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Acetylide complexes of diamagnetic gallium(III) and paramagnetic iron(III) porphyrins.

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Streszczenie

Five-coordinate f(TPP)GaHI(C=CR)} complexes (TPP is the dianion of tetraphenylporphyrin; R is n-Pr or Ph) and ((TTP)Fen,(C^CR)} (TPP is the dianion of tetrakis(p-tolyl)porphyrin); R = n-Pr or Ph) have been obtained by the reaction of {(P)MinCl} (M = Ga or Fe, P = porphyrin dianion) with the appropriate lithium acetylide. Red blocks of {(TPP)GaIH(C=CPrn)} (C49H35GaN4) crystallize in the orthorhombic space group Pnal\ with a = 12.032(2) A, b = 27.502(5) Á, and c = 11.138(2) Á at 120 K with Z = 4. Refinement of 487 parameters with2818 reflections gave R - 0.043 and /?w = 0.040. The complex consists of the five-coordinate gallium bound toa saddle-shaped porphyrin (Ga-N distance (average) = 2.047 Á) and a linear axial propyl acetylide (Ga-C distance = 1.949(6) A). The iron complexes decompose during attempts at chromatographic purifications and consequently have been examined only in solution. In noncoordinating solvents (toluene, dichloromethane) they exhibit1H NMR spectra that are characteristic of high-spin (S = 5/2) species rather than the low-spin (5 = '/2) ground state found

for similar complexes with axial alkyl or aryl substituents. Addition of tetrahydrofuran (THF) or pyridine (py)results in the conversion to low-spin (5 = */2), six-coordinate species, {B(TTP)Feln(C^CR)} (B = THF or py).The iron(III) acetylide complexes are not reactive toward dioxygen but are converted into {(TTP)FeMIBr} when treated with bromine in toluene solution.

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