MS78 STRUCTUROMES – STRUCTURES OF RIBOSOMES NUCLEOSOMES AND OTHER -OMES
Chairpersons: Anders Liljas, Wayne Hendrickson

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Nucleosome Core and Compact Nucleosome Array Structures
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The fundamental building block of chromatin is the nucleosome comprising 157-240 bp of DNA, two each of the four core histone proteins, and a single linker histone H1/H5. The nucleosome core is the greater part of the nucleosome and contains 147 base pairs of DNA wrapped in 1.67 left-handed superhelical turns around the histone octamer. Arrays of nucleosome in their most compact form constitute the "30 nm" chromatin fiber.

The crystal structure of the nucleosome core particle refined to 1.9 Å resolution reveals the details of DNA conformation as well as all the direct and water-mediated histone-DNA contacts. The acute DNA bending induced by the histone proteins results in an alteration of the form of the double helix every five base pairs along its superhelical path. Sequence-dependent DNA conformations are apparent.

Two nucleosome core particle structures containing different 146 base pair DNA sequences contain distinct regions in which the DNA is relatively over-twisted and stretched. These regions represent trapped-intermediates relevant to the "twist-defect diffusion" mechanism for nucleosome sliding and provide a means of buffering DNA linker length variation in the chromatin fiber.

The crystal structure of a tetranucleosome determined at 9 Å resolution comprises two stacks of two nucleosomes with three segments of linker DNA running between them. This structure is compatible with a two-start helix, but not with a one-start helix. A continuous fiber model built by stacking tetranucleosomes results in a nucleosome higher structure that is nearly fully compact.
Keywords: DNA, nucleosome, chromatin fiber

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Structural View of the Clamp-loading Mechanism onto DNA
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DNA replication is a highly coordinated process, which involves numerous proteins in nuclei. To promote the integrated system, proteins involved in this event constitute several kinds of molecular machinery. Archaeal systems generally exhibit attractive properties to study DNA metabolism; their DNA binding proteins are very similar to those from eukarya both functionally and structurally, irrespective of their morphological difference from eukaryotic ones, and hence they are good model systems for understanding eukaryotic DNA processing. We have been working on several DNA processing proteins from a hyperthermophilic archaeon, *Pyrococcus furiosus*.

Replicative DNA polymerase requires two essential protein factors, a sliding clamp and a clamp loader, for rapid and accurate DNA duplication. In eukarya and archaea, a homo-trimeric proliferating cell nuclear antigen (PCNA) and a hetero-pentameric replication factor C (RFC) function as the clamp and the clamp loader, respectively. The ATP-dependent clamp-loading mechanism is particularly intriguing, because it requires opening and resealing of the PCNA ring. We have determined the three-dimensional structure of an archaeal RFC–PCNA–DNA clamp-loading complex by electron microscopy-single particle reconstruction. Importantly, the structure of the complex presents the first direct view of a washer-like open conformation of the PCNA ring in contact with RFC. In combination with the two X-ray structural data reported previously, our EM model implies an intriguing clamp loading mechanism.
Keywords: DNA replication, clamp-loading complex, single-particle reconstruction

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X-ray Crystal Structures of the *E. coli* Ribosome
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We are using x-ray crystallography to probe the structural basis for the many aspects of protein biosynthesis that require the intact ribosome. Our goal is to make an atomic-resolution "movie" of a ribosome in the process of making a protein. We have obtained crystals of the entire *E. coli* ribosome that diffract x-rays to a resolution of 3.1-3.2 Å. Thus, we now have the means to determine the first atomic-resolution structure of the intact ribosome, the first frame of the movie. Moreover, we are using these crystals to probe in atomic detail the effects of antibiotics on the full ribosome and mutations in the ribosome that lead to antibiotic resistance or perturb key steps in translation. We are presently refining ribosome structural models at a resolution of 3.5 Å, the results of which will be presented.
Keywords: ribosome structure, antibacterials, protein synthesis

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Structural Analysis of the L7/12 Ribosomal Stalk
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The L7/12 stalk of the large ribosomal subunit encompasses protein L10 and multiple copies of L7/12 and is involved in translation factor related functions. We have determined crystal structures of *Thermotoga maritima* L10 in complex with L7/12 N-terminal domains and of an archaeal L10 N-terminal domain *in situ* on the 50S subunit. A mobile C-terminal α-helix of L10 harbors three consecutive binding sites for L7/12 dimers in *T. maritima* and two in *E. coli*, where the helix is shorter. The N-terminal domain of L10 recognizes the overall fold of the thiostrepton loop of 23S rRNA and interacts with L11. Together with structures of isolated L7/12, we devised a complete atomic model of the stalk and reinterpreted the morphology and dynamics of the stalk region as seen in electron microscopic reconstructions of ribosomes. Flexible hinges in both L10 and L7/12 lead to a high freedom of motion for the L7/12 C-terminal domains. Our structural data and analysis of L7/12 mutants by fast kinetics reveal that the L7/12 C-terminal domains can reach far out into solution to bind translation factors. They thereby promote factor recruitment to the ribosome. The L7/12 C-termini can then reach back towards ribosome-bound factors to stimulate GTP hydrolysis by stabilization of the factors' active GTPase conformation.
Keywords: ribosome structure, translation factor recruitment, GTPase activation

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Ribosomal Crystallography Reveals Co-Translational Trafficking by Eubacterial Trigger Factor
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The correct folding of newly synthesized proteins is a vital process in all kingdoms of life. It is coordinated and concerted by a set of molecular chaperones that direct the folding of nascent proteins towards their final functional state. Nascent chains emerging out of the ribosomal tunnel are highly prone to aggregation and degradation. Consequently, chaperone activity is initiated during translation.

Trigger factor (TF), the first chaperone in bacteria to encounter the

emerging nascent chain, binds to the large ribosomal subunit in the vicinity of the tunnel opening and forms a sheltered folding space. The 3.5Å crystal structure of the large ribosomal subunit of the eubacterium *Deinococcus radiodurans*, D50S, in complex with the TF binding domain (TFa) from *Deinococcus radiodurans*, reveals for the first time the molecular structure of **the entire** TFa bound to the bacterial ribosome. In comparison with structures of isolated TF/TFa molecules from different bacterial sources, the current structure displays a conformational change in the TFa domain that assures a degree of dynamic flexibility and may hint at mobility during early folding.

The signature part of TFa anchors on small exposed regions of ribosomal proteins L23 and L29 some 40Å away from the opening of the exit tunnel, in general agreement with the reported chimeric structure of the archaeal large ribosomal subunit (H50S) with the eubacterial TFa (Ferbitz et al., 2004). The chaperone TF does **not** exist in the archaeal kingdom, and therefore the similarity of its local interactions with both ribosomal systems highlights the high structural and sequence conservation of its contact region on the ribosome. Still, the archaeal ribosomal protein L23 lacks the sizable elongated loop, present in bacteria, which extends into the tunnel opening and can actively interact with the nascent protein passing through it. Our structure shows that TFa binds to two separate regions of L23 on both sides of the extended loop, thus linking the TF binding site with the ribosomal tunnel and enabling communication with the newly synthesized nascent chain.

Keywords: ribosome, chaperone, ribosomal tunnel

MS79 Inorganic and Mineral Structures Solved and Refined by Powder Diffraction Data

Chairpersons: Jordi Rius, Evgeny Antipov

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NMR and PXRD Analysis of the Structure of a New Hydrous Layer Silicate

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RUB-39 is a synthetic hydrous layer silicate with an unusual crystal structure. Upon heating neighboring layers condense to a framework silicate with empty zeolithic pores. The crystal structure RUB-39 (P 2/c, a = 7.3312(1) Å, b = 10.7238(1) Å, c = 17.5135(1) Å, ß = 115.6911(6) °) was solved from PXRD data and refined in its hydrated layered form with FULLPROF to final χ^2 = 3.1. The diffraction analysis also allowed for a detailed analysis of the organic species used as structure directing agent (SDA) which is intercalated between silicate layers.

^{1}H solid state NMR experiments were used to study the role of the intercalated water and SDA-cation. The low field signal at 16.7 ppm indicates a strong hydrogen bond which is connecting neighboring silicate layers. This is unusual and might explain the topotactic condensation reaction upon heating. The spectrum also reveals that molecular water in intercalated in the interlayer space which is released only at temperatures above 170 °C.

PXRD and NMR studies following the further heating of the material to temperatures above 250°C showed the breakdown of the SDA and the condensation process of the silicate. Attempts to isolate the SDA-free layered material failed so far since concomitant to the release of the intercalated molecules the condensation to the framework silicate sets in.

Keywords: powder XRD, solid state NMR, hydrous layer silicate

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Adventures in Solving Structures of Inorganic Compounds Using Powder Diffraction Data

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Anhydrous hygroscopic $Pd(NO_3)_2$ can be prepared by evaporating a commercial palladium nitrate solution to dryness in a vacuum oven at 50-60°C. It crystallizes in space group $P2_1/a$, with a = 10.0886(10), b = 5.395(6), c = 5.7484(5) Å, β = 97.377(7)°, V = 310.28(5) Å^3, and Z = 2. The Pd was placed at the origin, and the N and O atoms located by difference Fourier techniques. The structure consists of discrete planar $Pd(NO_3)_2$ molecules. The molecular solid is an insulator with a bandgap of ~2.3 eV.

An attempt to prepare a magnesium vanadate using hydrothermal techniques yielded a hygroscopic new compound. The pattern could be indexed in space group *Cmcm* with a = 6.3727(7), b = 13.5715(8), c = 6.3657(4) Å, and V = 550.56(8) Å^3. The structure was solved by direct methods and difference Fourier techniques. The Rietveld refinement clearly indicated that the compound $(Mg_{0.37}V_{0.63})O_{0.63}(SO_4)(H_2O)_{1.5}$ has a layered structure.

The powder pattern of the potential thermoelectric material $NaGe_4$ could be indexed on a primitive hexagonal cell. The structure was solved and refined in *P6/m* with a = 15.05399(5), c = 3.96845(2) Å, and V = 778.852(4) Å^3. It consists of a zeolite-like Ge framework, with partially-occupied sites in a large 24-ring channel. Progress on the crystal structure of the mineral charoite, $K(Ca,Na)_2Si_4O_{10}(OH,F)(H_2O)$, will also be discussed.

Keywords: palladium nitrate, vanadium sulfate hydrate, sodium germanium

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New Cathodes for Solid Oxide Fuel Cells studied by Powder Diffraction: the System (A1-xA'x)FeO3

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A new family of iron oxide perovskites $A_{1-x}A'_xFeO_3$ (A= Nd, Pr, La; A'= Ca, Sr) has been studied for its potential use as cathodes in solid oxide fuel cells (SOFC). This is part of a collaboration between Basque Country University and Ikerlan Technological Research Centre (MCC Group) towards the development of metal supported intermidiate temperature SOFC. Samples with a systematic change in doping level were prepared by the ceramic and combustion methods and characterised by laboratory X-ray powder diffraction and SEM. Electrochemical measurements were performed in the 600-850ºC temperature range on bulk and as electrodes in YSZ supported half-cells. A summary of these results will be presented in this work.

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Keywords: crystal structure determination X-ray powder data, perovskite structures, conductivity

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Crystal Structure of the Superconducting Layered Cobaltate $Na_xCoO_2{\cdot}yD_2O$

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Superconductivity in layered $Na_xCoO_2{\cdot}yH_2O$ occurs in CoO_2 sheets that have a quasi 2D triangular symmetry, analogous to that of geometrically frustrated systems. This is in stark contrast to the well know perovskite high-Tc cuprates, and makes the determination of the structural details of this novel superconductor essential in the understanding of its properties. However, due to the complexity of the intercalation of the H_2O between Na and CoO_2 sheets, details of the crystal structure of these materials have remained ambiguous. We have used electron and neutron powder diffraction to elucidate the structural properties of superconducting $Na_xCoO_2{\cdot}yD_2O$ over a wide compositional range. Our measurements show that superconducting samples exhibit a number of supercells ranging from 1/3a* to 1/15a*,