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Keywords: MOF, topology, MCR-reaction.

MS31.P23

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Crystal Structure of the CcbJ Methyltransferase from *Streptomyces caelestis* [1]

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CcbJ is an S-Adenosylmethionine (SAM) dependent methyltransferase from *S. caelestis* which catalyzes the final step in the biosynthesis of the antibiotic celesticetin. *S. caelestis* has exhibited the ability to synthesize different derivatives of celesticetin depending on the presence of different salicylic acid derivatives in the growth medium. [2] In order to understand how this organism is able to manage this, we have isolated, overexpressed, and purified the individual components of this pathway, including CcbJ [3].

The crystal structure of free CcbJ was determined by Multiwavelength Anomalous Dispersion and that of the CcbJ–SAM complex was determined by molecular replacement (using the free structure as the search model). In both structures CcbJ crystallized in the $C222_1$ space group with unit cell lengths of a = 168.02, b = 244.55, and c = 117.85. In both crystals, the asymmetric unit contained six monomers arranged as a dimer of trimers.

CcbJ possesses the class I SAM-dependent methyltransferase fold [4], [5]; modifications to the core fold include insertion of a four-stranded β-sheet, which serves as an active site cover, between αE and β5 and a short 3_{10} helix between β4 and αD, which forms part of the SAM binding cleft. There is also an extension to the N-terminus. These insertions match the general pattern seen in other small-molecule methyltransferases. Overall, CcbJ appears to be most similar to glycine N-methyltransferase (GNMT) which also has a similar active site cover and a 3_{10} helix in the SAM binding cleft. Aside from a similar overall shape, the active site of CcbJ is quite different from that of GNMT, having a much larger number of aromatic residues.

One of the most characteristic features of CcbJ is the great degree of flexibility exhibited by the residues in the N-terminal extension preceding αZ. In the free CcbJ structure, these residues were completely disordered in one of the six chains and none of the residues preceding Tyr-17 were visible. In the CcbJ–SAM complex, however, the entire extension was visible in all six chains. The newly ordered residues form an α-helix which passes between the active site cover and αB and forms part of the SAM binding site. Following this helix, the extension passes over part of the active site opening before entering helix αZ. The loop between these two helices contains several proline and glycine residues and is likely to be natively unstructured. This would probably allow it to adopt several different conformations which might allow it to accommodate the several different substrates observed *in vivo* [2].

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Keywords: antibiotic, flexibility, methylation

MS31.P24

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1 and 2-D metal-organic polymer of Sc(III) with sufonate-carboxylate ligand

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Reaction of scandium chloride with 3,5- disulfobenzoic acid[1] under hydrothermal conditions leads to the formation of two metal-organic polymer compounds **$[Sc_3(3,5\text{-}DSB)_2(HO)_3(H_2O)_2]$** (**1**) and **$[Sc_3(3,5\text{-}DSB)_2(HO)_2(H_2O)_2]$** (**2**) that crystallize in the triclinic space group P-1. In both cases, the oxygen atoms of the carboxylate group are linking two metallic centers in $\mu_1\,\mu_1\,\eta^2$ mode. Two hydroxyl groups act as bridges linking scandium (III) cations along [011] direction. In the axial positions the scandium is coordinated by the oxygen of the sulfonate group, which is in *anti* coordination $\mu_1\,\eta^1$ mode. Two water molecules are coordinated in equatorial and axial positions. This arrangement allows the formation of bidimentional polymeric structure arranged in layers along (100) plane. The difference between the compounds **1** and **2** is the substitution in the compound **2** of one bridge hydroxyl molecule by one oxygen atom of the sulfonate group ($\mu^1\,\mu^1\,\eta^2$) along the [011] direction.

The utilization of a auxiliary ligand 1,10-Phenanthroline[2], under conditions similar to previous leads to formation of **$[Sc(3,5\text{-}DSB)(Phen)(H_2O)]\cdot(H_2O)$.** In this compound the Sc(III) is hepta-coordinated by the phenathroline chelate, one water molecule and a carboxylate group in $\mu_1\,\mu_1\,\eta^1$ mode in equatorial position and with two sulfonate groups (*anti* $\mu^1\,\eta^1$ mode) in axial position. The *trans* disposition of the ligand allows the formation of polymeric chains that grow along the *c* axis in "ladder" form. The chains are linked by hydrogen bonds along *b* direction; π-π slipped stacking interactions between the rings of the phenanthroline give rise to 3D supramolecular structure.

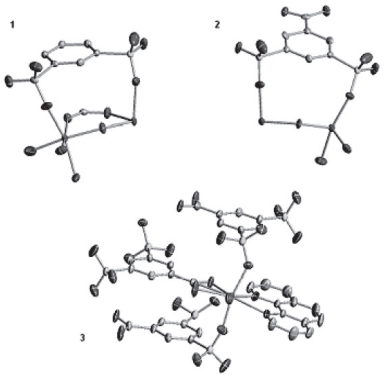

Fig 1. ORTEP drawing of the asymmetric units for **1-3** compounds; ellipsoids are displayed at the 50% probability level.

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Keywords: metal-organic polymer, scandium, supramolecular

MS31.P25

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Inhibition of MMP-1, 3, and 13 by Same Inhibitor – Structure Based Design Study

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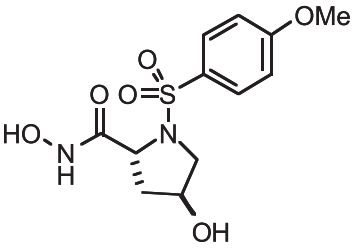
Matrix metalloproteinase (MMP) inhibitors are potential therapeutic agents for various diseases including cancer and osteoarthritis. Recent data from clinical trials with MMP inhibitors indicate that there is a great need for selective inhibitors. X-ray crystallography [1], [2], [3] has been used as a tool to help understand specific binding interactions of inhibitors to various MMPs. Large conformational changes have been noted when comparing the structures of the active MMP-3 catalytic domain and the one inhibited by a hydroxamic acid inhibitor. Both soaking and co-crystallization methods were used to generate the MMP-3/ inhibitor complex crystals for data collection. The same inhibitor has also been co-crystallized with MMP-1 and MMP-13. Comparisons of the structures of three inhibited enzymes, MMP-1, 3, and 13 show that MMP-3 and 13 are extremely similar. There are major differences in the binding pockets, especially in the S1' pocket between MMP-1 and MMP-3/13. These structural studies have helped design more selective inhibitors that can be used as therapeutic agents with improved safety profile.



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Keywords: osteoarthritis, drug, design

MS31.P26

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Understanding the Phases of DNAN

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Dinitroanisole (DNAN) is a key insensitive munition melt-phase ingredient that is currently featured in several melt-pour formulations developed by the U.S. Army. Current interest in DNAN has arisen due primarily to its ability to provide a less sensitive melt-cast medium than TNT, allowing for the development of less sensitive melt-cast formulations.

It is known in the literature that crystalline DNAN exists in two phases, A and B, that melt at 96(1)°C and 87(1)°C respectively. During this study, a third phase (C) was observed during a variable temperature study of the low melting point sample. It has also been observed that a spontaneous phase transition of B to A occurs under ambient conditions. Each phase has been isolated and detailed crystallographic studies of the three phases have been done, as well as some theoretical energy calculations. The transition from B to C is reversible and straightforward due to the ordering of a disordered nitro group at low temperature. The transition from B to A is far more complex, requiring a large molecular rotation of DNAN molecules.

Keywords: phase transition

MS31.P27

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Regioselective deacetylation based on teicoplanin-complexed Orf2* crystal structure

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Lipoglycopeptide antibiotics are more effective than vancomycin against MRSA as they carry an extra aliphatic acyl side chain on glucosamine (Glm) at residue 4 (r4)[1]. The biosynthesis of the r4 N-acyl Glc moiety at teicoplanin (Tei) or A40926 has been elucidated, in which the primary amine nucleophile of Glm is freed from the r4 GlcNac pseudo-Tei precursor by Orf2* for the subsequent acylation reaction to occur[2]. In this report, two Orf2* structures in complex with -D-octyl glucoside or Tei were solved. Of the complexed structures, the substrate binding site and a previously unknown hydrophobic cavity were revealed, wherein r4 GlcNac acts as the key signature for molecular recognition and the cavity allows substrates carrying longer acyl side chains in addition to the acetyl group. On the basis of the complexed structures, a triple-mutation mutant S98A/V121A/F193Y is able to regioselectively deacetylate r6 GlcNac pseudo-Tei instead of that at r4. Thereby, novel analogs can be made at the r6 sugar moiety.

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Keywords: regioselective, glycopeptide antibiotic, deacetylase.

MS31.P28

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Crystal structure of T-H protein complex of the glycine cleavage system

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Aminomethyltransferase, a component of the glycine cleavage system termed T-protein, reversibly catalyzes the degradation of the aminomethyl moiety of glycine attached to the lipoate cofactor of H-protein, resulting in the production of ammonia, 5,10-methylenetetrahydrofolate, and dihydrolipoate-bearing H-protein in the presence of tetrahydrofolate (THF). Several mutations in the human T-protein gene are known to cause non-ketotic hyperglycinemia. Previously we determined the