Rietveld refinement plots

In this file all the Rietveld plots obtained after the last refinement cycle are reported. The red dotted and the black continuous line are the experimental and the calculated diffractogram, respectively; the lower blue line is the difference curve; the vertical green bars indicate the position of the expected peaks for each phase.

According to the %Fe content with respect to the total (Fe + Ni) amount, and to the analysis temperature (in K), samples were named Fe40_303, Fe55_473, and so on.

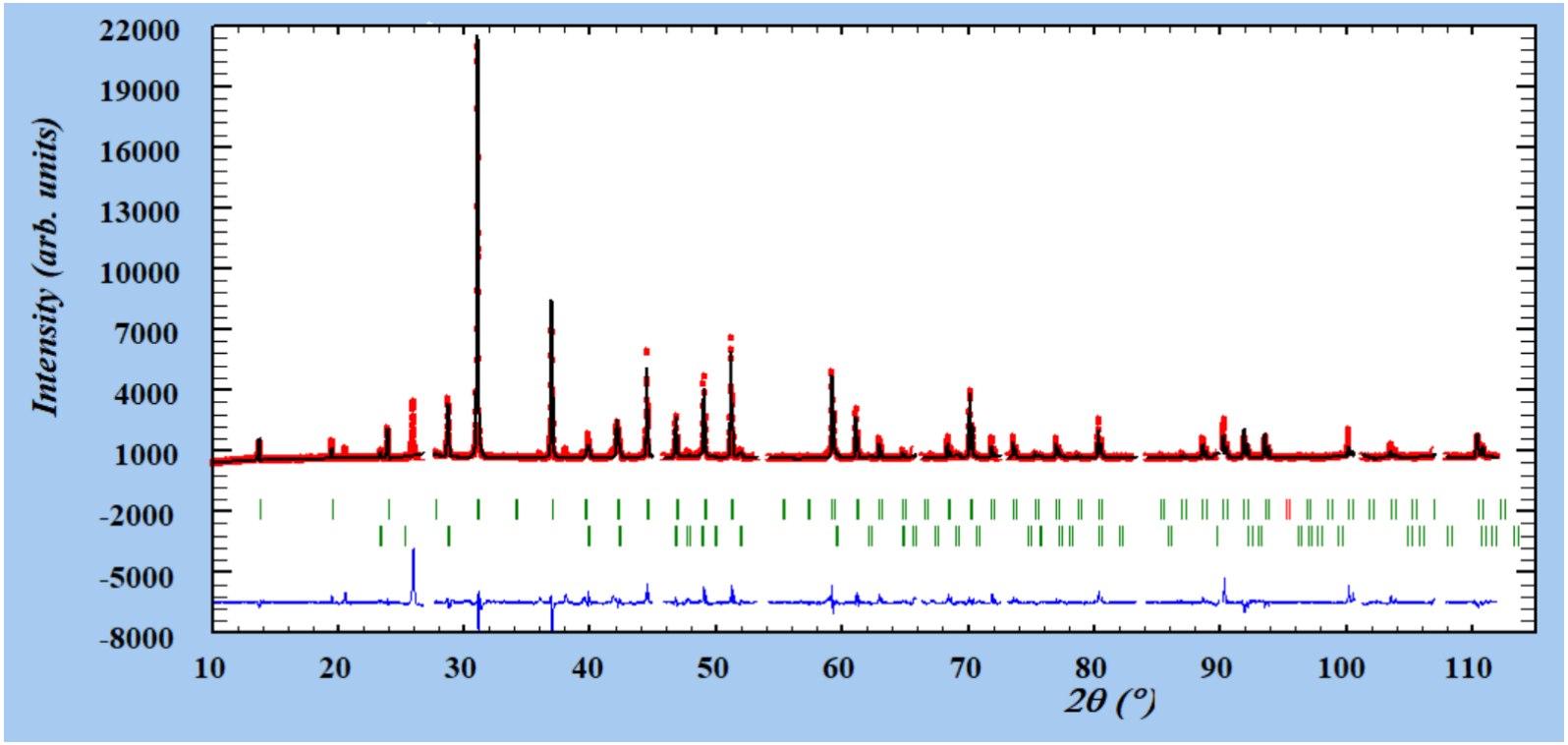

Figure S1 – Rietveld refinement plot for sample Sm40_303. Upper vertical bars: skutterudite; lower vertical bars: $Sb_{0.95}Sn_{0.05}$. Excluded regions are placed in correspondence of Bragg peaks of the internal standard Ge.

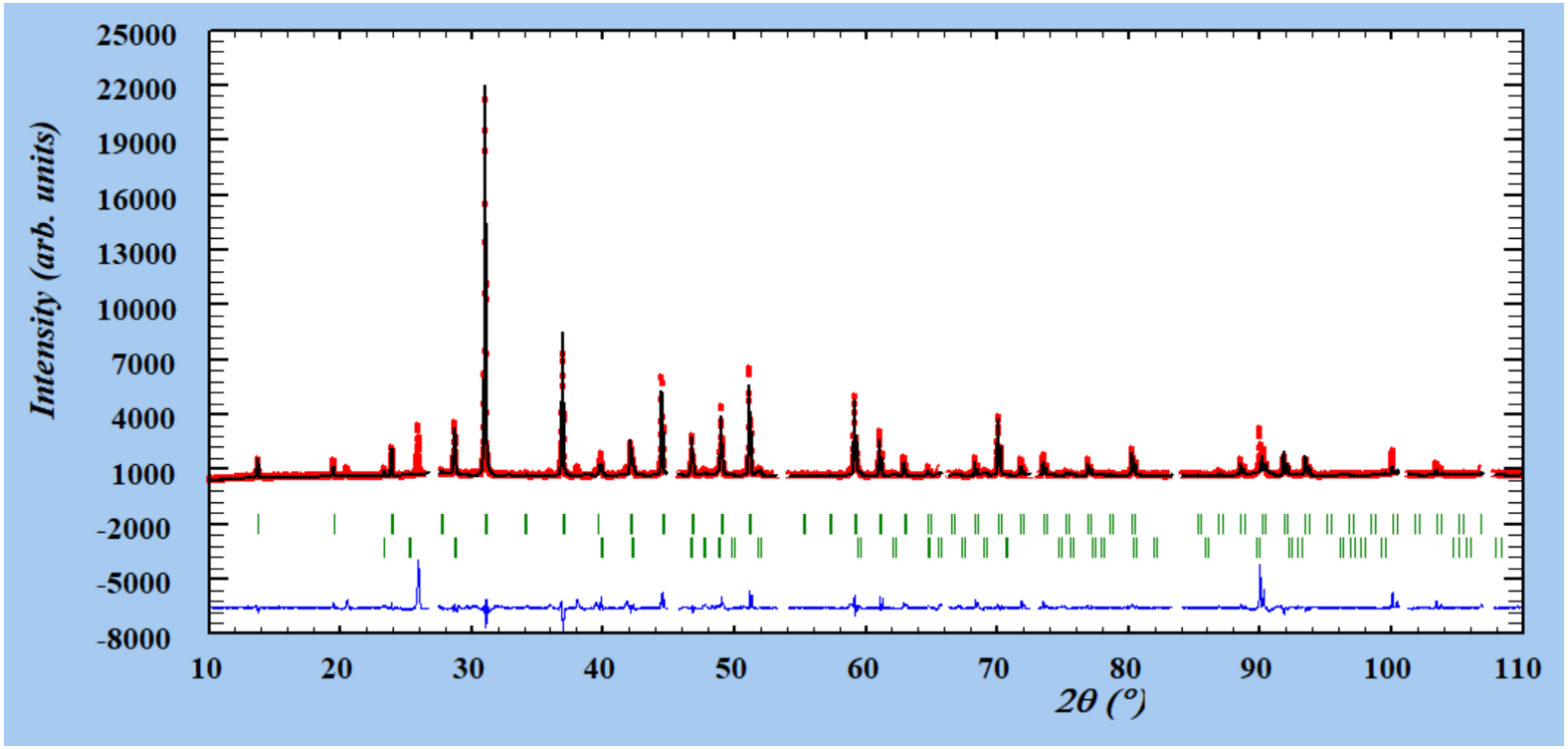

Figure S2 – Rietveld refinement plot for sample Sm40_373. Upper vertical bars: skutterudite; lower vertical bars: $Sb_{0.95}Sn_{0.05}$. Excluded regions are placed in correspondence of Bragg peaks of the internal standard Ge.

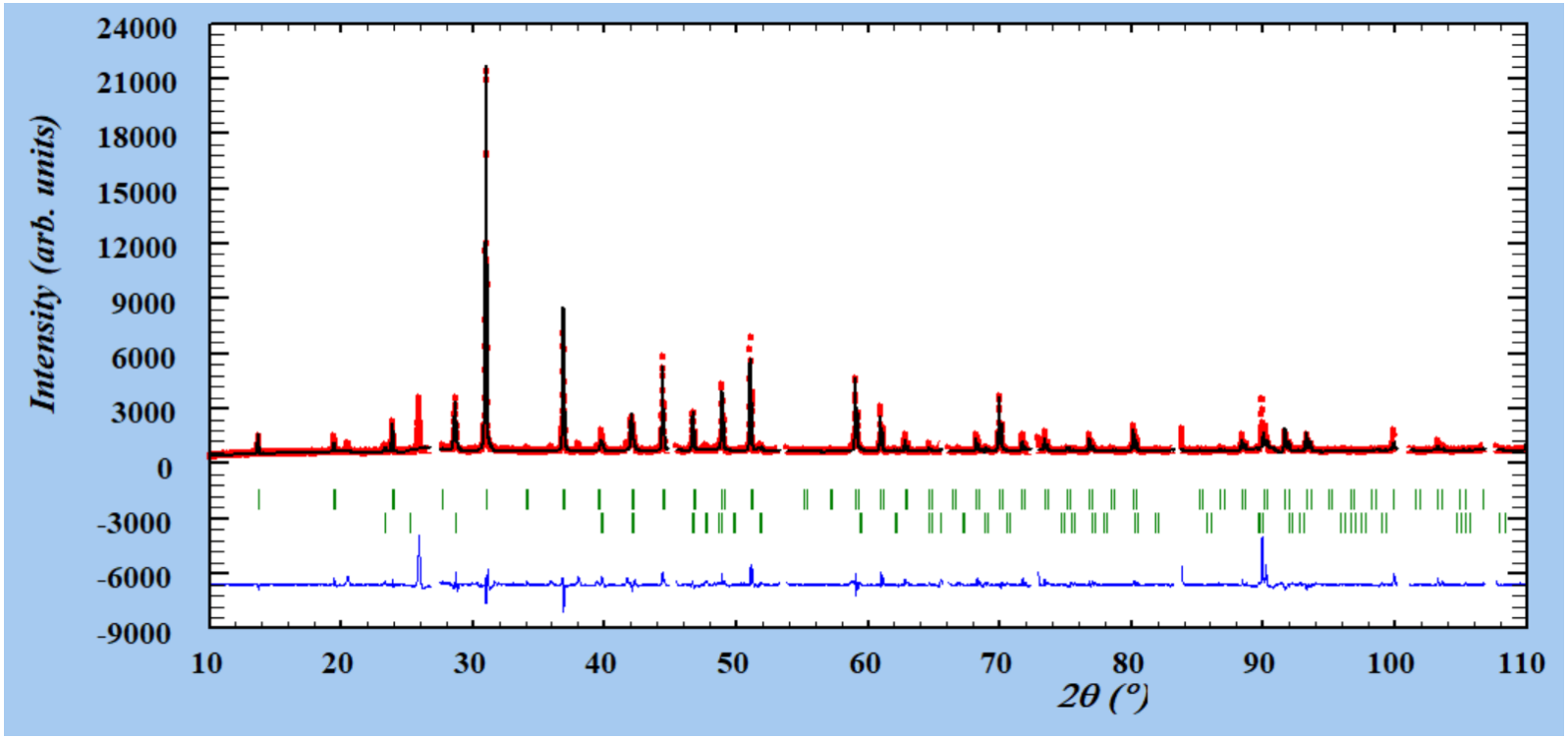

Figure S3 – Rietveld refinement plot for sample Sm40_473. Upper vertical bars: skutterudite; lower vertical bars: $Sb_{0.95}Sn_{0.05}$. Excluded regions are placed in correspondence of Bragg peaks of the internal standard Ge.

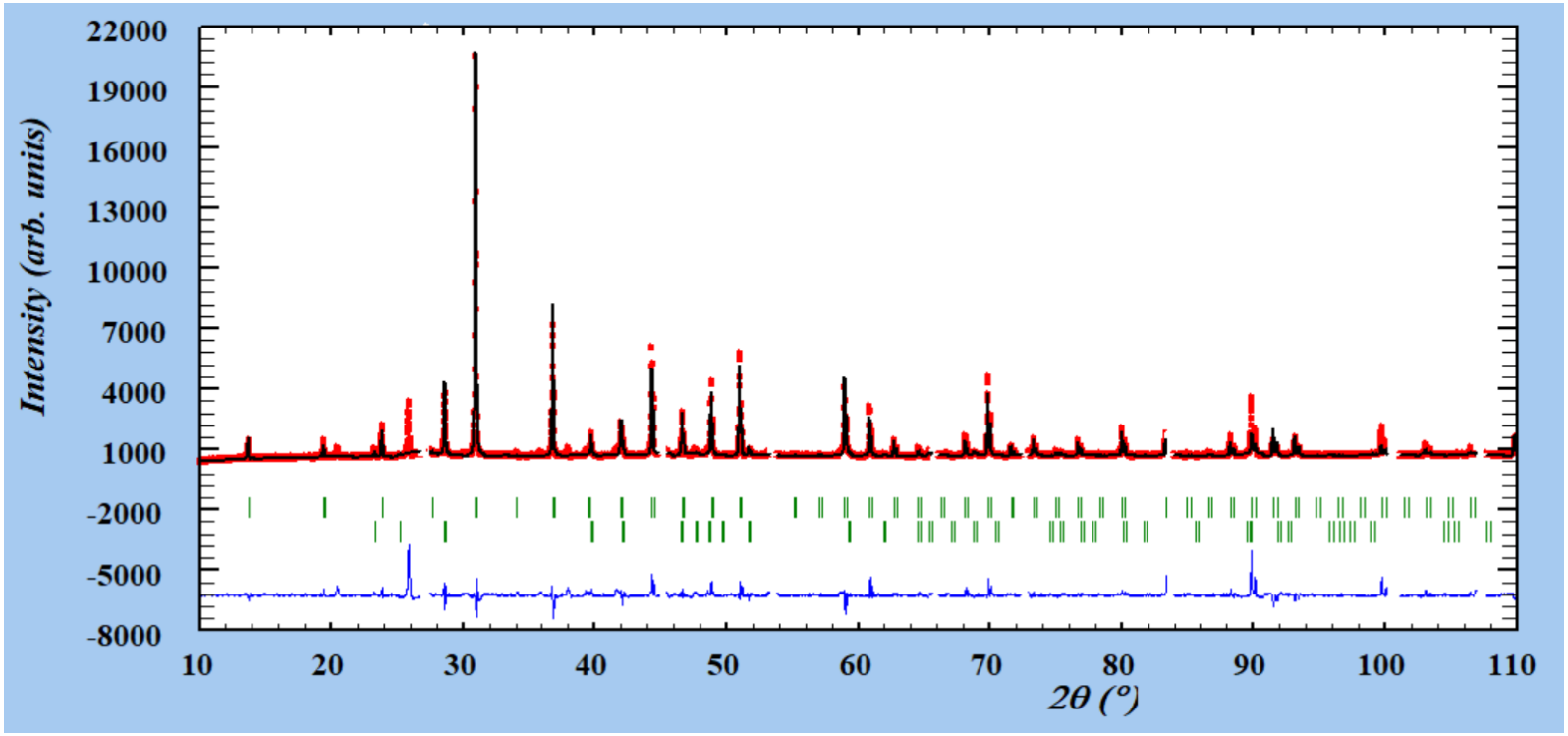

Figure S4 – Rietveld refinement plot for sample Sm40_573. Upper vertical bars: skutterudite; lower vertical bars: $Sb_{0.95}Sn_{0.05}$. Excluded regions are placed in correspondence of Bragg peaks of the internal standard Ge.

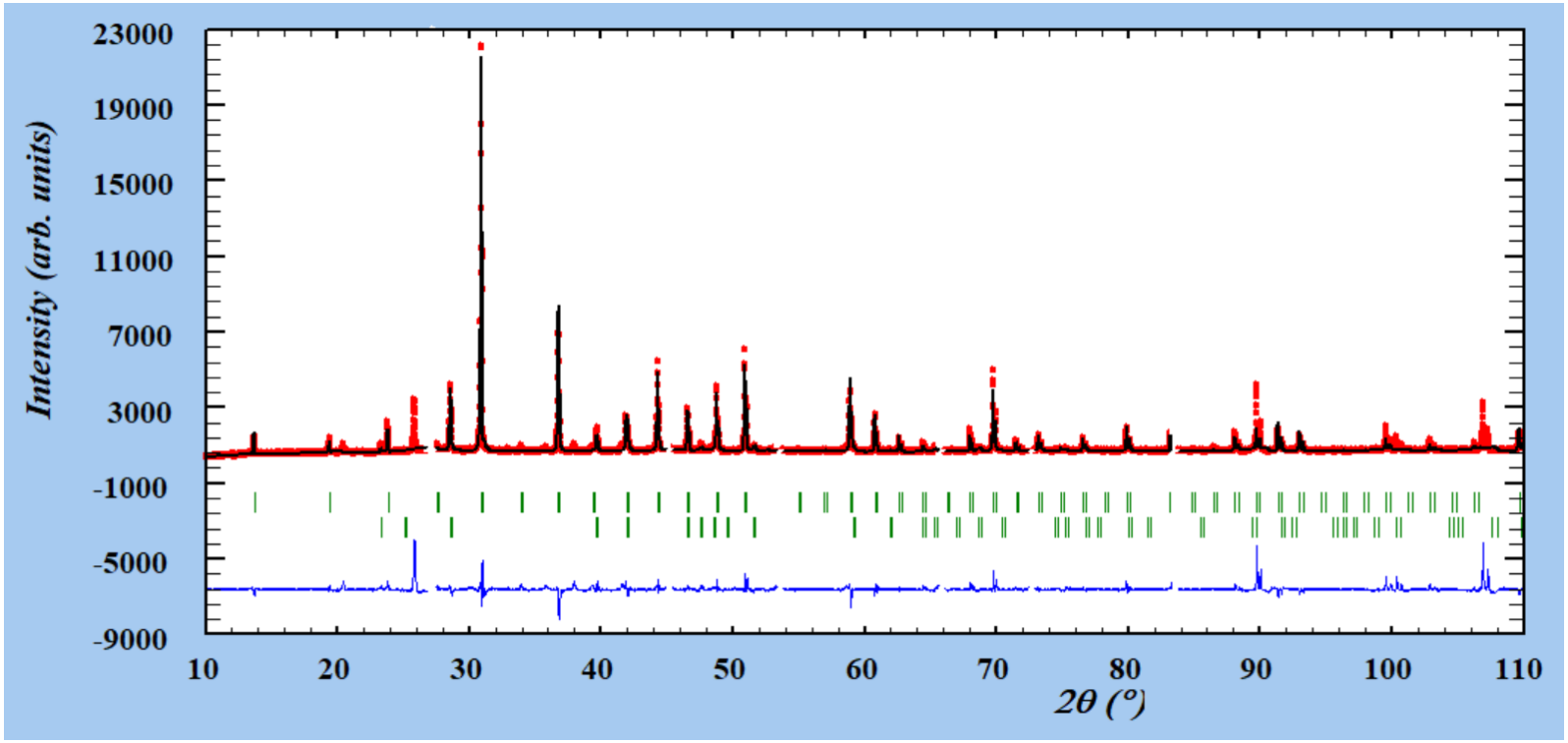

Figure S5 – Rietveld refinement plot for sample Sm40_673. Upper vertical bars: skutterudite; lower vertical bars: $Sb_{0.95}Sn_{0.05}$. Excluded regions are placed in correspondence of Bragg peaks of the internal standard Ge.

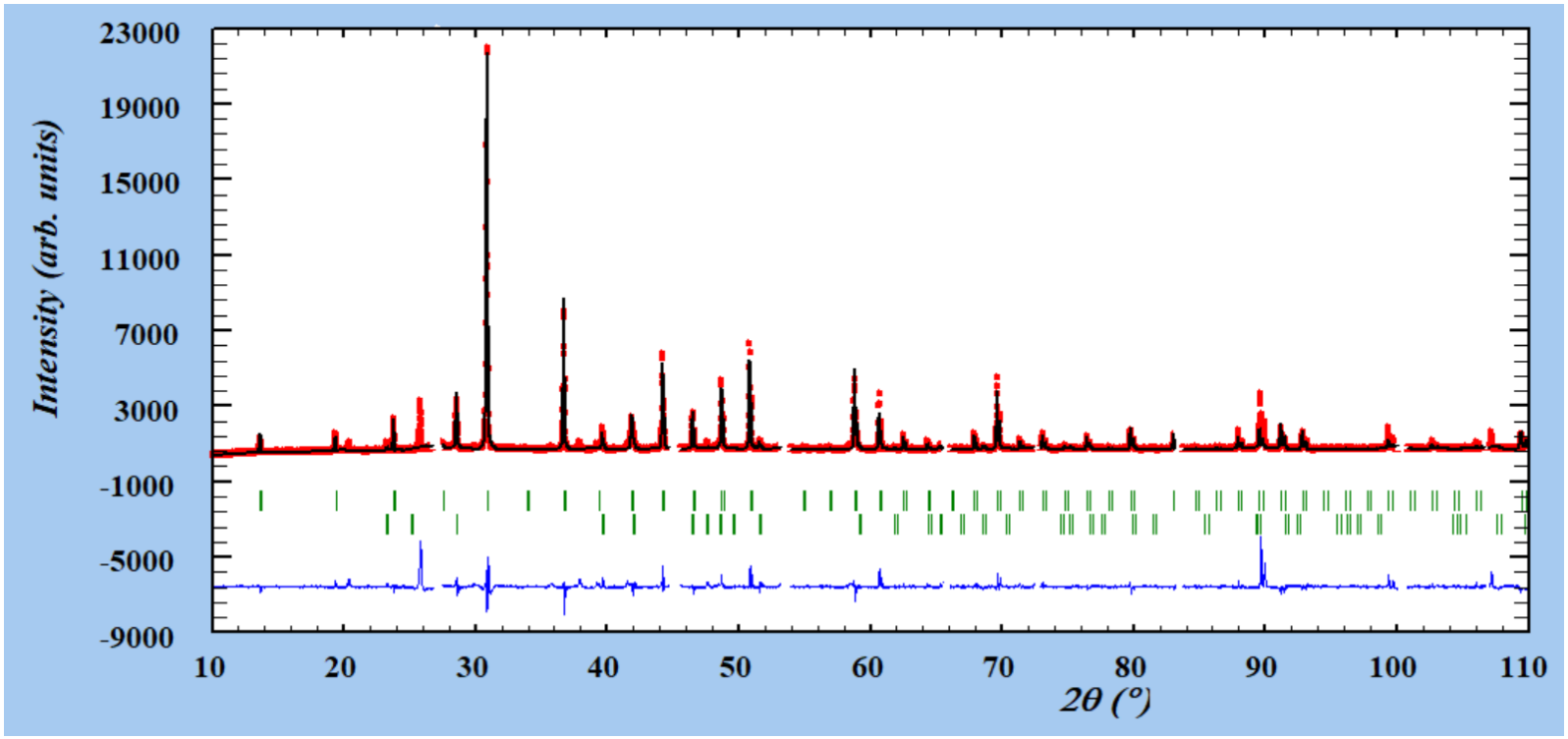

Figure S6 – Rietveld refinement plot for sample Sm40_773. Upper vertical bars: skutterudite; lower vertical bars: $Sb_{0.95}Sn_{0.05}$. Excluded regions are placed in correspondence of Bragg peaks of the internal standard Ge.

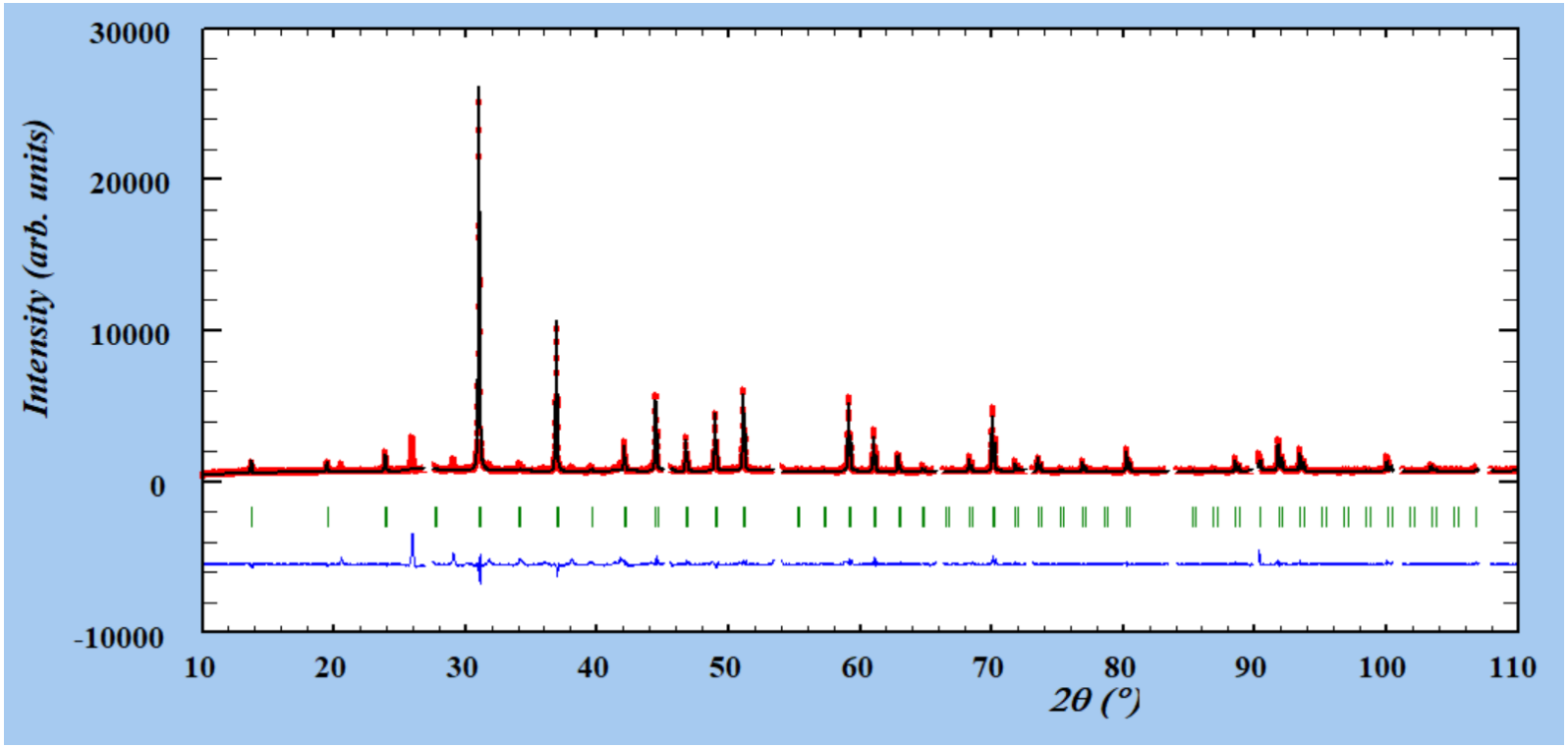

Figure S7 – Rietveld refinement plot for sample Sm55_303. Vertical bars: skutterudite. Excluded regions are placed in correspondence of Bragg peaks of the internal standard Ge.

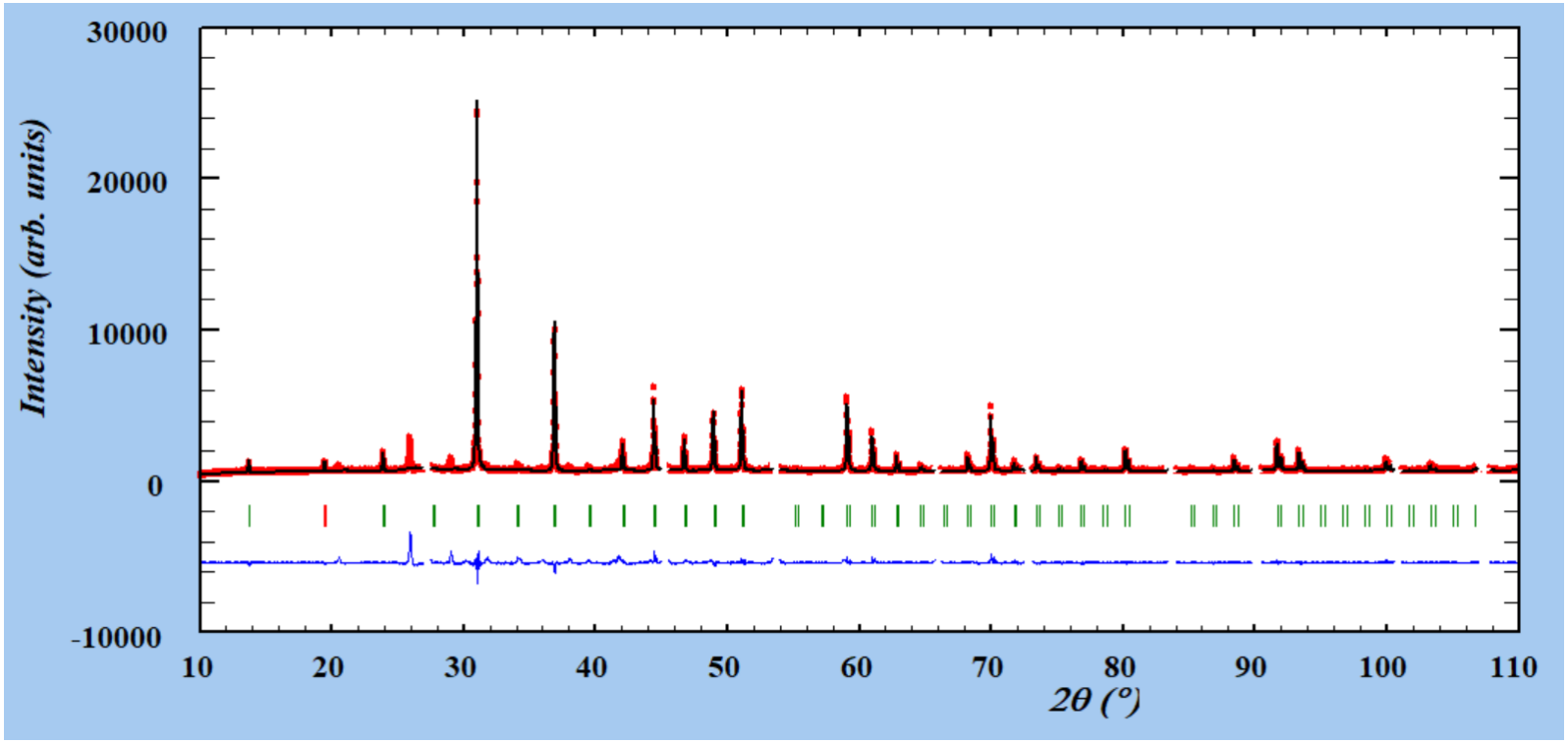

Figure S8 – Rietveld refinement plot for sample Sm55_373. Vertical bars: skutterudite. Excluded regions are placed in correspondence of Bragg peaks of the internal standard Ge.

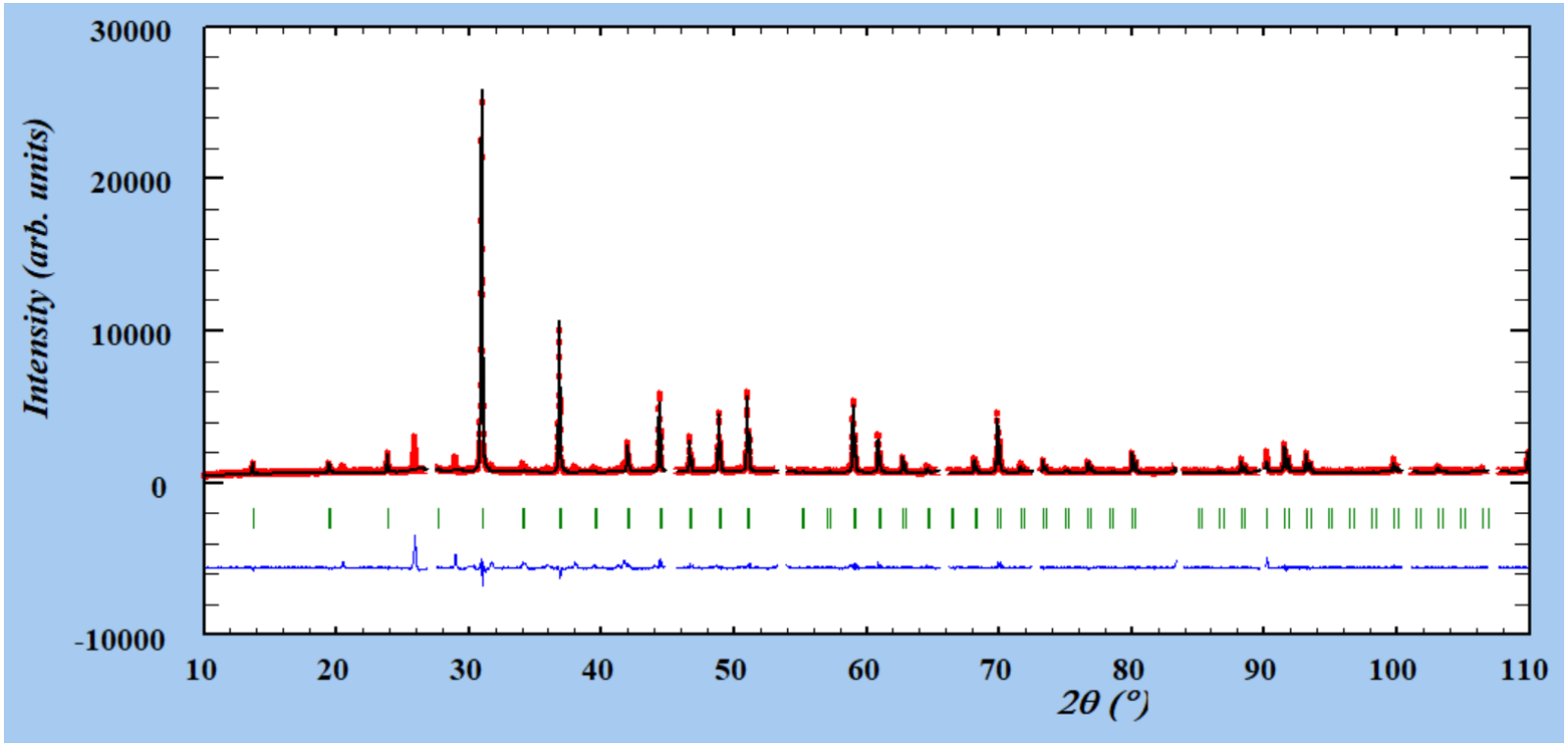

Figure S9 – Rietveld refinement plot for sample Sm55_473. Vertical bars: skutterudite. Excluded regions are placed in correspondence of Bragg peaks of the internal standard Ge.

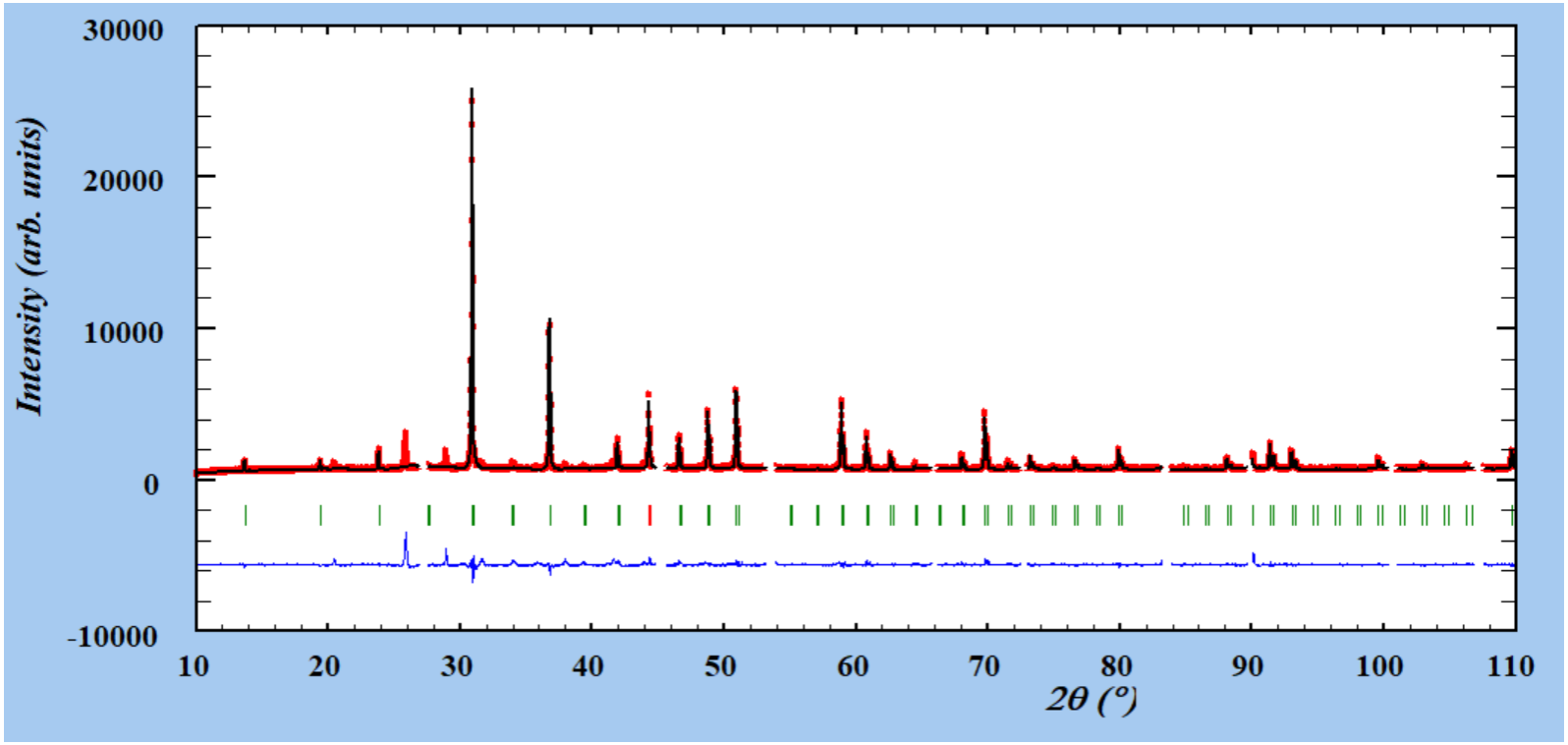

Figure S10 – Rietveld refinement plot for sample Sm55_573. Vertical bars: skutterudite. Excluded regions are placed in correspondence of Bragg peaks of the internal standard Ge.

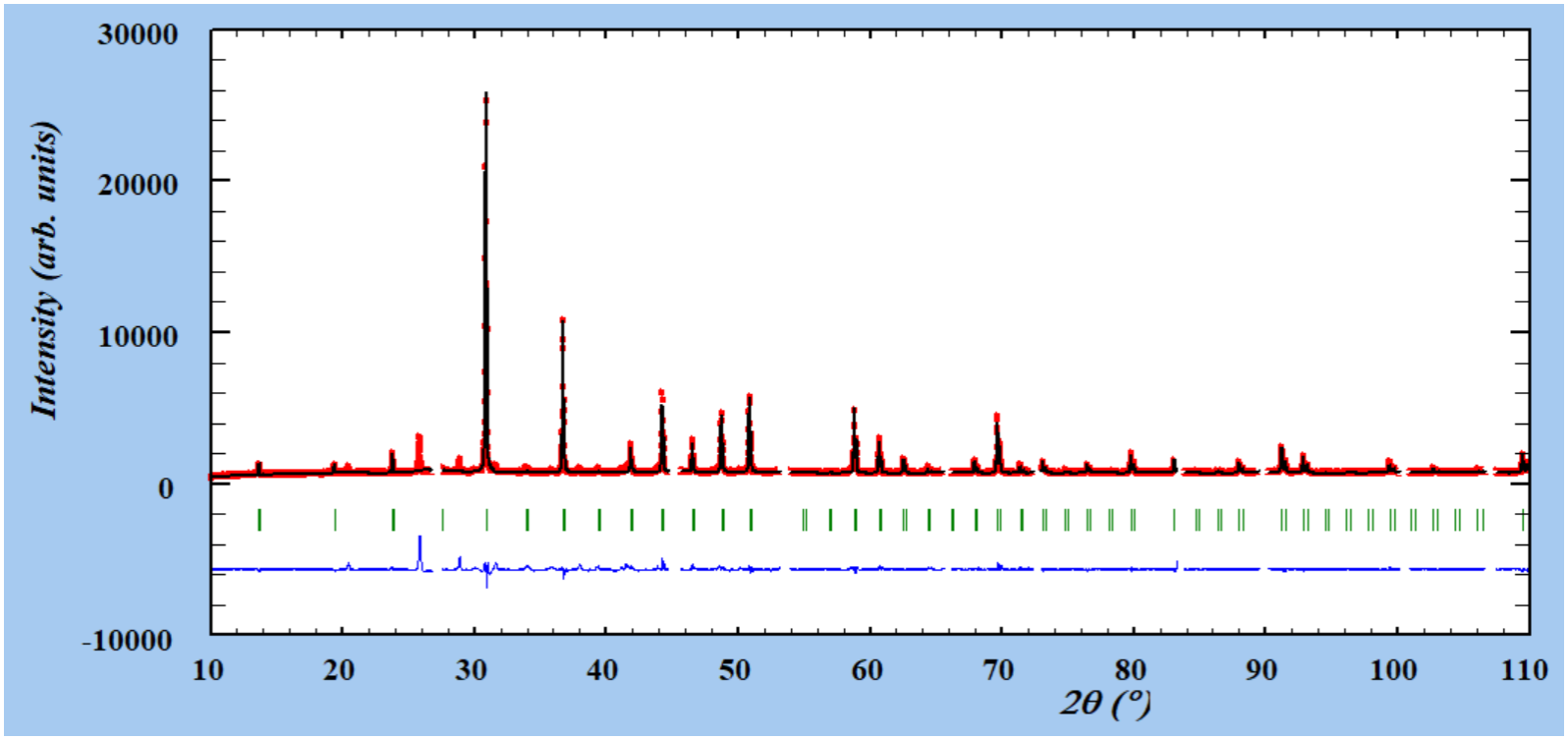

Figure S11 – Rietveld refinement plot for sample Sm55_673. Vertical bars: skutterudite. Excluded regions are placed in correspondence of Bragg peaks of the internal standard Ge.

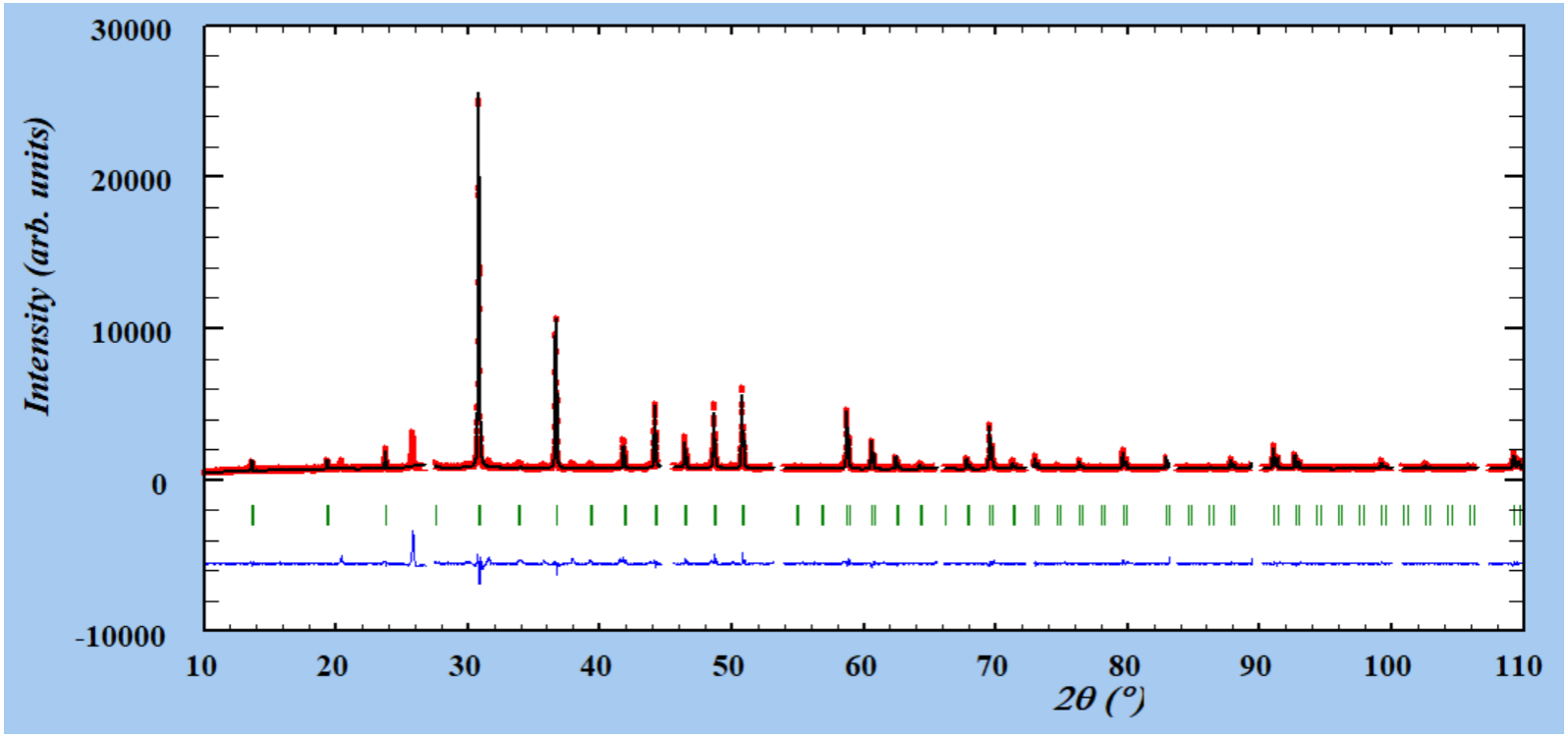

Figure S12 – Rietveld refinement plot for sample Sm55_773. Vertical bars: skutterudite. Excluded regions are placed in correspondence of Bragg peaks of the internal standard Ge.

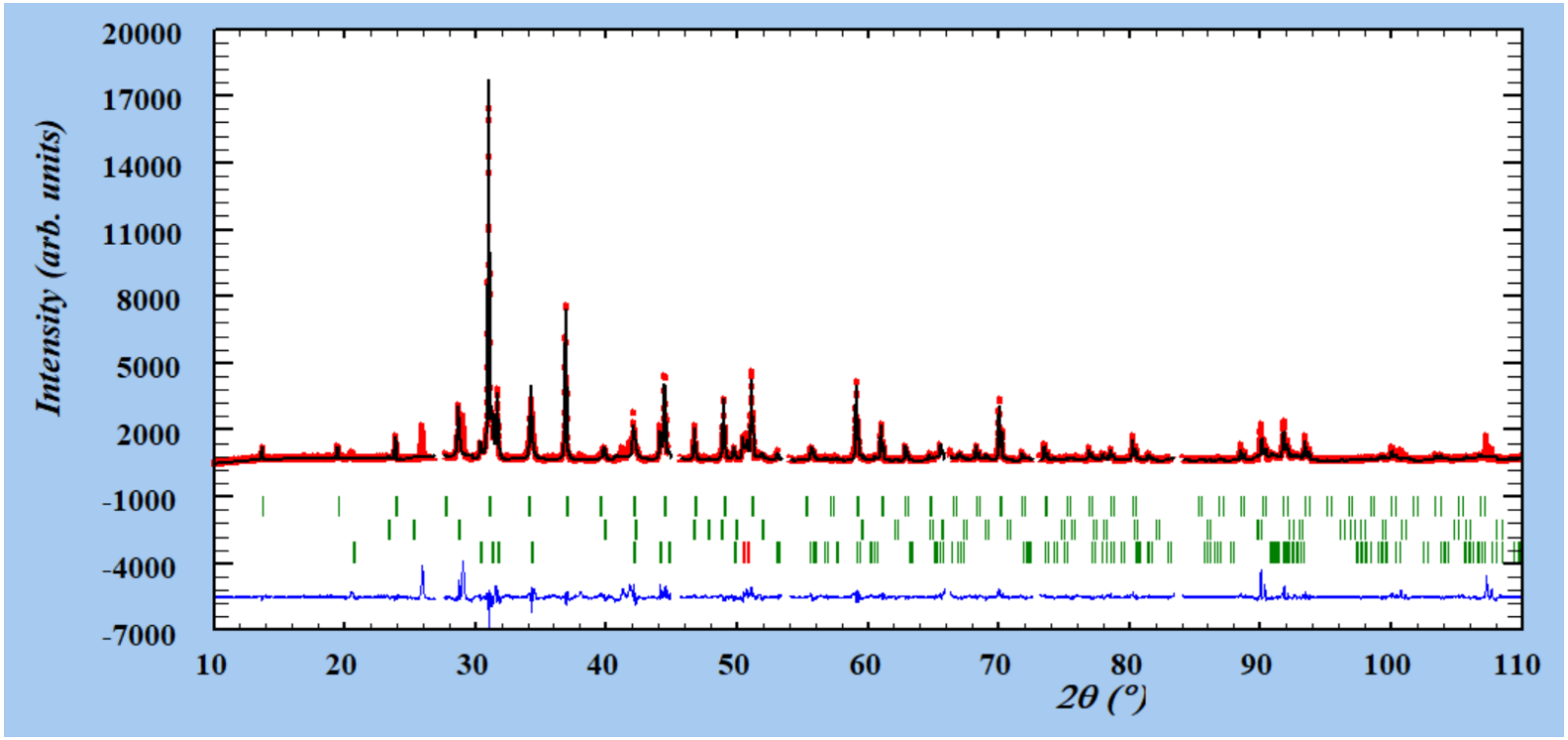

Figure S13 – Rietveld refinement plot for sample Sm60_303. Upper vertical bars: skutterudite; intermediate vertical bars: $Sb_{0.95}Sn_{0.05}$; lower vertical bars: $Fe_{0.66}Ni_{0.33}Sb_{2.}$ Excluded regions are placed in correspondence of Bragg peaks of the internal standard Ge.

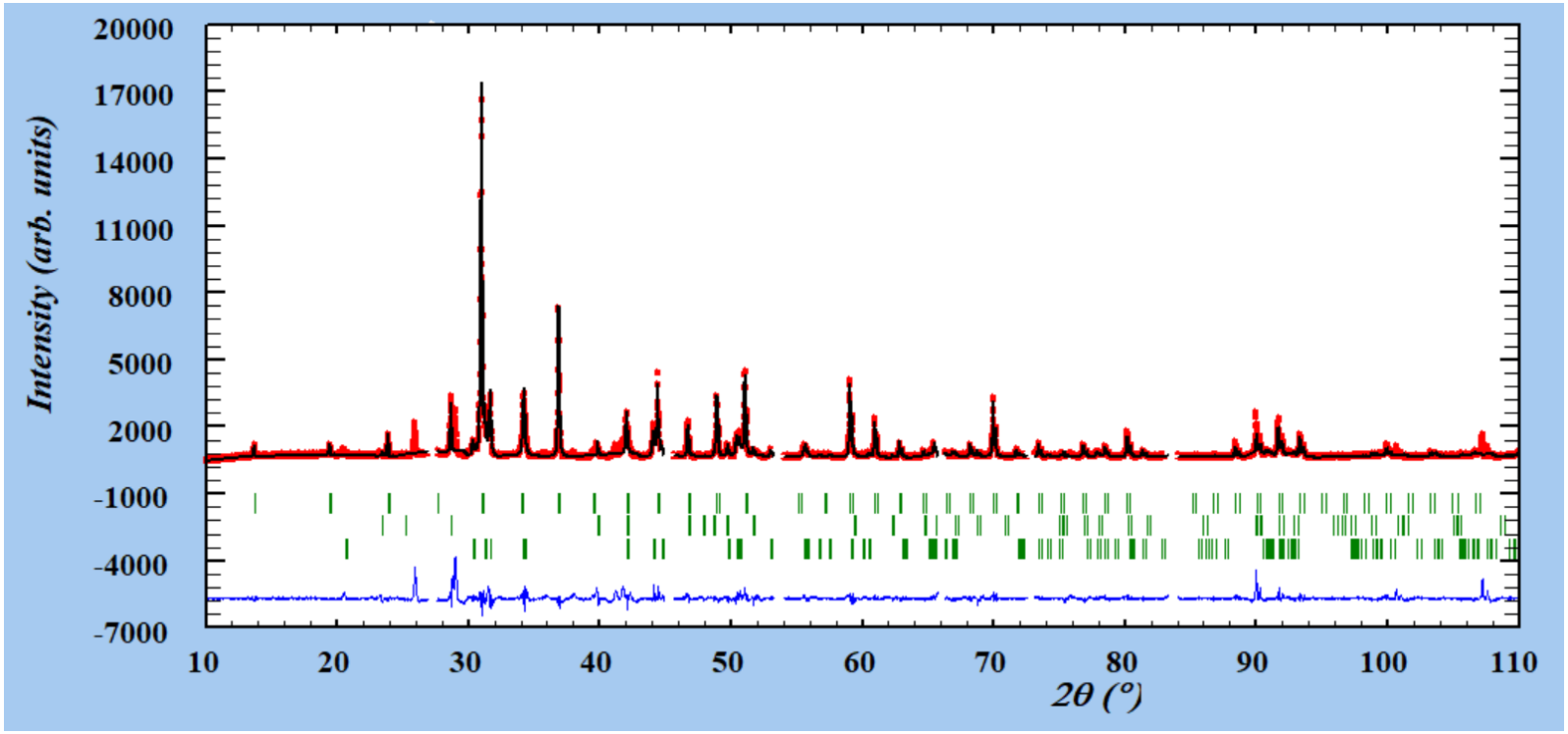

Figure S14 – Rietveld refinement plot for sample Sm60_373. Upper vertical bars: skutterudite; intermediate vertical bars: $Sb_{0.95}Sn_{0.05}$; lower vertical bars: $Fe_{0.66}Ni_{0.33}Sb_2$. Excluded regions are placed in correspondence of Bragg peaks of the internal standard Ge.

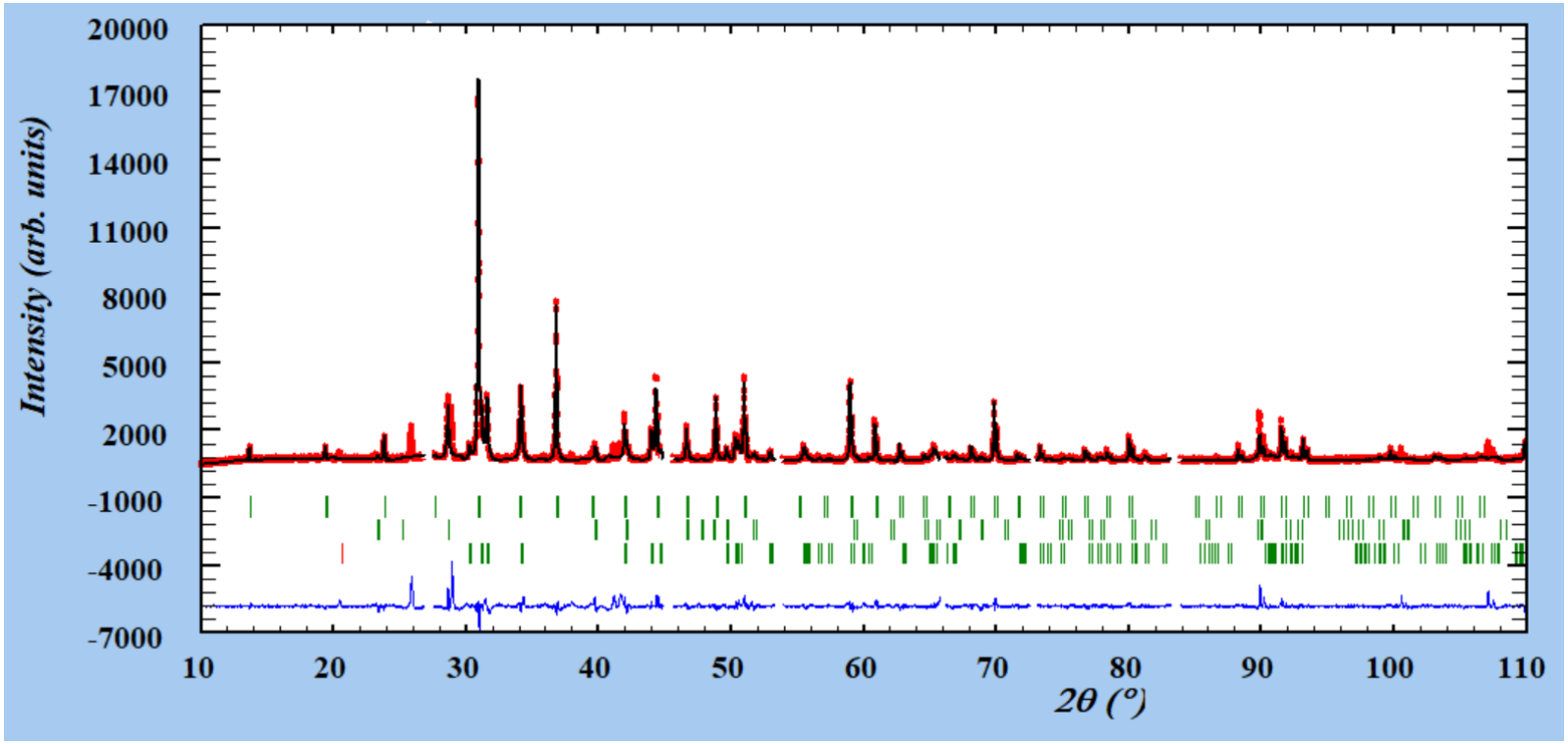

Figure S15 – Rietveld refinement plot for sample Sm60_473. Upper vertical bars: skutterudite; intermediate vertical bars: $Sb_{0.95}Sn_{0.05}$; lower vertical bars: $Fe_{0.66}Ni_{0.33}Sb_2$. Excluded regions are placed in correspondence of Bragg peaks of the internal standard Ge.

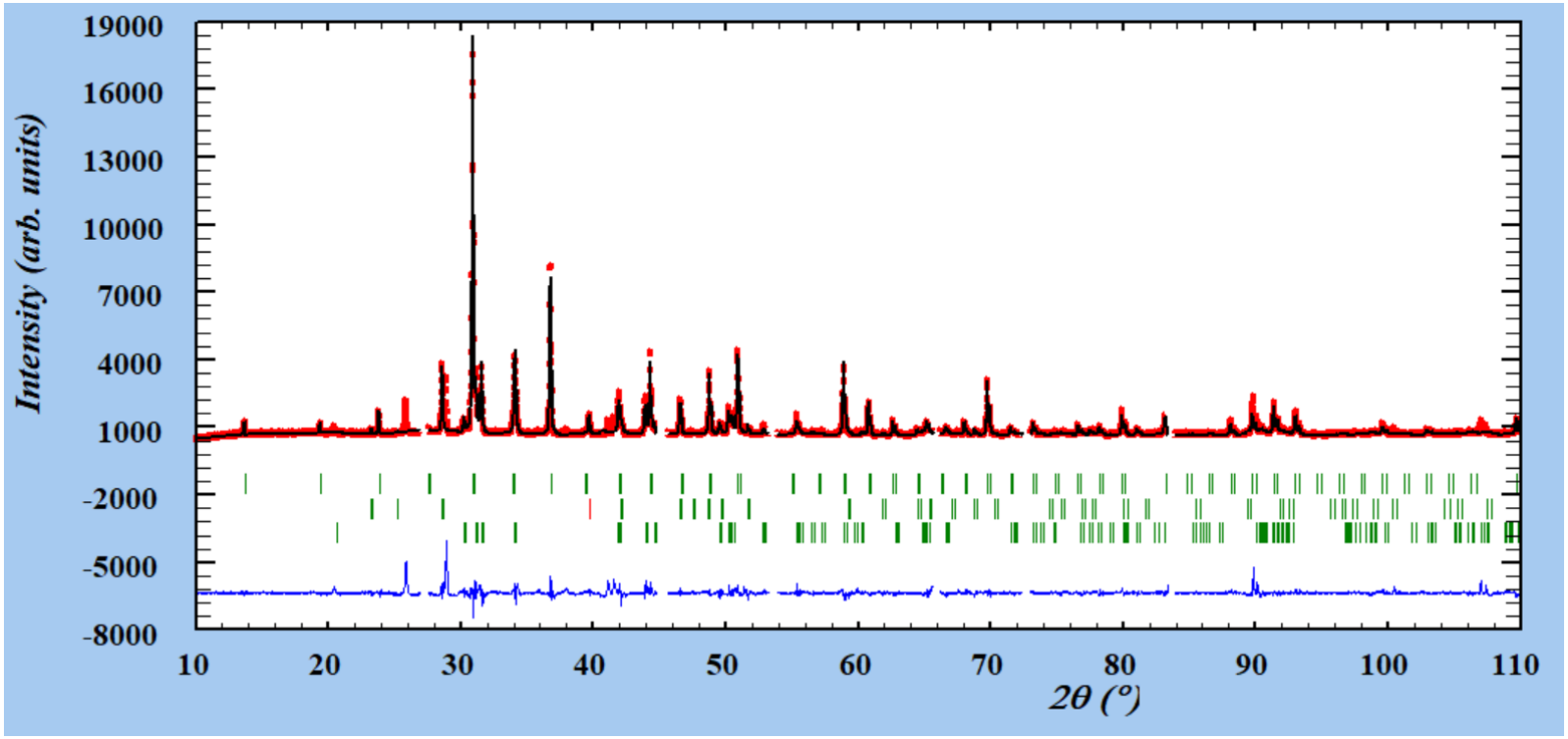

Figure S16 – Rietveld refinement plot for sample Sm60_573. Upper vertical bars: skutterudite; intermediate vertical bars: $Sb_{0.95}Sn_{0.05}$; lower vertical bars: $Fe_{0.66}Ni_{0.33}Sb_2$. Excluded regions are placed in correspondence of Bragg peaks of the internal standard Ge.

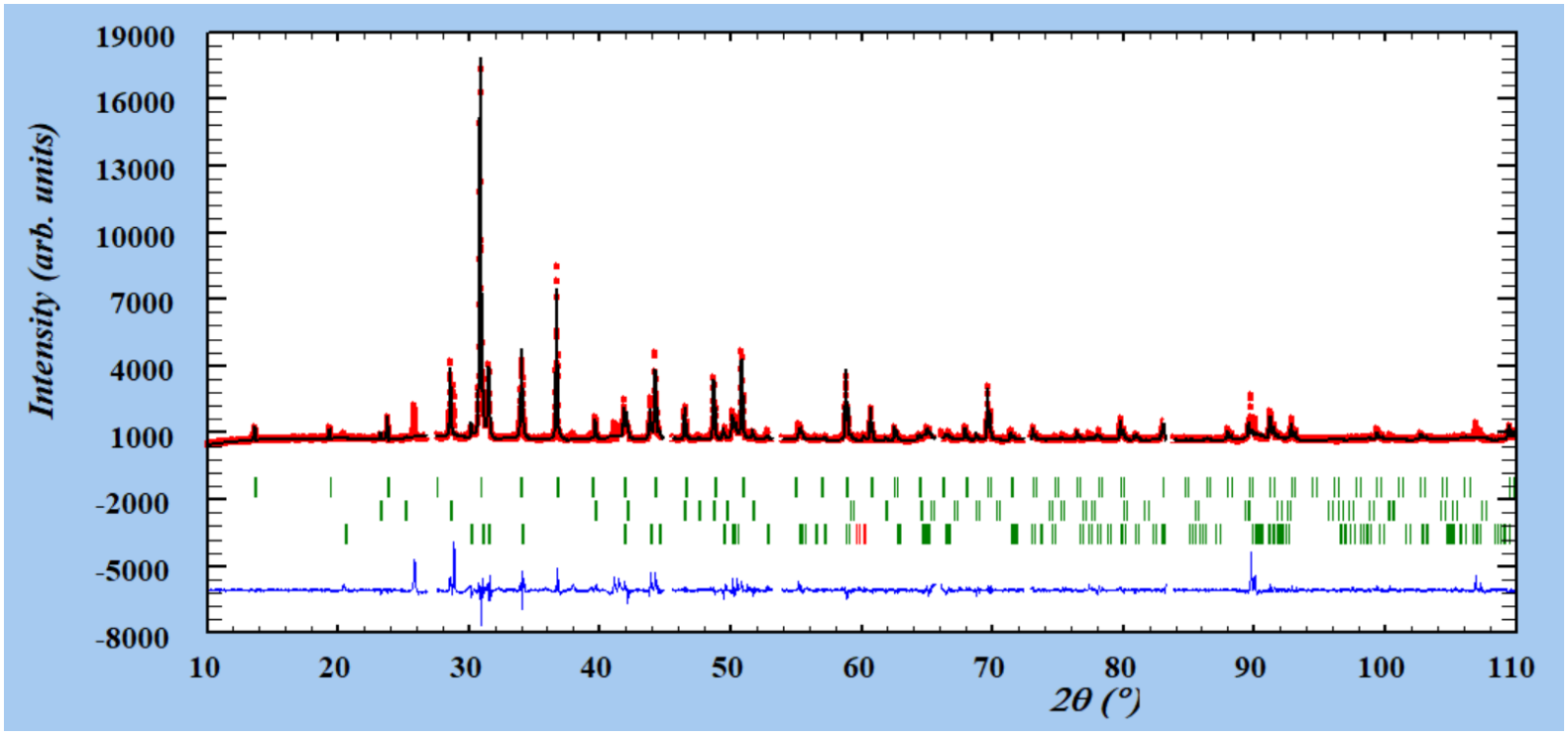

Figure S17 – Rietveld refinement plot for sample Sm60_673. Upper vertical bars: skutterudite; intermediate vertical bars: $Sb_{0.95}Sn_{0.05}$; lower vertical bars: $Fe_{0.66}Ni_{0.33}Sb_2$. Excluded regions are placed in correspondence of Bragg peaks of the internal standard Ge.

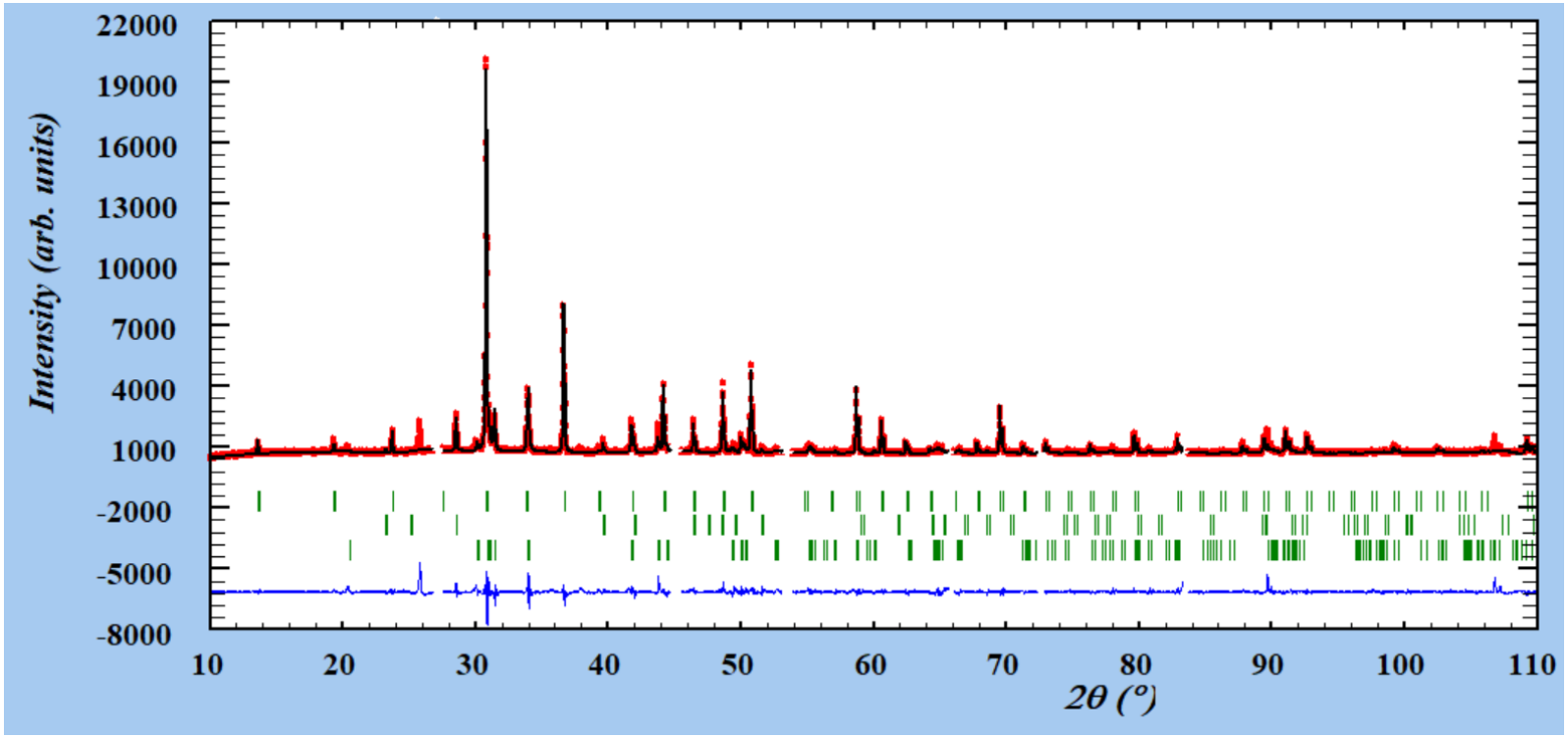

Figure S18 – Rietveld refinement plot for sample Sm60_773. Upper vertical bars: skutterudite; intermediate vertical bars: $Sb_{0.95}Sn_{0.05}$; lower vertical bars: $Fe_{0.66}Ni_{0.33}Sb_2$. Excluded regions are placed in correspondence of Bragg peaks of the internal standard Ge.