Multitechnique solution of a new structural type
Bi3MnO11.22(NO3)9.93
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The black fine powder of the new compound was obtained by
hydrothermal reaction; indexing of the electronic diffraction
patterns provided the hexagonal cell. A combination of direct
method of structure solution from powder (EXPO2004) and direct-
space method (FOX) using only synchrotron X-ray data allowed
to locate heavy atoms in the structure, but provided very diffuse
information about the light atoms. Further structure solution
and Rietveld refinement (FullProf) using neutron data led to the
non-centrosymmetric unit cell described in a space group
P3 (a = 4.9679(3) Å, c = 13.161(1) Å) with one NO3- group per cell,
significant distortion of Mn octahedra, and also random oxygen
vacancies around Bi(1) atoms (Figure). Bi(1) has either five ligands
or appears in a distorted octahedra; while Bi(2) and
Bi(3) atoms have umbrella-
like environment built of
three oxygen ligands. The
absence of the inversion
centre was confirmed by the
second harmonic generation
measurement; the presence of
NO3- groups in the structure
was confirmed by FT-1R
measurements and TG Mass
spectroscopy.

Keywords: electronic diffraction, X-ray diffraction, neutron
diffraction

The interaction between human rhinovirus 3C protease and stem loop D studied by solution scattering
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We present the results of recent small angle X-ray scattering and neutron contrast variation experiments that are part of a large effort
to determine the structure of the 3C protease (3Cpro) and stem loop
D (SLD) complex from human rhinovirus 14 (HRV-14). Human
rhinoviruses are members of the picornaviridae family of small
RNA viruses. Like all RNA viruses, picornaviridae achieve gene
expression by inducing the host cell to produce a single polypeptide
that is post-translationally cleaved to produce active viral proteins.
In HRV-14 the role of cleaving viral proteins from the host-
expressed polypeptide is performed by 3Cpro, which also acts as a
crucial component of the HRV-14 ribonucleaseprotein complex that
initiates transcription of the viral genome during replication. The
activity of this complex is dependent on the binding of 3Cpro to the
cloverleaf structure known as SLD near the start of the 5’ region
of the genome. While the structure of the 3Cpro-SLD complex is
unknown, individual structures of 3Cpro and SLD have been solved
by X-ray diffraction and NMR. We have studied a 1:1 3Cpro-SLD
complex using small-angle scattering and determined the ensemble
average structure of the complex in solution. Comparisons between
the structure of the complex and existing high resolution structures
of the individual components will provide insights into conformational
changes during the formation of the complex and assist in structure-
based drug design.

Keywords: neutron contrast variation, SAXS, RNA-protein
complexes

Inhibition of histidine kinase A in Bacillus subtilis: A
neutron contrast variation study
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We have used small-angle scattering with neutron contrast variation
to investigate the interaction between Histidine Kinase A (KinA)
and two of its inhibitors: Sda and KipI. In Bacillus subtilis, KinA
is responsible for initiating a phosphorelay that culminates in the
expression of genes controlling spore formation. The organism
expresses Sda if it fails to replicate its chromosome, thereby
preventing spore formation. Two Sda molecules then bind KinA
and inhibit the autophosphorylation reaction. We have shown
that Sda binds the dimerisation domain of KinA, which undergoes
a conformational change resulting in a compaction of the KinA
structure. While KipI is a much larger protein than Sda, and is
expressed under different conditions, two KipI molecules also
interact with the dimerisation domain of KinA resulting in the same
compaction. Our molecular models for the inhibitor complexes
(based on the crystal structures of homologous proteins) coupled with
bioinformatic analyses of these systems show that the compaction is
due to a collapse of the catalytic domains towards the dimerisation
domains, which prevents the catalytic domains from appropriately
accessing the target histidine. Our results also indicate that the
dimerisation domain (a four-helix bundle) operates as a conduit for
transmitting an inhibitory signal down the length of the molecule,
an observation that has implications for the histidine kinases that are
widely used in bacterial signal transduction.

Keywords: neutron contrast variation, small-angle scattering,
signal transduction

The KipI-KipA complex and histidine kinase regulation
in Bacillus subtilis
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The decision to sporulate in *Bacillus subtilis* is primarily governed by the histidine kinase KinA instigating a phosphorylating process resulting in the phosphorylation of the transcription factor Spo0A. The KipI protein is a regulator of this pathway, produced in response to low dietary nitrogen and high glucose conditions. KipI binds KinA, preventing the autophosphorylation required for the instigation of the phosphorylating. The KipA protein is expressed from the same operon as KipI and prevents its function as an inhibitor of KinA autokinase activity (an anti-anti-kinase). We have overexpressed and purified both proteins to homogeneity and demonstrated that they interact to form a complex. Biophysical investigation including small angle X-ray scattering and neutron contrast variation (using deuterated KipI) show that whilst the individual components are dimers at high concentration, when combined they form a 1:1 protein complex. The reconstructed shape of the KipI component closely resembles the structural envelope of a homologue ascribed as one subunit of an enzyme involved in urea metabolism — allophane hydrolyase. Although no structural model for KipA exists, at the amino acid level it resembles another allophane hydrolyase subunit, raising the question of the evolution and function of these ubiquitous folds. Implications as to how anti-anti-kinase activity is achieved are discussed.

Keywords: small-angle scattering, neutron contrast variation, signal transduction

**P02.16.49**  

**Synthesis, microstructure and catalytic property of nanocrystalline La$_1$-$\chi$Ce$_\chi$MnO$_3$**

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Manganites have attracted much attention of many researchers due to their fascinating physical and chemical properties, including catalytic one. In this study, Ce-doped nanocrystalline La$_1$-$\chi$Ce$_\chi$MnO$_3$ ($\chi$=0.05 - 0.2) powders have been prepared using sol-gel method at a relatively (800°C). The crystal structure was examined by X-ray powder diffraction (XRD). The change in morphology, particle size and its surface area were also investigated by FE-SEM, TEM, SEM, BET measurements. Warren-Averbach and Williamson-Hall methods were used for microstructural analysis. The catalytic oxidation over hydrocarbon of nanopowders has been investigated.

Keywords: Ce-doped nanocrystalline LaMnO$_3$, microstructure, catalytic property

**P02.16.50**  

**Site preference of Mn in Zn$_2$SiO$_4$ phosphor by combined Rietveld refinement**

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Zn$_2$SiO$_4$:Mn$^{2+}$ as a green-color emitting phosphor which have an emission peak around 517 nm under UV excitation (247 nm) has been synthesized in solid state reaction. The combined Rietveld refinement was carried out to determine the site preference of Mn$^{2+}$ ions in Zn$_2$SiO$_4$ phosphor. Of possible cation-disorder models, the best structural refinement result was obtained from a model that Mn$^{2+}$ ions substitute for Zn$^{2+}$ ions in ZnO$_2$ tetrahedra. The model proposed by the combined Rietveld refinement was corroborated by the first-principle pseudopotential calculation. The converged weighted R-factor, $R_p$, and the goodness-of-fit indicator, $S$ ($=R_p/R$) were 9.17 % and 2.40, respectively. The occupancies of Mn$^{2+}$ ions for two different Zn sites were 0.034(4) and 0.006(4), respectively. The refined model described a structure in space group $R3$ (No.148) with $Z = 18$, $a = b = 13.9611(1) Å$, $c = 9.3294(1) Å$ and $\gamma = 120^\circ$.

Keywords: phosphors, Rietveld analysis, X-ray neutron powder diffraction