

**Conversion of Li₂FeSbO₅ to the Fe(III)/Fe(V) Phase
LiFeSbO₅ via Topochemical Lithium Extraction**

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Published version

MARTÍNEZ DE IRUJO-LABALDE, X, SCRIMSHIRE, Alex, BINGHAM, Paul, SUARD, E and HAYWARD, MA (2022). Conversion of Li₂FeSbO₅ to the Fe(III)/Fe(V) Phase LiFeSbO₅ via Topochemical Lithium Extraction. *Chemistry of Materials*, 34 (5), 2468-2475.

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Conversion of $\text{Li}_2\text{FeSbO}_5$ to the Fe(III)/Fe(V) phase LiFeSbO_5 via topochemical lithium extraction.

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1. Thermogravimetric characterisation of $\text{Li}_{2-x}\text{FeSbO}_5$.

To establish the chemical composition of the delithiated phase, thermogravimetric data were collected as a sample of $\text{Li}_{2-x}\text{FeSbO}_5$ was heated to 1000 °C in air. PXRD data collected from the product of this process (Figure S1) indicated $\text{Li}_{2-x}\text{FeSbO}_5$ had decomposed to a mixture of:

96.1 % by mass FeSbO_4

0.80 % by mass LiSbO_3

3.1% by mass $\text{LiFe}_2\text{SbO}_6$

To give an overall composition of $\text{Li}_{0.08}\text{FeSbO}_{4.04}$.

When combined with the observed mass loss of 8.31% (Figure S2), this indicated a composition of $\text{Li}_{0.99(5)}\text{FeSbO}_5$.

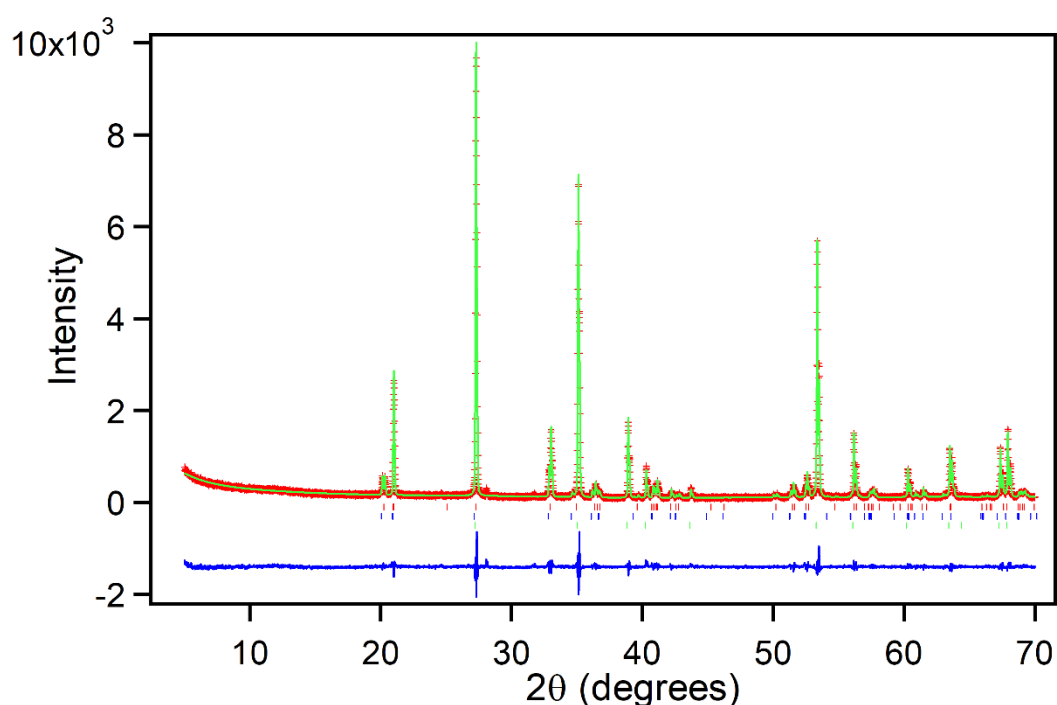


Figure S1. Fit to PXRD data collected from a sample of $\text{Li}_{2-x}\text{FeSbO}_5$ after being heated to 1000 °C in air in a thermogravimetric balance. Tick marks indicate peak positions for FeSbO_4 (Green), LiSbO_3 (blue) and $\text{LiFe}_2\text{SbO}_6$ (red).

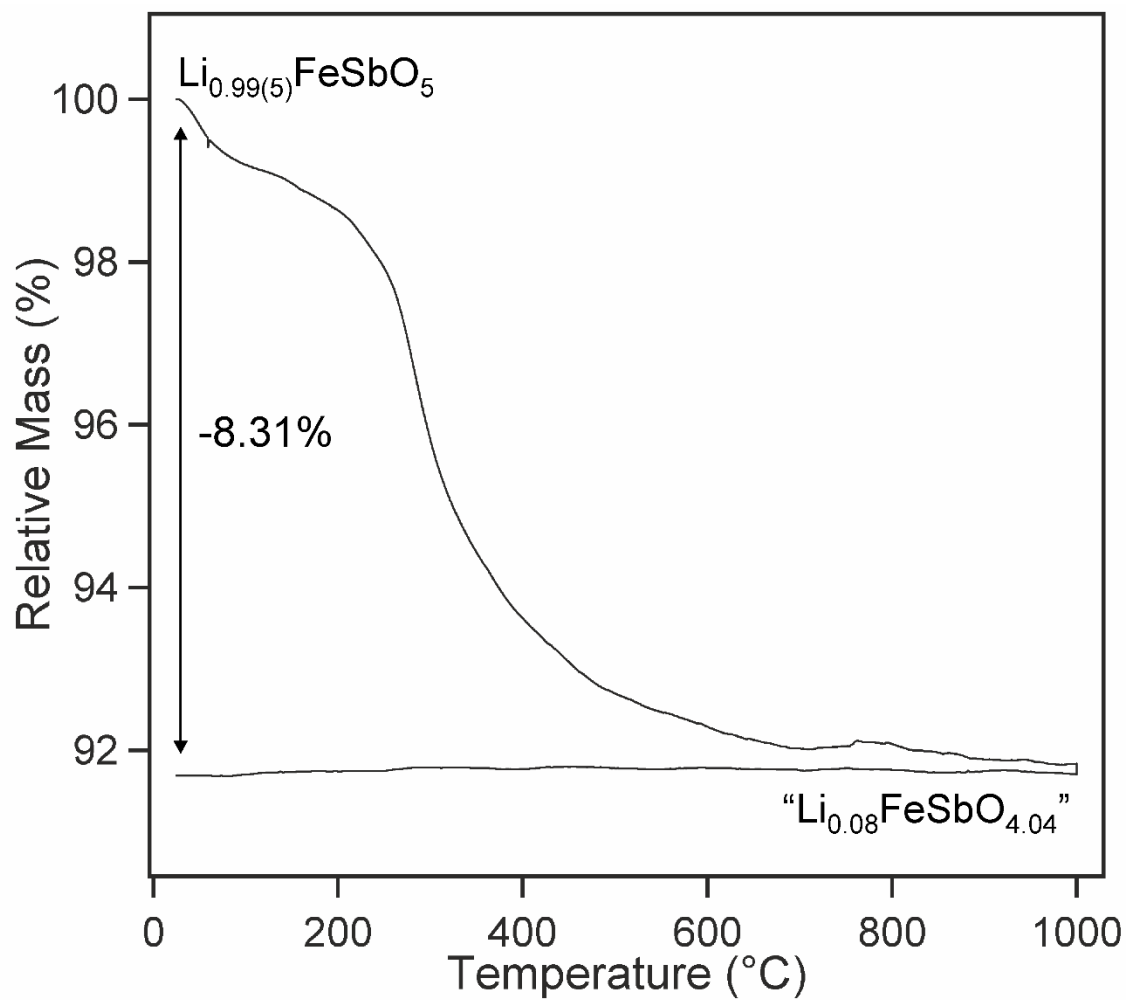


Figure S2. Thermogravimetric data collected as $\text{Li}_{2-x}\text{FeSbO}_5$ was heated in air to 1000 °C.

2. Structural characterization of Li₂FeSbO₅

Cation	Anion	Bond length (Å)	BVS
Li(1)	O(1) × 2	2.10(1)	Li+0.88
	O(4) × 2	2.45(3)	
	O(5) × 2	2.08(2)	
Li2	O(2) × 2	2.44(3)	Li+0.96
	O(3) × 2	1.91(3)	
	O(3) × 2	2.29(1)	
Li3	O1	1.92(3)	Li+0.79
	O2	2.19(3)	
	O3	2.72(3)	
	O4	2.31(3)	
	O4	2.27(3)	
	O5	2.29(3)	
Fe	O1	1.92(1)	Fe+2.94
	O3	1.93(1)	
	O5	1.76(1)	
	O5	1.91(1)	
Sb1	O(1) × 2	1.90(2)	Sb+4.99
	O(2) × 2	2.05(2)	
	O(4) × 2	2.00(1)	
Sb2	O(2) × 2	1.85(1)	Sb+5.42
	O(3) × 2	2.00(2)	
	O(4) × 2	2.02(2)	

Table S1. Selected bond lengths and bond valence sums from the refined structure of Li₂FeSbO₅.

3. Structural characterisation of LiFeSbO₅

Cation	Anion	Bond length (Å)	BVS
Fe1	O(2)	1.891(11)	Fe+4.04
	O(3)	1.909(13)	
	O(6)	1.907(10)	
	O(6)	2.154(12)	
	O(9)	1.822(20)	
	O(9)	1.931(10)	
Fe2	O(1)	2.223(26)	Fe+2.09
	O(4)	1.940(16)	
	O(5)	2.066(10)	
	O(5)	2.294(11)	
	O(10)	2.244(16)	
	O(10)	2.263(11)	
Sb1	O(1)	1.743(24)	Sb+5.48
	O(2)	2.037(12)	
	O(3)	2.187(14)	
	O(4)	2.088(14)	
	O(7)	1.929(11)	
	O(8)	2.030(11)	
Sb2	O(3)	2.306(13)	Sb+5.62
	O(4)	2.028(12)	
	O(5)	1.790(11)	
	O(6)	2.033(11)	
	O(7)	2.046(14)	
	O(8)	1.832(13)	

Table S2. Selected bond lengths and bond valence sums from the refined structure of LiFeSbO₅.