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| | Experiment title: Crystal Structure of Human Frequenin | Experiment number: LS1657 |
| Beamline: ID14-2 | Date of experiment: from: 3-3-00 to: 4-3-00 | Date of report: Aug00 |
| Shifts: 3 | Local contact(s): Sigrid STURMAN | <i>Received at ESRF:</i> |
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Report:

Human frequenin (1) crystals belong to three different crystal forms: (A) hexagonal needles, (B) thick and (C) thin plates that grow occasionally in the same drop. Crystals form A diffract to 2.8 Å resolution while crystal forms B or C diffract up to 1.9 Å resolution. Three complete data sets were collected for forms A and C (Table 1). Initial phases for form C crystals were obtained by molecular replacement using neurocalcin (PDB code 1BJF) as a search model with the AMoRe package, giving a correlation coefficient and an R-factor value of respectively 36% and 48% in the 15 Å to 4 Å resolution range. Rigid-body refinement was then performed on each subunit with CNS using data between 30 Å and 3 Å and gave an R-factor of 49%. For 3% of the reflections against which the model was not refined, R-free was 48%. Refinement of the molecular replacement model was performed at 2.1 Å resolution using CNS including bulk solvent and anisotropic B-factor corrections; the resulting 2Fo-Fc and Fo-Fc electron density maps were used to correct the model with the graphics program TURBO-FRODO. Addition of solvent molecules was automatically performed using CNS and were carefully examined on the graphics display. This model was then refined to 1.9 Å resolution using a new data set. However, no clear solution was found for crystal form A, suggesting that these crystals might have a twinning problem.

Table 1. Data collection and refinement statistics

| | form A | form C | form C |
|-----------------------|-----------------|-----------------|-----------------|
| Space group | P6 _x | P2 ₁ | P2 ₁ |
| Resolution (Å) | 2.85 | 2.1 | 1.9 |
| No. observations | 29 553 | 189 398 | 281 683 |
| No. unique | 4 916 | 24 748 | 35 881 |
| R _{sym} (%) | 6.0 (38) | 7.2 (35) | 6.0 (35) |
| I/σ(I) | 7.2 (1.9) | 6.6 (1.8) | 8.9 (2.0) |
| Redundancy | 2.7 | 2.5 | 3.3 |
| Completeness (%) | 96.4 (96.1) | 92.9 (80.4) | 99 (98.9) |
| Resolution (Å) | - | 30 – 2.1 | 20 – 1.9 |
| R-factor - R free (%) | - | 24.6 – 29.2 | 22 – 25.6 |

References:

- 1 Pongs, O *et al.* (1993) *Neuron* **11**, 15-28.