 3H_2O$ (**4**).

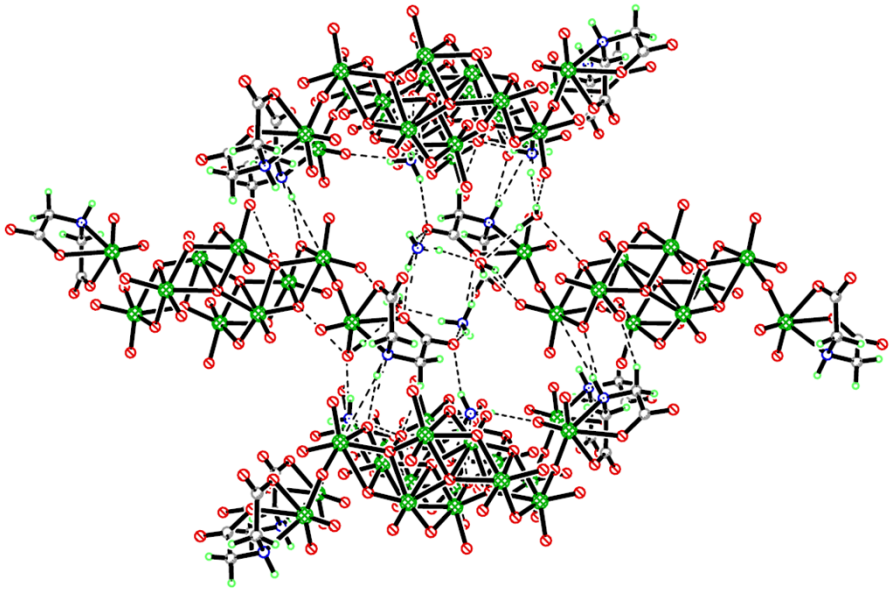


Figure S4. Perspective view of the 2D structure of $(NH_4)_8\{\gamma\text{-}Mo_8O_{26}[MoO_3(ida)]_2\} \cdot 6H_2O$ (**1**).

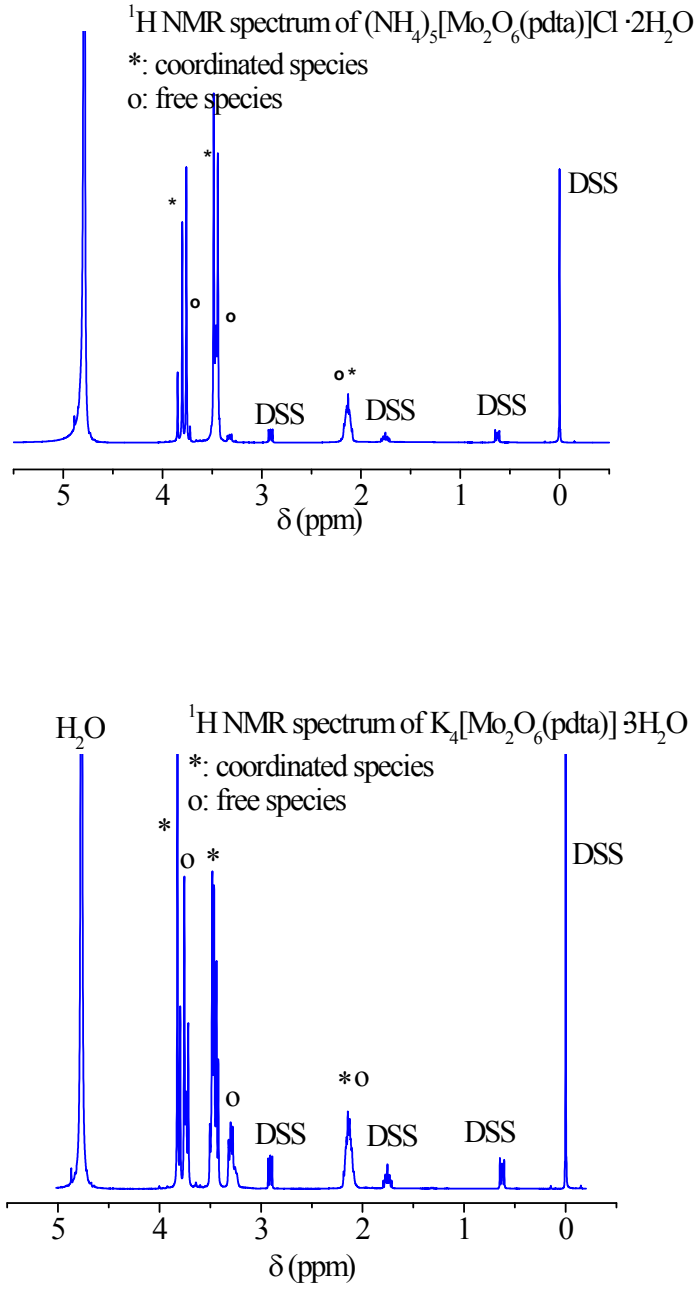


Figure S5. ^{1}H NMR spectra of the complexes $(NH_4)_5[Mo_2O_6(1,3\text{-pdta})]Cl \cdot 2H_2O$ (**5**) and $K_4[Mo_2O_6(1,3\text{-pdta})] \cdot 3H_2O$ (**6**); x indicates resonance signals of the coordinated ligand, o for the free ligand.

Table S1. Bond valence calculations of molybdenum complexes.[18]

Compounds	Mo1/Mo6	Mo2/Mo7	Mo3/Mo8	Mo4/Mo9	Mo5/Mo10
$(NH_4)_8\{\gamma\text{-}Mo_8O_{26}[MoO_3(ida)]_2\}\cdot 6H_2O$ (**1**)	6.142	6.018	5.924	5.865	5.933
$K_8\{\gamma\text{-}Mo_8O_{26}[MoO_2(ida)]_2\}\cdot 12H_2O$ (**2**)	5.270	5.205	5.996	6.024	6.052
$(NH_4)_{8n}\{\gamma\text{-}Mo_8O_{26}[Mo_2O_6(1,3\text{-pdta})]\}_n\cdot 30nH_2O$ (**3**)[13]	6.015	6.112	6.061	6.091	6.117
	6.119	6.080	6.034	6.083	6.070
$(NH_4)_4[MoO_3(ida)]_2\cdot 3H_2O$(**4**)	6.117	6.183			
$(NH_4)_5[Mo_2O_6(1,3\text{-pdta})]Cl\cdot 2H_2O$(**5**)	6.072	6.062			
$K_4[Mo_2O_6(1,3\text{-pdta})]\cdot 3H_2O$(**6**)	6.087	6.102			
$(NH_4)_{6n}(Mo_8O_{27})_n\cdot 4nH_2O$(**7**)	5.955	6.084	6.022	6.095	
$(NH_4)_{4n}(Mo_8O_{26})_n$(**8**)	6.033	5.982	6.004	5.922	

Table S2. Comparisons of the Mo–O and Mo–N distances (Å) in iminodiacetato molybdates.

Compounds	Mo-O_t	Mo-O_b	Mo-O_{c1}	Mo-O_{c2}	Mo-N	Ref
$(NH_4)_4[MoO_3(ida)]_2\cdot 3H_2O$(**4**)	1.736(2)		2.202(2)	2.226(2)	2.306(2)	
$(NH_4)_8\{\gamma\text{-}Mo_8O_{26}[MoO_3(ida)]_2\}\cdot 6H_2O$ (**1**)	1.705(5)	2.089(2)	2.211(5)	2.212(4)	2.285(5)	This work
$K_8\{\gamma\text{-}Mo_8O_{24}[MoO_3(ida)]_2\}\cdot 12H_2O$ (**2**)	1.713(2)	2.067(2)	2.123(2)	2.105(2)	2.342(2)	
$Na_2[MoO_3(ida)]\cdot 2H_2O$	1.746(2)		2.228(2)	2.228(2)	2.306(4)	[12]
$C_4H_{12}N_2[UO_2(ida)_2(OH)_2]\cdot 8H_2O$	1.781(8)		2.348(8)	2.341(9)	2.585(10)	[a]
$Pd(Hida)_2$			1.988(4)		2.061(4)	[b]
$Pt(Hida)_2$			2.014(9)		2.066(10)	[b]
$(pyH)[Fe(ida)_2]$			1.980(2)	1.984(2)	2.161(3)	[c]

a) M. A. Walters, V. Vapnyar, A. Bolour, C. Incarvito, A. L. Rheingold, *Polyhedron* 2003, **22**, 941–946.
b) A. J. Chakraborty, H. Mayer-Figge, W. S. Sheldrick, P. Banerjee, *Polyhedron*, 2005, **24**, 771–776.
c) J. Jiang, M. J. Sarsfield, J. C. Renshaw, F. R Livens, D. Collison, J. M. Charnock, M. Helliwell, H. Eccles, *Inorg. Chem*. 2002, **41**, 2799–2806.

Table S3. Comparisons of the Mo–O and Mo–N distances (Å) for propanediaminetetraacetato molybdates.

Compounds	Mo–O_t	Mo–O_b	Mo–O_{c1}	Mo–O_{c2}	Mo–N	Ref
$\{Na_8[Mo_{10}O_{32}(edta)](H_2O)_{35}\}_n$[12]	1.700(5)	1.812(4)	2.122(4)	2.173(5)	2.373(4)	[43]
$(NH_4)_{8n}[Mo_{10}O_{32}(1,3\text{-}pdta)]_n\cdot 30nH_2O$(**3**)[13]	1.711(7)	1.825(7)	2.118(8)	2.231(7)	2.342(8)	[43]
$(NH_4)_5[Mo_2O_6(1,3\text{-}pdta)]Cl\cdot 2H_2O$(**5**)	1.739(4)		2.198(4)	2.207(3)	2.347(3)	this work
$K_4[Mo_2O_6(1,3\text{-}pdta)]\cdot 3H_2O$(**6**)	1.735(4)		2.194(4)	2.221(4)	2.347(5)	
Average (Å)	1.713	1.864	2.143	2.187	2.368	