

***Synthesis of [CoAl<sub>2</sub>(acac)<sub>4</sub>(O<sup>i</sup>Pr)<sub>4</sub>] 5.***

Co(acac)<sub>2</sub> (0.451 g, 1.75 mmol) and Al(O<sup>i</sup>Pr)<sub>3</sub> (0.701g, 3.44 mmol) were dissolved in 4 ml of toluene and warmed up to reflux (with complete dissolution of the aluminium alkoxide). The pinkish purple solution thus obtained was cooled to +4°C in the refrigerator (2h) and then the acetylacetone (0.35 ml, ≈3.5 mmol) was added drop-wise by a syringe on vigorous stirring. No visible changes occurred with the solution. It was then evaporated to dryness *in vacuo* leaving (by microscopic observation) exclusively the cubo-octahedral crystals displaying the chemical analysis and spectral characteristics identical to those for **5**.

***Synthesis of [NiAl<sub>2</sub>(acac)<sub>4</sub>(O<sup>i</sup>Pr)<sub>4</sub>] 6.***

Ni(acac)<sub>2</sub> (0.364 g, 1.42 mmol) and Al(O<sup>i</sup>Pr)<sub>3</sub> (0.578g, 2.83 mmol) were dissolved in 3 ml of toluene and warmed up to reflux (with complete dissolution of the aluminium alkoxide). The bluish violet solution thus obtained was cooled to +4°C in the refrigerator (2h) and then the acetylacetone (0.28 ml, ≈2.8 mmol) was added drop-wise by a syringe on vigorous stirring. No visible changes occurred with the solution. It was then evaporated slowly to dryness *in vacuo* leaving (by microscopic observation) exclusively the bluish violet cubo-octahedral crystals (quantitative yield).

***Synthesis of [MgAl<sub>2</sub>(acac)<sub>4</sub>(O<sup>i</sup>Pr)<sub>4</sub>] 7.***

Mg(acac)<sub>2</sub> (0.644 g, 2.90 mmol) and Al(O<sup>i</sup>Pr)<sub>3</sub> (1.184g, 5.80 mmol) were dissolved in 5 ml of toluene and warmed up to reflux (with complete dissolution of the aluminium alkoxide). The colorless solution thus obtained was cooled to +4°C in the refrigerator (2h) and then the acetylacetone (0.58 ml, ≈5.8 mmol) was added drop-wise by a syringe on vigorous stirring. No visible changes occurred with the solution. It was

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then evaporated slowly to dryness *in vacuo* leaving (by microscopic observation)  
exclusively the colorless cubo-octahedral crystals (quantitative yield).