

**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 Gauss 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, part of which may be fixed in Rietveld refinement.

Further, we developed a Rietveld-analysis program for VEGA, RIETAN-96T, that includes the new profile function, transporting many new features from that for angle-dispersive X-ray and neutron diffraction. Results of some representative structure refinements will be presented.

Cole, I. & Windsor, C. G. (1980). *Nucl. Instrum. Methods*, 171, 107-113.  
Izumi, F. *et al.* (1987). *J. Appl. Crystallogr.* 20, 411-418.  
Kamiyama, T. *et al.* (1995). *Physica B*, 213&214, 875-877.

**PS10.08.13 INVESTIGATION OF STRUCTURE AND COMPOSITION OF  $\text{IrO}_2$  -  $\text{Ta}_2\text{O}_5$  SURFACE LAYERS.** J. Krysa<sup>1</sup>, J. Maixner<sup>2</sup>, <sup>1</sup>Department of Inorganic Technology, <sup>2</sup>Central Laboratories, Institute of Chemical Technology, Technicka 5, 166 28 Prague 6, Czech Republic

$\text{IrO}_2$  -  $\text{Ta}_2\text{O}_5$  coating is so far the best electrocatalyst for an oxygen evolution (1). Therefore, the structure and composition of the  $\text{IrO}_2$  -  $\text{Ta}_2\text{O}_5$  thermally deposited surface layers on Ti substrate were investigated by X-ray diffraction and electron microscopy.

X-ray diffraction revealed the crystalline form of  $\text{IrO}_2$  and the amorphous form of  $\text{Ta}_2\text{O}_5$  in the coating. The average coating thickness was calculated from the decrease of Ti substrate intensities using 2 methods and it is proportional to the deposited iridium content. The X-ray intensities of  $\text{IrO}_2$  coating show the same dependence.

From analysis of microphotographs of  $\text{IrO}_2$  -  $\text{Ta}_2\text{O}_5$  surface layer it is apparent that the mixture of both oxides is uniformly distributed over the surface area.

(1) R. Mráz and J. Krysa, *J. Appl. Electrochem.*, 24, 1262 (1994)  
(2) R. Kuzel Jr.: Difpatan, Program for X-ray analysis, Charles University, Czech Republic

**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.

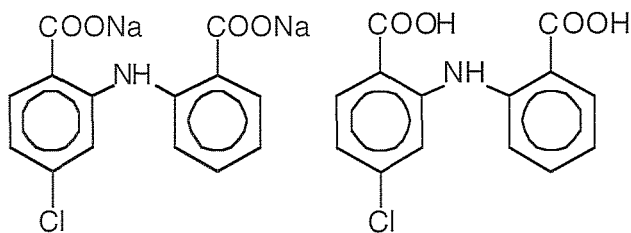
The phase systems analysed in this work include (i) igneous, metamorphic and volcanic rocks, (ii) sedimentary rocks, and (iii) massive sulphide ores. The Rietveld results are compared with values obtained by (i) optical modes for the medium to coarse grained samples obtained by point counting, and (ii) 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.

The presence of severe preferred orientation in some phases required the collection of X-ray data using Debye-Scherrer geometry with samples packed in 0.5mm capillaries. While preferred orientation was virtually eliminated by this method, care had to be taken to ensure that the sample examined (<2mg) adequately represented the bulk material.

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 Córdomb, 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,  $\text{C}_{14}\text{H}_{10}\text{ClNO}_4$ , and lobenzarit disodium (CCA),  $\text{C}_{14}\text{H}_8\text{ClNNa}_2\text{O}_4$ , 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  $\text{Cu K}\alpha 1$  radiation, and evaluated by indexing programs. The source of initial cells was the indexing program DICVOL91. The triclinic cell found for lobenzarit acid was  $a = 16.580(1)\text{\AA}$ ,  $b = 16.389(1)\text{\AA}$ ,  $c = 4.9023(3)\text{\AA}$ ,  $\alpha = 71.674(6)^\circ$ ,  $\beta = 92.609(6)^\circ$ ,  $\gamma = 97.127(6)^\circ$ ,  $Z = 4$ ,  $D_x = 1.544 \text{ Mg/m}^3$ . The triclinic cell found for CCA was  $a = 18.041(5)\text{\AA}$ ,  $b = 11.461(4)\text{\AA}$ ,  $c = 5.936(2)\text{\AA}$ ,  $\alpha = 66.80(3)^\circ$ ,  $\beta = 103.61(3)^\circ$ ,  $\gamma = 113.13(3)^\circ$ ,  $Z = 4$ ,  $D_x = 2.159 \text{ Mg/m}^3$ . The final results were checked using the program NBS\*AIDS83.



Lobenzarit disodium

Lobenzarit acid