

$\text{Ni}(\text{CN})_2\text{-CN-}]_n$  with trans-positioned bridging cyanide ligands in both cation and anion. The water molecules of crystallization are located between the chains and link them by hydrogen bonds. In the crystal structure of **4** the chains are built up of  $[\text{Ni}(\text{cyclam})]^{2+}$  cations linked by cyanide anions solely; as a consequence the chains formed are positively charged and the positive charge is counterbalanced by uncoordinated  $\text{NO}_3^-$  anions. The water molecules of crystallization are involved in a hydrogen bonding system linking the chains, anions and water molecules.

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**Keywords:** cyanide complexes, cyclam complexes, nickel compounds

#### FA4-MS34-P12

**Powder diffraction as a tool for studying clusters and molecules in solution** Antonio Cervellino<sup>a</sup>, Antonietta Guagliardi<sup>b,c</sup>, Norberto Masciocchi<sup>b</sup>, Alessandro Ceriotti<sup>d</sup>, <sup>a</sup>Swiss Light Source, Paul Scherrer Institut, Switzerland, <sup>b</sup>Dipartimento di Scienze Chimiche ed Ambientali, Università dell'Insubria, Como, Italy <sup>c</sup>Istituto di Cristallografia, Consiglio Nazionale delle Ricerche, Bari, Italy, <sup>d</sup>Dipartimento di Chimica Inorganica, Metallorganica e Analitica, Università di Milano, Italy

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The brilliance of third-generation synchrotron sources allows nowadays the detection of a meaningful scattering signal from extremely small atomic arrangements (including nanostructures and even relatively small molecules in suspension or solution), provided a certain level of contrast with the solvent. [1] This makes the method especially suited for high nuclearity metal carbonyl clusters. Although the resulting relatively smooth diffraction patterns are not yet amenable for direct structure solution, it is nowadays easy – using total scattering techniques, like PDF or the Debye Function Analysis [2] method – to compare the experimental pattern with calculated ones, derived from structure models of the solvated molecule (ideally, in its gaseous form). These models can be inferred in other ways, most commonly from structure solution of the same substance in crystallized form. The door is then open for using Reverse Monte Carlo-based algorithms for modifying and relaxing the initial structure model, aiming at improving the knowledge of the molecular conformation (in the solvated form) through a better match with the recorded pattern. The theoretical and experimental requirements of the method are discussed, with reference to some metal-organic small- and medium-sized clusters. [3]

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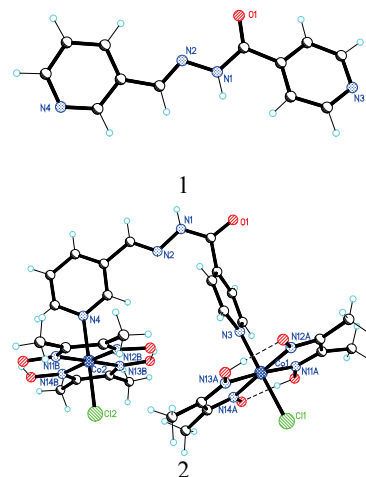
**Keywords:** powder diffraction, pair distribution function, crystallographic and NMR solution state structures

#### FA4-MS34-P13

**A new Schiff Base as a bridge in binuclear Cobalt(III) Dioximate.** Olga Ciobanica<sup>a</sup>, Paulina Bourosh<sup>b</sup>, Vasile Lozan<sup>a</sup>, Ion Bulhac<sup>a</sup>, Yurii Simonov<sup>b</sup>  
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By condensation of isonicotinic acid hydrazide with 3-pyridine aldehyde the new Schiff Base HL (**1**) was obtained. The interaction of  $[\text{CoCl}(\text{DmgH})_2\text{H}_2\text{O}]$ , (where  $\text{DmgH}_2 = \text{dimethylglyoxime}$ ) with HL the binuclear complexes with composition  $[(\text{CoCl}(\text{DmgH})_2)_2\text{HL}]$  (**2**) was synthesized. Both compounds were characterized by using the IR-, UV/Vis- and NMR-spectroscopy. Single crystals were grown by slow evaporation of neat methanol solution of the ligand and complexes. The X-ray analysis of single crystal HL shows that free molecule adopts *E*-conformation around central bond  $\text{N1-C}$ . Torsion angle  $\text{N2N1CO1}$  is  $6^\circ$ . The HL has a lot of active center, which can be used for coordination to the metal atom,  $\text{N1}$ ,  $\text{N2}$ ,  $\text{N3}$ ,  $\text{N4}$  and  $\text{O1}$ . Crystal structure of **2** shows that the HL is coordinated to two  $[\text{CoCl}(\text{DmgH})_2]$  neutral residues through the nitrogen atoms of N-heterocyclic, HL acting as a bridging bidentate neutral ligand. At the same time the HL for coordination through the two terminal nitrogen atoms in the coordination process changes its conformation to *Z*, making some turns around the ordinary bond, particularly around the bond  $\text{N1-C}$ . The torsion angle of  $\text{N2N1CO1}$  in HL coordinated to **2** is  $174^\circ$ . In the binuclear unit, each  $\text{Co(III)}$



cation is hexacoordinated by five nitrogen atoms and one Cl in a square-bipyramidal environment. Its basal plane is made up by a bidentate  $\text{DmgH}^-$ , an anion  $\text{Cl}^-$  and a bidentate HL ligand occupy the apical sites. Two  $\text{DmgH}^-$  residues are bound via  $\text{O-H}\cdots\text{O}$  hydrogen bonds, typical for all *bis*-ligand com-

plexes of  $\alpha$ -dioximes [1,2]. The  $\text{O}\cdots\text{O}$  distances range from 2.45 to 2.50 Å and correlate with the hydrogen bonding in the octahedral complexes of  $\text{Co(III)}$ , containing the  $[\text{Co}(\text{DmgH})_2]$  core. The  $\text{N}_4$  equatorial atoms are almost planar ( $\pm 0.023$  Å for  $\text{Co1}$  and  $\pm 0.014$  Å for  $\text{Co2}$ ) and the deviation of the cobalt atoms from this plane are  $-0.053$  Å and  $-0.030$  Å, respectively. The axial  $\text{Co-N}$  distances 1.945 and 1.964 Å are significantly longer than the equatorial  $\text{Co-N}$  distances (the average distances  $\text{Co-N}$  1.893 and 1.882 Å),  $\text{Co-Cl}$  2.228 and 2.230 Å.  $\text{Co-Co}$  distance in the complex is equal to 7.211 Å. The crystal packing is discussed.

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