

different electronic and structural effects when compared with the analogues. Very little has been done on poly(pyrazolyl)borate systems bearing electron withdrawing substituents. The electron withdrawing groups in polyfluorinated ligands commonly improve the volatility, oxidation resistance, thermal stability, and solubility of metal complexes. To our knowledge, no poly(azolyl)borates containing a -NO<sub>2</sub> function have been prepared, presumably due to difficulties in the synthesis of ligands having both a hydride and a nitro group. However, a poly(azolyl)borate containing a -NO<sub>2</sub> substituent could be of interest due to its high coordinative flexibility from κ<sup>4</sup>- to μ<sup>4</sup>-N<sub>2</sub>O<sub>2</sub> coordination ability.

We report here on the syntheses and structural investigations of main-group metal elements Ca, Ba and Sr with the hydrotris(3-methylpyrazolyl)borate, the hydrotris(1,2,4-triazolyl)borate and the new hydrotris(3-nitro-1,2,4-triazolyl)borate, an emerging category of electron withdrawing substituted scorpionate ligands.

**Keywords:** metalloorganic chemistry, pyrazolyl, crystal structures

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#### Synthesis and Crystal Structure of [Co(HL)L]·CH<sub>3</sub>OH (H<sub>2</sub>L = Pyridoxal S-methylisothiosemicarbazone)

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Brown single crystals of the title compound, C<sub>21</sub>H<sub>29</sub>CoN<sub>8</sub>O<sub>5</sub>S<sub>2</sub>, were prepared by the reaction of MeOH solutions of Co(Oac)<sub>2</sub>·4H<sub>2</sub>O and H<sub>2</sub>L·H<sub>2</sub>O in mole ratio 1 : 1. The complex has a *mer*-octahedral configuration with two non-equivalent tridentate ligands with O,N,N donors: one of the ligands having deprotonated isothiosemicarbazido fragment, whereas for the other additional deprotonation involves the pyridine nitrogen. The compound crystallizes in the P 2<sub>1</sub>/c space group with a = 11.375(3) Å, b = 14.263(5) Å, c = 15.854(6) Å, β = 99.63(2)°, V = 2535.9(15) Å<sup>3</sup>. X-ray diffraction data were recorded on an Enraf-Nonius CAD-4 diffractometer with graphite monochromated Mo Kα radiation (λ = 0.71069 Å). Anisotropic refinement of all non-hydrogen atoms converged to R = 0.0567 for 4966 independent reflections and 356 parameters.

**Keywords:** cobalt(III) complex, pyridoxal S-methylisothiosemicarbazone, mer-octahedral configuration

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#### Novel Five-membered B-N-C Heterocycles

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Stable N-heterocyclic carbenes [1] have found a multitude of applications as catalyst components due to their remarkable ligand properties [2]. Their growing significance prompted us to investigate the stability of carbenes with inorganic backbones, as well as influence of the inorganic backbone on the ligand properties of the carbon ligand. To date, only one N-heterocyclic carbene with an inorganic backbone (:C(NR)<sub>2</sub>PR') has been reported [3].

The tremendous importance of cyclopentadienyl (Cp) as a ligand in organometallic chemistry has prompted the design and synthesis of numerous five-membered, anionic heterocyclic analogs involving besides C also B, N, P, O and S. To our knowledge not more than three carbon atoms have been replaced by heteroatoms in heterocyclic analogs of Cp. We decided to investigate the synthesis, stability and coordination chemistry of Cp analogs derived from 1,2-diaza-3,5-diborolidine, where two C<sub>2</sub> fragments have been replaced with (B,N) pairs. The structures of novel five-membered B-N-C heterocycles will be presented.

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Despagnet-Ayoub E., Grubbs R. H., *J. Am. Chem. Soc.*, 2004, **126**, 10198.

**Keywords:** coordination complexes, heterocycles, catalysts

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#### A new Trinuclear Europium(III) Complex [Eu<sub>3</sub>(C<sub>10</sub>H<sub>9</sub>N<sub>3</sub>O<sub>2</sub>)<sub>4</sub>(C<sub>10</sub>H<sub>9</sub>N<sub>3</sub>O)(CF<sub>3</sub>CO<sub>2</sub>)<sub>6</sub>](C<sub>6</sub>H<sub>6</sub>)·2H<sub>2</sub>O

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The new trinuclear Europium complex consists of composite ligands which include four di-2-pyridylamine N,N' dioxide, one di-2-pyridylamine N-oxide and six trifluoroacetate groups. All europium atoms are nine-coordinate: the coordination sphere of the Eu<sub>2</sub> and Eu<sub>3</sub> ions is completed by the amine N atom and two O atoms of each di-2-pyridylamine N,N' dioxide, and the carboxylate O atoms of three trifluoroacetate groups. The middle Eu<sub>1</sub> ion, which has no contact with trifluoroacetate groups, is coordinated by di-2-pyridylamine N,N' dioxide molecules and a di-2-pyridylamine N-oxide molecule. There are triple bridge bonds through the O atoms of the di-2-pyridylamine N,N' dioxide between all adjacent Eu ions, thus forming a discrete trinuclear complex. The Eu<sub>1</sub>...Eu<sub>2</sub> and Eu<sub>1</sub>...Eu<sub>3</sub> distances are 3.831 and 3.783 Å, respectively.

The benzene molecule and two water molecules should be included in this structure, because the complex single crystal has been prepared by solvothermally synthesized method.

Crystal Data: monoclinic; P2(1)/c; a=22.084(4) Å, b=16.973(3) Å, c=21.398(4) Å; β= 97.14(3)°; V=7959(3) Å<sup>3</sup>; Z= 4; Final R indices [I>2σ(I)]: R1 = 0.0570, wR2 = 0.1430; Goodness-of-fit on F<sup>2</sup>: 1.022.

**Keywords:** crystalline structure, rare-earth materials, pyridine complexes



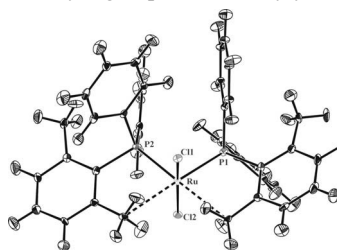
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#### Non Classical vs. Classical Metal...H<sub>3</sub>C-C Interactions: A Neutron Diffraction Study of a 14-Electron Ruthenium(II) System

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A neutron diffraction study establishes the precise nature of the δ agostic interactions in the complex RuCl<sub>2</sub>[PPh<sub>2</sub>(2,6-Me<sub>2</sub>C<sub>6</sub>H<sub>3</sub>)]<sub>2</sub> (**1**). By contrast to the classical agostic bonding, it is shown that two ortho-methyl groups of the xylyl substituents interact with the



unsaturated metal centre through two C-H bonds each. The result is also substantiated by the NMR data in solution. [1], [2] Re-examination of all the X-ray structures with β, γ, δ and ε M...H<sub>3</sub>C-C moieties as well as DFT calculations on models of **1** allow to conclude that the agostic interactions span the range between the classical (M...η<sup>2</sup>-HC) and the non-classical (M...η<sup>3</sup>-H<sub>2</sub>C) types, depending on the number of atoms between the metal and the methyl group.

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**Keywords:** neutron structure determination, hydrogen bonding of coordination compounds, databases