MS45-P3 **Setting high resolution limits for anisotropic data** or **The effect of high resolution cutoff schemes on refinement quality for strongly anisotropic data** Melanie Vollmar,[a] Garib Murshudov[b], Frank von Delft,[a] *[a]University of Oxford, Structural Genomics Consortium, Old Road Campus Research Building, Oxford, OX3 7DQ, U.K.,[b]MRC Laboratory of Molecular Biology, Hills Road, Cambridge, CB2 0QH, U.K.*
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Most diffraction data are anisotropic and often strongly so, which can significantly affect the ability to interpret derived electron density, making model building and structure refinement very challenging. There is no consensus on how anisotropy should be treated at the resolution limit: imposing isotropic resolution limits either excludes many reflections with good signal, or includes reflections without signal.The problem has various approaches both at data integration and structure refinement. Data integration with HKL2000 effectively imposes anisotropic resolution limits, while MOSFLM/SCALA and XDS both apply isotropic resolution limits, yet generate significantly differing estimates of standard deviations of intensitiesin regions where diffraction is weak.These standard deviationsnot only inform subsequent refinement, but the various programs treat these estimates very differently, leading to very different electron density. A popular variation is to modify measured amplitudes using the Diffraction Anisotropy Server in an attempt to compensate for the anisotropy (http://services.mbi.ucla.edu/anisoscale/).

We thus systematically examined the various approaches using a series of strongly anisotropic datasets with resolution below 2.6A. By assessing the residual density after refinement in REFMAC5, we compare the overall outcome of refinement and structure completeness when applying anisotropic, conservative isotropic and generous isotropic resolution cutoffs.In addition, we assess the use of different estimates of standard deviation from various integration/scaling packages, as well as anisotropic B-factor sharpening applied both by the Diffraction Anisotropy Server and within REFMAC5.Finally, standard deviationsare treated in a variety of ways during refinement, including use of intensities rather than amplitudes. A comparative analysis of the different approaches will be presented.

Keywords: anisotropy; resolution limits; refinement

MS46-P1 **Quantification of TiO$_2$ nanoparticles in samples of crystalline TiO$_2$.** Renie Birkedal, Keld Alstrup Jensen, *National Research Centre for the Working Environment, Denmark*
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It is an interesting challenge to detect and quantify small amounts of nanomaterials in a mixture using X-ray powder diffraction. An even greater challenge is to determine the relative amounts in a mixture of two identical phases with differentcrystallite sizes. This work describes the use of X-ray powder diffraction to determine the weight fraction and crystallite sizeofnanocrystallineanatasemixed with microcrystalline anatase. Rietveld refinement of X-ray diffraction data may be used to quantify the ratio between phases in a mixture. A rule of thumb says that quantification is possible for materials if there is more than 2 w% of the material in the mixture. This is of course depending on the instrument used. High resolution instruments at a synchrotron may be used for quantification of even smaller amounts. The materials themselves are also significant; mixtures of highly crystalline materials and few overlapping reflections may be quantifiedin even lower amounts, but if there are many overlapping reflections quantification is much more challenging.A material in the nano-size range has broad reflections; the smaller the crystallites, the wider the reflection. If the mixture consists of only one polymorph but with two different sizes of crystals, all reflections will overlap.The question is; can these reflections be separated and the right ratio calculated?The topic of this poster is the search for the limits of reliable quantifications of the ratio between known mixtures of "nano" anatase crystals and large crystals of anatase. Four different programs have been used, TOPAS 4.1 from Bruker[1], GSAS [2], PowderCell[3] and Fityk[4]. The first 3 programs all have all had the structure of anatase, whereas the last, Fityk, has been used model free and only to calculate the area of the reflections, whereby the ratio between the phases have been calculated.The calculations have been performed using data from an in-house Bruker D8 diffractometer. The powder samples used have been mixtures of crystals around 200 nm together with crystals of either 7 nm, 26 nm or 27 nm.EU has defined "nanomaterial" as a "material containing particles, in an unbound state or as an aggregate or as an agglomerate and where 50% or more of the particles in the number size distribution, (has) one or more external dimensions is in the size range 1 nm – 100 nm" [5]. This means, that a mixture of 200 nm crystals and 20 nm crystals is a "nanomaterial" if there is more than 0.1 w% of the 20 nm crystals.

[1] Bruker AXS, TOPAS Version 4.1; Copyright 1999, 2008 Bruker AXS.
[2] Larson, A.C. and Von Dreele, R.B., 2004. General Structure Analysis System (GSAS).*Los Alamos National Laboratory Report* (2004) LAUR 86-748.
[3] PowderCell for Windows, Version 2.4 by W. Kraus and G. Nolze,Federal Institute for Materials Research and Testing, Germany.
[4] Fityk; M. Wojdyr, (2010) *J. Appl. Cryst.* **43**, 1126.[5] Nanomaterials, Definition of a nanomaterial; http://ec.europa.eu/environment/chemicals/nanotech/index.htm

Keywords: crystallite size; quantitative analysis by XRD;Rietveld refinement