CLAIMS

1. Process for the recovery of metals M from a Li-containing starting material, wherein M comprises Ni and Co, comprising the steps of:

Step 1: Providing said starting material, comprising Li-ion batteries or their derived products;

Step 2: Removing Li in an amount of more than the maximum of (1) 30% of the Li present in said starting material, and (2) a percentage of the Li present in said starting material determined to obtain a Li:M ratio of 0.70 or less in a subsequent acidic leaching step, by using either one or more of:

- a pyrometallurgical smelting process using slag-formers, thereby producing one or more of a Li-bearing slag phase and Li-fumes, and a Li-depleted Ni-Co-bearing phase susceptible to be acid-leached;
- a thermal treatment process using a reducing agent, thereby producing a Ni-Co-bearing residue containing at least one water-soluble Li-compound, and removing said at least one Li-compound by washing with an aqueous solution, thereby obtaining a Li-depleted Ni-Co-bearing residue susceptible to be acid-leached;
- a hydrometallurgical leaching process using an aqueous or acidic solution, thereby leaching Li from said starting material, wherein Ni and Co are at least partially insoluble, and solid-liquid separation, thereby obtaining a Li-depleted Ni-Co-bearing residue susceptible to be acid-leached;

Step 3: subsequent leaching using relative amounts of Li-depleted Ni-Co-bearing material, obtained in step 2, and a mineral acid, thereby obtaining a Ni- and Co-bearing solution; and,

Step 4: crystallization of Ni, Co, and optionally Mn, wherein step 4 is performed by lowering the temperature of the Ni- and Co-bearing solution and/or by removing water from the Ni- and Co-bearing solution and/or by solvent displacement crystallization, characterized in that the process is free from a solvent extraction or ion exchange step in which Ni and/or Co are extracted from the Ni- and Co-bearing solution.

2. Process according to claim 1, wherein thermal treatment process of step 2 is performed at a temperature above 250 °C.

3. Process according to claim 1 or 2, wherein the step of removing said at least one water-soluble Li-compound by washing with an aqueous solution is performed at a pH of 2.5 or higher.

4. Process according to any one of claim 1 to 3, wherein said at least one water-soluble Li-compound of step 2 comprises one or more of Li_2O, Li_2CO_3, $LiHCO_3$, LiCI, Li_2SO_4, LiF, LiOH.

5. Process according to any one of claim 1 to 4, wherein the mineral acid is one or more of HNO_3, H_2SO_4 and HCl.

6. Process according to any one of claims 1 to 5, wherein the Ni-Co-bearing phase of the pyrometallurgical smelting process of step 2 is comminuted to a particle size distribution with an average diameter of 10 mm or less, preferably 1 mm or less, and more preferably 0.5 mm or less.

7. Process according to any one of claims 1 to 6, wherein the Ni- and Co-bearing solution from step 3 also contains Cu and/or Fe and/or Al, wherein the process comprises a purification step between step 3 and step 4, wherein Cu and/or Fe and/or Al are removed from the Ni- and Co-bearing solution.

8. Process according to any one of claims 1 to 7, wherein the Ni- and Co-bearing solution from step 3 also contains Mn, wherein the process comprises a Mn-removal step between step 3 and step 4, wherein Mn is removed from the Ni- and Co-bearing solution.

9. Process according to any one of claims 1 to 8, wherein before step 4 the ratio of Ni to Co in the Ni- and Co-bearing solution is adjusted to a desired value by addition of Ni and/or Co in dissolved form or as one or more compounds which are soluble in the Ni- and Co-bearing solution.

10. Process according to any one of claims 1 to 9, wherein before step 4 the ratio of Ni to Co to Mn in the Ni- and Co-bearing solution is adjusted to a desired value by addition of one or more of Ni, Co and Mn in a dissolved form or as one or more compounds which are soluble in the Ni- and Co-bearing solution.

11. Process according to any one of claims 1 to 10, wherein the Ni- and Co-bearing solution contains more Ni than Co, when expressed in g/l.

12. Process according to any one of claims 1 to 11, wherein the Ni- and Co-bearing solution contains more Ni than the sum of Co and Mn, when expressed in g/l.

13. Process according to any one of claims 1 to 12, comprising a purification step between step 3 and step 4, wherein Co is selectively removed from the Ni- and Co-bearing solution, resulting in a Co-depleted, Ni-bearing solution, suitable for obtaining Ni crystals.

14. Process according to any one of claims 1 to 13, wherein the starting material contains Ni, Co, Li, Al, F, and one or more of Cu and Fe, wherein the pyrometallurgical smelting process of step 2 is a reducing smelting of the starting material with fluxing agents, wherein the Ni-Co-bearing phase is an alloy comprising a major part of Ni, Co, Cu and at least part of the Fe and, wherein the alloy is depleted in Li, Al and F.