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## ANALYTICAL CHALLENGE

By Dr. Jan Krasoń

From the beginning of our endeavor we were aware of the past research and exploration efforts for sediment-hosted precious metal deposits, particularly in the US. We suspected that in the black shales gold and PGM may be accumulated in a form of sub-micron size particles, which may form microclusters.

- Microclusters may be associated with sulfides, oxides, iron silicates, clays, organic matter and evaporitic salts.
- Complex clusters have been proposed to explain the positive correlation between precious metals and base metals found in the perfect crystal structure of same sulfides (Novogradova, M. i., 1994).
- Particularly gold and PGM particles larger than 0.01 µm have metallic properties unless under the influence of interfere in elements contained in many iron-bearing rocks and minerals.
- Many occurrences of microfine, particularly gold in the sediment-hosted ores have been observed worldwide. Gold particles tend to very in shape from rounded to octahedral crystals and dendrites, platelets or irregular forms with an increase in size.
- Per analogy of the potential hosting rocks, that we have already sampled, particularly of clays and black shales, especially clays like bentonite; we anticipate micron and colloidal gold and PGM.
- We have also found that the standard fire assays yield blanks, and wet chemical extraction yield only trace of gold on the untreated sample material.
- Although it is known that the presence of arsenic and antimony form spies compounds during the oxidation part of standard assay, precious metals are absorbed into said compounds and lost in the slag, thus the fire assays of clay and shales usually indicate the samples are barren.
- Routine atomic absorption tests also fail to indicate the presence of gold and PGE, because their complexity by organic compounds not dissolved by acid solutions.
- Assays of carbon-rich and bituminous black shales are also difficult because of the organometallic metal sources. Therefore, values can easily be overlooked.

Facing of all the above, we have analyzed our black shales samples by ICP-MS. Although by this analytical method, in some of our black shale samples ICP-MS detected as much as:

Pd – 18.5, 16.4, 15.5, 7.29, 1.41 ppm; in the same samples, respectively: Pt – 0.18, 0.14, 0.14, 0.11, 0.02 ppm; Rh – 0.06, 0.05, <dl, 0.11, <dl; Ir – 0.07, 0.06, 0.06, <dl, <dl; Os – all <dl; Ru – 0.28, 0.26, <dl, 0.61, 0.61, 0.12 ppm; Au – 0.20, 0.22, 0.15, 0.11, 0.02 ppm; Ag – 1.41, 1.50, 0 – 1.36, 0.63, 0.20 ppm.</li>

The samples analyzed by the same laboratory using ICP-MS reported respectively:

 Pd – 0.32, 2.39, 2.87, 0.97, 2.45 ppm; in the samples respectively: Pt – <dl, 0.02, 0.04, 0.04, 0.03 ppm; Rh – all <dl; Ir – all <dl; Os – all <dl; Ru – 0.03, 0.06, 0.20, 0.02, 0.13 ppm; Au – 0.0.7, 0.05, 0.22, 0.33, 0.05 ppm; Ag – 0.06, 0.08, 0.16, 0.09, 0.08 ppm. Than we were warned that: "notwithstanding the above, Au, and Pd values reported by ICP-MS should be treated with caution as interferences are common and may lead to spurious results. In this regard, values should be confirmed by alternative methods (e.g. INAA and/or Fire Assay).

We did 20 samples, but because negative analytical results by INAA method, we were also warned that it could happen because interference of some base metals.

After all of the above considerations and efforts, with commonly applied procedures of the sample preparation, we have proceeded with the alternative method of metals concentration.

Pre-concentrated 61 samples have been analyzed by Eltron Research & Development, Inc., in Boulder, Colorado.

- The surface of the samples was examined using a JEOL 5610 Scanning Electron Microscope.
- Images of metal beading on the surface were captured at x 500. APGT Avalon 4000 Series EDX was used to identify elemental composition of the beading and substrate material.
- Interference with silicon and sulfur caused difficulty in detecting the presence of trace amounts of the PGM. When these metals were present in larger amounts there was a discernible peak.
- However, for many samples this isn't the case. The computer analysis list quantifiable amounts for these metals because it is integrating parts of the silicon and/or sulfur peaks.
- As pointed out above, all numbers calculated by the software are standard-less values and can only be used for comparison amongst samples under the same conditions.

Particularly considering the latter statement, for verification of the Eltron's results we have delivered 51 samples for SEM/EDX analysis by Aspex Corporation, in Delmont, Pennsylvania.

- Aspex analyzed our samples with pre-concentrated metals using the ASPEX Explorer.
- Chemical composition data was acquired using a SDD detector with 30 mmsq-Ultra Thin Window SDD detector.
- Automated particles analysis was performed using the Automated Feature Analysis (AFA<sup>™</sup>) component of the Perception software suite.
- Reports were prepared using ASPEX Tabular Reporter<sup>™</sup> and ASPEX Image Reporter<sup>™</sup>.

Examples of the analytical results of some samples, with micro-photographic documentation by ELTRON Research & Development, Inc., and by ASPEX Corporations are documented in following two PowerPoint Presentations prepared by Dr. Jan Krasoń of Geoexplorers International, Inc.:

- 1. *"PROPOSAL FOR THE EXPLORATION PROJECT OF THE SEDIMENT-HOSTED PRECIOUS METALS IN THE WESTERN UNITED STATES".*
- 2. "ANALYTICAL RESULTS OF THE BASE AND PRECIOUS METALS FROM KGHM POLISH COPPER FLOTATION TAILINGS".