s10.m1.p3 **Structure determination of two organometallic complexes from powder data with grid-search techniques.** E. Dova, K. Goubitz., R. Driessen, E. Sonneveld, V. Chernyshev, H. Schenk. *Laboratory for Crystallography, IMC, University of Amsterdam, Nieuwe Achtergracht 166, 1018WV Amsterdam, The Netherlands.*
Keywords: structure determination, powder data, grid search.

The structures of the organometallic complexes $[Re(PPh_3)_2(CO)_2(DBQ)]^+.PF_6^-$ (DBQ = 3,5-di-*tert*.butyl-benzoquinone) and $[Fe(teec)_6]^{2+}.2BF_4^-$ (teec = 1-(2-chloroethyl)-tetrazole) have been determined from Guinier-camera powder data (?=1.54056 Å, with 4º<2?<82º and 4º<2?<81º for the second complex and a scan step size of 0.01º), using the grid-search techniques performed by the program MRIA[1,2]. The unit cells of both structures were determined with the program ITO[3]. In both cases, first the organometallic moieties were located, then they were kept fixed and the PF_6^- and BF_4^- respectively were searched for. After the determination of the position of the whole molecule, torsion-angle variations were applied to determine more accurately the position of the rings and some other smaller fragments. Then, Rietveld Refinement (RR) on basis of Guinier data was performed. The background parameters were refined (fifth order polynomial) and a split-type pseudo-Voigt profile was assigned to the intensities. Zero correction, cell-parameter refinement and texture correction (symmetrized harmonics expansion) were applied. Any attempt to refine the coordinates led to a distorted model. High-resolution data, which give narrower peaks (thus less overlap), were expected to improve the refinement. For the second complex synchrotron data have been collected at the ESRF (BM1 Swiss-Norwegian beam line) with ? = 0.99452 Å. The refinement is in progress and gives indeed better results with the coordinates being refinable. Some crystal data and the R-factors in the several stages are listed in Table 1. It is remarkable how the torsion-angle search improves the starting model used for the refinement. More details and information about these structures will be given in the near future elsewhere [4,5].

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s10.m1.p4 **Magnetic textures – Analysis of Easy Magnetization Direction; Application to Powder Pattern Decomposition.** R. Cerny, *Lab. of Crystallography, University of Geneva, 24 quai Ernest-Ansermet, CH-1211 Geneva, Switzerland.*
Keywords: texture, powder pattern decomposition, image plate.

Powders of magnetically ordered compounds can be easily textured in a magnetic field if they show an easy magnetization direction (e.m.d.). In the case that e.m.d. is a unique crystallographic direction, the texture is of a simple uni-axial type with the e.m.d. as the texture axis. It can be easily detected by an analysis of the intensity distribution on Debye-Scherres circles (DSC) or by Rietveld refinement. An analysis of crystallographic directions that can be detected unambiguously as the e.m.d. by the texture analysis is given for all crystal systems. The method was applied to test compounds in hexagonal and trigonal crystal systems showing axial (*c*-axis, $SmCo_5$), basal ($TbCo_5$) or intermediate ($Yb_{12}Fe_{64}Al(Ga)_{24}$) e.m.d. Powders of test compounds were enclosed in a spinning capillary and exposed to a magnetic field of 0.3 – 0.5 T as created by a permanent magnet $Nd_2Fe_{14}B$ oriented perpendicular to the capillary axis. Complete DSC were measured using an image plate.

Textured powder patterns can be used for the separation of overlapping diffraction peaks[1]. As large structures as a zeolite with 69 non-H atoms in the asymmetric unit can be determined *ab initio* from powder data using the structure amplitudes obtained by the texture decomposition of powder patterns[2]. A simple empiric function for the preferred orientation correction, which assumes the uni-axial texture, can be used in the texture decomposition of powder patterns[3].

The images of textured DSC of magnetically aligned test compounds were used to test the method. An integration of the images in different azimuth directions benefits from a partially removed DSC overlap due to the preferred orientation. The images were integrated in 7 different azimuth directions and resulting powder patterns were decomposed using the texture method[3].

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