QUANTITATIVE PHASE ANALYSIS OF ONE POWDER SAMPLE USING THE RIETVELD METHOD AND X-RAY POWDER DIFFRACTION DATA.

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EXPERIMENTAL METHODS

The sample "73397 Idaho gold and silver sample" was reduced to the optimum grain-size range for quantitative X-ray analysis (<5 μm) by grinding under ethanol in a vibratory McCrone Micronising Mill for 7 minutes. Fine grain-size is an important factor in reducing micro-absorption contrast between phases.

Step-scan X-ray powder-diffraction data were collected over a range 3-80°2θ with CoKα radiation on a standard Siemens (Bruker) D5000 Bragg-Brentano diffractometer equipped with an Fe monochromator foil, 0.6 mm (0.3°) divergence slit, incident- and diffracted-beam Soller slits and a Vantec-1 strip detector. The long fine-focus Co X-ray tube was operated at 35 kV and 40 mA, using a take-off angle of 6°.

RESULTS

The X-ray diffractogram was analyzed using the International Centre for Diffraction Database PDF-4 using Search-Match software by Siemens (Bruker). X-ray powder-diffraction data were refined with Rietveld program Topas 3 (Bruker AXS). The results of quantitative phase analysis by Rietveld refinements are given in Table 1. These amounts represent the relative amounts of crystalline phases normalized to 100%. The Rietveld refinement plot is shown in Figure 1.

The pattern shows a hump between about 6 and 8º2θ that likely corresponds to minor amounts of either amourphous or nanoscale material (disordered clays?).

Table 1. Results of quantitative phase analysis (wt. %) – Met-Solve Lab. Inc.

Mineral	**Ideal formula**	**73397 Idaho gold & silver**
Quartz	SiO_2	51.0
Clinochlore	$(Mg,Fe^{2+})_5Al(Si_3Al)O_{10}(OH)_8$	1.5
Muscovite	$KAl_2AlSi_3O_{10}(OH)_2$	14.4
K-feldspar	$KAlSi_3O_8$	14.2
Plagioclase	$NaAlSi_3O_8 – CaAl_2Si_2O_8$	15.5
Calcite	$CaCO_3$	2.2
Cu, elemental	Cu	0.3
Al, elemental ?	Al	0.8
Total		100.0

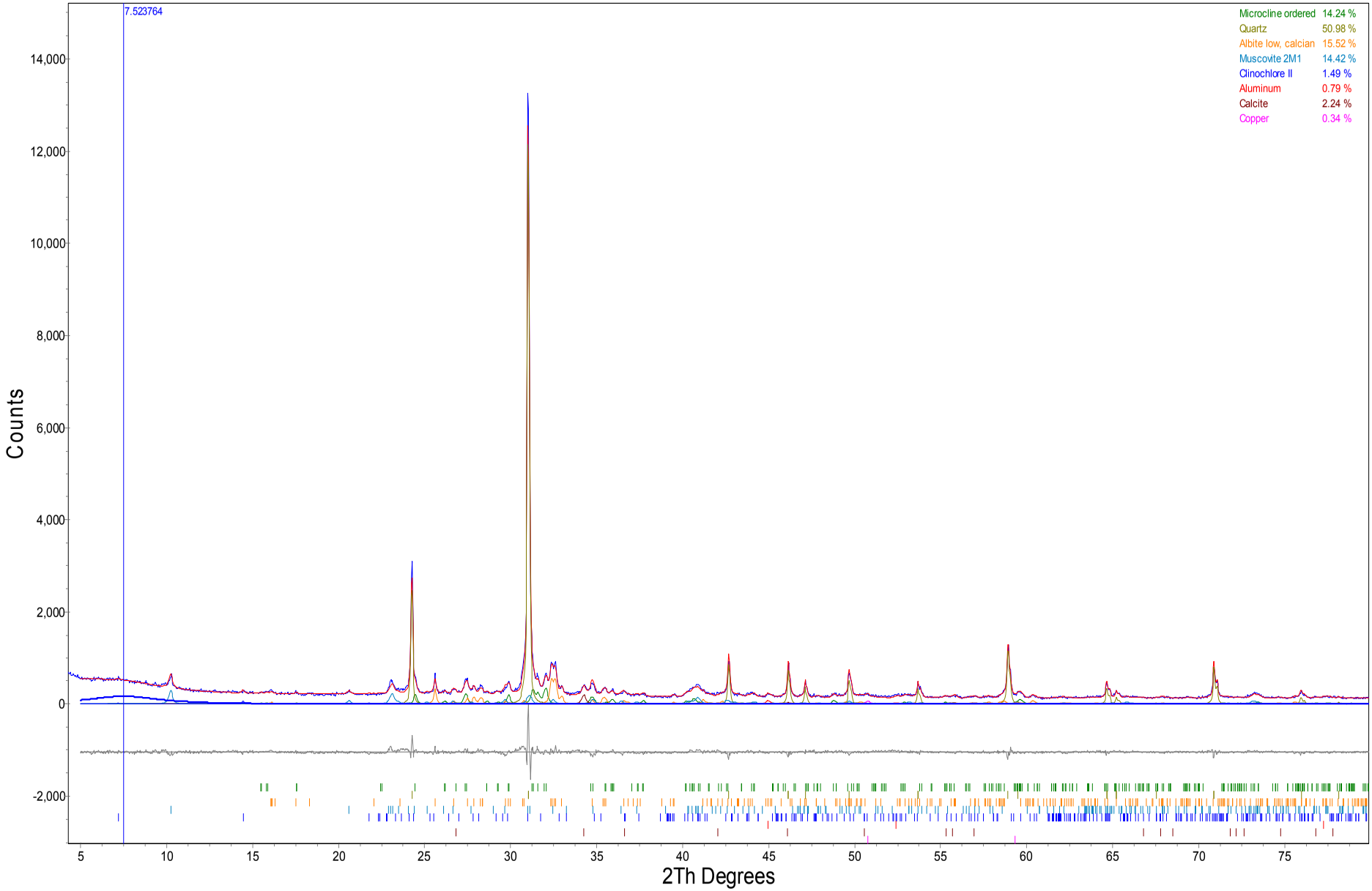

Figure 1. Rietveld refinement plot of sample **Met-Solve 73397 Idaho gold and silver sample** (blue line - observed intensity at each step; red line - calculated pattern; solid grey line below – difference between observed and calculated intensities; vertical bars, positions of all Bragg reflections). Coloured lines are individual diffraction patterns of all phases.