PS10.08.12 A RIETVELD-REFINEMENT PROGRAM FOR THE TOF NEUTRON POWDER DIFFRACTOMETER VEGA. F. Izumi, National Institute for Research in Inorganic Materials, 1-1 Namiki, Tsukuba, Ibaraki 305, Japan; K. Oikawa and T. Kamiyama, Institute of Materials Science, University of Tsukuba, Tennodai, Tsukuba, Ibaraki 305, Japan.

A time-of-flight (TOF) neutron powder diffractometer, VEGA (Kamiyama et al., 1995), has been operated at KENS for over two years in place of the previous one, HRP. VEGA was designed in such a way that one-dimensional position-sensitive detectors are spread two-dimensionally all over three detector banks. This novel geometry makes it possible to enhance the efficiency of data collection without lowering resolution.

A FORTRAN program for Rietveld analysis of TOF neutron diffraction data measured on HRP (Izumi et al., 1987) contained a profile function optimized for HRP, i.e., linear combination of two Cole-Windsor functions (Cole & Windsor, 1980) in a (1-R):R ratio. Preliminary Rietveld refinements of intensity data taken on VEGA with this program indicated that the above profile function no longer fits observed diffraction profiles very well. In particular, observed intensities tend to exceed calculated ones (Gaussian) in leading edges.

The fit between observed and calculated patterns was considerably improved if the leading part is represented by a pseudo-Voigt function instead of the Gaussian function. Then, we determined the dependence of seven parameters in this new profile function on d. The resulting seven equations contains 18 refinable profile parameters, and these were updated during the refinement. Not only are more accurate phase abundances produced, but supplementary information is obtained about individual phase chemistry.

PS10.08.15 IMPROVED MODAL ANALYSIS FROM X-RAY POWDER DIFFRACTION DATA. W.G. Mumme, G. Tsambourakis, R.J. Hill and I.C. Madsen, CSIRO Division of Minerals, P.O. Box 124 Port Melbourne 3207 Australia

Mineral phase abundances have been determined on a range of naturally occurring rock types by Rietveld analysis of X-ray and neutron powder diffraction data.

The phase systems analysed in this work include (i) igneous, metamorphic and volcanic rocks, (ii) sedimentary rocks, and (iii) massive sulphide ores. The Rietveld results are compared with values obtained by (i) optical modes for the medium to coarse grained samples obtained by point counting, and (ii) normative calculation from bulk chemistry. For the sulphide ores, the X-ray results have been improved by the inclusion of a correction for microabsorption effects.

One of the major benefits of a Rietveld based approach over traditional quantitative XRD methods is that the crystal structure can be refined during the analysis. Since this involves the unit cell dimensions and contents, the "calibration constant" (ZMV) can be automatically updated during the refinement. Not only are more accurate phase abundances produced, but supplementary information is obtained about individual phase chemistry.

PS10.08.16 CRYSTAL DATA AND X-RAY POWDER DIFFRACTION DATA OF LOBENZARIT ACID AND LOBENZARIT DISODIUM. Héctor Novoa de Armas*, Rolando Pellón Cóndom, Center of Pharmaceutical Chemistry, P. O. Box 16042, Havana, Cuba, Ramón Pomés Hernández and Julio Duque Rodríguez, National Center for Scientific Research. P. O. Box 6990, Havana, Cuba

Lobenzarit acid, C₃H₃O₂CINN₄, and lobenzarit disodium (CCA), C₄H₄CINNN₂O₄, have been investigated by means of X-ray powder diffraction. The 4-chloro-2,2' iminodibenzoic acid (lobenzarit acid) is an intermediary compound in the CCA synthesis, which is a powerful anti-rheumatic drug. The unit cell dimensions were determined from diffractometer methods, using strictly monochromatized Cu Kα1 radiation, and evaluated by indexing programs. The source of initial cells was the indexing program DICVOL91. The triclinic cell found for lobenzarit acid was a = 16.580(1)Å, b = 16.389(1)Å, c = 4.9023(3)Å, α = 71.674(6)°, β = 92.609(6)°, γ = 97.127(6)°, Z = 4, Dx = 1.544 Mg/m³. The triclinic cell found for CCA was a = 18.041(5)Å, b = 11.461(4)Å, c = 5.936(2)Å, α = 66.80(3)°, β = 103.61(3)°, γ = 113.13(3)°, Z = 4, Dx = 2.159 Mg/m³. The final results were checked using the program NBS*AIDS83.

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From a microphotograph of the superimposed layer, it is apparent that the mixture of both oxides is uniformly distributed over the surface area.

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