**s7.m6.o3** **Anomalous Dispersion Experiments in Microporous Materials Crystallography.** V. Kaucic[1], N. Zabukovec Logar[1], M. Helliwell[2], and J.R. Helliwell[2], [1]National Institute of Chemistry, Hajdrihova 19, 1000 Ljubljana, Slovenia, [2]Department of Chemistry, University of Manchester, Manchester M13 9PL, UK.
Keywords: microporous materials, anomalous dispersion, area detector.

Knowledge about the framework and extra-framework cation positions in microporous zeolite-like materials (e.g.: $Co^{2+}$, $Ni^{2+}$, in metal-substituted aluminophosphates, aluminosilicates etc.) offers a crucial insight into their catalytic properties. Isomorphous substitution of transition-metal ions into Al, Si, P or some other non-oxygen framework sites gives rise to acid and redox active centres, whose determination present a very challenging crystallographic problem (low relative occupancies, elements close together in atomic number). Anomalous dispersion methods using synchrotron X-ray sources, have already been successful in the structure determination of some of these materials (e.g. cation distribution in $FeNi_2BO_5$, the Zn and Na extra-framework distribution in ZnNaY). The use of IP or CCD area detectors in recent years greatly reduces data collection time and consequently the chance of errors due to sample decay or beam instabilities. The problems that arise are due to small apertures of current detectors and long wavelengths that are often needed in the experiments (e.g. NiK edge 1.488 Å, CoK edge 1.608 Å, or MnK edge 1.896 Å). The resolution limits of the data therefore tend to be rather low. For example, in our five-wavelength study of cobalt zincophosphate CoZnPO-CZP[1], the resolution limits were only 1.44 Å for the two data sets collected at the ZnK edge, and 1.8 Å for the two data sets collected at the CoK edge using 180mm MAR IP. In order to improve the resolution of the required data sets at these or even longer wavelengths, some adaptations in detectors geometry are needed. The best choice seems to be a cylindrical IP holder realising $2\theta_{max}=180°$. Cipriani et al.[2] made a prototype of the cylindrical IP detector for neutron and X-ray diffraction patterns. Recently, Takagi et al.[3] reported a new operating image-plate reader for various sizes and shapes up to 40x40cm$^2$, with IP lying on the breathnite cylinder with diameter of 20 cm.

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**s7.m6.o4** **Use of map connectivity properties in macromolecular *ab-initio* phasing.** N.L. Lunina, V.Y. Lunin, A.G. Urzhumtsev, *Institute of Mathematical Problems of Biology, RAS, Pushchino 142290 Russia; LCM³B, Facultés des Sciences, Université Henri Poincaré, Nancy I, France.*
Keywords: connectivity, *ab-initio* phasing, macromolecules.

Depending on the resolution the regions of high values in properly phased macromolecular Fourier syntheses present different types of information. In particular, the properly phased low resolution syntheses reveal huge 'blobs' corresponding to molecules as a whole. At an intermediate resolution the region of high synthesis values looks like a long continuous chain corresponding to the backbone of the polypeptide chain. These properties disappear if wrong phases are used to calculate Fourier synthesis and the high density regions reveal a number of 'drops' or endless strands which continue through the whole crystal.

The first step of the suggested phasing procedure consists in: generating a number of random phase sets; calculation of corresponding Fourier syntheses (with the use of the experimental magnitudes); determination of the number of separated connected components in high values region; and selecting phase sets which result in the desired connectivity properties. At the second step cluster analysis methods are applied to chose a small number of clusters of close phase sets among the selected phase sets. The phase sets averaged through different clusters may be used as nodes in multi-solution phasing strategy or some additional criteria may be applied to select the most reliable phases.

The testing of the suggested procedure with low resolution experimental data sets (about 20 reflections per molecule in the unit cell) for several structures with already known atomic models allowed to get 70-80% correlation between the ab-initio phased synthesis and the exactly-phased one. An image of such quality allows to determine molecular positions in the unit cell and define the envelopes which at low resolution correspond to a joint region of all molecules in the unit cell rather then to an individual molecule.

The developing of the procedure for medium resolution phasing is in progress. The attempt of ab-initio phasing for a relatively small Protein G (the atomic model is known) resulted in the ab-initio synthesis with the following correlation to the exact one: 59% at 7Å resolution (117 independent reflections); 50% at 5.4Å resolution (243 reflections) and 33% at 4Å resolution (580 reflections). The quality of this synthesis allows to identify beta-structure elements. The ab-initio phasing for a larger molecule (Elongation Factor G) resulted in the 20Å resolution synthesis (84 reflections) with the correlation 57% to the exact one.

The developed procedure was applied for ab-initio phasing the Low Density Lipoprotein particle at 27Å resolution (about 800 reflections).