PS02.09.11 DEVELOPMENT OF NEW METHOD FOR STRUCTURE DETERMINATION BASED ON XANES ANALYSIS. Yu.F.Migal, Dept.of Physics, Don State Technical University, Gagarin Sq. 1, Rostov-on-Don 344010, Russia

Maxima in X-ray Absorption Near Edge Structure corresponding to one-electron quasi-stationary states (shape resonances) contain various information about objects under investigation. Positions and widths of the maxima depend strongly on arrangements and types of atoms, the presence and nature of defects, the distribution of electric charge, etc. This is why the study of Xanes can be a useful tool for the structure determination, particularly in the case of unordered systems.

A scheme of extracting information from experimental data related to shape resonances was proposed by the author recently [1, 2]. It is based on the S-matrix pole equation within the muffin-tin approach [3]. The system of square-well potentials with depths depending on the orbital quantum number l is used [4].

The scheme has been tested by the determination of geometric parameters of the free molecules N_2, NO_2 and CH_3 NO_2 and the solid compound $NaNO_2$. It was shown that this scheme enables to determine internuclear distances up to 1% and valence angles up to 3%.

The method proposed can be applied to investigate parameters of molecules adsorbed on solid surfaces. For example, the adsorption of the O_2 molecule on the Cu(100) surface is studied. By using the oxygen K spectra the most probable location of the molecule, the molecule-surface distance and the change of the O-O distance caused by the adsorption are found.

[1] Migal Yu.F. J.Phys.B: At.Mol.Opt.Phys. 27 (1994) 1515-1524.
[2] Migal Yu.F. Physica B 208&209 (1995) 77-78
[3] Migal Yu.F. J.Phys.B: At.Mol.Opt.Phys. 26 (1993) 2755-2766.
[4] Migal Yu.F. J.Phys.B: At.Mol.Opt.Phys. 26 (1993) 2767-2775.

PS02.09.12 EXPERIMENTAL TRIPLET PHASES FOR DISCRIMINATING BETWEEN SIMILAR STRUCTURE MODELS. Mo, F., Mathiesen, R. H., Nyborg, T., Dept. of Physics, Norwegian Univ. of Science and Technology - NTNU, N-7034 Trondheim, Norway

Physically estimated triplet phases (PETP) from 3-beam diffraction experiments appear uniquely suited to identify the correct or «best» structure model among several possible and closely related ones. The experimental work involves measurement of a selected set of triplets of which the model sensitive ones are used for model discrimination. A model sensitive triplet contains at least one structure factor phase which is significantly influenced by the change in structure. This novel application of PETP's is being explored in a study of the complex α-D-glucose • NaCl • H_2O (2:1:1) in space group $P3_1$ with V = 4.180 Å^3. A structure (model I) has been published [1], however, at least one alternate solution (model II) exists [2]. We have refined both models and identified the difference in structure. The phases for the strong F_o are closely similar in the two models, thus for the 522 $F_o > 50$ the mean difference, $\Delta\phi$, is 3.80°. For reflections of intermediate strength, e.g. the group $10 < F_o < 40$, $\Delta\phi$ shows a pronounced bimodal distribution, 99.4% falling in one of the two ranges 0 - 30° and 150 - 180°. For the very weak reflections the $\Delta\phi$ histogram is more uniform but peaked in the range 150 - 180°. A total of 89 triplet phases, including both model sensitive and insensitive ones, have been estimated from measurements on seven different crystals. The PETP's for each crystal are internally fully consistent with either Model I or Model II, but the set of crystals define two distinct groups. Thus, the results so far indicate that both structure models are present, but do not coexist in the same crystal specimen.

[1] Ferguson, G., Kaitner, B., Connett, B. E. & Rendle, D. F. (1991). *Acta Cryst.* B47, 479-484.
[2] Fröhlich, R. Personal communication.

PS02.09.13 USE OF HARKER SECTIONS IN A DENSITY MODIFICATION METHOD FOR DIRECT SOLUTIONS OF CRYSTAL STRUCTURES. By M.Shiono, Department of Physics, Kyushu University, Higashi-ku, Fukuoka, Japan

A real space phase refinement procedure has been developed, which is based on elimination of negative density (Shiono et al. (1992) Acta Cryst. A48, Refaat et al. (1993) Acta Cryst. A49). This method can produce ab initio solutions of crystal structures by employing multi-solution procedure. In order to give more power to this method, modified Patterson-Harker Sections are used as a mask of electron density.

Harker sections include self-interatomic vectors of component atoms and this fact gives useful informations to us for solving crystal structures. However, if the structure is complex, it is very difficult to interpret Harker sections since selfinteratomic vectors seriously overlap each other and, in addition, non-self-interatomic vectors may appear in the Harker sections. For above reason, instead of searching self-interatomic vectors, we find regions in which electron density is 0 or very small. In order to use this information as a constraint of density modification, Harker sections are modified as described in following example.

The method has been applied to ab initio phase determination of a protein, Ribonuclease Apl (Space group $P2_1$). For this space group, the Harker section appears at y=1/2 and self-interatomic vectors are (2x,1/2,2z). The 2-dimensional mask is constructed as follows. Firstly, Harker section is calculated from all observed E's. Secondly, the Harker section is shrunk to 1/4 in area and then 4 pieces of them are placed in the original size of plane. We thus get a section which contains 4 shrunk Harker sections. Finally, negative density in the section is set to 0. This protein has been solved by iterative use of this section as a mask of density.

PS02.09.14 A NEW SOLUTION TO THE PHASE PROBLEM. Gu Xu, Dept. of Materials Sci. & Eng., McMaster University, Hamilton, Ontario, L8S 4L7, Canada

A new solution is proposed to solve the 'phase problem', which has in the past prevented people from using X-rays to determine non-crystalline structure, and made phase determination tedious, if not impossible, for bio-molecular crystals. It involves only current X-ray diffraction technology, with the sample attached to a single crystal to form an assembly. The X-ray intensity diffracted by the assembly will then produce a convolution function which contains the cross modulation of both the unknown and crystal density functions. With the known crystal structure, the electron density of the amorphous or biological specimen can thus be resolved by a simple mathematical image inversion algorithm to obtain the atomic arrangements in 1, 2 or 3 dimensions. In principle, this method uses the same idea of a reference object of known structure (the single crystal) to probe the unknown, as the holographic method now widely used in optical systems. However, no reference beam is needed in this method, thus no macroscopic coherent X-ray is necessary. Moreover, the Bragg diffraction peaks from the single crystal can be omitted from the measurement, which provides the scheme with the same noise tolerance as used in the usual amorphous diffraction experiment.