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Structure of a polymeric Ce(III) complex containing pyridine-2,6-dicarboxylic acid

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Coordination polymers of rare earth elements have received significant attention in recent years because of their potential optical, magnetic, and porous properties, especially for the carboxylates [1–4]. The large ionic radii of rare earth elements together with the coordination flexibility of lanthanide ions can lead to unusual structural topologies and new framework families [5].

Here we report the synthesis, characterization and crystal structure of the polymeric $\{[\text{Ce}(\text{pydc})_3(\text{H}_2\text{O})_8] \cdot 2\text{H}_2\text{O}\}_n$ complex (**I**), obtained from the reaction of Cerium(III) nitrate with the pyridine-2,6-dicarboxylic acid, dipicolinic acid (pydcH₄) and 2-aminopyrimidine (ampy) in 1:1:1 molar ratio in basic media. Polymeric complex was characterized by FT-IR spectroscopy and X-ray diffraction. X-ray crystal structural analysis reveals that the compound belongs to the triclinic space group $P\bar{1}$ with cell parameters $a = 10.407(4)$ Å, $b = 11.795(5)$ Å, $c = 13.770(6)$ Å, $\alpha = 93.852(2)^\circ$, $\beta = 111.142(2)^\circ$ and $\gamma = 111.105(2)^\circ$. The molecular structure consists of two crystallographic independent pairs of Ce^{III} ions in the unit cell (Fig. 1). The coordination polyhedron of the ten-coordinated Ce1 ion is a distorted bicapped square antiprism comprised of O1 and O2 bridged atoms from pydc ligand coordinated to the Ce1 ion of the neighboring unit, N1, O3, O4 atoms of the one dipicolinate ligand, bridged O5 atom of pydc ligand coordinated to the Ce2 ion of another neighboring unit, O13, O14, O15 and O16 atoms of the water molecules. The Ce2 ion is in slightly distorted tricapped trigonal prism being coordinated by N2, O6, O7 and O8, O11 and N3 atoms from two dipicolinate ligands, bridged O11 atom from pydc linked to the Ce2 ion of the neighboring unit, O17 and O18 atoms of the water molecules. In the crystal structure, a wide range of noncovalent interactions consisting of hydrogen bonding (of the types of O–H⋯O and C–H⋯O) and ion pairing interactions connect the various components into a supramolecular structure.

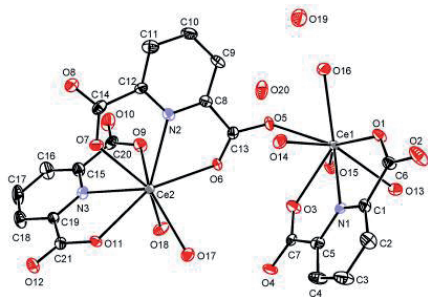


Fig. 1. ORTEP plot showing the coordination environments of cerium ions in **I**.

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Reciprocal space statistical thermodynamics of inhomogeneous disordered systems

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Within the last decades a lot of efforts were made in order to understand the nature of dynamical heterogeneities in disordered systems, e.g. supercooled liquids, amorphous alloys. Nevertheless the nature of the amorphous state formation, a fundamental problem of condensed matter physics, is not completely understood.

It has been recently shown [1] that the dynamic origin of heterogeneous dynamics might be a result of critical-like fluctuations of static structural order. This is a concept that counters the currently accepted trend.

Here we present a reciprocal-space approach for inhomogeneous systems, based on the ring approximation [2], [3]. This concept deals with cooperative reciprocal-state modes and takes into account their statistical coupling, both in atomic interaction and in mutual arrangement of particles, much unlike traditional real-space statistical thermodynamic approaches.

Using the ring approximation in the framework of kinetic atomic density functional approach [4], we describe configurational self-organization in the amorphous state. The Fourier transform of the relaxation time is given by the following expression:

$$\tau(\mathbf{q}, T) \sim -kT/[D_0(T)q^2 V_{\text{eff}}(\mathbf{q}, T)], \quad (1)$$

where \mathbf{q} is a reciprocal lattice vector, temperature-dependent coefficient $D_0(T)$ accounts diffusive properties of the system, $V_{\text{eff}}(\mathbf{q}, T)$ is the “renormalized” effective pairwise interaction potential [1], and it is taken into account that interference of atomic interaction modes with different wave vectors takes place. In the mathematical expression above it is also assumed that the temperature range is significantly lower than the structural instability point, also called spinodal point.

Equation (1) leads to the conclusion that the dominant mode, corresponding to the minimal negative value of $V_{\text{eff}}(\mathbf{q}, T)$, develops the most intensively. For the Dzugutov-like potential, widely used in simulations of the amorphous state, the function $V_{\text{eff}}(\mathbf{q})$, exhibits a flat region of the dominant modes for a certain range of potential parameters. Consequently, a set of these modes possesses the same value of relaxation time. This corresponds to formation of a highly inhomogeneous structural state. Interference of the dominant modes leads to the long-living ordered structural states, e.g., clusters, recently revealed by X-ray cross-correlation analysis [5] of coherent scattering data.

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Poster Sessions

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Some Examples of Polyoxometalates Crystal Structure Defects

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The study of Molybdenum and Tungsten polyoxometalate complexes is of fundamental importance in the context of both oxidation catalysis and biology. The possibility to build complexes with different structures and to attach to them varied organometallic molecules is related to oxygen and electron transport during enzymatic reactions [1].

X-ray crystal structure solution and refinement of poly-oxometalate single crystals often involves some difficulties usually related to different disorders in their crystal packing. The structural framework of these compounds is formed by joining complexes W, Mo – O, which can be presented in variety of different shapes, like spheres, ellipsoids, wheels and others. The complexes can be connected to each other by a network of solvent water molecules and cationic counter ions or by different organic or organometallic groups. Large accessible voids between the complexes usually allow inserting additional atoms or groups. The sufficiently weak bonds between different structural groups and the inherent high symmetry of the polyoxometalate can cause several types of disordering. Metal atoms of the complexes may also be partially substituted by other metal atoms, which in turn can be disordered distributed inside of the complexes.

Different types of disordering and partial or mixed occupancies at certain positions can cause small changes in the symmetry of the crystals, which can give rise to supersymmetry or even to an aperiodic structure. Lack of small reflections with high resolution can prevent from finding correct symmetry group and thereafter from obtaining structure solution.

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Supercell approximation for an incommensurately modulated crystal of profilin:actin

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A crystal structure from an incommensurately modulated protein crystal has never been reported, mainly due to a lack of methods for solving these unique structures. Here we report the first modulated protein structure that was determined using q-vectors to accurately integrate the diffraction data followed by a commensurate supercell approximation. Crystal modulation is characterized by a loss of short-range translational symmetry, where a single unit cell is no

longer sufficient to accurately describe the structure. Such a loss of periodicity is often caused by dynamic processes within the crystal arising from, for example positional modulations. Experimentally, the incommensurately modulated state is characterized by the appearance of distinct satellite reflections surrounding the main Bragg reflections on the diffraction pattern that cannot be indexed with a supercell. In order to fully describe the modulated structure, and hence the dynamic processes within the crystal, one must explore higher-order space over multiple unit cells. By careful examination of atomic positions over higher dimensional space, a modulation function can be calculated that traces the atomic disorder. Such a function is periodic, but incommensurate with the crystal lattice. Another form of modulation occurs when the atomic disorder is commensurate with the main lattice. In this case, the intensities can be indexed as a large, or 'supercell'. In the case of incommensurately modulated profilin:actin crystals, the data was not able to be indexed as a supercell by use of existing software. The diffraction data had to be indexed and integrated using the q-vector approach. Data were observed to 2.4 Å and were processed with a single q-vector with *EVAL15* software. The main and satellite reflections were indexed and described by an orthorhombic main unit cell of a=37, b=71, c=185 Å and the q-vector $q=0.2829 \dots b^*$. From this q vector we know that the modulation repeats at approximately, but not exactly, three and a half unit cells in the b direction. To approximate the supercell, the data was then reindexed using a supercell approximation (assuming the q vector is close to 2/7; then $h=h$, $k=7k+2m$, $l=l$). The supercell dimensions were a=37, b=498, c=185 Å with space group $P2_12_12_1$. A previously refined periodic PA structure was used as a search model and molecular replacement using *MOLREP* produced a ribbon structure with 6 PA copies. Analysis of electron density maps made clear the position of the 7th copy, which was fit manually. Then refinement was performed with *REFMAC* using 7 rigid groups for each actin and one for profilin. In the resultant map there was interpretable density for ATP. In this preliminary structure the motion appears to involve a rotation in the ac plane and is in general agreement with the analysis of the average structure, which by analysis of Fourier electron density maps indicate major motion in actin subdomains 2 and 4. The position of profilin seems to change the least. The structural information gleaned from this approximation will help bootstrap the solution of the incommensurate structural refinement in (3+1)D superspace.

Keywords: **Modulation, Aperiodic, Profilin:Actin,**

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Service crystallography at the university of santiago de compostela

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The latest technological developments in X-Ray production, optics and detection, the automation of crystallization, sample handling and conditioning, and the improvement in computing facilities combined with new methodologies have converted crystallographic analysis to routine in many cases, mainly for small and medium-size molecules. The standardization of these techniques allowed their use by non-specialized users inside Departments and to be included in the general services offered by Universities and other research institutions. However, there are still many situations, even in simple systems, where the assistance of an expert crystallographer is required.

We describe here our experience with the set up and development of the X-Ray Unit at the University of Santiago de Compostela (USC)