The trinuclear platinum(II) complex in Vèzes' red salt as a building block for coordination polymers

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Supporting Information

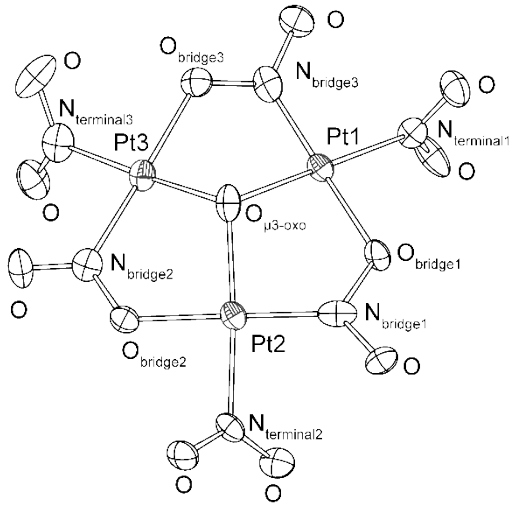
Contents

Table S1. Crystallographic parameters.

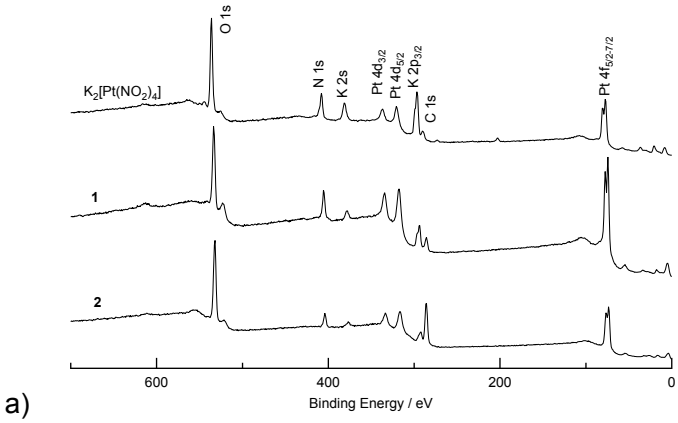
	2	**3**	**4**	**5**
Formula	$C_{26}H_{60}K_2N_6O_{29}Pt_3$	$C_{27}H_{54}Ag_2N_6O_{26}Pt_3$	$C_{15}H_{30}Ag_2N_6O_{20}Pt_3$	$C_{10.5}H_{21}Ag_2N_6O_{16.5}Pt_3$
Fw	1584.27	1679.77	1415.46	1296.34
T / K	100(2)	100(2)	100(2)	100(2)
Space Group	$P\bar{1}$	$P\bar{1}$	$P2_1/n$	$Pbcn$
a / Å	8.2798(2)	15.8083(3)	8.170(2)	17.5819(6)
b / Å	17.4801(5)	16.2005(3)	21.7158(4)	20.2747(8)
c / Å	18.1002(5)	18.9885(3)	18.2590(4)	15.0045(6)
α / °	95.484(7)	68.585(5)	90	90
β / °	100.669(7)	84.549(6)	92.200(7)	90
γ / °	90.825(6)	87.459(6)	90	90
V / Å^3	2561.2(1)	4506.5(2)	3240.3(1)	5348.6(4)
Z	2	4	4	8
Data	9170	16233	5850	4893
Parameters	578	1157	417	352
$R1$, $wR2$[a] [$I>2\sigma(I)$]	0.0885, 0.2281	0.0644, 0.1474	0.0439, 0.1121	0.0804, 0.1897
$R1$, $wR2$[a] (all data)	0.1208, 0.2502	0.0994, 0.1673	0.0610, 0.1251	0.1260, 0.2232
GoF	1.053	0.986	1.165	1.0075

[a] $R1 = \Sigma\, ||F_o| - |F_c|| \,/\, \Sigma\, |F_o|$; $wR2 = \{ \Sigma\, [\, w(F_o^2 - F_c^2)^2] \,/\, \Sigma[w(F_o^2)^2]\}^{1/2}$.

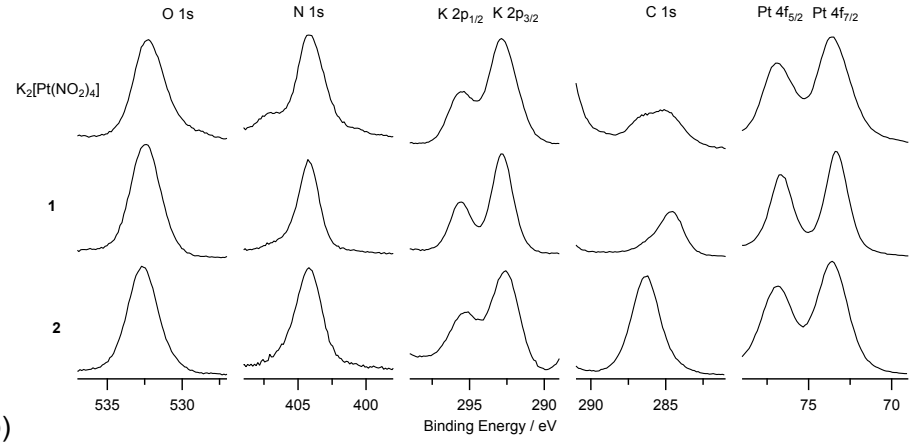
Table S2. Coordination lengths (Å) of $[\mathbf{Pt3}]^{2-}$ units in **2**–**5**.



	2	**3** [Pt(1)-Pt(3), Pt(4)-Pt(6)]		**4**	**5**
Pt(1)-Pt(2)	3.2657(10)	3.2365(7),	3.1895(7)	3.2285(6)	3.2194(14)
Pt(2)-Pt(3)	3.2522(10)	3.2413(7),	3.2196(7)	3.2274(7)	3.2390(15)
Pt(3)-Pt(1)	3.2317(10)	3.2058(7),	3.2179(7)	3.2296(6)	3.2381(16)
Pt(1)-$O_{\mu3\text{-oxo}}$	2.009(10)	2.006(7),	2.004(7)	2.022(7)	2.028(16)
Pt(1)-$O_{bridge1}$	2.012(11)	1.998(8),	1.992(8)	2.032(8)	2.016(16)
Pt(1)-$N_{bridge3}$	1.892(18)	1.942(9),	1.929(10)	1.972(10)	1.92(2)
Pt(1)-$N_{terminal1}$	1.984(15)	2.005(9),	1.999(9)	2.004(10)	2.00(3)
Pt(2)-$O_{\mu3\text{-oxo}}$	2.046(12)	2.010(7),	2.027(7)	1.993(7)	1.985(16)
Pt(2)-$O_{bridge2}$	1.976(11)	2.020(8),	2.012(8)	2.006(8)	1.959(17)
Pt(2)-$N_{bridge1}$	1.873(18)	1.938(10),	1.951(10)	1.976(10)	1.91(2)
Pt(2)-$N_{terminal2}$	2.006(15)	2.022(10),	2.013(11)	2.003(10)	2.04(3)
Pt(3)-$O_{\mu3\text{-oxo}}$	1.979(10)	2.037(7),	2.030(7)	2.008(8)	2.013(16)
Pt(3)-$O_{bridge3}$	2.015(13)	2.030(8),	2.017(7)	2.010(8)	2.007(15)
Pt(3)-$N_{bridge1}$	1.978(16)	1.942(9),	1.946(10)	1.943(10)	1.97(2)
Pt(3)-$N_{terminal3}$	1.999(16)	2.004(11),	2.000(10)	1.999(10)	2.05(3)



a)



b)

Figure S1. XPS spectra of **1** and **2** together with those of reference crystals of $K_2[Pt(NO_2)_4]$. The N 1s peaks in **1**, **2**, and $K_2[Pt(NO_2)_4]$ and the C 1s peak (284.69 eV) in **1** were used for correction because a strong C 1s peak arises from the 18-crown-6 ether in **2** and adventitious hydrocarbons are not usable for comparison between **1**, **2**, and $K_2[Pt(NO_2)_4]$.

Table S3. Binding energies (eV) for crystals **1**, **2**, and the reference compound $K_2[Pt(NO_2)_4]$.

	$K_2[Pt(NO_2)_4]$	**1**	**2**
O 1s	532.3	532.5	532.6
N 1s	(404.2)[a]	404.2	(404.2)[a]
K $2p_{1/2}$	295.5	295.6	295.2
K $2p_{3/2}$	292.8	292.8	292.6
C 1s	286.5-285.0	(284.6)[b]	286.3
Pt $4f_{5/2}$	76.6	76.4	76.9
Pt $4f_{7/2}$	73.6	73.3	73.6

a) Binding energies of $K_2[Pt(NO_2)_4]$ and **2** are corrected using the peak for N 1s in **1**.

b) Adventitious hydrocarbon, corrected using the literature value.*

* Koizumi, A.; Yamauchi, K.; Sato, M.; Takano, M. *J. Surf. Anal. Soc. Jpn.* **2008**, *15*(1), 126–26.

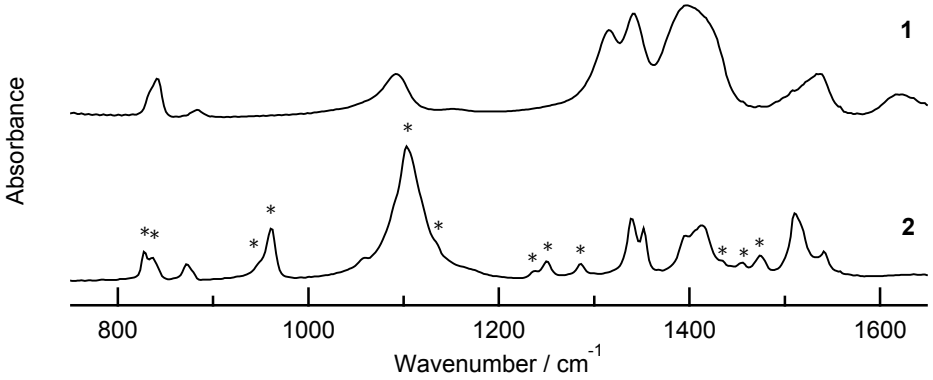


Figure S2. IR spectra of **1** and **2** compressed into KBr pellets.

Table S4. Vibrational frequencies and assignments for **1** and **2**.

1	**2**	Assignment
1535	1540, 1510	$\nu_a(NO_2$, bridging)
1398	1413, 1396	$\nu_a(NO_2$, terminal)
1340, 1315	1338	$\nu_s(NO_2$, terminal)
1151	[a]	ν(N−O, bridging)
1091	[a]	$\nu_s(NO_2$, terminal)
883	871	δ(ONO, terminal)
841	(827) [a]	δ(ONO, bridging)

a) Obscured by bands of $[K(18\text{-crown-8})]^+$ cation.[**]

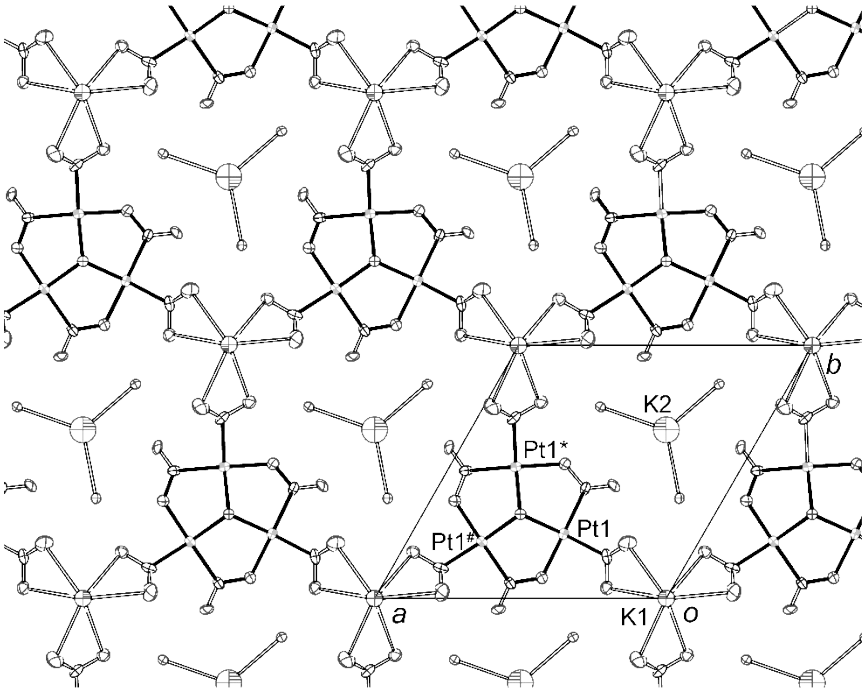


Figure S3. ORTEP diagram of the tentative crystal structure of **1**. Trigonal *P*3, $H_2K_2N_6O_{16}Pt_3$ = 1005.55, $a = b = 11.838(5)$ Å, $c = 3.2760(5)$ Å, $V = 397.6(2)$, $T = 100$ K, $Z = 1$, CuKα ($\lambda = 1.54187$ Å), 4,346 data collected, 916 unique, 85 parameters refined, $R1 = 0.0712$, $wR2 = 0.1785$, GoF = 1.142, refined on F_o^2 with 4-component inversion twinning (see text).

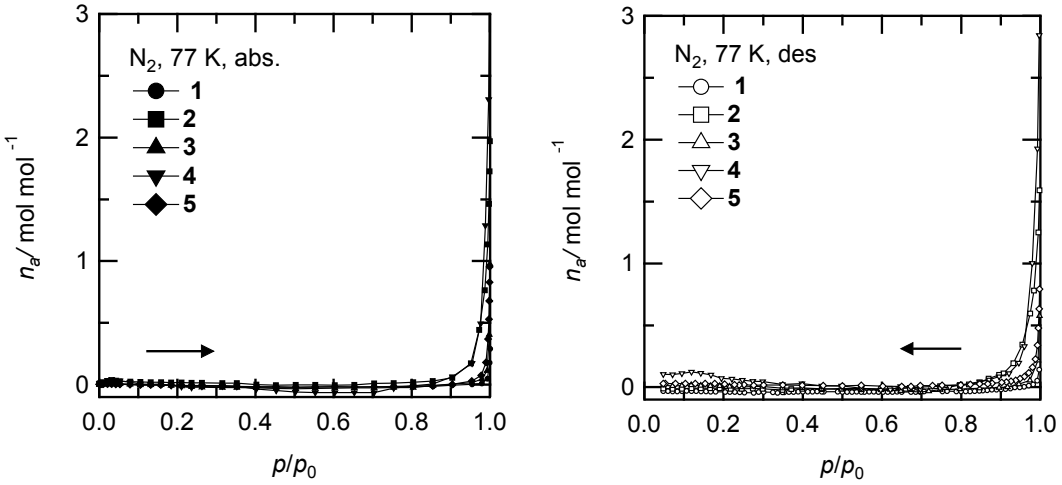


Figure S4. N_2 isotherms of dried crystals of **1**–**5** obtained at 77 K.

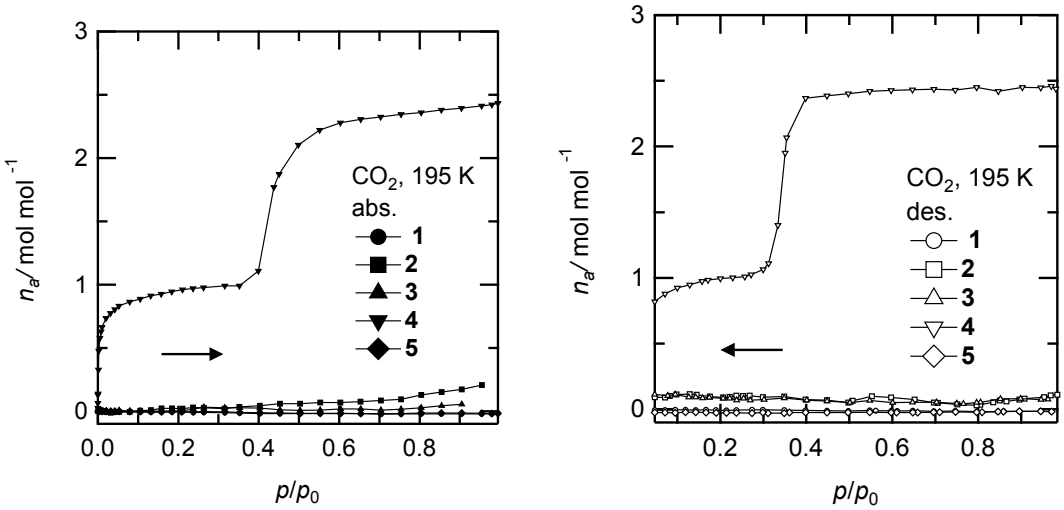


Figure S5. CO_2 isotherms of dried crystals of **1**–**5** obtained at 195 K.

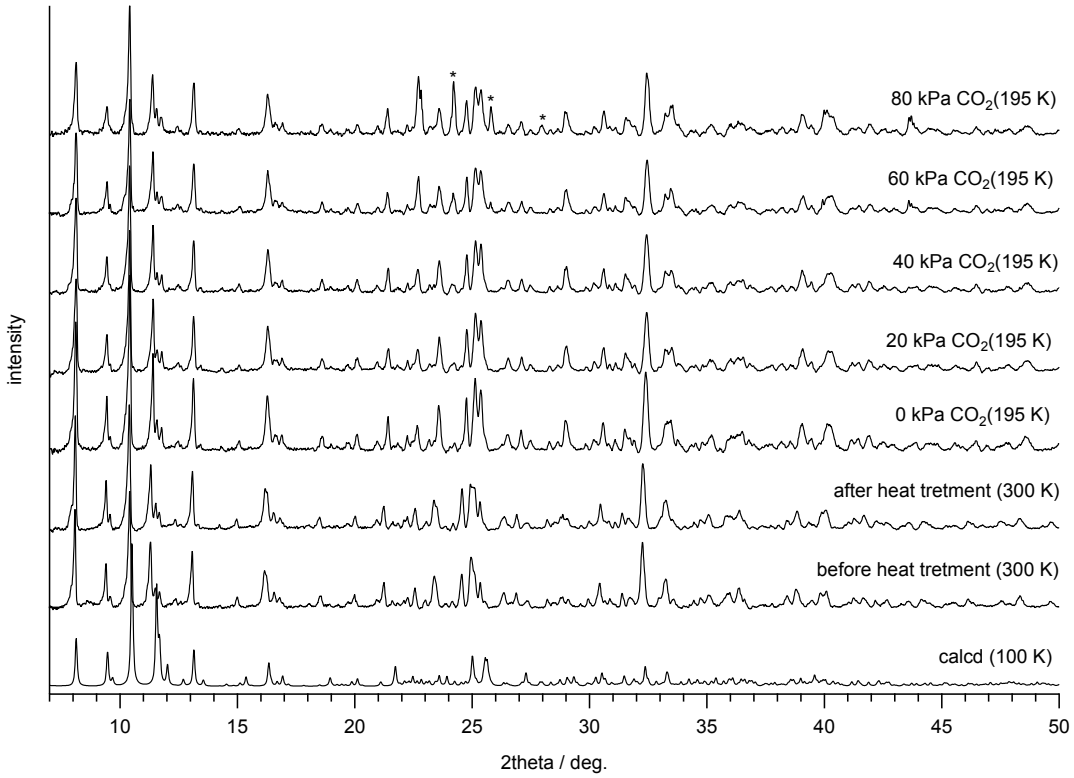


Figure S6. XRD patterns of dried crystals of **4** under controlled CO_2 pressure. Peaks at 2θ = 22.7, 24.2, and 25.8° are artefacts by icing (probably impurity of CO_2 gas).

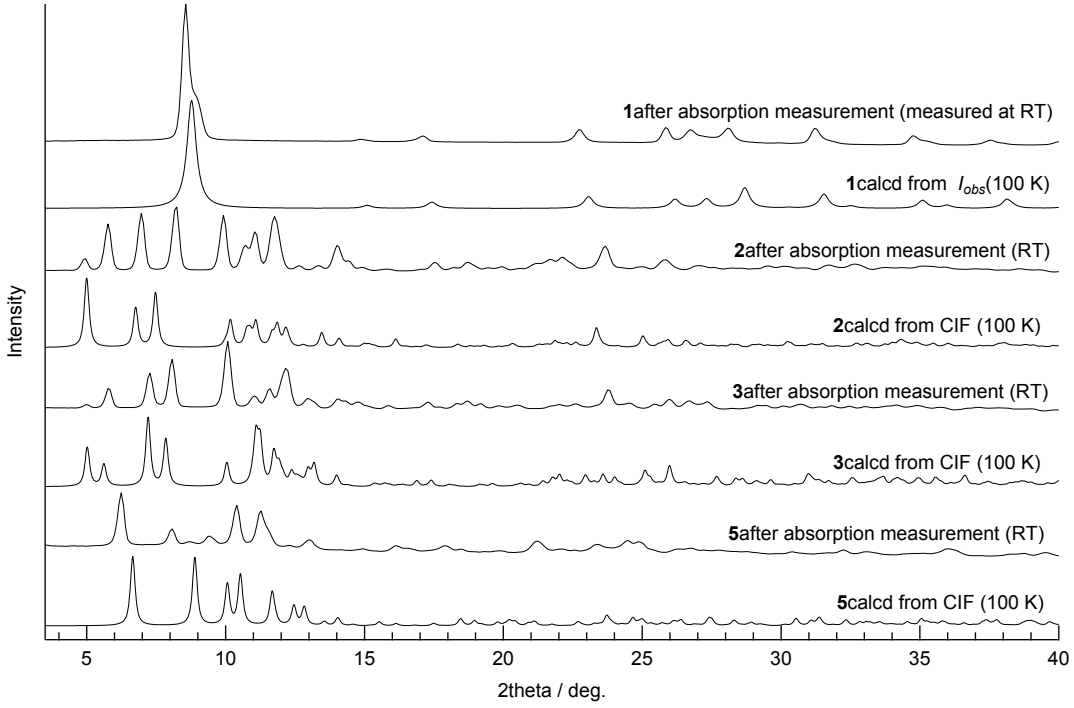


Figure S7. XRD patterns of dried crystals of **1**–**3**, and **5** with calculated patterns from the CIFs.