

o.m12.p1 Investigations of two nickel phosphine complexes . D.D.Ellis, A.L. Spek, *Bijvoet Center for Biomolecular Research, Crystal and Structural Chemistry, Utrecht University, Padualaan 8, 3584 CH Utrecht, The Netherlands.*

Keywords: phosphine, database analysis.

Under fairly harsh reaction conditions two nickel(I) complexes were formed as by-products in recrystallisation processes.¹ The crystallographic properties of [NiCl(PPh₃)₂].thf and the trigonal form of [NiCl(PPh₃)₃] have been determined using single crystal diffraction. The nickel centre, in both cases, has a *d⁸* configuration and a formal oxidation state of (I).

Other low valent three coordinate nickel(0, II) species have been synthesized using sterically demanding phosphine ligands, even so, trivalent monomeric nickel complexes are relatively uncommon. Three coordinate trigonal planar geometries exist in nickel(0, II) complexes but, to our knowledge, only one other trigonal planar nickel(I) species, [Ni{N(SiMe₃)₂} (PPh₃)₂],² has been structurally characterized.

Tetrahedral complexes, in general, are more numerous than the three coordinate analogues, but again only a few nickel compounds have been reported. [NiCl(PPh₃)₃] is isomorphous to a range of [(Ph₃E)₃MX] species (M = transition metal; E = P, As or Sb, X = for example halides, SCN), and exists in a second form as the toluene solvate.³ Statistical comparisons between the trigonal and triclinic configurations, as well as numerical comparisons of the geometric parameters of [NiCl(PPh₃)₂].thf and the trigonal form of [NiCl(PPh₃)₃], will be presented.⁴

This poster will also show the conformational properties of the triphenylphosphine groups coordinated to trigonal planar and tetrahedral metal complexes.

o.m12.p2 Crystal structures of heteronuclear Cu/Zn complexes with ethylenediamine obtained by direct synthesis. O.V. Prima, S.R. Petrusenko, V.N. Kokozay. *Department of Chemistry, Kiev Shevchenko University, Vladimirskaya St. 64; 01033 Kiev, UKRAINE Fax: +380 44 296 2467 E-mail: kokozay@chem.kiev.ua*

Keywords: crystal structure, ethylenediamine, mixed-metal complexes.

The synthesis of a coordination compound from a zero-valent metal is a fascinating and promising approach to the production of complexes of unusual stoichiometry and structure [1]. Using this approach to synthesis of heteronuclear complexes we succeeded in obtaining a number of mixed-metal Cu/Zn complexes under simple one-step interaction of copper powder and zinc oxide in the dimethyl sulfoxide (dmsO) solutions of ammonium salts and ethylenediamine (en).

The two novel complexes [Cu(en)₂ZnCl₄].dmsO (**1**) and [Cu(en)₂Zn(CH₃COO)₄] (**2**) were characterized by X-ray crystallography. Crystal structures were solved by direct method and refined by a full-matrix least-squares technique. The crystallographic computations were performed with SHELXS-86 and SHELXL-93.

Crystal data:

(1) C₆H₂₂Cl₄N₄OSCuzn, monoclinic, space group *P2₁/c*, a = 11.820(2) Å, b = 8.247(2) Å, c = 18.169(4) Å, β = 94.33(3)°, V = 1766.1(7) Å³, Z = 4, R = 0.0675 and wR = 0.1616 for 2977 independent reflections with I > 2σ_I.

(2) C₁₂H₂₈N₄O₈Cuzn, monoclinic, space group *C2/c*, a = 14.213(3) Å, b = 14.840(3) Å, c = 9.334(2) Å, β = 94.60(3)°, V = 1962.4(7) Å³, Z = 4, R = 0.0480 and wR = 0.1233 for 1456 independent reflections with I > 2σ_I.

Both complexes possess 1D crystal structures which consist of alternating Cu(II) and Zn(II) ions bridged by chloride (**1**) or acetate (**2**) ligands. The chains are strengthened with numerous H-bonds.

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