At the moment we are not able to establish if compound \( \text{I} \) would be a monohydride or a dianhydride. Infrared and NMR spectra suggest that \( \text{I} \) may rather be a monohydride. On the contrary the low magnetic moment (0.7 \( \mu_B \)) of \( \text{I} \) do not support spectroscopic data. Then the following questions need an answer: is \( \text{I} \) a mixture of isostructural \( \text{I}^{+} \) monohydride and \( \text{I}^{2-} \) dianhydride complexes? Is it a low \( \mu_B \), sometimes, a peculiar characteristic of paramagnetic hydrides? At present definite evidences on the basis of the IR and NMR spectra are not in favour to \( \text{I} \) as non classical dianhydride \( \text{I}^{2-} \) complex.

**PS-07.05.09 CRYSTAL STRUCTURE OF \{[Ni(CHT)]\}_2-(ClO_4)_2. A NICKEL(II) DIMER WITH CARBONYL-OXIME PYRIMIDINE BRIDGES.** By R. Kivioja* and H. Klings, Division of Inorganic Chemistry, University of Helsinki, Finland; A. Romero, J. M. Dominguez-Vera and G. Solano, Departamento de Química Inorgánica, Facultad de Ciencias, Universidad de Granada, Spain.

Studies of magnetic exchange interactions in polymeric metal complexes are of continuing interest to coordination chemists. One of the reasons for this is to seek new bridging systems which can mediate a strong antiferromagnetic spin-coupling between two different paramagnetic centers in order to obtain ferromagnetic materials. Spin exchange through oxime bridges is known antiferromagnetic and is able to cause complete or nearly complete spin-coupling even at room temperature.

In attempts to prepare oxime bridged Ni-Cu-Ni complexes by reacting \([\text{Cu}_2(\text{ClO}_4)_2]\) with \([\text{Ni}(\text{CHT})][(\text{ClO}_4)]_2\) an unexpected product, analysed as \([\text{Ni}(\text{CHT})][\text{ClO}_4]_2\), was formed. This paper deals with the structure of this unusual compound.

Abbrications: HLT is \( \text{N},\text{N}-(3,3\text{-dimethyl}-5\text{-nitrooxo}-1\text{-ylamine})\)oxamocarbonyl and HLT is \( \text{N},\text{N}-(1\text{-ylamine})\)oxane and HLT is \( \text{N},\text{N}-(1\text{-ylamine})\)oxane.

Crystal data: monoclinic with \( \alpha=10.083(6) \), \( b=12.22(5) \), \( c=15.773(5) \), \( \beta=98.31(3) \), \( V=3449(2) \), \( a_2 \), \( R=0.065 \) (Re=0.044) for 2965 reflections and 415 parameters.

![Fig. 1. (Ni(CHT)2)2](image1.png)

**PS-07.05.10 Eclipsed Cofacial Dimers of Metal Complexes Containing Bi13-3Al31 and B13-3Al11 Bonds. Crystal Structure of \( \text{Bi}(\text{seimquinacridon}-4-	ext{dioxido})\text{carbeno}(\text{rhodium})\text{(1)} \) and \( \text{Bi}(\text{seimquinacridon}-4-	ext{dioxido})\text{carbeno}(\text{iridium}) \).** By Mei-Chi Liu* and Shue-Ming Fen, Department of Chemistry, National Taiwan University, Taipei, Taiwan.

In this paper, we report two novel crystal structures of metal complexes of semiquinacridone-tetragonal ligands which contain synergic metal-metal and ligand-metal bonds.

- The reaction of \( \text{Bi}(\text{III})\text{CO}_{3}[\text{Cl}]_2 \) with o-phenylenediamine yields a green solution, from which the dark green crystals are obtained. It was characterized by IR, EA, and X-ray diffraction as \( \text{Bi}([\text{II})(\text{III})\text{CO}_{3}][\text{o-phenylenediamine}]_2 \). The green crystals were found to crystallize in the monoclinic space group \( \text{P}_{2}1/c \), \( a=12.46(9), b=9.73(8), c=24.15(4) \), \( \beta=112.81(1) \), \( R=0.046 \), \( R_{w}=0.046 \) for 896 observations and 235 parameters.

- The reaction of \( \text{Bi}(\text{III})\text{Co}_{3}[\text{Cl}]_2 \) with o-phenylenediamine yields a green solution, from which dark green crystals are obtained. It was characterized by IR, EA, and X-ray diffraction as \( \text{Bi}([\text{III})(\text{III})\text{CO}_{3}][\text{o-phenylenediamine}]_2 \). The green crystals were found to crystallize in the monoclinic space group \( \text{P}_{2}1/c \), \( a=12.46(9), b=9.73(8), c=24.15(4) \), \( \beta=112.81(1) \), \( R=0.046 \), \( R_{w}=0.046 \) for 2900 reflections and 35 parameters. These two structures have a number of unusual features:
  1. They contain \( \text{Bi}_{13}-\text{Bi}_{31} \) and \( \text{Bi}_{13}-\text{Al}_{11} \) bonds.
  2. The two semiquinacridone ligands are stretched in an eclipsed fashion and are separated by only 3.0 Å.
  3. The ligands have disordered bond character and contain a \( \beta \) type bond between the two parallel semiquinacridone ligands.

**PS-07.05.11 CRYSTAL STRUCTURE OF A BARIUM FLUORINATED ALKOIDE B₄₀(OH)(OCHCF₃)₂₉ (THF)₂ H₂O FOR USE AS A PRECURSOR FOR HIGH Tc SUPERCONDUCTORS.** By H. Vincent*, ENSP/LMGP BP 46, 38402 St Martin d'Hères, F. Labize and T. Hubert, Labo. de Chimie Moléculaire, Université de Nice, 06934 Nice, France.

MOCVD (Metal-Organic Chemical Vapor Deposition) is potentially well suited for the obtaining of thin films, but needs volatile precursors. To date, \( \beta \)-diketonates based on bulky or fluorinated ligands have generally been used as a source of metal oxides. For heavy electropositive metals such as barium, problems due to a poor volatility and/or stability are often encountered and result in a lack of reproducibility for the deposits.
Metal alkoxides might represent an alternative, especially as they are generally more attractive for the design of mixed metal species. Barium alkoxides involving fluorinated groups, namely perfluorooctylalkoxides (OC(=CF(CH3)2)3) (OM) have been obtained either by direct reaction between the metal and the alcohol or by alkoxysus reactions. Crystal structure and chemical formula of one of them, crystallized from hexane/methylalcohol (THF), have been determined by single crystal x-ray diffraction at 298 K.

Crystal data are as follows: monoclinic symmetry; space group P2₁/m; cell parameters at 290 K: a = 24.667(7) Å, b = 13.925(3) Å, c = 25.856(9) Å, β = 113.15(3)°; asymmetric unit: one Ba5(OS)2(OCH(CF3)2)2(THF)4 H2O molecule + one not bonded THF molecule; Z = 4. The molecular structure consists of a classical pentanuclear cluster of 5 barium atoms, arranged in a square based pyramid. A 4,5-oxide ligand lies at the center of the pyramid squared basis; 5 μ-Ο-Ο-ligands, 4 μ-Ο-ligands, 4 THF and 1 H2O molecules are surrounding the cluster. Thus, each barium atom has 7 to 11 oxygen or fluor atoms as neighbors. Such a high coordination number for barium atoms explains the relative stability of this alkoxide.

Dimensions are compatible. This would further enhance the necessary communication for the inconsequential modulation to be observed. Resolution of the structure options requires an ability to model the anions. The anions were initially included in the model, as a series of spherically averaged SO3CH3 spaced at 1.5c apart with a larger Uiso thermal parameters. Subsequent to this fractional oriented anions streaked along c have been used. The progress of refinement has been monitored by assessing data of different index condition. Refinement was kept in control using RAI/SF90 to refine rigid body thermal parameters and impose localized symmetry constraints. The anions appear to have restricted orientation and be clustered around the same 2 heads as the cations, 1/2c apart in the average structure. Refinement is continuing. The observed inconsequential modulation appears to be the result of the c-axis length being larger than required to have anions touching. This allows extra species, presumably solvent, to be fitted into the channels.

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