

Supplementary Information for Dalton- B805848B

Assembling metal phosphonates in the presence of monodentate-terminal and bidentate-bridging pyridine ligands. Use of non-covalent and covalent-coordinate interactions to build polymeric metal-phosphonate architectures

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Table S1. Comparison of structural parameters for **1** and **2** with other cubane-phosponates in the literature.

Compound	Space group	M–O, Å	P–O, Å	M···M face diagonal, Å	P···P face diagonal, Å	M···P face diagonal, Å	M···P body diagonal, Å	M ⁿ⁺ ionic radius, Å [#]	Ref
[^t BuPO ₃ BEt] ₄	I4 ₁ /a	1.47	1.50	4.12	3.92	2.85	4.93	0.25	a
[^t BuPO ₃ B ^s Bu] ₄	Pī	1.47	1.50	4.13	3.93	2.85	4.94	0.25	b
[^t BuPO ₃ BPh] ₄	P2 ₁ /n	1.46	1.50	4.15	3.93	2.84	4.92	0.25	c
[^t BuPO ₃ Al ⁱ Bu] ₄	I4 ₁ /a	1.76	1.52	4.56	4.42	3.17	5.50	0.53	d
[PhPO ₃ Ga ^t Bu] ₄	Pī	1.85	1.51	4.69	4.42	3.22	5.57	0.61	e
[^t BuPO ₃ Cu(3,5- <i>t</i> -Bu ₂ PzH)] ₄	C2/c	1.95	1.53	3.77	4.43	3.11	5.15	0.71	f
[Ph ₃ CPO ₃ Co(py)] ₄	I4 ₁ /a	1.94	1.53	4.29	4.82	3.06	5.52	0.72	g
[^t BuPO ₃ Co(2-apy)] ₄	I4 ₁ /a	1.93	1.52	4.29	4.77	3.14	5.56	0.72	this work
[^t BuPO ₃ Zn(2-apy)] ₄	I4 ₁ /a	1.93	1.52	4.31	4.72	3.19	5.54	0.74	this work

Ionic radius for Mⁿ⁺ in the tetrahedral geometry.

(a) M. G. Walawalkar, R. Murugavel, H. W. Roesky and H.-G. Schmidt, *Organometallics* 1997, **16**, 516.

(b) M. G. Walawalkar, R. Murugavel, H. W. Roesky and H.-G. Schmidt, *Inorg. Chem.*, 1997, **36**, 4202.

(c) K. Diemert, U. Englert, W. Kuchen and F. Sandt, *Angew. Chem., Int. Ed. Engl.*, 1997, **36**, 241

(d) Y. Yang, H.-G. Schmidt, M. Noltemeyer, J. Pinkas and H. W. Roesky, *J. Chem. Soc., Dalton Trans.*, 1996, 3609.

(e) M. R. Mason, M. S. Mashuta and J. F. Richardson, *Angew. Chem.* 1997, **109**, 249; *Angew. Chem., Int. Ed. Engl.* 1997, **36**, 239.

(f) V. Chandrasekhar, L. Nagarajan, R. Clérac, S. Ghosh and S. Verma, *Inorg. Chem.* 2008, **47**, 1067.

(g) V. Baskar, M. Shanmugam, E. C. Sanudo, M. Shanmugam, D. Collison, E. J. L. McInnes, Q. Wei and R. E. P. Winpenny, *Chem. Commun.* 2007, 37.

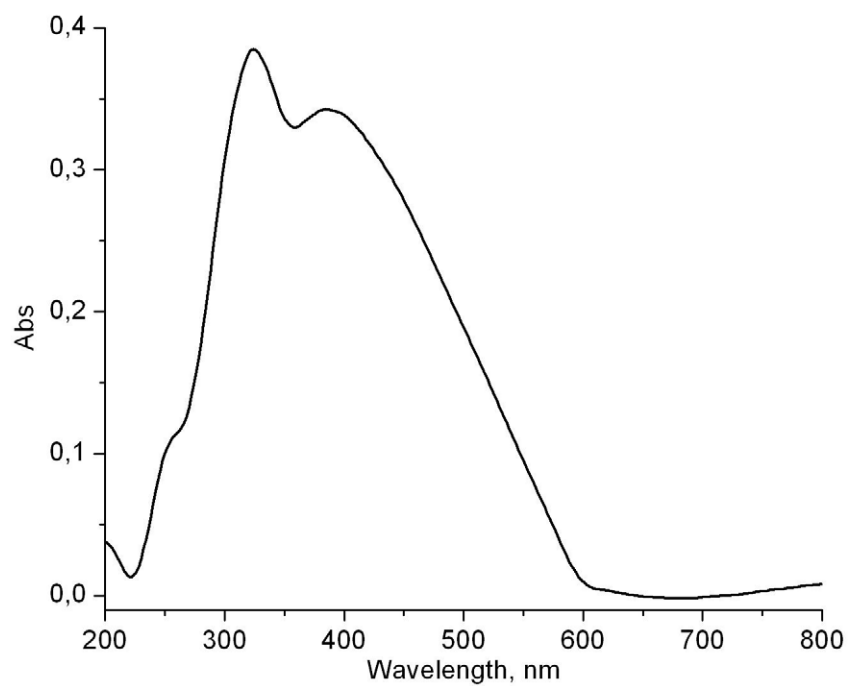


Figure S1. Solid State UV-vis spectrum of **1**.

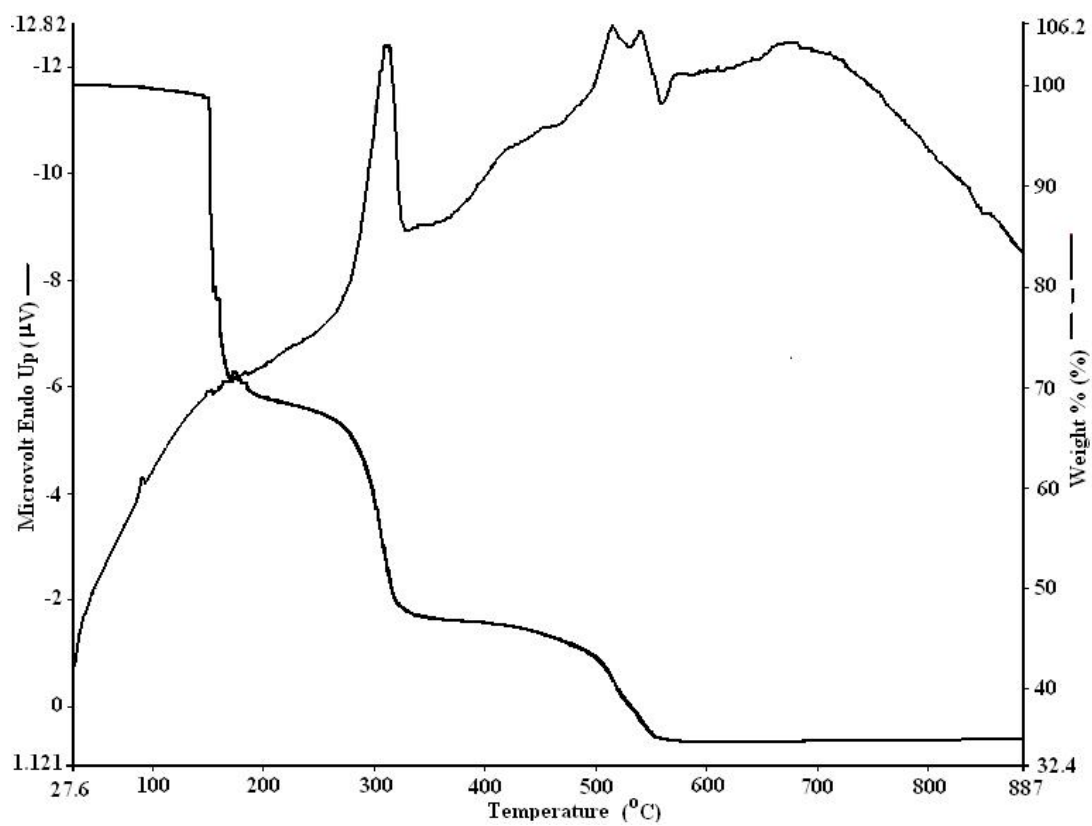


Figure S2. Thermal decomposition of **1** at N₂ atm.

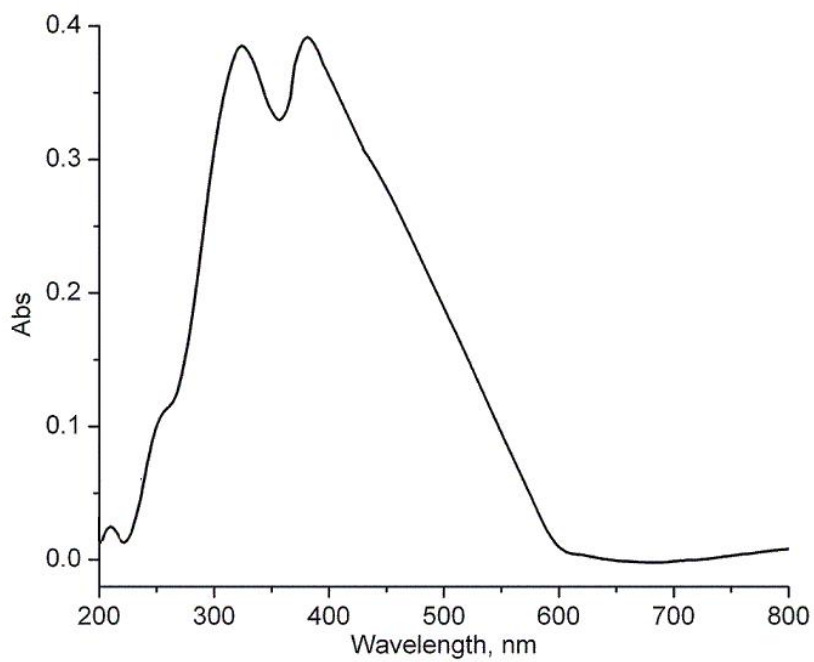


Figure S3. Solid State UV-vis spectrum of **2**

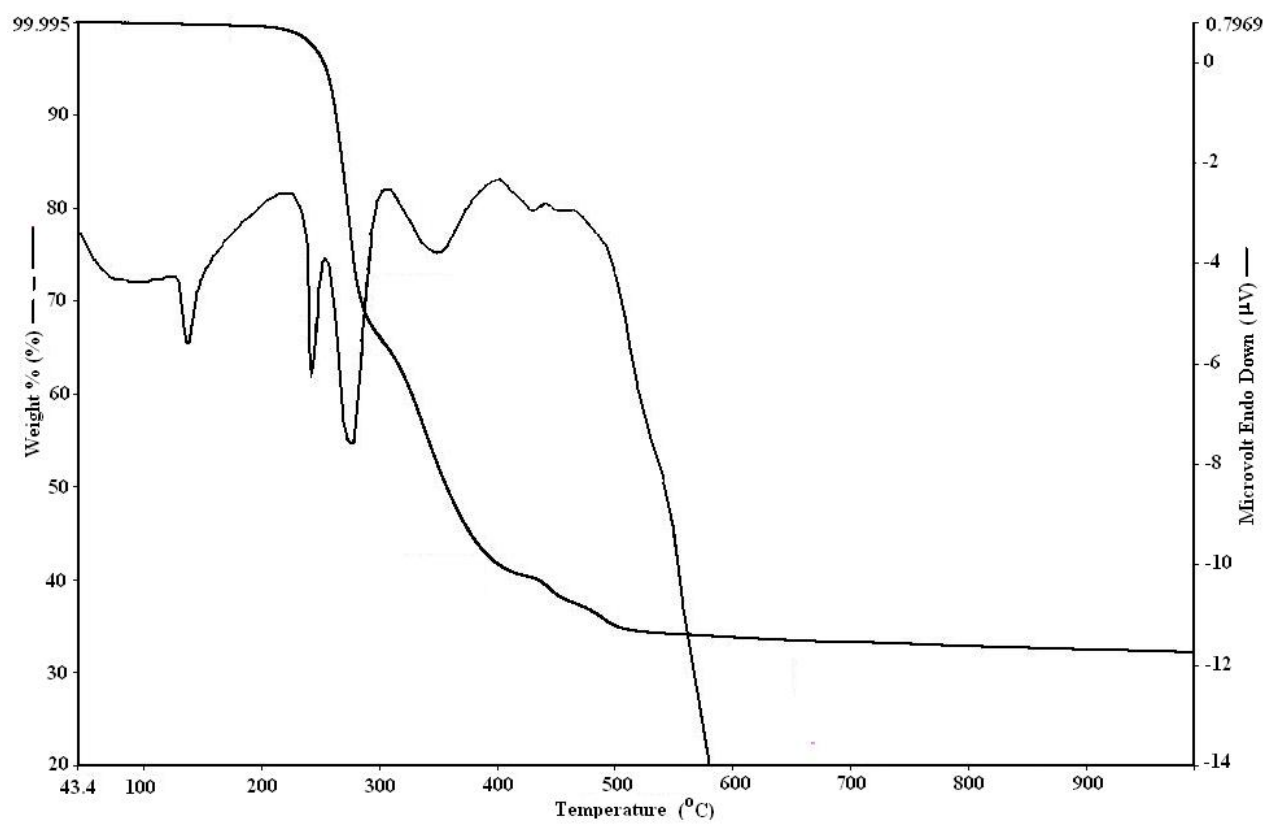


Figure S4. Thermal decomposition of **2** at N₂ atm.

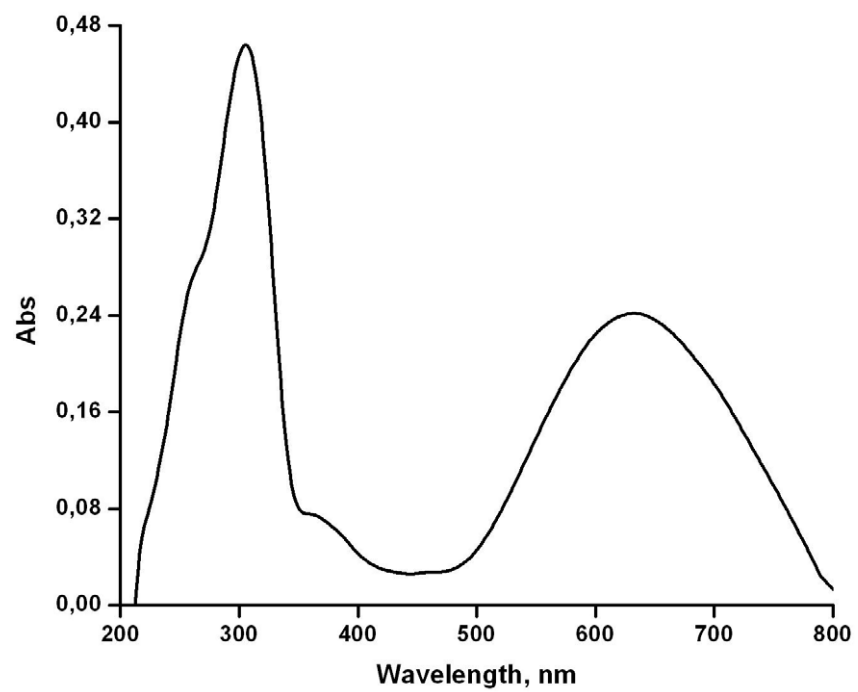


Figure S5. Solid State UV-vis spectrum of **3**.

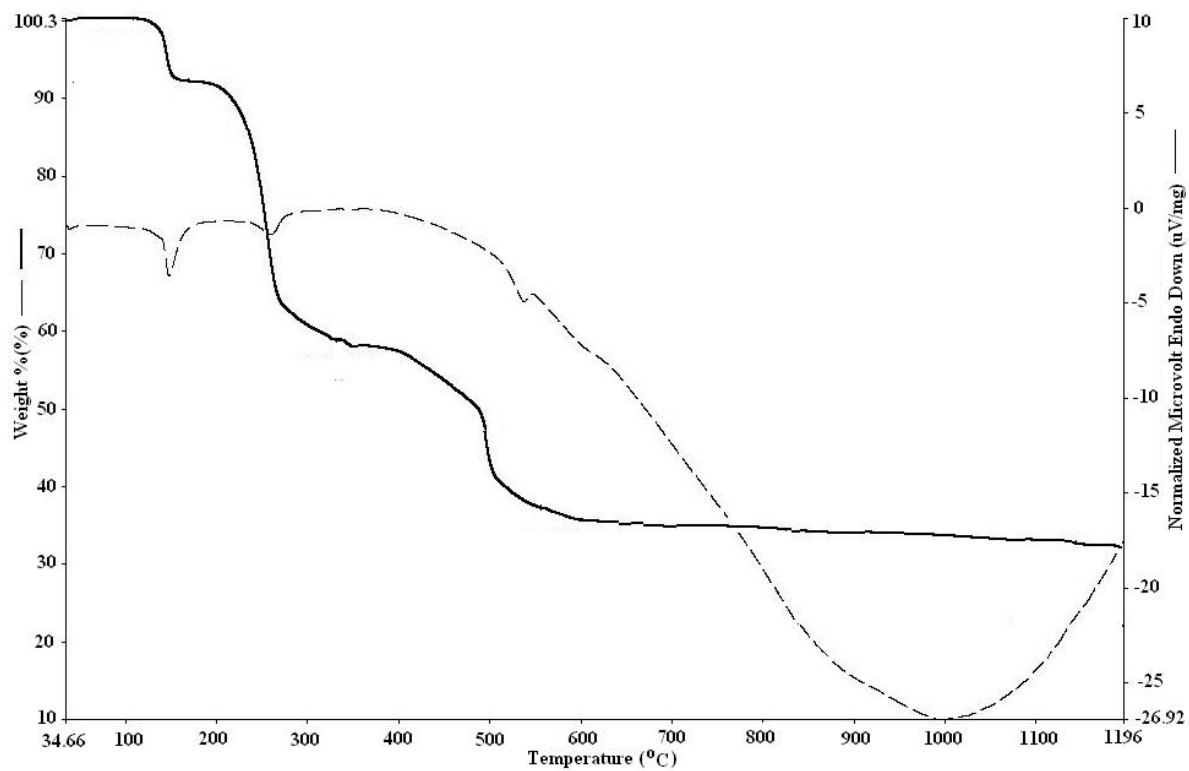


Figure S6. Thermal decomposition of **3** at N₂ atm.

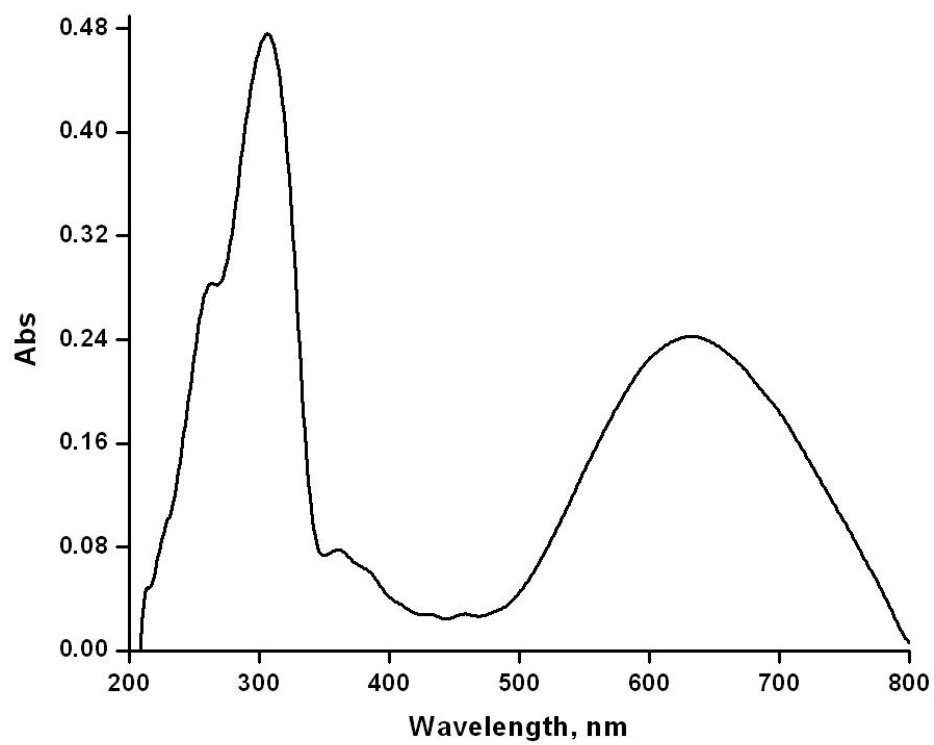


Figure S7. Solid State UV-vis spectrum of **4**.

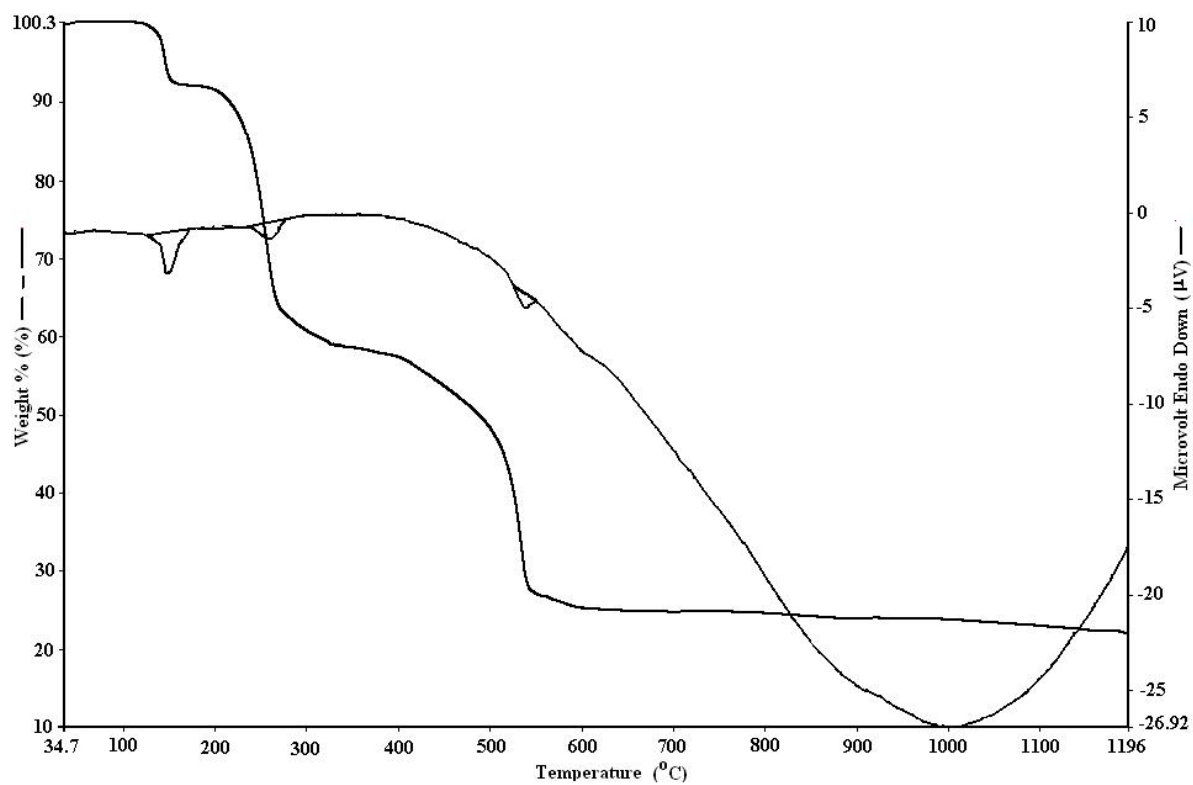


Figure S8. Thermal decomposition of **4** at N₂ atm.

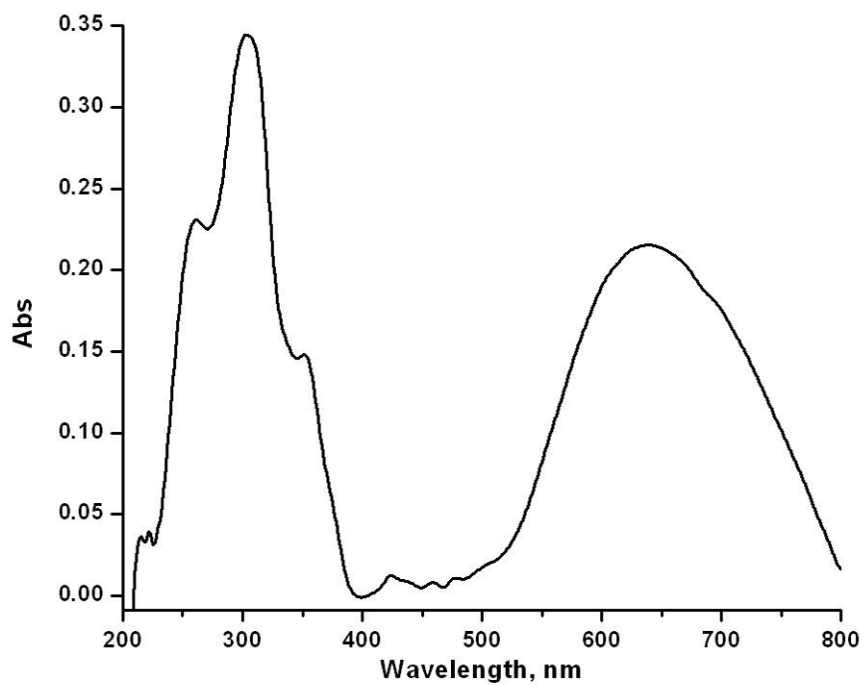


Figure S9. Solid State UV-vis spectrum of **5**.



Figure S10. Thermal decomposition of **5** at N₂ atm.