Electronic Supplementary Material (ESI) for CrystEngComm.
This journal is © The Royal Society of Chemistry 2018

Supporting Information

A lead-iodide based single crystal semiconductor: exploring multi-orientation photoconductive behaviour via intervening isopropyl viologen component between the inorganic $[Pb_2I_6]^{2-}_n$ wires

Yifang Wang,[a] Jie Zhang,[a] Jia Huang,[a] Haoyu Zhang[a] and Zhiyong Fu *[a,b]

General methods and materials

All of the reagents were purchased from commercial channels and used without further purification. ATA Instrument Q600 SDT thermogravimetric analyser was used to obtain the thermogravimetric analysis (TGA) curve in N_2 100 mL min^{-1} at a rate of 10 °C min^{-1}. The X-ray powder diffraction (XRD) data were collected in the angular range of 2θ = 5°- 50° with a Bruker D8 Advance X-ray diffractometer using CuKα radiation (λ= 1.5406 Å). UV-Vis diffuse reflectance spectra were carried out using a HITACHIU-3010 spectrometer, and a $BaSO_4$ plate was used as a 100% reflectance standard. IR-spectra were characterized by a Bruker Tensor 27 FTIR spectrometer in the range of 4000- 400 cm^{-1} using KBr pellets. The C, H and N elemental analyses (EA) were performed on a Vario EL III elemental analyzer.

Synthesis of compound 1

A mixture of PbI_2 (231 mg, 0.5 mmol), H_3BO_3 (30.9 mg, 0.5 mmol), 4,4'-bipyridine (187.4 mg, 1.2 mmol), HI (3 mL, 1.5 mmol, 57 wt. % in water), isopropanol (3mL) and H_2O (9 mL) was stirred for 30 min in air, and then transferred to and sealed in a 23mL Teflon-lined steel bomb, which was heated at 170 °C for 72 h and then cooled to room temperature at a rate of 5 °C h^{-1}, Finally, dark red crystals were collected by filtration, washed with distilled water for 3 times and then dried at room temperature for 12 h (226.5 mg, 63.9 % based on PbI_2). Elemental Anal. Calc. (%) for $C_{16}H_{22}N_2Pb_2I_6$ (1418.13): C 13.55, H 1.56, N 1.97; found: C 13.92, H 1.02, N 1.84. IR data : 3107 (w), 3047 (m), 2979 (w), 2927 (w), 2856 (w), 1633 (s), 1556 (m), 1500 (w), 1440 (s), 1377 (m), 1342 (m), 1288 (w), 1232 (m), 1174 (s), 1085 (s), 968 (w), 823 (s), 767 (w), 713 (w), 561 (w), 505 (w).

X-ray single crystal diffraction

The data were measured on a Rigaku R-AXIS SPIDER IP diffractometer with graphite monochromated Mo/Kα radiation (λ = 0.71073 Å). Data were collected at 298K, using the ω- and φ-scans to amaximum θ value of 25.02°. Absorption corrections were performed using a multi-scan method. The structures were solved by direct methods with SHELXS-97 and refined by full-matrix least-square technique on F^2 with SHELXL-97.

CCDC 1820029 contains the supplementary crystallographic data for this paper. These data can be obtained free of charge from the Cambridge Crystallographic Data Centre via www.ccdc.cam.ac.ukf/data_request/cif.

Table S1. Crystal data for 1.

Empirical formula	$C_{16}H_{22}N_2Pb_2I_6$
Formula weight	1418.13 g /mol
Crystal system	Monoclinic
Space group	*P* 2_1/n
Unit cell dimensions (Å, °)	a = 7.8674(6), α = 90
	b = 17.7109(16), β = 98.763(2)
	c = 21.9456(18), γ = 90
Volume (Å^3)	3022.2(4)
F (000)	2456
Goodness-of-fit on F^2	0.980
Final R indices [I>2σ (I)]	R_1[a] = 0.0382, wR_2[b] = 0.0828
R indices (all data)	R_1[a] = 0.0581, wR_2[b] = 0.0951

[a] $R_1 = \sum (\|F_0\| - \|F_c\|) / \sum |F_0|$, [b] $\omega R_2 = \left\{ \sum \omega \left[\left(F_0^2 - F_c^2 \right)^2 \right] / \left[\left(F_0^2 \right)^2 \right] \right\}^{1/2}$

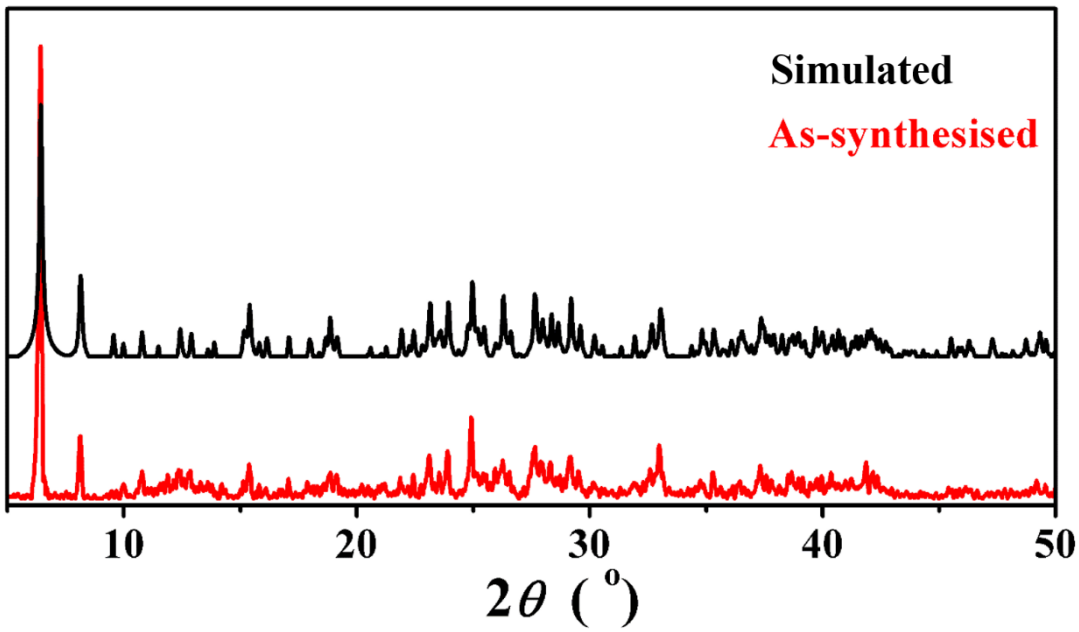

Fig. S1 PXRD spectra of compound **1.**

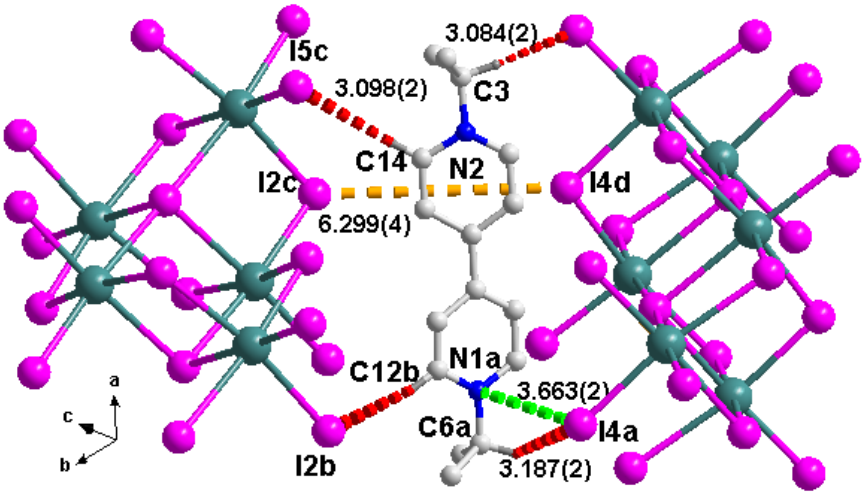

Fig. S2 Crystal structure of **1** showing interactions between IV^{2+} and $[Pb_2I_6]^{2-}{}_n$ chains.

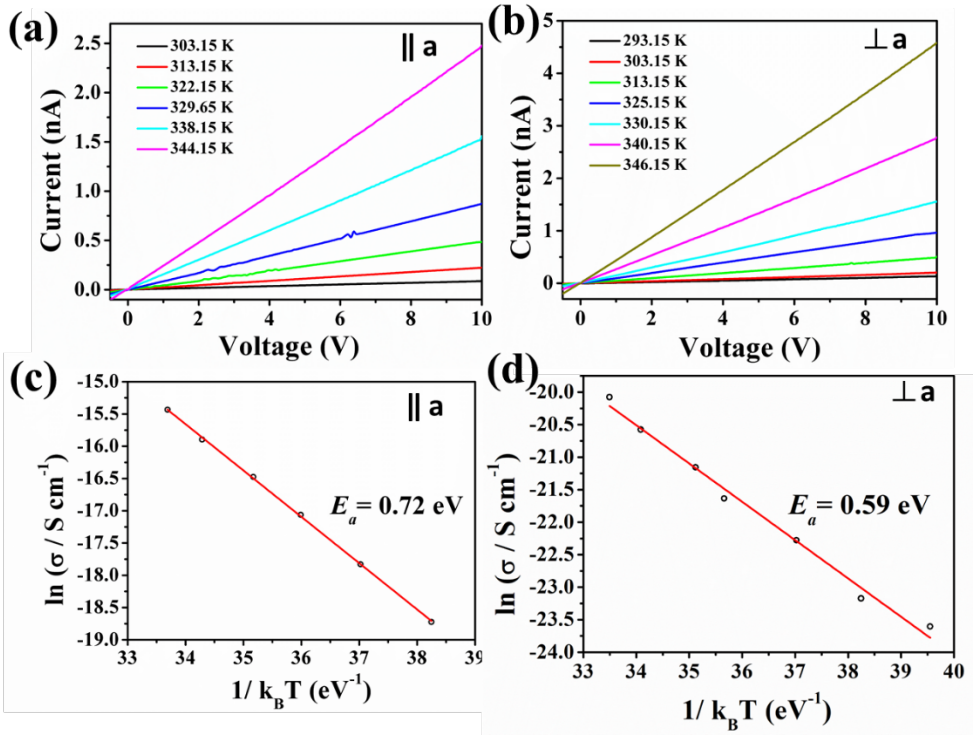

Fig. S3 Temperature-dependent I–V curves along the *a* direction (a) and perpendicular to the *a* direction (⊥*a*) (b). Arrhenius plots along the *a* direction (c) and perpendicular to the *a* direction (⊥*a*) (d). *Ea* is the activation energy.

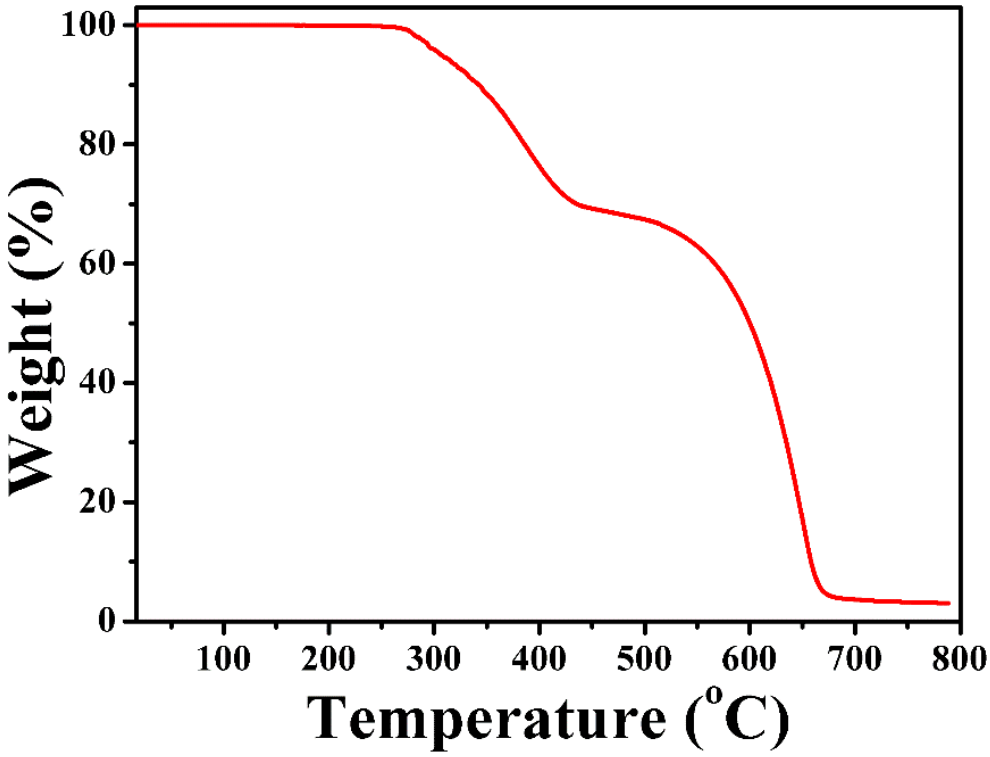

Fig. S4 TG curve of **1.**

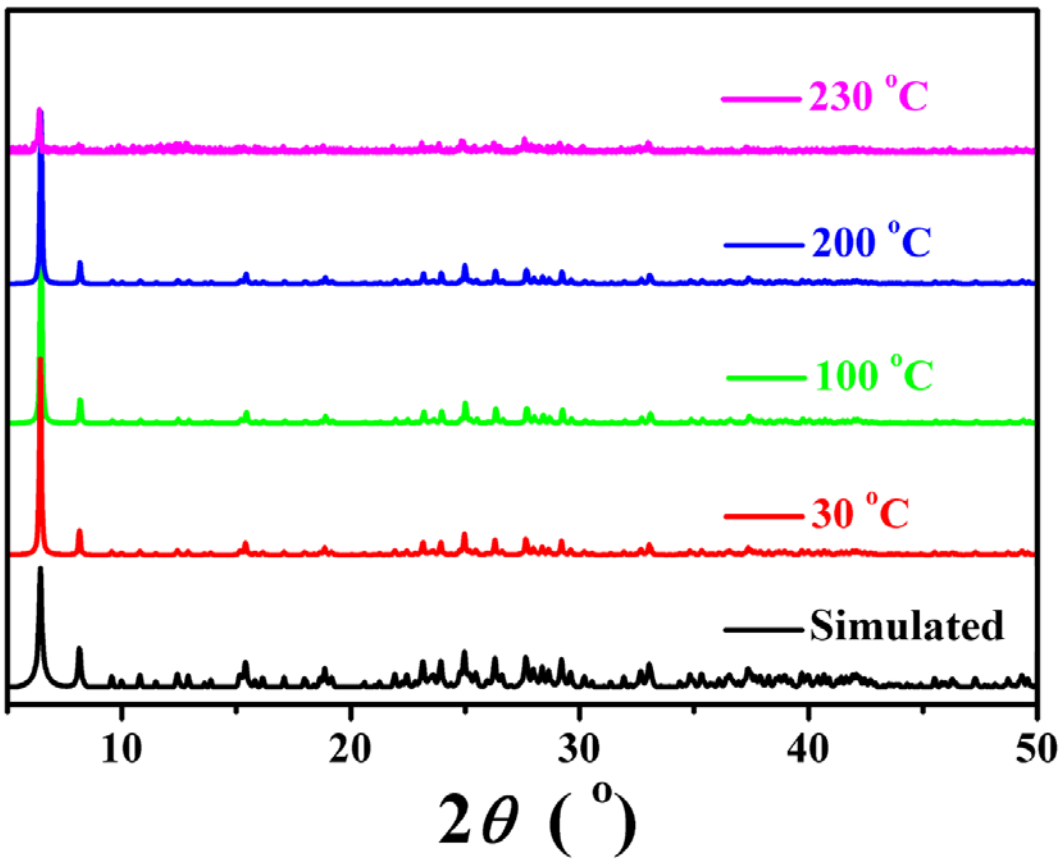

Fig. S5 Powder XRD patterns of samples calcined at different temperatures.

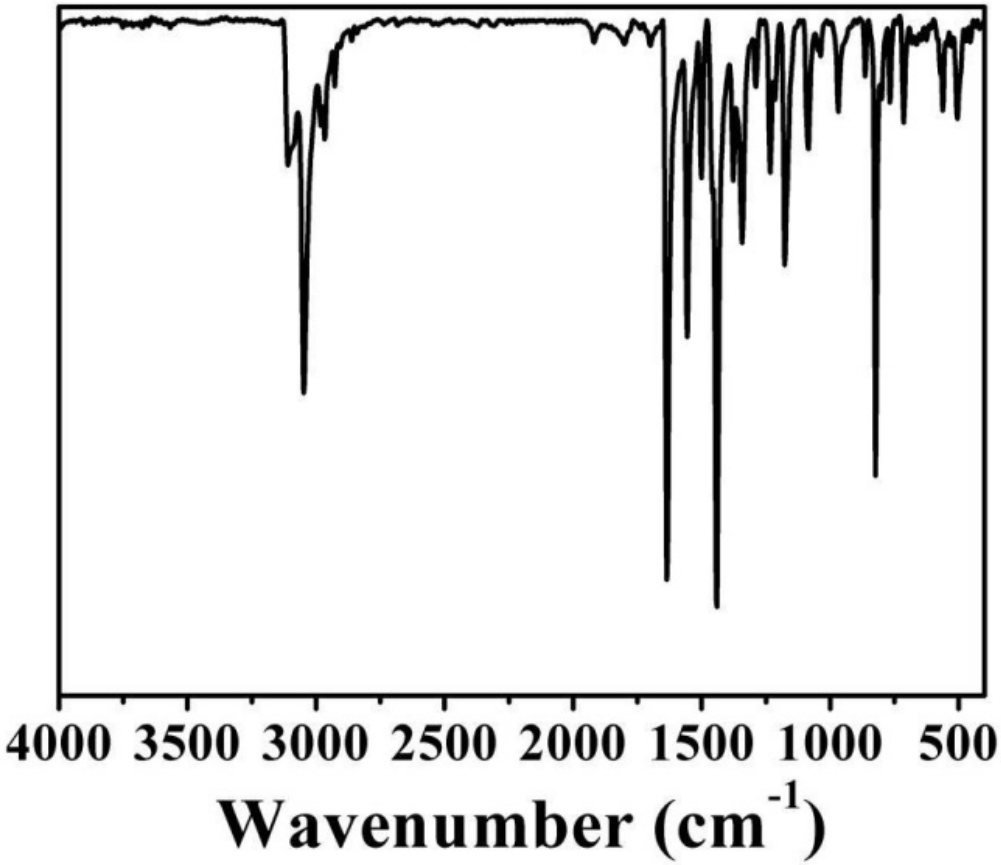

Fig. S6 IR spectrum of **1**