

***In situ* Raman spectroscopic analysis of the lithiation and sodiation of antimony microparticles**

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Table A1. Table of CHN results for sol-gel synthesised and commercial Sb.

	Sol-gel synthesised Sb (wt. %)	Commercial Sb (wt. %)
C	23.3	0.5
H	0.7	-
N	3.6	-

Table A2. Table of discharge, charge and irreversible capacity of sol-gel synthesised and commercial antimony vs. lithium.

Sol-gel synthesised Sb capacities / mAhg ⁻¹			
Cycle number	Discharge	Charge	Irreversible
1	1082	744	338
2	685	640	45
10	603	596	8
60	566	564	2
120	550	549	2
Commercial Sb capacities / mAhg ⁻¹			
Cycle number	Discharge	Charge	Irreversible
1	853	622	231
2	650	588	62
10	597	563	35
60	451	444	7
120	265	262	3

Table A3. Table of discharge, charge and irreversible capacity of sol-gel synthesised and commercial antimony vs. sodium.

Sol-gel synthesised Sb capacities / mAhg ⁻¹			
Cycle number	Discharge	Cycle number	Irreversible
1	722	600	122
2	546	511	35
10	513	504	10
60	484	478	5
120	470	465	5
Commercial Sb capacities / mAhg ⁻¹			
Cycle number	Discharge	Cycle number	Irreversible
1	930	625	305
2	619	575	45
10	544	528	16
60	118	114	4
120	93	90	3

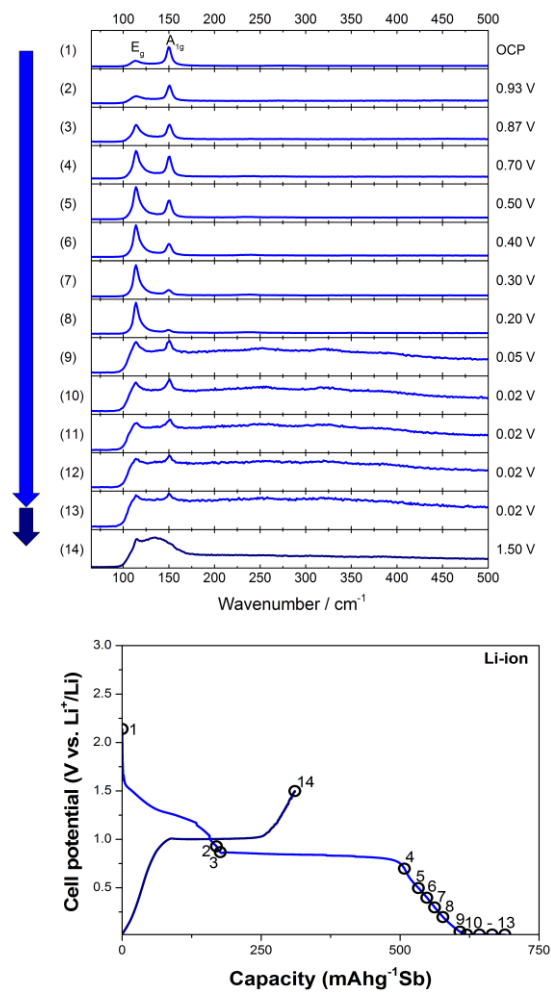


Figure A1. *In situ* Raman spectroscopy data for **commercial** antimony vs. lithium over 1 charge/discharge cycle at 60 μA g⁻¹.

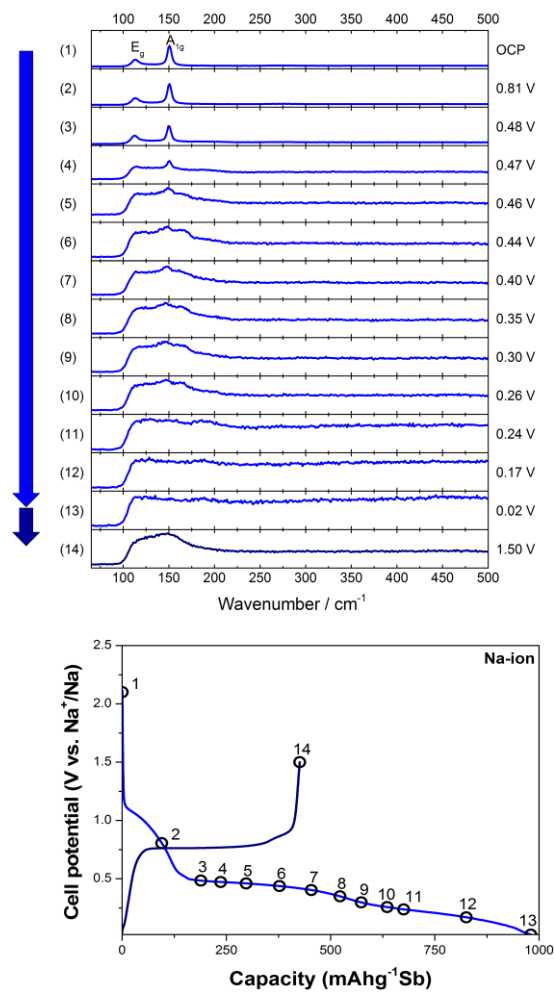


Figure A2. *In situ* Raman spectroscopy data for **commercial** antimony vs. sodium over 1 charge/discharge cycle at $60\mu\text{A g}^{-1}$.

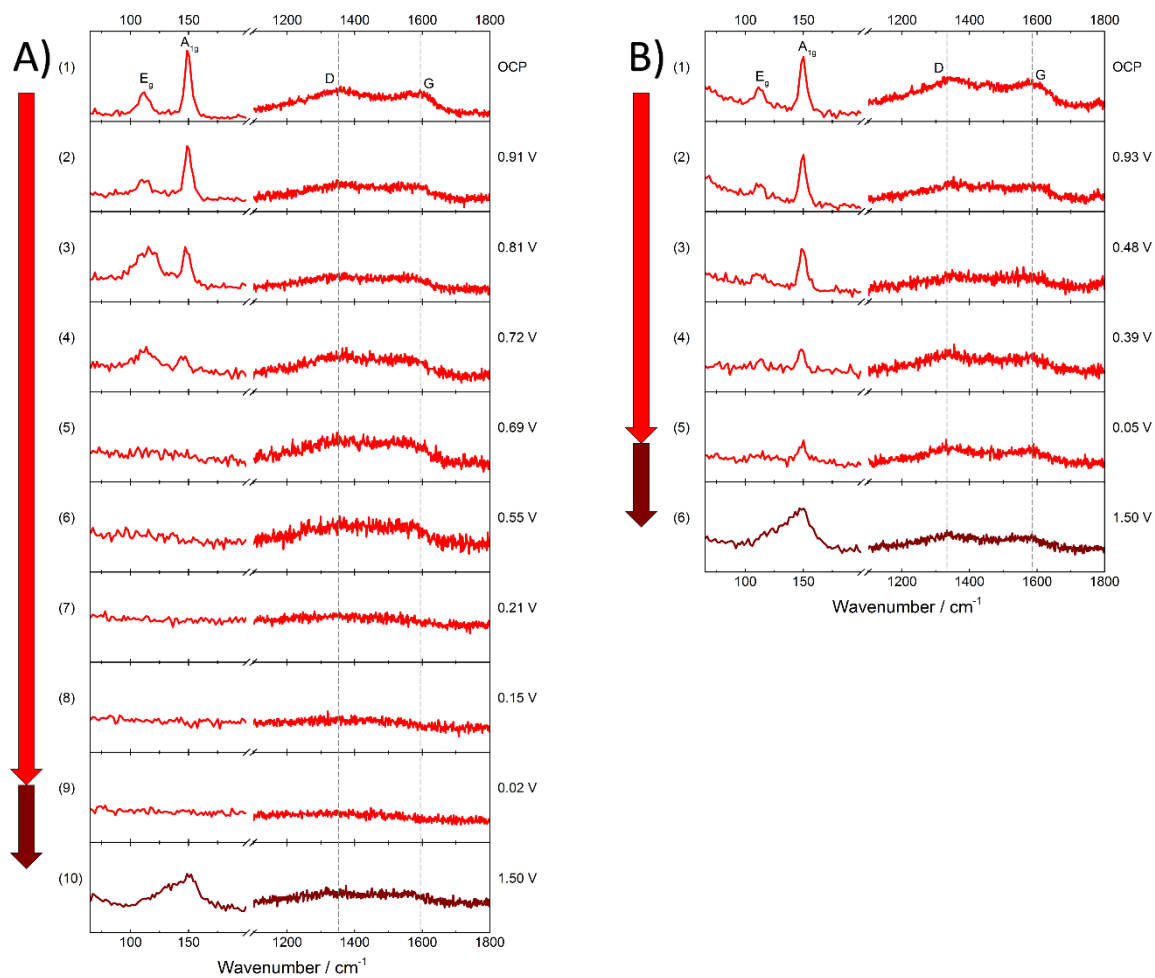


Figure A3. *In situ* Raman spectroscopy data for sol-gel synthesised antimony vs. A) lithium and B) sodium, including carbon bands.