

Extreme Performances of the Rietveld Method in the Structural Characterization of New Minerals

SOKOLOVA, E.V. and KABALOV, Yu.K. Faculty of Geology, Moscow State University, Moscow, Russia.

The Rietveld method helps to find a structure solution when single crystal technique does not work. The last two decades have seen great advances in our ability to extract detailed crystal structure information from powder diffraction data, e.g., to do systematic structure refinement. We present a detailed overview of Rietveld structure refinement results from powder X-ray data for new minerals characterized by a very poor crystallinity, heavy twinning or/and multiphase impurities.

Our own experience is based on the studies of crystal structures of five new minerals with a complicated chemistry: turkestanite $\text{Th}(\text{Ca}, \text{Na})_2(\text{K}_{1-z})[\text{Si}_8\text{O}_{20}]$, $z=0.47$, belovite-(La) $\text{Sr}_3\text{Na}(\text{La}, \text{Ce})[\text{PO}_4]_3(\text{F}, \text{OH})$, zlatogorite CuNiSb_2 , kapitsaite $(\text{Ba}, \text{K}, \text{Pb})_4(\text{Y}, \text{Ca})_2\text{Si}_8(\text{B}, \text{Si})_4\text{O}_{28}\text{F}$, potassicferrisadanagaite $(\text{Ca}, \text{Na}, \text{Mn})_2(\text{Mg}, \text{Fe}^{2+}, \text{Mn}, \text{Fe}^{3+}, \text{Al}, \text{Ti})_5(\text{Si}_{5.33}\text{Al}_{2.67})\text{O}_{22}[(\text{OH}, \text{F}, \text{O})]_2$.

For triclinic kapitsaite (V_0 1243.6(9) \AA^3 ; cations range from Pb, $Z=82$ to B, $Z=5$; number of refined parameters increases to 177) final R_{wp} 0.03 and R_F 0.02, i.e. comparable with single crystal R -values. In this case final refinement results should be better but interatomic distances are less reliable than those derived from single crystal data. However, site occupancies are in a good accordance.

The observed powder pattern of turkestanite contains only 3 visible reflections that do not fit the calculated pattern: impurity of thorite $(\text{Th}, \text{U})\text{SiO}_4$ of 1.34 wt % has been determined by a multiphase Rietveld refinement, R_{wp} 0.04.