reproducible as tetragonal bipyramides. Diffraction images of different crystals show split or smeared spots but symmetry is clearly visible. Indexing works properly using different programs suggesting a primitive tetragonal lattice with a = b = 66.8 Å and c = 113.2 Å and one molecule in the asymmetric unit. Merging statistics and automatic space group assignment in various program packages suggests point group 422 with a $4_{1/3}$ screw axis along the fourfold and also a twofold screw axis (space group $P4_12_12$ or $P4_32_12$). But some strong violations of systematic absences at low resolution along the fourfold and one twofold axis indicate that space group $P4_{1/3}2_12$ may not actually be correct. Despite this, a three-wavelength SeMet-MAD dataset was measured with a resolution of 2.8 Å and useable anomalous signal to about 3.5 Å. SHELXD [1] consistently found a solution for the Se-substructure in space group $P4_12_12$ (4 out of 5 Se atoms with CC all of 52.9 and CC weak of 33.6). After solvent flattening with SHELXE [1], the experimental electron density revealed the expected three helical bundle. No improvement was achieved in further attempts of density modification using different programs from the CCP4 program suite [2].

Using the Se positions as anchors for sequence assignment, around 80 amino acids (out of a total of 221) could be built, another 40 could be placed with some uncertainty. The free R-factor stalled at ~46 % with a FOM of ~50 %, and little difference density that would have allowed to extend the model. This model was taken for molecular replacement with PHASER [3] into the single datasets of the MAD experiment and other datasets from the native protein. Single solutions were found but with rather low Z-scores of ~3.6 (rotation function) and ~6 (translation function). We also tried molecular replacement with lower symmetry down to $P4_1$, $P2_12_12$ and $C222_1$ as well as experimental phasing in these space groups. But this did not result in better phases that allowed extending the structure or refining it to lower R-factors.

Apparently, this is a case of severe pseudosymmetry as refinement of an initial model in the suggested high symmetry spacegroup $P4_12_12$ did not work. Pseudo-merohedral twinning in a lower symmetry space group seems possible but analysis with PHENIX Xtriage [4] did not detect any indication for this. Experimental phasing in space groups $P2_1$ or P1 did not work due to lower multiplicity of the data. After all, other crystallographic or biochemical approaches may be required to determine the structure of this protein.

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Low energy SAD experiments performed at the photon factory BL-1A

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Recent developments in SAD (single anomalous dispersion) phasing techniques facilitate to solve macromolecular crystal structures using light atoms such as sulfurs or phosphors. Longer wavelength beam ranging from 1.7 Å to Cr K-edge (~2.3 Å) has been mainly used in data collection to enhance the weak anomalous signals. The method would be attractive for de-novo structure solutions without any derivatives, including membrane proteins or macromolecular complexes, for which heavy atom or selenomethionine derivative crystals are difficult to prepare.

We have developed a synchrotron beamline dedicated to long wavelength SAD experiments. The beamline is designed to take full advantage of a long wavelengh X-ray beam at around 3 Å to further enhance anomalous signals. The light source is an in-vacuum short gap undulator optimized at around the wavelength with the fundamental harmonics to obtain maximum brilliance. The vacuum section of the beamline has only one terminal beryllium window, followed by a diffractometer equipped with a helium cryostream and a specially designed helium chamber to minimize the attenuation of the lower energy beam and background noises. A cryo-cooled channel-cut monochromator and bimorph KB focusing mirrors compose a simple optics to deliver a focused beam with a good stability. The focused beam size (FWHM) at the sample position is 70 μm (H) x 10 μm (V), and the measured beam intensity is in the order of 10^{10} photons/sec on the area of 10 μm square.

Diffraction experiments using the wavelength of 2.7 Å was performed against various protein or nucleic acid crystals. X-ray detector was the commonly used CCD area detector (ADSC Quantum 270). Some of the crystals are mounted in the 'mother-liquor free' condition following the method developed by Hokkaido University [2][3] to decrease the background noise and anisotropic absorptions. Fully automated structural solutions were obtained for some crystals. In the presentation, the effectiveness of the mounting method, the helium cryostream and variable beam sizes are discussed. The exploration of the parameters used for phasing (resolution cut-off, number of frames, etc.) will be presented in the relationship with the radiation damage.

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Coordinated ligand effects in the substitution kinetics of $[Re(CO)_3]^+$ complexes

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The coordination chemistry of rhenium gained a lot of interest in the last few years, since Alberto's *fac*-$[^{99m}Tc(H_2O)_3(CO)_3]^+$ complex were remarkably synthesized from $[^{99m}TcO_4]^-$ in water and under mild conditions [1].

Synthetically, a lot of work has been published on $[M(CO)_3]^+$ (M = Re, Tc) systems with a huge variation in ligand systems [2 – 7]. The aqua ligands on the starting synthon, $[Re(CO)_3(H_2O)_3]^+$, can readily be substituted by a variety of combinations of ligands to produce potential radiopharmaceuticals with many different characteristics.

Our interest lies with the *fac*-$[Re(CO)_3]^+$ moiety and adopting the [2+1] approach [8]. The solid state behaviour and different effects like charge of the complexes and the influence of coordinated bidentate ligands on the rate of substitution can be explored.