Ultra-rapid microwave synthesis of Li3-*x*-*y*M*x*N (M= Co, Ni and Cu) nitridometallates.

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**SUPPLEMENTARY INFORMATION**

Table S1 Lattice parameters and cell volumes obtained from indexed powder X-ray data for samples (1-7).

|  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- |
| Sample | **M** | x | a/ Å | c/ Å | V/ Å3 |
| **1** | Ni | 0.1 | 3.668(2) | 3.8416(5) | 44.77(2) |
| **2** | Ni | 0.22 | 3.688(1) | 3.741(4) | 44.05(5) |
| **3** | Ni | 0.29 | 3.697(6) | 3.72(2) | 44.08(2) |
| **4** | Co | 0.09 | 3.667(3) | 3.8267(8) | 44.56(4) |
| **5** | Co | 0.21 | 3.678(2) | 3.804(3) | 44.57(4) |
| **6** | Co | 0.3 | 3.697(7) | 3.772(2) | 44.65(9) |
| **7** | Cu | 0.11 | 3.670(3) | 3.806(7) | 44.32 (4) |
|  |  |  |  |  |  |

Table S2 Thermal displacement parameters for atoms in samples 1-7.

|  |  |  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- | --- | --- |
| **Atoms** | ***U*a x 100****/A2** | **1** | **2** | **3** | **4** | **5** | **6** | **7** |
| **M/Li(1)** | ***Ui / Ue*** | 2.64 | 1 | 0.14 | 1.58 | 3.90 | 2.49 | 1.33 |
| (0,0,0.5) | ***U11*** | 3.8(9) | - | 0.2(1) | 2.1(3) | 4.6(7) | 3.9(4) | 2.5(3) |
|  | ***U33*** | 0.2(9) | - | 0.1(3) | 0.4(4) | 2.31(3) | 1.4(1) | 1.5(3) |
| **N** | ***Ui / Ue*** | 1.55 | 2.35 | 2.98 | 1.21 | 1.58 | 2.33 | 1.69 |
| (0,0,0) | ***U11*** | 1.33(6) | 1.56(5) | 1.96(5) | 1.00(5) | 1.38(7) | 1.99(4) | 1.08(4) |
|  | ***U33*** | 1.9(1) | 3.9(1) | 4.97(2) | 1.60(7) | 2.0(1) | 2.98(7) | 2.9(8) |
| **Li(2)** | ***Ui / Ue*** | 3.59 | 4.28 | 3.82 | 2.37 | 2.31 | 3.12 | 4.79 |
| (0.3333, 0.6667, 0) | ***U11*** | 1.6(3) | 2.7(3) | 2.0(2) | 1.5(2) | 1.5(3) | 2.3(2) | 1.6(2) |
|  | ***U33*** | 7.5(9) | 7.3(9) | 7.4(8) | 4.0(4) | 3.9(7) | 4.7(4) | 9.8(8) |
|  |  |  |  |  |  |  |  |  |

a *U11* = *U22* = 2 *U12*and *U13* = *U23* = 0.



Fig. S1 Photograph showing a reaction in progress demonstrating the occurrence of an orange-yellow glow following Li evaporation.



Fig. S2 First two charge/discharge cycles of Li2.7-*x*Ni0.3N (3) between 0 and 1.4 V at a C/30 rate.



Fig. S3 Discharge capacity for the first five cycles of Li2.7-*x*Ni0.3N (3) tested galvanostatically against lithium metal between 0 and 1.4 V at a C/30 rate.



Fig. S4 First two charge/discharge cycles of Li2.8-*x*Co0.2N (5) between 0 and 1.4 V at a C/30 rate.



Fig. S5 Discharge capacity for the first five cycles of Li2.8-*x*Co0.2N (5) tested galvanostatically against lithium metal between 0 and 1.4 V at a C/30 rate.