

MS40 P01

Synthesis, characterisation and X-ray study of Ni(II) complex : [Ni(H₂P₂O₇)(C₁₂H₈N₂)₂].2.5H₂O Namouchi-Cherni Saoussen & Ahmed Driss *Laboratoire de Matériaux et Cristallographie, Département de chimie, Faculté des Sciences, 2092 El Manar, Tunis, Tunisia.*
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Keywords: Structure compounds, characterization method, magnetic crystal structures.

Investigation of novel organic phosphate complexes represents the intense interest of chemical research because of their diverse practical applications.

This complex has been characterized by X-ray diffraction, IR spectral analysis and UV-vis spectroscopic. The natural elements in the complex are detected by the EDX on a scanning electron microscope. The thermal analysis (TG, DTA) shows that the dehydration of compound occurs in agreement with the structural features.

The title complex was prepared by hydrothermal crystallization in a sealed vessel from ethanolic solutions of nickel nitrate, 1,10-phenanthroline hydrate, phosphoric acid and water.

The structure was determined by X-ray diffraction on single crystal. This compound crystallizes in the triclinic space group P-1 with $a = 10.285(2)$ Å, $b = 10.510(3)$ Å, $c = 12.775(3)$ Å, $\alpha = 88.06(2)^\circ$, $\beta = 77.87(2)^\circ$, $\gamma = 89.26(2)^\circ$, $V = 1349.2(5)$ Å³, $Z = 2$ and refined to $R = 0.042$ and $wR = 0.012$.

Crystal structure of [Ni(H₂P₂O₇)(C₁₂H₈N₂)₂].2.5H₂O is composed of nickel complexes and water molecules. Each complex is connected to two others by means of hydrogen bonding and weak π - π stacking interactions, to form a bi-dimensional framework, which delimits b-axis tunnels, where water molecules are located.

The Ni^{II} ion is coordinated in a distorted octahedral arrangement by one O and three tertiary N atoms in equatorial positions, and by O and N atoms in axial positions.

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MS40 P02

A Refinement Target Directly Using the Experimental Phase Information from SIRAS experiment Pavol Skubak, Navraj S. Pannu. *Department of Biophysical Structural Chemistry, Leiden University.*
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Keywords: automatic model building and refinement; direct use of experimental phase information; multivariate refinement target

Previously, the direct use of prior phase information from a single-wavelength anomalous diffraction (SAD) experiment with a multivariate likelihood function applied to automated model building with iterative refinement was proposed [1] and shown to extend the limits for successful model building [2].

We have derived a multivariate likelihood function that directly incorporates the experimental phase information from a single isomorphous replacement with anomalous scattering (SIRAS) experiment, accounting for the correlations between the observations and the models. The implementation of the derived likelihood function requires multi-dimensional numerical integration, leading to the necessity of approximations in order to achieve an efficient implementation. Based on the properties of the three-dimensional integral of the SIRAS function, we have proposed an algorithm for the evaluation of this integral and we have implemented the algorithm in REFMAC5. The algorithm and the performance of the SIRAS function have been tested on several SIRAS datasets, yielding satisfactory results.

[1] Skubak P.; Murshudov, G.N.; Pannu, N.S. (2004). *Acta Cryst.*, D60, 2196-2201.

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MS40 P03

Crystal Structure Of 2-[2-(Aminophenoxy) Ethoxy]-N-[(1e)-2,3,5,6,8,9,11,12-Octahydro-1,4,7,10,13-Benzopentaoxacyclopentadecim-15-Ylmethylene]Aniline

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The title compound, C₂₉H₃₄N₂O₇, is a substituted monotopic benzo-15-crown-5-ether ligand. It belongs to the space group $P2_1/a$ with cell parameters $a = 8.9857(6)$, $b = 25.0353(17)$, $c = 12.6979(9)$ Å and $\beta = 109.730(1)^\circ$. The intermolecular N-H...O hydrogen bonds are dominantly effective in stabilizing the crystal structure. The N-H...O intermolecular hydrogen bonds link the molecules, forming infinite one-dimensional chains running approximately parallel to the a -axis. The relative macrocyclic inner-hole size is estimated to be 1.44 Å. The substituent and benzocrown ether precursors about the C=N imine bond reveals a *trans* planar (1*E*) configuration.

MS40 P04

AMPLIMODES: a computer program for the symmetry-mode analysis of structural phase transitions. Danel Orobengoa, Cesar Capillas, Mois I. Aroyo, J. Manuel Perez-Mato, *Depto. de Fisica de la Materia Condensada, Universidad del Pais Vasco 48080 Bilbao, Spain.* E-mail: bcborgud@ehu.es

Keywords: structural phase transitions, symmetry-mode analysis, Bilbao Crystallographic Server

The main signature of a displacive phase transition is the appearance of a symmetry-breaking distortion (with respect to the high symmetry phase) that is mainly caused by the freezing of the so-called *primary* mode, associated with the order parameter. In general, *secondary* modes are also triggered at the transition and can have non-zero amplitudes in the distorted structure. The symmetry-mode analysis of a structural phase transition results in the calculation of the amplitudes of the symmetry modes frozen in the distortion, *i.e.* to the determination of the

eigenvectors of both the primary and the secondary modes present in the distortion.

The aim of this contribution is to report on the development of the computer program AMPLIMODES that carries out a symmetry-mode analysis of a displacive phase transition. Starting from the experimental structures of the high- and low symmetry phases, the program determines the global structural distortion that relates the two phases. The symmetry-modes compatible with the studied symmetry break are calculated by a dedicated module of the software package SYMMODES [1]. The orthonormalization of the symmetry modes permits the decomposition of the global distortion into symmetry-mode contributions, and the determination of the corresponding eigenvectors. The normalization of the eigenvectors allows the comparison of their amplitudes,

and hence indicates their importance in the mechanism of the studied phase transition.

The presentation of the program AMPLIMODES, includes illustrative examples of symmetry-mode analysis of structural phase transitions. The computer program and the corresponding documentation can be found on the Bilbao Crystallographic Server [2] which is a free web site with crystallographic databases and programs available at <http://www.cryst.ehu.es>.

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