MS46-05 **An alternative method for quantitive XRPD analysis; partial least square regression.** Céleste A. Reiss[a], Detlef Beckers[a], Thomas Degen[a], Dorith Stauch-Steffens[b], [a]*PANalytical B.V., Almelo, The Netherlands,* [b]*University Bonn, Institute for Pharmaceutical Techology, Germany*
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The quantification of crystalline phases in mixtures, or of polymorphic impurities in active ingredients, or the quantification of an amorphous content in a crystalline matrix have an increasing importance in the pharmaceutical industry. Polymorphism and the stability of amorphous regions in a crystalline matrix are directly related to product stability, dissolution rates and processability of the pharmaceutical excipients and active substances. It is therefore important to control them in all process steps. In this presentation we investigate the applicability of PLS (Partial Least Square) regression methods to the quantification of crystalline phases and the determination of the crystallinity of organic compounds by X-ray powder diffraction. As a model substance we used lactose to create calibration curves based on predetermined mixtures of highly crystalline and amorphous substances as well as mixtures of different crystalline (pseudo-)polymorphs of lactose. The results are compared with traditional analytical X-ray powder diffraction methods, namely the Rietveld analysis, the FULLPAT [1],[2] (full pattern quantitative analysis for X-ray diffraction) method and calibration models based on measured peak intensities.

[1] Smith D.K., Johnson G.G.Jr., Scheible A., Wims A.M., Johnson J.L. and Ullmann G., (1987). *Powder Diffr.* 2, 73-77.
[2] Chipera S.J. & Bish D.L., (2002) *J. Appl. Cryst.* 35, 744 -749.

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MS47-01 **Current understanding of X-ray induced radiation damage on macromolecular crystals.** Kristina Djinović-Carugo[a], *Department of Structural and Computational Biology, Max F Perutz Laboratories, University of Vienna, Campus Vienna Biocenter 5, 1030 Vienna, Austria*
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Radiation damage to macromolecular crystals is an inherent problem of X-ray crystallography, especially at the highly brilliant synchrotron sources. The first systematic study on radiation damage in protein crystals was carried out in 1962 at room temperature on myoglobin crystals by Blake and Phillips. During the last ten years the topic of radiation damage in macromolecular crystallography has become an increasing concern for structural biologists. It has become clear that, even with the crystalline sample kept at 100?K during the data collection, not only do the deleterious effects of damage affect the chances of successful structure solution, but they can also compromise the biological information that may be inferred from the results. An overview will be given on the current understanding of X-ray induced radiation damage and explored practical ways of mitigating its effects. Finally, a study will be reported on UV-vis micro-spectrophotometry/X-ray crystallography systematic study of a wide range of potential soaked-in scavengers to assess their capacity to: (i) alleviate photo-reduction of metal centres, (ii) to reduce global and specific radiation damage effects in presence of cryoprotectants and high concentrations of ammonium sulphate employed as a common precipitating agent. The study showed that due to the fast initial reduction of metal-centres it seems improbable that any concentration of the tested scavengers can efficiently protect the metal centres of metalloproteins from X-ray induced photo-reduction.

Keywords: X-ray induced radiation damage