Supporting information

The hydrothermal synthesis, crystal structure and electrochemical properties of MnSb2O4

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1. High resolution PXRD

Low temperature patterns were collected at 105, 150, 200, 250, 300 and 350 K. High temperature was collected at HT1: 300, 380, 420, 460, 520, 560, 620, 660 K and HT2: 300, 380, 420, 560, 660, 760 and 850 K. The wavelength was calibrated with a NIST CeO_2 standard. The wavelength was 0.500345(2) Å, 0.500256(2) Å and 0.500176(2) Å during three different beamtimes.

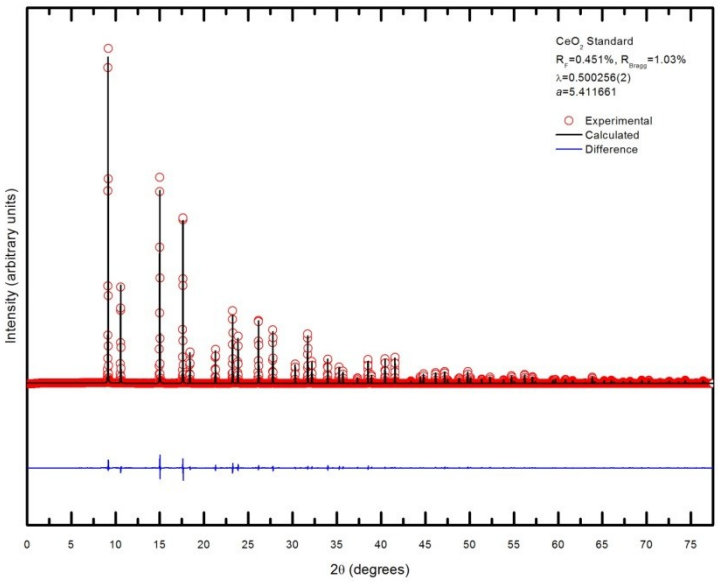

Figure S.1: Wavelength calibrations at the BL44B2 beamline using CeO_2.

In figure S.2 the cell axes for three measurements of two samples of $MnSb_2O_4$. The left and middle part shows the same trend is reproduced when re-measured. The right part shows a different sample reproducing the same trend, but to a different degree. Reliability factors and atomic displacement parameters from the Rietveld refinements are given in table S.1 and S.2.

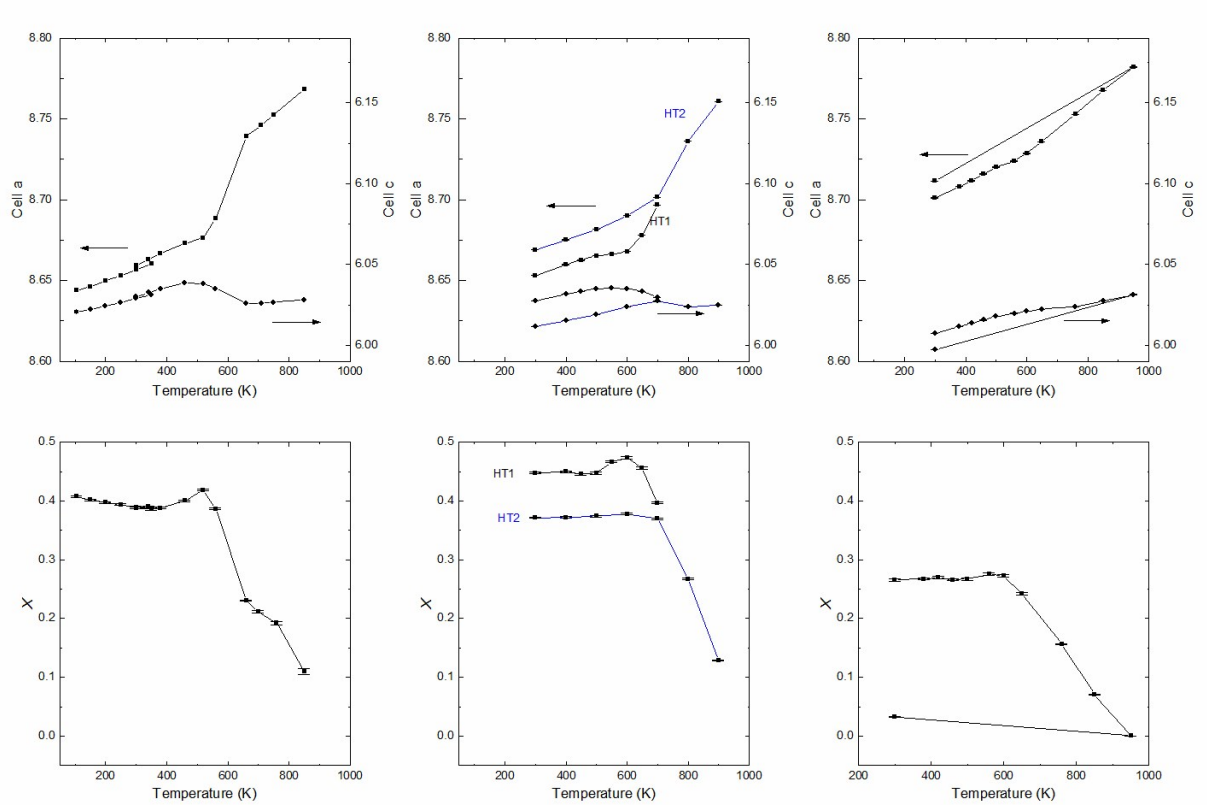

Figure S.2: Cell axes and *X* peak profile parameter for three samples measured. Left: 105-350 K (glass), 300-850 K (quartz). Middle: 300-660 K (HT1) and 300-850 K (HT2). Right: 300-950 K and 300 K.

Table S.1: Reliability factors from 105 – 850 K

T (K)	R_p	R_{wp}	R_{exp}	χ^2	R_B	R_F
LT						
105	10.1	10.8	4.2	6.597	3.38	2.41
150	10.2	10.9	4.21	6.66	3.29	2.48
200	10.5	11.1	4.26	6.805	3.4	2.53
250	10.6	11.3	4.29	6.882	3.25	2.71
300	10.9	11.4	4.33	6.909	3.25	3.04
350	10.9	11.3	4.37	6.706	3.36	3.12
HT1						
300	11.4	10.4	10.1	1069	4.43	3.41
380	11.4	10.3	10.2	1008	3.85	3.81
420	11.6	10.2	10.2	1000	3.86	3.7
460	11.6	10.3	10.4	0.9795	4.43	4.25
520	11.6	10.1	10.4	0.9434	5.67	4.51
560	12.2	10.7	10.4	1064	8.37	5.48
620	12.6	11.3	10.3	1213	18.2	5.6
660	11.2	10.1	10	1003	4.72	5.86
HT2						
300	10.2	9.88	9.04	1194	3.61	3.18
380	10.3	9.81	9.14	1154	3.57	3.65
460	10.6	9.83	9.24	1132	3.61	3.75
560	10.7	9.83	9.46	1081	3.77	5.02
660	10.6	9.45	9.59	0.971	3.7	5.8
760	9.23	8.36	8.16	1050	2.51	6.85
850	11.5	10.1	8.24	1.502	2.64	12.2

Table S.2: Atomic displacement parameters for $MnSb_2O_4$ (LT, HT1 and HT2).

Temperature (K)	U_{eq}(Mn) (Å²)	U_{eq}(Sb) (Å²)	U_{eq}(O1) (Å²)	U_{eq}(O2) (Å²)
LT				
105	0.003(4)	0.0118(7)	0.008(2)	0.007(2)
150	0.0012(4)	0.0130(7)	0.009(2)	0.008(2)
200	0.0020(5)	0.0145(8)	0.010(2)	0.007(2)
250	0.0032(5)	0.0160(8)	0.011(2)	0.008(2)
300	0.0043(5)	0.0177(8)	0.014(2)	0.011(2)
350	0.0050(5)	0.0195(9)	0.015(2)	0.012(2)
HT1				
300	0.0036(5)	0.0191(9)	0.014(2)	0.014(2)
400	0.0050(5)	0.0216(9)	0.018(2)	0.019(2)
450	0.0058(5)	0.0229(9)	0.014(2)	0.019(2)
500	0.0064(5)	0.024(1)	0.018(2)	0.021(3)
550	0.0076(6)	0.026(1)	0.018(2)	0.022(3)
600	0.0090(6)	0.028(1)	0.020(2)	0.025(3)
650	0.0115(6)	0.028(1)	0.019(2)	0.027(3)
700	0.0137(6)	0.0273(9)	0.021(2)	0.030(3)
HT2				
300	0.0049(4)	0.0147(7)	0.011(2)	0.009(2)
400	0.0067(5)	0.0173(7)	0.013(2)	0.011(2)
500	0.0090(5)	0.0204(8)	0.016(2)	0.015(2)
600	0.0114(5)	0.0232(8)	0.019(2)	0.018(2)
700	0.0137(5)	0.0261(8)	0.024(2)	0.024(2)
800	0.0210(4)	0.0278(6)	0.027(1)	0.029(2)
900	0.0263(5)	0.0315(6)	0.033(2)	0.037(2)

2. Evolution of cell parameters from SCXRD

The cell parameters of a $MnSb_2O_4$ crystal is plotted against temperature in Figure S.3. The data was acquired in one series from 100-475 K and subsequently from 475-100 K, and normalized to the first temperature at 300 K. Fluctuations in the cell parameters are either due to a systematic error in the setup or changes to the sample (decomposition/gas uptake). All measured diffraction data have been measured using the same experimental setup (SuperNova diffractometer), with the same measuring strategy and the same orientation of the crystal.

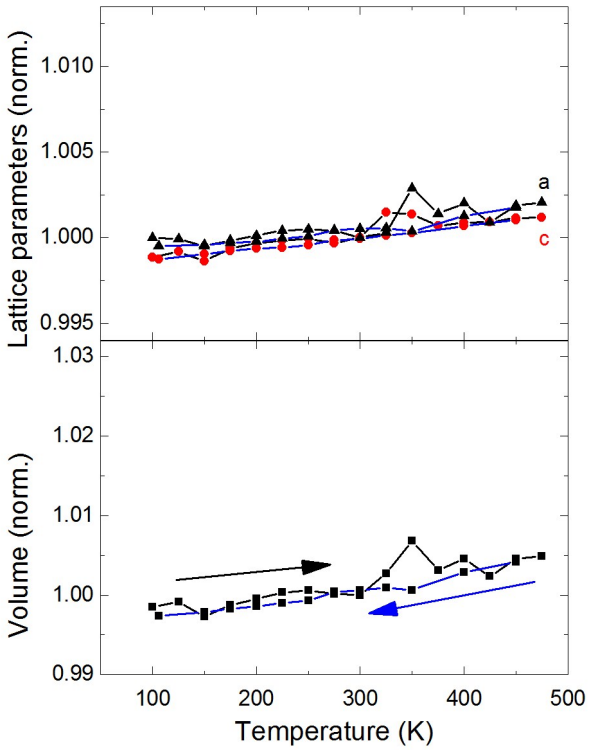

Figure S.3: Normalized cell parameters for $MnSb_2O_4$ as function of temperature. Diffraction data have been measured from 100 K up to 475 K (black lines) and then also upon cooling the sample down to 100 K (blue lines).

3. Thermogravimetry

Figure S.4 shows PXRD obtained of $MnSb_2O_4$ that was heated up to 1000 K with oxygen present and three standard PXRD patterns to describe all reflections.

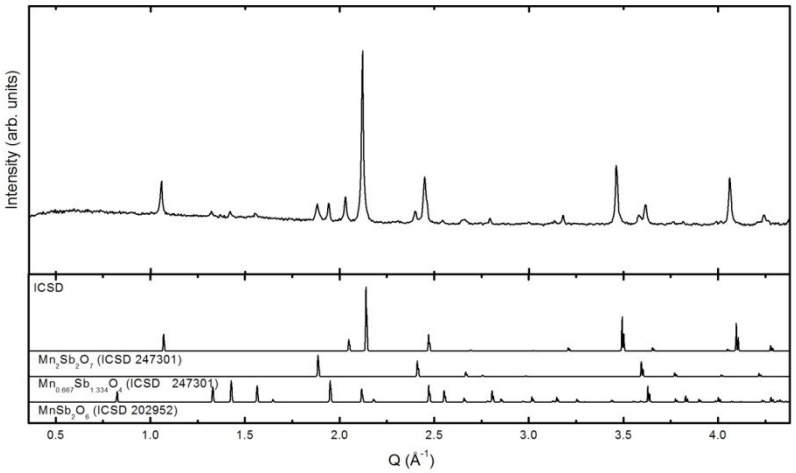

Figure S.4: PXRD of $MnSb_2O_4$ after being heated to 1000 K with oxygen present and simulated patterns from ICSD. Oxidation has occurred with a mass increase of 8%.

4. FTIR

Figure S.5 shows FTIR of the as-synthesised $MnSb_2O_4$ particles and particles that were washed with deionised water.

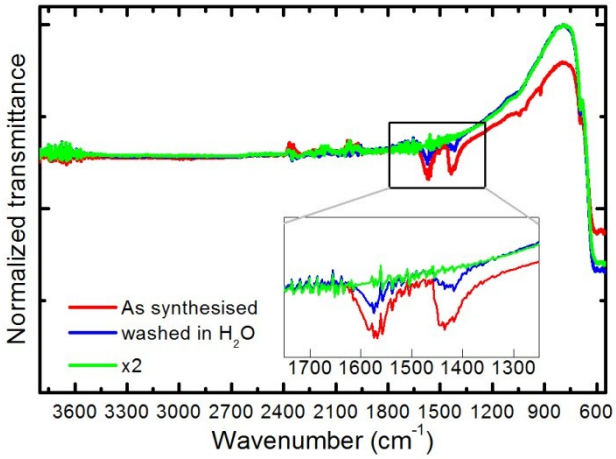

Figure S.5: FTIR of $MnSb_2O_4$ before/after wash/decant procedure.

5. Na-ion half-cell electrochemical performance

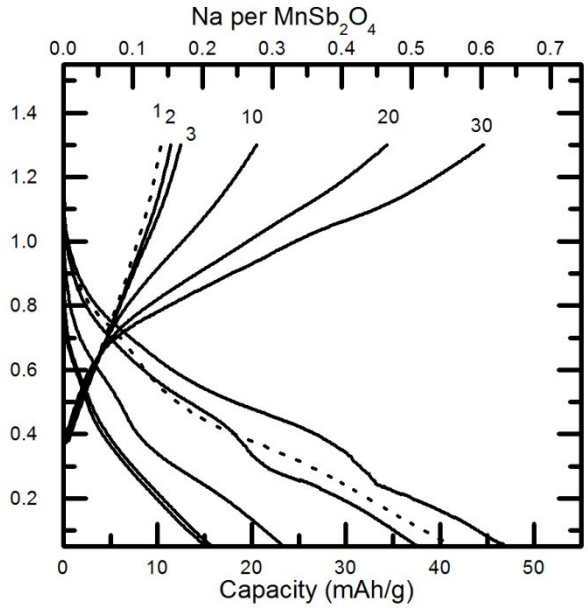

Figure S.6: Potential (vs. Na/Na^+) vs. capacity for $MnSb_2O_4$ under galvanostatic charge/discharge at 30 mA/g between 0.05-1.3 V.