s13.m37.p7 **Kinetics of the Single-To-Single Crystal Isomerisation Reactions of (η^5-C_5H_4Me)$W(CO)_2$(L)I, (L = $P(O^iPr)_3$, PPh_3).** Demetrius C. Levendis[a], Robert S. Bogadi[a], Muhammad Bala[a], Bhekie B Mamba[a] and Neil J. Coville[a], [a]*Molecular Sciences Institute, School of Chemistry, University of the Witwatersrand, PO WITS, 2050, South Africa. E-mail: demi@aurum.chem.wits.ac.za*

Keywords: Solid-state; Isomerization; Kinetics

The *cis-trans* isomerization in crystals of tungsten complexes, (η^5-C_5H_4Me)$W(CO)_2$(L)I (L = PPh_3 (**1**), $P(O^iPr)_3$ (**2**)) was studied using single-crystal and powder XRD, microscopy and thermal analysis. A complete crystal structure analysis at seven stages of the thermally initiated reaction in 1 showed that the variation in the site-occupancy with reaction time at 100°C (Fig. 1) follows an exponential decrease, similar to that observed [1] in crystals of the Re complex (η^5-C_5H_4Me)Re(CO)($P(OPh_3)Br_2$.

The reaction direction is *trans (diag)* to *cis (lat)* for **1**, L = PPh_3, the direction most frequently observed; for 2, L = $P(O^iPr)_3$, the isomerization proceeds in the opposite direction. The direction of the isomerization reaction is related to the melting points of the *cis* and *trans* isomers. Furthermore, the rate of *cis-trans* conversion in 1 is coupled to the rotation of the cyclopentadienyl group and/or the disorder of the methyl group.

Previous crystal structure determinations of the *cis* and *trans* isomers of (η^5-C_5H_4Me)$W(CO)_2(PPh_3)$I [2] showed that only a limited amount of isomer conversion can be accommodated in the crystal of the *trans* isomer prior to crystal fragmentation. These new studies show that this observation may depend on the environment, such as whether one single crystal or several crystals, some in contact, are heated simultaneously. Also, the structure of the final *cis* product obtained by heating a single crystal of **1**, i.e. after 320 hours (Fig.1) differs considerably from that of the recrystallised *cis* compound. Preliminary XRD results indicate that there is a likely phase transformation between these two polymorphs.

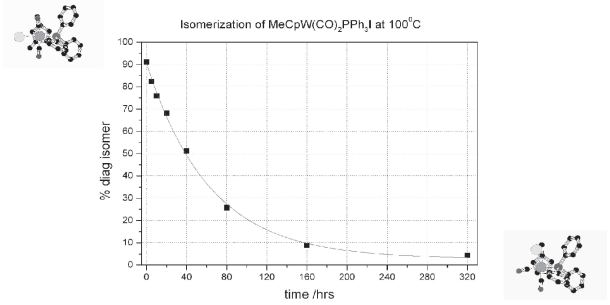

Figure 1. Isomerization kinetics for a single crystal of (1)

[1] R.S. Bogadi, D.C. Levendis and N.J. Coville, *Journal of the American Chemical Society*, 2002, **124**, 1104-1110.
[2] O. G. Adeyemi, , U.B. Eke, L. Cheng, L.M. Cook, D.G Billing, D.C. Levendis, B.B Mamba and N.J. Coville, *Journal of Organometallic Chemistry*, submitted

s13.m37.p8 **The Effect of the Low Magnetic Field on the Quality of Tetragonal Lysozyme Crystals Grown with Paramagnetic Salts.** Ana Belén Moraleda Merlo[1,2], José Antonio Gavira[1], Fermín Otálora[1], Vivian Stojanoff[2]. [1]*LEC (IACT) Campus Fuentenueva (Facultad de Ciencias) 18002 Granada Spain;* [2]*Brookhaven National Laboratory, National Synchrotron Light Source, Upton, NY 11973, US. E-mail: anabelen@ugr.es*

Keywords: Macromolecule structure; Crystal quality; X-ray diffraction

The effect of magnetic fields on the quality of bio-molecular crystals has been observed by several groups [1]. [2] used low magnetic fields 1.6T, for the first time, to grow Tetragonal Hen Egg White Lysozyme (HEWL) crystals by the batch methods. These authors observed when a magnetic field was applied during the entire crystallization process, about the 95% of the crystals were oriented.

Here we report on the effect of low inhomogeneous magnetic fields on the crystallization of tetragonal HEWL crystals. Crystals were grown by the counter diffusion method in the presence of different paramagnetic salts at room temperature from Sigma Lysozyme stock without any further purification. The counter diffusion apparatus were kept at 0 T or in an inhomogeneous field for 24 hours and for 7 days. Crystals, obtained in the 0 field condition were used for control.

Crystals were inspected visually under a light microscope. The quality of the crystals was determined from X-ray diffraction studies performed at room temperature. The crystals were not removed from their original growth containers for these studies. The mosaicity, lattice parameters and diffraction power were monitored by the Rotation Method. Rocking curve measurements were performed on all crystals with the fine slicing data collection technique. X-ray imaging techniques were employed to observe the structural formation of defects in selected crystals. In general there were no significant differences observed between the control (0 T) crystals and crystals grown in low magnetic field. Crystals however followed the orientation of the magnetic field.

[1] Sazaki et al 1997, Wakayama *et al* 1998, Takato *et al*., 2000.
[2] Wakayama *et al* 1998.