Supplementary Experimental Details

Synthesis of 1
Hmpo (0.196 g, 2 mmol) and tbutylbenzoic acid (0.376 g, 2 mmol) were mixed in methanol (50 cm³) and added to an aqueous solution of nickel (II) tetrafluoroborate (50% b/w, 0.929 g, 2 mmol). The light apple green solution was stirred for 5m. before addition of sodium methoxide solution (4 cm³, 0.5 mol/dm³, 2 mmol) and refluxing for 24h. The solvent was then removed in vacuo leaving a lime green solid that was dissolved in ethyl acetate (25 cm³), from which crystals of 8 grew over ten days, that were suitable for X-Ray diffraction from a synchrotron source. Yield, 65mg, 7% by % Ni. Elemental analysis: Calculated (found): C 49.63 (48.76); H 5.34 (5.33); N 7.12 (7.07); F 5.23 (6.19), Na 1.59 (1.60); Ni 16.17 (15.81).

Synthesis of 2
Hmpo (0.196 g, 2 mmol) and tbutylbenzoic acid (0.376 g, 2 mmol) were mixed in methanol (50 cm³) and added to an aqueous solution of nickel (II) tetrafluoroborate (50% b/w, 0.929 g, 2 mmol). The light apple green solution was stirred for 5m. before addition of sodium methoxide solution (4 cm³, 0.5 mol/dm³, 2 mmol) and excess sodium azide (0.65g, 10mmol), which turned the solution notably darker and further stirring for 72h. The solvent was then removed in vacuo at room temperature leaving a green solid that was dissolved in ethyl acetate (25 cm³), and filtered to leave an apple green solution. From this solution crystals of 12 grew over a period of one week, that were suitable for X-Ray diffraction from a synchrotron source. Yield, 47 mg, 7% by % Ni. Microanalysis suggests that some ethyl acetate is lost in powder form, and that only four molecules remain. Elemental analysis for the molecule without these four ethyl acetate molecules C67H101N56Na2Ni8O20: Calculated (found): C 31.85 (31.51); H 4.03 (3.94); N 31.05 (29.99).

Synthesis of 3
Hmpo (0.196 g, 2 mmol) and benzoic acid (0.376 g, 2 mmol) were mixed in methanol (50 cm³) and added to an aqueous solution of nickel (II) tetrafluoroborate (50% b/w, 0.929 g,
2 mmol). The light apple green solution was stirred for 5m. before addition of sodium methoxide solution (4 cm$^3$, 0.5 mol/dm$^3$, 2 mmol) and excess sodium azide (0.65 g, 10 mmol), which turned the solution notably darker and further stirring for 72h. The solvent was then removed in vacuo at room temperature leaving a lime green solid that was dissolved in ethyl acetate (25 cm$^3$), and filtered to leave an apple green solution. From this solution crystals of 13 grew over a period of one week, that were suitable for X-Ray diffraction from a synchrotron source. Yield, 83 mg, 13% by % Ni. Microanalysis indicates that the terminal ethyl acetates are lost in powder form and replaced by water molecules. Elemental analysis for replacement with water molecules as C$_{54}$H$_{82}$N$_{56}$Na$_2$Ni$_8$O$_{22}$: Calculated (found): C 27.22 (26.72); H 3.47 (3.36); N 32.91 (32.98).