Supporting information:

Spontaneous Redox Synthesis of the Charge Transfer Material $TTF_4[SVMo_{11}O_{40}]$

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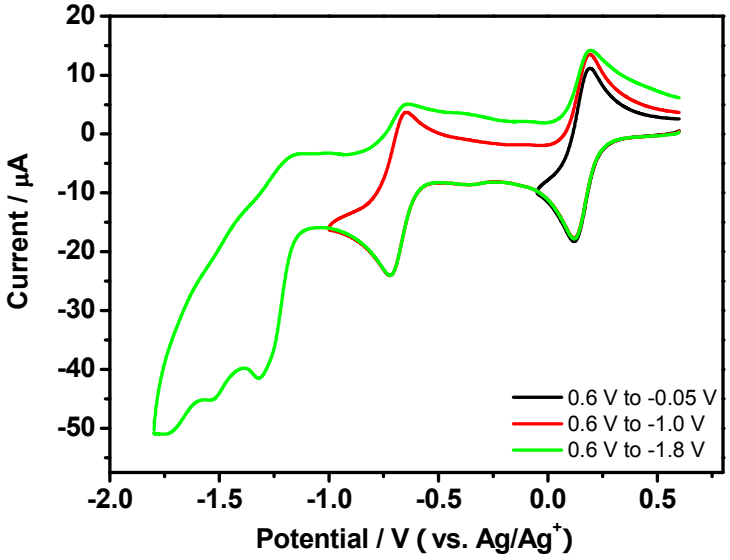

Figure S1. Cyclic voltammograms for reductions of 1 mM $(n\text{-}Bu_4N)_3[SVMo_{11}O_{40}]$ in MeCN (0.1 M $(n\text{-}Bu_4N)PF_6$). Electrode: glassy carbon (diameter 3 mm). Scan rate = 0.1 V s^{-1}.

Table S1: Bond distances [Å] and angles [°] of hydrogen bonds for relevant crystallographic interactions for $TTF_4[SVMo_{11}O_{40}].2CH_2Cl_2.\ 2H_2O$*.

D–H----A	d (D–H) Å	d (H-A) Å	d (D-A) Å	＜(DHA) °	Symmetry code
C11–H11----O5	0.930	2.473	3.068	122.0	½–x, 1.5-y, z-½
C6–H6----O6	0.931	2.391	3.229	149.8	x, y, z
C7–H7----O7	0.931	2.438	3.171	135.8	½–x, 1.5-y, 1–z
C8–H8----O8	0.929	2.455	3.333	157.5	x, y, z
C1–H1----O9	0.931	2.500	3.255	138.5	½-x, y-½, 1.5-z
C5–H5----O9	0.930	2.622	3.342	134.8	½-x, 1.5-y, 1-z
C5–H5----O10	0.930	2.466	3.219	152.5	½-x, 1.5-y, 1-z
C13–H13B----O13	0.971	2.481	3.394	156.7	-x, 1-y, 1-z
C13–H13B----O15	0.971	2.527	3.257	132.0	-x, 1-y, 1-z
C2–H2----O16	0.930	2.580	3.328	128.1	-x, y, 1.5-z,
C11–H11----O16	0.930	2.413	3.190	141.0	x-½, 1.5-y, ½+z
C11–H11----O20	0.930	2.436	3.186	137.8	½-x, y-½, ½-z

*The identity of each atom is shown in Figure 5. D and A represent the donor and acceptor atoms, respectively.

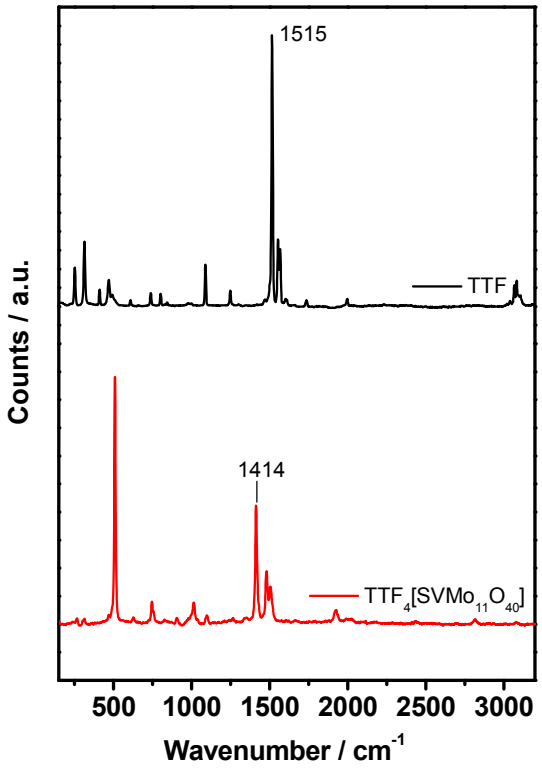

Figure S2. Raman spectra for TTF and $TTF_4[SVMo_{11}O_{40}]$ solids.

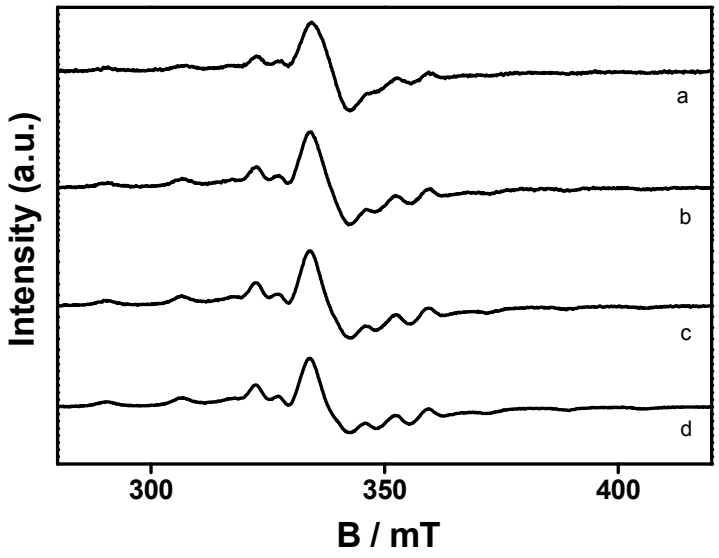

Figure S3. EPR spectra of $TTF_4[SVMo_{11}O_{40}]$ solid (prepared by the solution phase reaction) in the temperature range 290 K to 110 K: (a) 290 K, (b) 210 K, (c) 150 K, (d) 110 K. Spectrometer setings: a) - d) microwave frequency 9.436 GHz; microwave power 0.105 mW; receiver gain 5.0 x 10^4; 100 kHz modulation amplitude 1.0 G; scan range/time 1400 G/84 s.; time constant 41s.

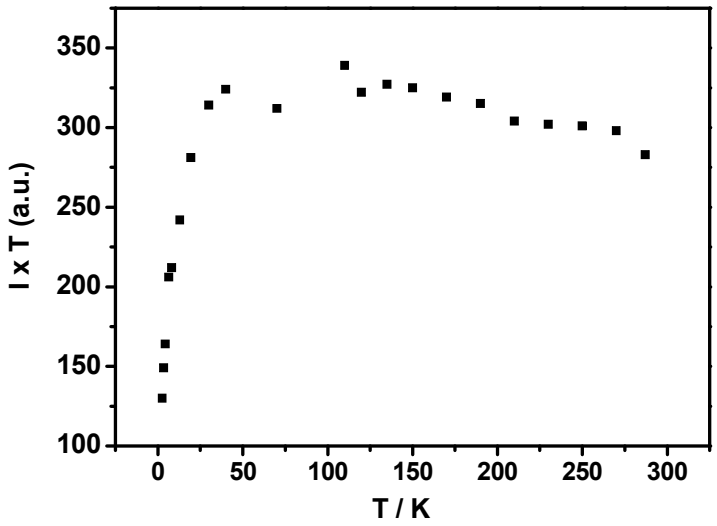

Figure S4. Temperature dependence of the product $I \times T$ for the $TTF_4[SVMo_{11}O_{40}]$ solid, derived from EPR spectra, where I is the resonance intensity and T is the temperature.