



VUORIMIESYHDISTYS-  
BERGSMANNAFÖRENINGEN r.y.



# 13. Geokemian Päivät 2018 – 13<sup>th</sup> Finnish Geochemistry Days 2018

28.–30.11.2018  
Oulun yliopisto, Oulu  
Finland



## **Tiivistelmät - Abstracts**

Toimittaneet – Edited by  
Kirsi Luolavirta, Pertti Sarala



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Julkaisija - Publisher: Vuorimiesyhdistys, Sarja B, Nro B99, Oulu 2018  
Taitto – Layout Kirsi Luolavirta, Pertti Sarala

ISBN 978-952-9618-96-5  
ISSN 0783-1331



# 13. Geokemian Päivät 2018 – 13th Finnish Geochemical Meeting 2018

28.–30.11.2018 Oulun yliopisto, Linnanmaa – University of Oulu, Linnanmaa Campus

## SEMINAARIOHJELMA – SEMINAR PROGRAMME

### Keskiviikko – Wednesday 28.11.2018

12.15-17.00 Lyhyt kurssi geokemiallisista on-site analysointimenetelmistä - Short course on geochemical on-site analytical techniques (in English). Sali – Lecture room L4 Kouluttajat – Lecturers: Bruno Lemière (BRGM), Pertti Sarala (Oulun yliopisto). Tilaisuus on ilmainen ja suunnattu erityisesti opiskelijoille – The course is free of charge and aimed particularly at students.

Sisältää – Includes: Introduction to the on-site analysers, pXRF in exploration using till and other sediments, pXRD in mineral identification, on-site analytical technologies for water, pLIBS in mineral exploration and on-site laboratory technologies.

### Torstai – Thursday 29.11.2018

- 8.30-9.00 Ilmoittautuminen – Registration, Luentosali – Lecture room L4
- 9.00-9.05 Avaus - Opening
- 9.00-9.35 Bruno Lemière, Keynote: Field portable geochemical techniques and site technologies for mineral exploration
- 9.35-9.50 Hokka, J. & Virnes, L. Litho-geochemical pXRF study on the Virtasalmi Cu deposit, eastern Finland
- 9.50-10.05 Kinnunen, J. et al. Practical usage of portable measurement devices in exploration
- 10.05-10.20 Koskinen, H. & Sarala, P. Portable X-Ray Diffraction (pXRD) analyser as an on-site surficial geological exploration tool
- 10.20-10.40 Kahvitauko - Coffee break
- 10.40-11.05 Nuottimäki, K. et al. Applying pXRF analyser for the soil geochemical baseline studies
- 11.05-11.20 Nissinen, T. et al. On-site analysis of metal concentrations of natural waters
- 11.20-11.35 Boman, A. et al. Development of field methods for identification of acid sulfate soils
- 11.35-11.50 Mattbäck, S. et al. Quantification of metals and acidity leached from coarse-grained acid sulfate soil materials in western Finland
- 11.50-12.00 Huomion osoitukset - Honour
- 12.00-13.00 Lounas - Lunch
- 13.00-13.15 Dalhem, K. et al. A miniaturised distillation method for determining reduced sulfur species
- 13.15-13.30 Sapon, S. et al. Applying heatmap plots in interpreting urban geochemical data in the city of Oulu

- 13.30-13.45 Junttila, H. Hyperspectral imaging and its' geological applications – a review
- 13.45-14.00 Rönqvist, J. et al. The portable hyperspectral measurement device Halo and how to use it in exploration
- 14.00-14.15 Holma, M. Muography: a method using cosmic-ray muons for imaging density contrasts in soil and bedrock formations in 2D, 3D and 4D
- 14.15-14.30 Woodard, J. et al. Geochemistry and U-Pb zircon age of the Pha Aok Granite, SE Cambodia
- 14.30-14.50 Kahvitauko - Coffee break
- 14.50-15.20 Peronius, A. Mikro- ja makrokultahippujen kustannustehokkaat tutkimusmenetelmät
- 15.20-15.35 Sääsö, J. Metallisen kullan esiintyminen moreenissa Iso-Kuotkon alueella
- 15.35-15.50 Panttila, H. Vesinäytteenottoa multikopterilla
- 15.50-16.05 Karinen, J. et al. Arctic Steel and Mining – In the search of critical minerals
- 16.05-16.20 Sarala, P. et al. Upscaling deep buried geochemical exploration techniques into European business – UpDeep
- 16.20-16.45 Posterit ja näyttely – Posters and exhibition
- Pietilä, R. et al. Biochar as dry cover material and greening solution in mine waste areas
- Mueller, M. The Mustajärvi Orogenic Gold Deposit, Central Lapland Greenstone Belt, Finland
- Luolavirta, K. & Hanski, E. Application of in-situ LA-MC-ICP-MS isotopic analysis in tracking down petrogenesis of magmatic rocks and related ore deposits: Kevitsa case study
- Kärenlampi, K. Utilization of handheld XRF in identification of A-type granites and Nb-Zr-REE mineralized glacial boulders
- Rantala, N. & Tolkkinen, M. Maaperän mikrobien vaikutus purojen rantavyöhykkeisiin
- 17.00-20.00 Buffet-illallinen OMS-tutkimuskeskuksessa - Buffet dinner and refreshments, OMS Research Center, PR141

### **Perjantai – Friday 30.11.2018**

Indika-projektin loppuseminaari, Luentosali – Lecture room L2

(Suomenkielinen – In Finnish)

- 9.00-9.30 Sarala, P. & Taivalkoski, A. Indikaattorimineraalit kriittisten metallien etsinnässä - Indika-projekti
- 9.30-9.50 Karinen, J. et al. Knelson-konsentraattori kriittisten mineraalien tutkimuksessa

- 9.50-10.10 Lukkari S. & Lehtonen, M. Kriittisten mineraalien indikaattorien tunnistaminen laboratoriomenetelmin
- 10.10-10.30 Taivalkoski, A. et al. Kriittisten mineraalien kenttärikastusmenetelmät ja tulosten vertailu
- 10.30-10.50 Kahvitauko - Coffee break
- 10.50-11.10 Koskinen, H. pXRD-analysointori kriittisten mineraalien indikaattorien tunnistamisessa
- 11.10-11.25 Strand, K. Hyperspektrimenetelmän sovellukset kriittisten mineraalien etsinnässä
- 11.25-11.40 Heinonen, H. et al. pXRF mobiilitiedonsiirto kenttämittauksissa
- 11.40-12.00 Sarala, P. Indika-projektin tulosten yhteenveto
- 12.00-13.00 Lounas - Lunch
- 13.00-15.00 Geoalan osaajat työelämässä (Geologiliitto) (Suomenkielinen – In Finnish)
- Useita esityksiä monipuolisesti geoalan ammattilaisilta liittyen koulutukseen, uravalintoihin ja tehtäväkenttään erilaisissa työyhteisöissä. Tilaisuus on kaikille avoin, mutta erityisesti toivotaan opiskelijoiden tulemaan kuulolle ja keskustelemaan sekä kyselemään ammatinvalintaan liittyvissä asioissa.

### Julkaisu - Publication

Luolavirta, K. and Sarala, P. (eds.) 2018. 13. Geokemian Päivät 2018 – 13th Finnish Geochemistry Days 2018, 28.-30.4.2018, Oulun yliopisto, Oulu, Finland: Tiivistelmät - Abstracts. Vuorimiesyhdistys, Sarja B 99, 44 p.

### Järjestäjät - Organisers



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## **Tiivistelmät - Abstracts**





## Development of field methods for identification of acid sulfate soils

Anton Boman<sup>1</sup>, Miriam Nystrand<sup>2</sup>, Krister Dalhem<sup>2</sup>, Fanny Bollström<sup>2</sup>, Jaakko Auri<sup>3</sup>, Mirkka Hadzic<sup>4</sup>, Raimo Ihme<sup>4</sup> and Peter Österholm<sup>2</sup>

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Human disturbance of acid sulfate soils cause a lot of stress on the environment due to acidic and metal-rich runoff (e.g. Boman et al. 2010; Österholm & Åström 2002). Also in infrastructure developments there are corrosion problems associated with the acidic runoff from acid sulfate soils. The present method (incubation-pH) used to recognize acid sulfate soils in Finland is time consuming and may therefore slow down the progress of various projects by several months.

The main goal of this project (“Maastokäyttöisten tunnistusmenetelmien kehittäminen happamille sulfaattimaille - Tunnistus”, funded by the European Regional Development Fund 2017-2020) is to develop laboratory and field methods that are easy and safe to use and that rapidly enables reliable recognition of acid sulfate soils in the field. Another goal is to develop methods in order to assess the acidifying potential of acid sulfate soils. The acidifying potential can be utilized to assess the acidity and metal load and to assess the need for mitigation methods such as liming. The developed methods will be important tools for environmental authorities in order to improve decision making regarding land use on e.g. peat production areas, farmlands and in infrastructure developments.

The current field method for identifying acid sulfate soils is to measure the soil pH in the oxidised material. If the pH is below 4 for mineral soil materials or less than 3 for peat, the soil is considered an acid sulfate soil. However, acid sulfate soils are commonly in different stages in terms of time of leaching and oxidation and quite often the soil pH in the oxidised layer is not below the critical limit (i.e. 4 or 3). In such cases, samples of the reduced parent material (i.e. not oxidised yet) is collected and the sample is naturally oxidised (incubated) in the laboratory for at least 8 weeks but generally up to 16 weeks. If the pH drop after the incubation period is less than 4 for mineral soil material and less than 3 for peat, including a drop in pH with more than 0.5 units compared with initial pH, the material is considered hypersulfidic and the soil is classified as an acid sulfate soil.

The method development is divided into two parts of which the first part is focusing on methods for the recognition of acid sulfate soils in the field and the second part is focusing on methods for characterizing the environmental risk (i.e. leaching of acidity and metals) associated with these soils. A flow chart of the methods to be tested is presented in Fig. 1.

Central to many of the experiments is to use 30% hydrogen peroxide in order to speed up the oxidation and to analyze the leachate for electrical conductivity (electrode), sulfate (portable sulfate analyzer), and amount of acidity produced (titration with KCl and NaOH). In the field, 30% hydrogen peroxide is used to indicate acid sulfate soils if the resulting pH in the leachate solution is acidic enough (pH < 2.5). For sulfide concentration, a so called RISA-analyzer developed in Australia is being tested alongside traditional sulfur speciation (Dalhem et al. 2016). The sulfide concentration itself is not enough for classification of acid sulfate soils, but can be used to show if the pH drop during oxidation (either natural or during

incubation) is due to sulfide oxidation or among others the influence of organic acids. However, if the sulfide (and/or sulfate) concentrations are high, generally also the potential acidity is high (thus indicating a potential high environmental risk). The use of a handheld XRF for the determination of total sulfur in the field will also be tested.

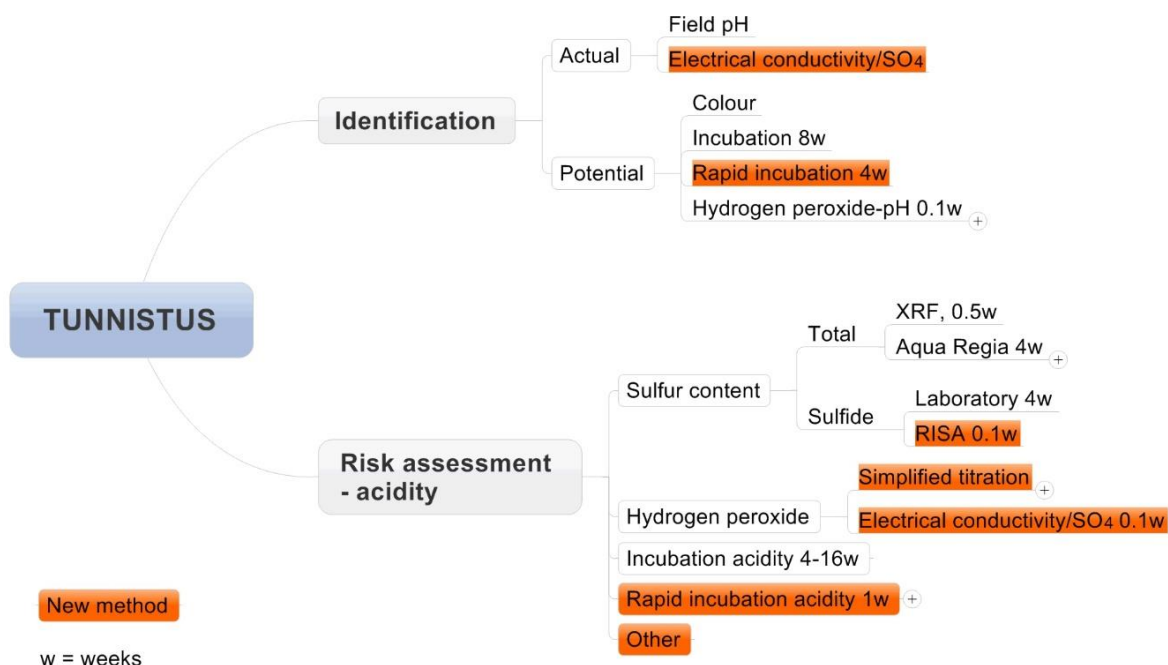


Fig. 1. Flow chart of the methods that are tested in the project.

## References

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Dalhem, K., Mattbäck, S., Österholm, P. & Boman, A. 2016. A miniturized distillation method for determining reduced sulfur species in sulfidic soil materials. 8th International Acid Sulfate Soils Conference, 07.17.2016-23.07.2016. College Park, Maryland, USA.

Österholm, P. & Åström, M. 2002. Spatial trends and losses of major and trace elements in agricultural acid sulphate soils distributed in the artificially drained Rintala area, W. Finland. *Applied Geochemistry* 17, 1209-1218.

## A miniaturised distillation method for determining reduced sulfur species

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In order to assess the risks of sulfidic soil materials (e.g. marine sediments, peat, dredge materials, mine tailings) on the environment and infrastructure, it is crucial to quantify the different sulfur species that are the major culprits for acidification and metal leaching. Predominately these are reduced species such as metastable iron sulfides (FeS<sub>1.1</sub>) and pyrite (FeS<sub>2</sub>) that, when subjected to oxidising conditions, produce sulfuric acid which acidifies the surrounding and enhances chemical weathering. Sulfur speciation methods are, however, time consuming, expensive and labour intensive. With existing methods (e.g. Backlund et al. 2005) it is possible to separate and quantify reduced sulfur species from fine-grained (d<sub>50%</sub> grain size < 63 µm) hypersulfidic material (i.e. soil material with sulfidic S ≥ 100 mg kg<sup>-1</sup> and a pH < 4.0 upon oxidation (Sullivan et al., 2010)) with good precision and accuracy. Recent studies (Mattbäck, Boman & Österholm, 2017) have brought to attention coarse-grained (d<sub>50%</sub> grain size ≥ 63 µm) soil material with low sulfidic S that have an acidifying effect on the local environment due to their low buffering capacity, as well as hypersulfidic peat which can produce significant amounts of acidity if excavated. A distillation method for quantifying low amounts of sulfidic S with good precision and accuracy for risk analysis of low sulfidic soil materials is therefore needed. Previous methods (e.g. Burton et al., 2005) are also inadequately designed to dissolve sulfur species from peat and other organic material. We present a versatile miniaturized sulfur speciation distillation method for reduced sulfur species that is inexpensive to manufacture, easy to use, has a low detection limit and can be used to analyse several samples simultaneously.

The miniaturised distillation apparatus (Fig. 1) is constructed from simple 50 ml polypropylene centrifuge tubes. Gas-tight centrifuge tube caps are fitted with acid resistive tubing plugged with ports and luer-lock stopcocks, which allow delivery of reagents to the reaction chamber, and transport of liberated H<sub>2</sub>S gas to the collection chamber using N<sub>2</sub> gas as a transport medium. The centrifuge tubes are readily sterilized and can be discarded after use, thus reducing time spent cleaning expensive glassware, and can sustain over boiling point temperatures, thus enabling shorter reaction times (Sullivan, Bush & McConchie, 2000) compared to cold purge-and-trap methods (Backlund et al., 2005). Smaller sample sizes than previous methods are possible due to the miniaturised setup and a modified analytical procedure enables sequential distillation of reduced sulfur species from a single 0.5 – 5 g sample. Acid volatile sulfide (AVS), comprised mainly of metastable iron monosulfides and greigite, is first liberated using 6 M HCl, followed by hot chromium reducible sulfur (HCrS) using 3 M CrCl<sub>2</sub> for dissolving pyrite. Elemental sulfur (ES) can be dissolved using an organic solvent and separately liberated using the HCrS method. The liberated H<sub>2</sub>S from AVS, HCrS and ES procedures is collected as ZnS in an alkaline medium and the quantities of the reduced sulfur species is then determined using an iodometric titration procedure.

Extensive experiments for determining reduced sulfur species in fine-grained hypersulfidic material, coarse-grained sulfidic material, sulfidic peat material and a laboratory graded pyrite standard show good precision and low quantifiable detection limits, thus enabling more precise risk assessments for a broad range of sulfidic materials.

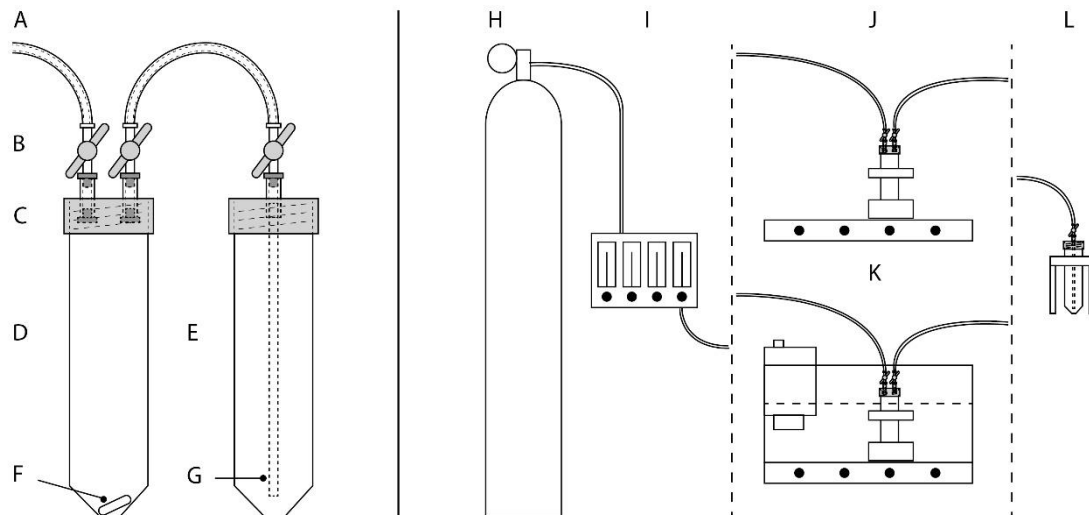


Fig. 1. Schematic of reaction and collection vessels (left) and distillation setup (right). A: tubing for gas-flow, B: luer-lock stopcocks, C: polypropylene screw caps, D: 50 ml polypropylene centrifuge tube as reaction vessel, E: 50 ml polypropylene centrifuge tube as collection vessel, F: stirring magnet, G: glass pipette, H: N<sub>2</sub>-gas bottle, I: flow meter, J: distillation setup with reaction vessel on a stirring plate, K: distillation setup with reaction vessel in a hot water bath on a stirring plate, L: collection vessel.

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## **Lithochemical pXRF study on the Virtasalmi Cu deposit, eastern Finland**

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Field-portable X-ray fluorescence (pXRF) analyzers have become widely used tools, for example, in collecting primary exploration data and distinguishing different minerals. In brownfield exploration targets, where pre-existing drill-core data can be re-measured with a high density and in a relatively reliable manner using pXRF, it can help in improving the understanding of local geology and exploration targeting.

In this study, historical drill cores were re-examined to test the use of a pXRF analyzer in detecting the geochemical halos around the Virtasalmi Cu deposit and to demonstrate that the analyzer characterizes the mineralization intercepted in drill cores. A total of 304 in-situ, non-destructive measurements from five drill holes were conducted to observe the elemental dispersion from distal to proximal zones. From the results, the main lithological units can be discriminated. The data display a gradual increase in CaO, and high concentrations of chalcophile elements, such as Cu, were detected with pXRF. pXRF data were shown to be reliable and can be used for different modelling purposes, as well as in mineral systems studies. Additional geological background for this work was provided by a master's thesis project where the Virtasalmi Cu deposit and the Virtasalmi mine was reconstructed in a modern 3D-modelling program to investigate the geometry and distribution of the deposit. The new Cu mineralization model is based on a digitized dataset of 239 historical drill holes and around 3000 Cu-analyses.

## **Muography: a method using cosmic-ray muons for imaging density contrasts in soil and bedrock formations in 2D, 3D and 4D**

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Various geophysical methods are useful for making rough assessments of mineralogical and geochemical compositions of unexposed rock formations. For instance, magnetic susceptibility is correlated with the magnetite content of a rock body, while the density value can be related, e.g., to the ore grade or igneous petrology. The density values can be calculated from the core or bulk samples or determined using geophysical borehole density logging tools (for example, gamma-gamma scattering, photoelectric absorption, microgravity). The relative densities of subsurface formations can also be determined by a technique called muon radiography, or simply muography. This emerging geo-particle physical imaging method is based on elementary particles called muons. Muons are sub-atomic particles similar to the electrons but over 200 times heavier and originate naturally from the upper layers of the atmosphere as it is constantly interacting with primary cosmic radiation (or “cosmic rays”). Muons are a major contributor of secondary cosmic rays and rain downwards in large numbers and with high frequency as cascading particle showers. Although muons have a lifetime (or rather half-life) of only 2.2 microseconds, many of them eventually reach sea level due to their velocities close to the speed of light. Cosmic-ray muons are extremely penetrating particles and some of them, albeit a very minor number, can travel thousands of meters below the Earth’s surface until stopped or decaying to other particles. The fundamental properties of muons are exploited in muography.

In the recent years muography has experienced an impressive development and has found applications in many different fields including nuclear non-proliferation, spent nuclear fuel monitoring, imaging of inaccessible sections of industrial plants, cargo scanning, archaeology and volcano imaging. The utilization of muons requires no radiological sources. The muon absorption rate is utterly independent of the chemical composition of the rock (Tanaka 2013). Hence the attenuation of cosmic-ray muons passing through any matter can be used to estimate the density of any objects they pass through. The positions and directions of individual muons traversing the object under investigation are recorded before and after they enter and exit the object. The observed muon data can then be inverted by dedicated algorithms to 3D density contrast maps. In the Earth sciences, the only requirement of imaging is that the detector is positioned behind the object of interest. In some applications, like in long-range volcano imaging, the detector can be set on the slope of the volcano. In other applications the detector must be positioned in underground tunnels to generate a 3D density contrast map of the volume of rock between the sky and the detector. As technological breakthroughs are currently made to improve particle detectors and miniaturize electronics, muon detectors fitting all standard-size boreholes will soon enable the use of muography in a much greater scale and in more varied applications, than ever before. Also the ever increasing processing power will promote further development of muography and lead to new applications that simply were not technically feasible before.

There are many potential application areas for muography in the Earth sciences. These include density mapping of lithological boundaries and faults, structural imaging of folded lithologies, imaging of layering in sedimentary deposits and igneous intrusions, weathering crust studies, imaging of internal structures of soil formations, and ore exploration. In the

latter the natural resources related to density variations are the most oblivious targets for muographic imaging. However, with the increasingly sophisticated simulation algorithms it is also possible that the density anomalies in the hydrothermal alteration zones become muographically recognizable in the near future.

One of the unique attributes of muography is that it enables monitoring of temporal changes (so-called 4D) in densities. This is why muography is one of the few available methods to effectively study, for example, eruption dynamics of volcanoes or permanent monitoring of their activity levels. The hydrological recharging of aquifers or migration of water in faults and soil layers can also be studied with time-lapse muography (4D imaging). Whenever possible, muon-based relative densities can be combined with gravimetric data, or any other type of geophysical or geological data. If available, direct density measurements can be used as fixed point values for certain parts of the transilluminated volume of rock or soil improving the density calibration even further.

The future of muography as an imaging method capable of resolving internal structures of large-size targets by taking the advantage of high penetrating power of cosmic-ray muons appears to be a promising one. If the recent developments made in the fields of detectors and electronics together with inversion and simulation algorithms continue still further, muography will have a good chance to become one of the very standard methods used in the Earth science studies in both practical and scientific purposes. There are, after all, not many methods available for imaging internal structures of large-scale geological bodies.



Fig. 1. A borehole muon detector in action in a groundwater production well in Kurikka, Finland. This particular muon monitoring system is equipped with solar panels and external modem antenna for remote control and data transferring purposes. Figure courtesy by Muon Solutions Oy.

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## Hyperspectral imaging and its' geological applications – a review

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Hyperspectral imaging technology has existed for about 30 years, starting from remote sensing applications. As sensor technologies have developed, one can image smaller objects with greater accuracy and resolution. Also the size of the sensors has decreased, there is already portable imaging spectrometers available.

This presentation will concentrate on different types of geological applications of hyperspectral imaging. There will be five examples presented from different fields, based on literature.

Example 1 is in the field of economic geology. Porphyry copper deposits can be found and characterized by hyperspectral imaging. Even in an extent that first indications of e.g. porphyry deposit can be done from air or space, finding the alteration halo using hyperspectral remote sensing technologies (Pour & Hashim, 2012). The boundaries of the deposit can be characterized on site with a portable field spectrometer, and when the drilling program starts and the drillcore are produced, the alteration patterns of white micas, detected from hyperspectral data, will bring towards the mineralization. Furthermore, using relationships of Cu-grade and alteration zones, the NIR based sorting methods can be used in processing of the ore (Dalm et.al. 2014).

Example 2 was taken in as REE minerals are considered critical in many countries. Turner et.al. (2015) have shown that concentration of lanthanides in a given mineral is affecting to the absorption features of REE-bearing minerals, which have distinctive spectral features on VNIR-region of infrared spectrum.

In sedimentary geology hyperspectral imaging has been used for e.g. identifying sub-annual sediment structures like lake sediment varves and their biogeochemistry in sub-varve scale (e.g. Grosjean et. al. 2014). Example 3 gives also insight to prediction of nutrient composition of soils.

Example 4 is about classification of hypabyssal kimberlites by SWIR and LWIR imaging. Tappert et. al. (2015) created dilution maps of kimberlites using hyperspectral core imaging and created an automated data processing method to identify undiluted kimberlites, micro- and macrodiluted kimberlites and crustal rocks.

Example 5 gives an approach to detecting and classifying materials from drill core from Stillwater complex on VNIR-range, in which there is not necessarily diagnostic absorption features present. The study was to examine imaging spectroscopy for detecting sulphide minerals by their color and brightness intensity (albedo). (Bolin & Moon, 2003)

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## Arctic Steel and Mining – In the search of critical minerals

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Arctic Steel and Mining (ASM) is one of the research groups of Lapland University of Applied Sciences (Lapland UAS) in the field of Industry and Natural Resources. ASM is specialized in performing mineralogical studies and applied steel tests in arctic conditions. The group has been founded in 2008 as the research group of metallurgical studies. During that time the steel test laboratory of ASM was founded in public funded project M-Lab. Later there occurred a need to study abrasive wear of steels caused by different minerals. After that, the investments for mineralogical laboratory were made in the project MinLappi. The mineralogical laboratory of ASM has been utilized extensively in education as well as special RDI-services for companies including sample preparation and mineralogical analyses.

ASM has constant project partnerships with companies in TEKES-, ERDF- and ESF-funded research projects. One of the most remarkable of these projects is INDIKA, which is led by GTK. In that project, the main goal is to find indicator minerals for critical metals and find study concept to detect and separate these minerals.

Studied soil samples were collected from Vuotso, Sokli and Vuolijoki. Element compositions of soil samples were measured by portable XRF-analysator (pXRF) in the field. The pXRF recognized most of the REEs and it seemed to be a good method for preliminary compositional analysis. The Knelson separator of Lapland UAS was one of the methods used in gravity separation tests. Knelson was suitable for field use and a major part of the separation tests were carried out near the study area itself. Because of that, it was possible to separate necessary mineral concentrates in the field rather than carry heavy soil samples to the laboratory.

The mineral contents of Knelson-concentrates were measured in the mineralogical laboratory of ASM. Qualitative mineral content analyses were carried out by Raman-microscope. Raman seemed to be a quick study method, because it does require only minimal sample preparation and one analysis takes only a minute. Raman was found to be especially suitable to find indicator minerals containing light elements.

Quantitative mineralogical analyses were carried out by FE-SEM EDS-microscope (Fig. 1). It seemed to be statistically reliable measuring up to 10 000 grains per sample. The accuracy of FE-SEM is down to 1  $\mu\text{m}$ , so it was possible to observe compositional variations inside a single mineral grain. In FE-SEM, it is also possible to pick minerals of one specific density for one analysis. That turned out to be a huge advantage when searching some peculiar types of indicator minerals, like minerals containing heavy or light REEs only.

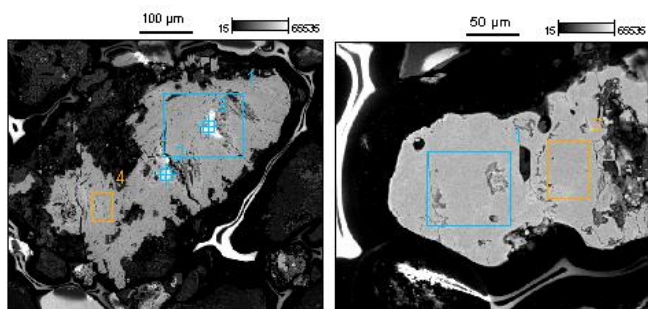


Fig. 1. FE-SEM EDS-images of monazite (left) and pyrochlore (right).

## Practical usage of portable measurement devices in exploration

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Mawson Resources is an exploration company focusing on its flagship property Rompas – Rajapalot in northern Finland. Rompas – Rajapalot project is located at the northern parts of the Peräpohja belt, a Paleoproterozoic supracrustal formation. The Peräpohja belt is bound to the south by Archean rocks of the Pudasjärvi complex and to the north by Central Lapland granitoid complex. Rocks of the northern part of the belt are composed of various mafic volcanic rocks and mafic dykes, calcareous and clastic metasediments, including quartzites and micaceous schists (Ranta 2018).

At the Rompas-Rajapalot area, rock exposure is variable. Most of the bedrock is covered by till and glacial sediments. Even on the areas with better exposure, exposure is selective and certain rock types are overexposed compared to the true extent of the units. Rocks at the area are also highly altered and complexly folded, making exploration and map production a complex task (Cook & Hudson 2018, Vanhanen et al. 2015).

Since 2010 the company has successfully used multiple tools in the field and with the core logging. One beneficial tool in the field has been a gamma spectrometer. Gamma spectroscopy has been used extensively for many years and it is a great tool to assist with mapping and alteration in the field. Portable XRF has been used also to help to identify rock types and minerals, for example, discriminating albite from K-feldspars and sulphide-associated metal enrichment.

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## **Portable X-Ray Diffraction (pXRD) analyser as an on-site surficial geological exploration tool**

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With modern field analysers, such as portable XRD and XRF devices, samples can already be analysed mineralogically and geochemically in the field. With the support of advanced electron optical methods, minerals can be identified fully or semi automatically. Those methods speed up the exploration work process and improve the cost-efficiency of exploration. In this study, we demonstrate the use of Olympus Terra pXRD (Fig. 1) in critical mineral exploration in two test areas in northern Finland: Mäkärä Au-REE mineralization and the Sokli area (phosphorus-rare earth elements, P-REE deposit), northern Savukoski. The first results show that the mineralogy analysed from the till concentrates using pXRD fits relatively well to the known mineralogy of the study areas. Furthermore, the identified minerals are comparable with the results produced by laboratory XRD and FE-SEM, although there are further needs to improve data interpretation and quality. This makes pXRD analysers useful in the mineralogical work and mineral exploration even directly in the field.

The Indika project was funded by the European Regional Development Funding (ERDF) and professional associations (Geological Survey of Finland, University of applied sciences and the Oulu Mining School of the University of Oulu) and industry sector' companies.



Fig. 1 Olympus Terra pXRD analyzer in field use

## Utilization of portable XRF in identification of Nb-Zr-REE mineralization in rocks

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Portable (or handheld) X-ray fluorescence (pXRF) spectrometers have become widely used in the field of geological research, exploration and mining (Sarala, 2016; Young et al., 2016). Recent developments in X-ray tube and detector technologies have also enabled the determination of many critical elements (e.g., Nb, Ta, Zr, Hf, Co, V, REEs etc.) in geological materials by pXRF (Gallhofer and Lottermoser, 2018).

In this study, pXRF analyses of Nb-Zr-REE mineralized rocks collected from the Otanmäki area in central Finland are reported, and the capability of an energy-dispersive pXRF spectrometer to quantify the Nb, Zr and REEs is assessed. A Hitachi Hi-Tech X-MET 8000 (Fig. 1) pXRF unit equipped with a 50 kV X-ray tube was used in the study. The factory delivered calibration in the X-MET 8000 unit was able to quantify Nb, Zr and five of the lightest REEs (Y, La, Ce, Pr and Nd). The analyses from the studied samples were done from diamond saw cut rock slabs (samples 1-8; Fig. 1). A certified geological reference material (REE-1, Natural Resources Canada) was also analysed. An average result of six different measurement points (randomly selected) and a 60 second measurement time per point was used (Table 1). The reference results for the same samples were verified in a certified commercial laboratory (Bureau Veritas) by ICP-MS analyses after pulverization, fusion with lithium borate and dissolution in ACS-grade nitric acid.

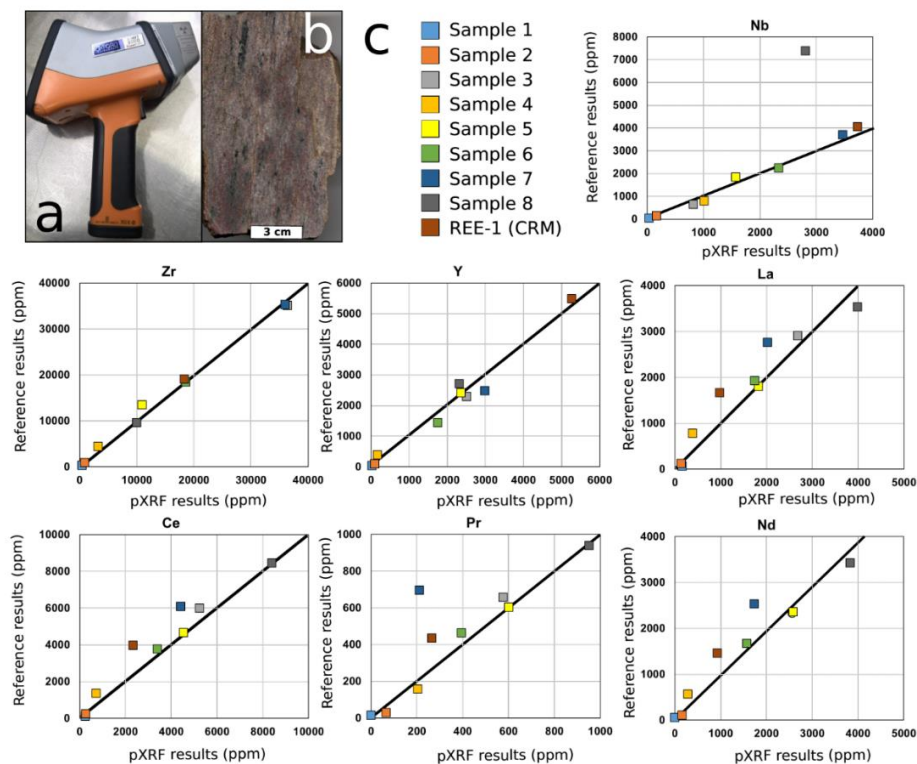


Fig. 1. (a) X-MET 8000 energy-dispersive pXRF. (b) a rock slab (sample 3) cut by diamond saw. (c) pXRF results (x-axis) vs reference results (y-axis) for Nb, Zr, Y, La, Ce, Pr and Nd.

The studied samples (1-8) were felsic meta-igneous rocks, which contained bands or dissemination of Nb-Zr-REE-bearing silicate (e.g. allanite, zircon, titanite) and oxide (e.g. pyrochlore) minerals. The pXRF analyses for Nb, Zr and REE are well or fairly well comparable with laboratory ICP-MS analyses (Fig. 1 and Table 1). Divergent results for some samples (e.g. Nb in sample 8) were likely due to heterogenous distribution of Nb-Zr-REE-bearing minerals in the samples (e.g. in bands) and more single measurements could have improved the average result. The analysis of the REE-1 (powder) indicated that in comparison to CRM recommended values, the precision and accuracy of pXRF was in acceptable limits for Zr, Nb and Y, but not for La, Ce, Pr and Nd (Table 1). The cause for the inaccurate quantification was not obvious, but it could be due to non-optimal factory calibration attributes for powdered samples, matrix effects or spectral interferences.

Table 1. Average results of pXRF analyses (in ppm) from samples 1-8 (rock slabs), REE-1 CRM (powder) and reference results (ICP-MS, Bureau Veritas; BV) for each sample.

Sample	1	BV Ref.	2	BV Ref.	3	BV Ref.	4	BV Ref.	5	BV Ref.
Nb	35	41	167	143	819	639	1009	792	1571	1836
Zr	399	339	906	931	36368	35121	3229	4423	10925	13494
Y	29	32	98	88	2512	2292	172	384	2365	2419
La	159	60	136	123	2684	2905	388	776	1821	1799
Ce	247	118	270	247	5247	6000	727	1358	4540	4663
Pr	0	14	66	28	577	657	204	157	601	603
Nd	0	53	161	104	2567	2330	285	566	2587	2353
Sample	6	BV Ref.	7	BV Ref.	8	BV Ref.	CRM REE-1	BV Ref.	CRM Recom. values	
Nb	2334	2238	3471	3696	2809	7381	3718 ± 89	4046	4050	
Zr	18602	18446	35981	35280	9980	9603	18196 ± 523	19343	19100	
Y	1751	1438	2985	2480	2318	2709	5212 ± 208	5580	5480	
La	1741	1929	2024	2761	3987	3534	1122 ± 120	1583	1661	
Ce	3391	3775	4422	6091	8398	8447	2682 ± 190	3824	3960	
Pr	395	463	212	696	952	938	317 ± 173	429	435	
Nd	1575	1667	1738	2534	3829	3422	1175 ± 247	1433	1456	

The results of this study indicate that the pXRF can provide information about the Nb, Zr and REE contents in rocks reliably, cost-efficiently and fast, as the chemical information produced by pXRF is available in minutes. However, the pXRF determinations should not be taken as a correct result without a doubt, and laboratory analyses and in-house standards or suitable CRMs have to be used to understand possible sources of error in pXRF analysis.

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## **LIBS**

Laser-induced breakdown spectroscopy was introduced later and brought big promises in overcoming the three classical limitations of pXRF: distance to sample, lower atomic weights, and radioemission permits. However, most current applications of LIBS fall short of the reliability of pXRF on heavier elements. LIBS is indeed the only option for Li and elements lighter than Mg, and some manufacturers claim it can analyse all the elements, but it often needs to be used in combination with pXRF to cover exploration needs. An advantage of LIBS over pXRF is the ability to carry out focused spot measurements and micromapping. Their safety constraints (laser) are less strict than those implied by radiation mitigation for pXRF, hence easier permitting for field operation.

## **pXRD**

XRD instruments are field-portable but bulkier than handhelds. Their applications are constrained by the limitations of optical geometry. They can fill a significant role in exploration mineralogy, especially through the recognition of hydrothermal alteration zones and secondary minerals.

## **FTIR**

Infrared handheld spectrometers can be used for the detection or semi-quantification of organic substances and specific minerals (nIR instruments for humidity and asbestos, mIR instruments for extended mineralogy and organics). They need the development of exploration-oriented libraries. They have a potential for hydrothermal alteration recognition and mapping. Their operation is hampered by water contents and by dark substances, such as organic matter.

## **μRaman**

Micro-Raman handheld spectrometers can also be used for organic substances and minerals, and are less sensitive to water and darkness, but their operation and spectral interpretation are still experimental for exploration applications.

## **Water analysis**

Fluid analyses are not as common as analyses of solid materials, but there are such technologies as ASV, polarography, and ion exchange electrodes allowing analysis of commodity or other important elements. Current applications for water comprise:

- accurate measurement of geochemical properties that are not stable with time and transport,
- fast decision-making measurements and analyses: wastewater treatment or discharge monitoring, including process monitoring and alarm networks,
- catchment mapping and reconnaissance (both for baseline and exploration surveys),
- screening of samples to select which ones will be analysed in the lab,
- improved measurements and analyses for site investigations: catchment or pit lake reconnaissance and monitoring, groundwater logging and monitoring.

Possible issues about sample preparation are less important for water samples than for solid samples, as lab-ready preparation on site is mandatory, and homogeneity is easier to ensure. They are not yet sensitive enough for advanced hydrogeochemical exploration purposes.

## **Exploration site technologies**

Heavier equipment can be used at a mining exploration camp, even if not on the outcrop. This includes:

- laboratory devices adapted for mobile or rough conditions (XRF, XRD, AAS,...)

- combinations of field sensors around a core scanner, allowing the fast processing of cores at the drilling site, before logging or splitting them for laboratory analyses. Core scanners include hyperspectral, gamma and XRF systems, to build extensive core data sets,
- combination of field sensors or devices around a sample preparation system, such as cuttings separated from drilling fluids (an example is the Lab-at-Rig® technology (CSIRO, Imdex and Olympus within Deep Exploration Technologies CRC).

## **APPLICATIONS**

All these technologies are aimed at providing decision-support results while drilling, or while performing geochemical surveys, in order to optimise the exploration strategy in quasi-real time. Handheld instruments operate on outcrops, or on samples submitted to a very basic preparation, using field-portable devices such as battery-operated mills. Field-transportable lab instruments may be hosted by portable cabins or lab trucks at a camp.

There are many discussions about the quality of field analyses vs. laboratory analyses, with often non-neutral contributions from instrument suppliers and from the laboratory profession. Sample preparation and sample representativeness issues are by far more important than instrument limitations to interpret discrepancies. Documented evidence of field-lab concordance is frequently published, usually by geochemists which paid attention enough to sample preparation and representativeness.

Benefits of field analysis comprise in-situ analysis, without transport issues; ASAP, DSP or Triad (adaptation of sampling strategy along results collection or dynamic sampling plans: Crumbling, 2001; US-DOE, 2001), increase of data density and quality (through larger data sets) and better representation of transient phenomena.

Possible pitfalls include improper interpretation of field data as a result of insufficient understanding of the sampling and measurement processes, use of heterogeneous data sets collected by different methods, and the lack of critical examination of data.

Field and on-site results do not need to achieve absolute accuracy. They only need to achieve the level of confidence expected from the decision (Ramsey & Boon, 2012). Field techniques produce usually denser data sets than more expensive laboratory analyses, which may compensate for lower accuracy. Most mineral exploration decisions are based on flexible thinking rather than on a preset framework of investigations. One of the key benefits of real-time analyses, or short delay analyses (less than a day) is the possibility to adjust sampling plans, test hypotheses based on ongoing results, and make fast decisions on exploration work - especially drilling and sampling. This is particularly important for remote locations, where sample logistics to the laboratory may become long and demanding.

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## **Application of in-situ LA-MC-ICP-MS isotopic analysis in tracking down petrogenesis of magmatic rocks and related ore deposits: Kevitsa case study**

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Multicollector-inductively coupled plasma-mass spectrometry (MC-ICP-MS) is an efficient method for high-precision measurements of the isotopic ratios of many elements in geological samples. MC-ICP-MS coupled with laser ablation (LA) enables in-situ analysis of solid samples and determination of the isotopic composition of specific mineral(s) enriched in an element of interest (Sr isotopes in carbonates, plagioclase and apatite, Hf isotopes in zircon, S isotopes of sulfur phases etc.). From a practical point of view, in-situ analysis requires little sample preparation (e.g., thin section) and hence, it is a rather fast and low-cost method to study the isotopic characteristics of various minerals and materials. Consequently, the in-situ analysis using LA-MC-ICP-MS has been quickly developing and presently applied in various fields of geosciences.

Considering petrogenesis of igneous rock suites, aside from radiometric dating, in-situ analysis of minerals in thin sections allows to (i) examine the textural setting of the analyzed minerals, (ii) target the analysis on pristine domains of minerals and hence avoid effects of alteration, microscopic inclusions and fractures, (iii) detect sample-scale disequilibrium among individual minerals, mineral populations or between different mineral phases, and (iv) analyze the same mineral for different parameters. In whole-rock isotope data, the aforementioned pieces of information are inaccessible but can be of significant importance in evaluating complex syn- and post magmatic processes. Furthermore, in the case of magmatic ore deposits, in-situ analyses can be done on minerals that are co-genetic with mineralization, providing important information on ore-forming processes.

As an example of applying in-situ analytical techniques to ore geology, we report on the extensive in-situ Sr and S isotope investigation that has been carried out on the petrogenesis of the Kevitsa intrusion and related Ni-Cu-(PGE) sulfide ore (Luolavirta et al. 2018b). The Kevitsa deposit is hosted by ultramafic olivine-pyroxene cumulates and shows a wide range of Ni tenors from <4 wt.% (uneconomic false ore and contact mineralization) to 4–7 wt.% (regular ore, main economic mineralization) and up to 40 wt.% (Ni-PGE ore) (Mutanen 1997; Santaguida et al. 2015).

Luolavirta et al. (2018b) reports highly radiogenic  $^{87}\text{Sr}/^{86}\text{Sr}_{(i)}$  values for intercumulus plagioclase in the Kevitsa ultramafic cumulates ( $>0.7045$ , estimated DM @2.06Ga is  $\sim 0.702$ ) and  $\delta^{34}\text{S}$  values of pyrrhotite (and pyrite) that are generally higher than +2‰. These isotope data imply involvement of crustal material in the genesis of the Kevitsa intrusion and its ores. Also, the  $^{87}\text{Sr}/^{86}\text{Sr}_{(i)}$  values show differences in different parts of the intrusion, varying in the ore-bearing domain from 0.7050 to 0.7109 with stratigraphic fluctuations but being systematically more homogeneous in the “unmineralized” parts of the intrusion (0.7047–0.7060). The isotope data suggest that dynamic magma emplacement of variably contaminated magma was important in the generation of the economic ore, whereas the evolution of the adjacent part of the intrusion was far less vigorous. Furthermore, notable isotopic differences are observed between different ore-types (Table 1), indicating that more than one magma type was involved in the genesis of the Kevitsa ores. The obtained Sr isotope compositions of the Kevitsa ore types are in line with whole-rock  $\epsilon_{\text{Nd}}$  values reported by Huhma et al. (2018). The Ni-PGE ore type is enigmatic due to its isotopic characteristics

indicating significant incorporation of crustal materials in the magma, yet the host rocks to this ore type record the most primitive mineral compositions observed in the Kevitsa intrusion (Mutanen 1997; Yang et al. 2013; Luolavirta et al. 2018a). The Sr and S isotope compositions show no correlation, which is explained by bulk assimilation of the silicate magma in a deeper staging magma chamber and variable assimilation of sulfur en route into the Kevitsa magma chamber.

Table 1. Selected isotopic and mineral compositional characteristics of the Kevitsa ore types.

	<b>Regular ore</b>	<b>Ni-PGE ore</b>	<b>False ore</b>	<b>Reference</b>
$\delta^{34}\text{S}$ ‰ (median)	+4.0	+2.7	+6.5	Luolavirta et al. 2018b
$^{87}\text{Sr}/^{86}\text{Sr}_{(i)}$	0.706–0.707	0.709–0.711	0.706–0.707	Luolavirta et al. 2018b
$\epsilon_{\text{Nd}}$	-3.4	-6.4	-3.4	Huhma et al. 2018
<b>Olivine Fo%</b>	77–84	84–90	76.5–83	Mutanen 1997; Yang et al. 2013; Luolavirta et al. 2018a

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## Quantification of metals and acidity leached from coarse-grained acid sulfate soil materials in western Finland

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Since the national mapping of acid sulfate (a.s.) soils in Finland began 2009, coarse-grained (d<sub>50%</sub> grain size  $\geq 63\mu\text{m}$ ) hypersulfidic material (i.e. soil material with sulfidic S  $\geq 100\text{ mg/kg}$  and a pH  $< 4.0$  upon oxidation; Sullivan et al., 2010; IUSS Working Group WRB, 2014) attracted research attention due to many unknowns related to their classification and characterization.

We quantified the acidity potential and the metals leached from some of these materials by conducting leaching experiments (Figure 1) using a modified version of European norm EN 12457-2:2002 (cf. Åström & Björklund, 1996; Mattbäck et al. in prep), after the samples had undergone a 16-week incubation procedure (cf. Creeper et al., 2012). During and after the incubation period, the samples were leached with deionized water, and the leachates were recovered. The hypotheses were that (1) the electrical conductivity (EC) could be used for the quick assessment of the amount of acidity and metals released from soil materials to the liquid phase upon oxidation, and (2) the coarse-grained a.s. soils release less acidity and metals than fine-grained a.s. soils. Thus, EC in the leachates were compared with corresponding pH, acidity and element concentrations of different types of a.s. soil materials. For determination of the acidification rate, and the amount of acidity in the oxidized soil, duplicate soil samples were analysed for the titratable incubation acidity (TIA; Österholm & Nystrand, 2016) at specific time intervals (in parallel to the leaching experiments). The aqua regia leachable trace and major elements in the soil samples were analysed with ICP-OES and reduced sulfur species were quantified using a distillation sulfur speciation method (Dalhem et al., 2016).

In accordance with previous studies (Mattbäck et al., 2017), sulfur has been extensively leached from the oxidized portions of the coarse-grained a.s. soil profiles, which therefore lack sulfuric horizons (i.e. pH  $< 4.0$ ). The EC of the leachates from coarse-grained a.s. soils was significantly lower than that of fine-grained a.s. soils; and as expected higher than for coarse-grained non-a.s. soils, which shows that the leaching of potentially harmful elements from coarse-grained a.s. soils is less than from fine-grained a.s. soils.

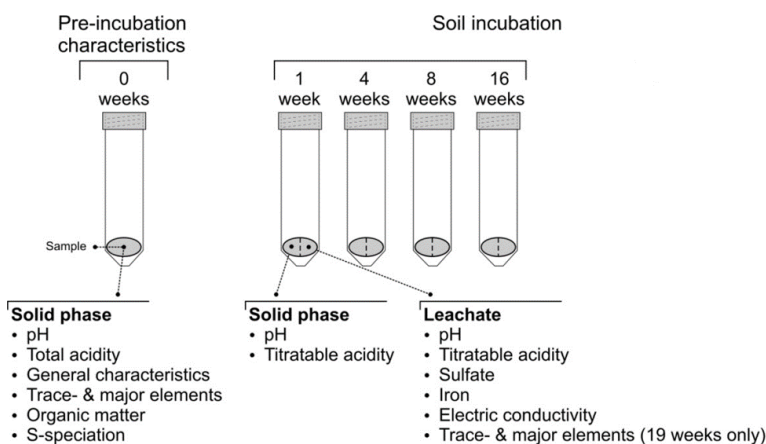


Fig. 1. Analytical framework of the incubation experiments.

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## **The Mustajärvi Orogenic Gold Deposit, Central Lapland Greenstone Belt, Finland**

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The Mustajärvi gold deposit is situated on the southern border of the Paleoproterozoic Central Lapland Greenstone Belt, in proximity to the flatly dipping transcrustal Venejoki thrust system. The local geology comprises Sodankylä group Honkavaara formation siliciclastic metasediments and Savukoski group (ultra) mafic volcanics. The metasedimentary rock package consists of banded arkose quartzites and intermediate & mafic tuffites. The mafic to ultramafic volcanics encompass komatiites (talc-chlorite schists), komatiitic & tholeiitic basalts. Both rock units are roughly dipping SSE with an average dip of 55°. The overall contact between the rock units is gradual, with strong interlayering between the rock types in proximity to the contact zone. Within the domain of the known gold occurrence, thick lenses of (ultra) mafic volcanics occur in the siliciclastic metasediment unit. Due to the competency contrasts, this likely resulted in the formation of a smaller E-W trending fault that hosts the known mineralization which consists of a set of parallel auriferous quartz-pyrite-tourmaline veins that show a dip similar to the host rocks. This small fault is interpreted to be a third-order splay of the NE trending, second-order, Mustajärvi fault zone, situated at the regional contact between both rock units, delineated by ground magnetic- and IP-resistivity surveys. The Mustajärvi fault itself likely represents a splay from the first order, transcrustal, Venejoki thrust system. Strong IP-chargeability anomalies within the interpreted Mustajärvi fault zone and high Au -anomalies in bottom-of-till samples that are spatially closely associated with the fault indicate that not only the third-order splay, but also the Mustajärvi fault zone is mineralized, constituting a highly prospective target for economic gold mineralization. With both fault zones likely being connected at depth, the known occurrence might only represent an outcropping, smaller, lode of a potentially larger ore body.

Due to intense regional alteration, the mineralization-related alteration is ambiguous and mostly difficult to determine. Regional alteration comprises pervasive, intense albitization and moderate carbonatization and sericitization of the matrix minerals. Moreover, thick quartz-albite ± carbonate veins are common in the entire rock package. Despite the intense background alteration, it is possible to attribute a spatial alteration sequence to the ore-forming fluids that is distinctively different from background alteration (Fig. 1).

Additional to the alteration assemblages shown in Figure 1, different types of veins indicate nearby mineralization. Within the intermediate to proximal alteration zone, non-mineralized quartz-pyrite ± albite + carbonate veins are typical, with pyrite commonly being the main constituent of the vein. With decreasing distances to the mineralized body, the thickness and abundance of the veins appears to increase along with anomalous enrichment of gold and tellurium in pyrite. Moreover, magnetite-pyrite-carbonate veins occur in the proximal alteration zone, which can be either barren, anomalous for gold or clearly mineralized. Also, hematite-pyrite-quartz veins with anomalous gold concentrations of 0.3 ppm Au were observed in proximity to the mineralization.



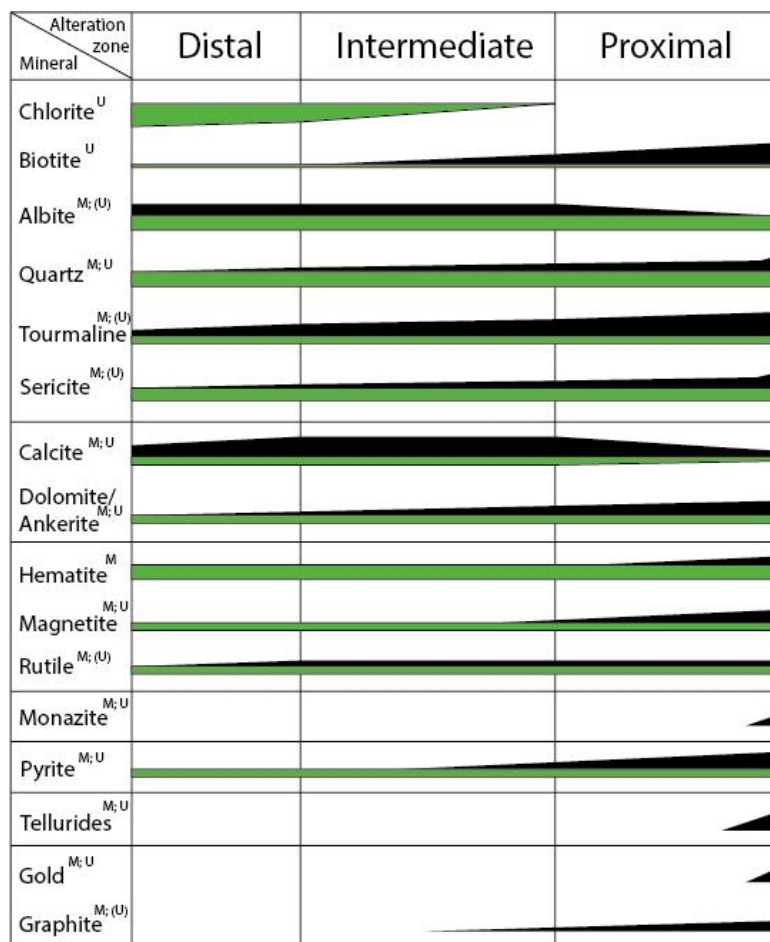


Fig. 1. Schematic overview of the paragenetic alteration sequence around the known mineralization. The letters M and U respectively stand for “Metasediments” and “Ultramafics” indicating in which rock unit the alteration occurs. Black represents alteration that is most likely related to mineralization; green represents regional background alteration. The data is based on outcrop and drill core observations.

The mineralized veins at Mustajärvi consist of quartz, pyrite and tourmaline with accessory muscovite and rare monazite. Quartz is partly strongly deformed and shows multiple recrystallisation features. Pyrite amounts on average to around 25 % of the vein with grain sizes ranging between 100 µm to 1 cm. The amount of tourmaline in the vein varies strongly and ranges from 5 % to as much as 35 %. Tourmaline is spatially closely associated with pyrite. In unoxidized parts gold is hosted by 1-10 µm micro-inclusions of calaverite (Au-telluride) and montbrayite (Au-Bi-telluride) in pyrite. Other associated telluride minerals are the Ni-telluride melonite, the Bi-telluride tellurobismuthite and the Bi-Se-telluride kawazulite. Most telluride minerals occur as a set of parallel strings or in clusters. No spatial relationship between the telluride minerals has been observed. In heavily weathered parts of the mineralization, pyrite is oxidised, resulting in the destruction of tellurides and the subsequent remobilization and precipitation of gold as native Au mainly the cracks of oxidised pyrite. More rarely, also Ag-Au-Se solid solutions exist in the oxidised pyrite, which possibly represent a transitional stage of weathering. Relating to the gold mineralogy, geochemical analyses show that the mineralization is highly enriched in Te, Bi, Se and Co. Other common pathfinder elements, like Ag and As, clearly correlate with gold but are not significantly enriched, W and Sb only correlate weakly and are also only slightly anomalous.

## On-site analysis of metal concentrations of natural waters

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On-site and real-time monitoring of low metal concentrations from waters is still problematic. The laboratory devices remain to be the primary techniques utilized for this purpose. The measurement includes sampling on-site, delivery of the samples to laboratory, preparation of the samples and measurement with inductively coupled plasma mass spectrometer or optical emission spectrometer (ICP-MS or ICP-OES). These measurements are expensive, because of various steps, valuable devices and working hours of educated personnel required. Moreover, real-time information from the metal concentrations of waters cannot be obtained this way, but results must be waited at least for days and typically weeks.

In the present study, novel multimetal analysis method for metal concentrations of water samples was developed. The method is based on nanotechnology enhanced x-ray detection of metals. The core of the technology is a metal collecting filter build from a specific hybrid nanomaterial. The filter has a size of slightly smaller than ten euro sent coin (Fig. 1A). A water sample is pumped through the filter, which collects metal ions from the solution. The metals are concentrated to 1000 – 10000 higher ratio in the filter compared to the original water sample. The metals can be then measured straight from the filter with fast and affordable X-ray fluorescence technique (XRF) and original metal concentration of water can be calculated.

The metal collecting filters were prepared by attaching developed hybrid nanomaterial particles (Riikonen et al. 2018) on a filter support to form about 200 µm permeable layer. Lake water from Kuopio Savilahti bay was pre-filtrated through 0.45 µm filter. After this, lake water was spiked with several different concentration of one selected metal between 50 ppb – 10 ppm and pH was set to about neutral. 10 ml of metal spiked lake water sample was filtrated through metal collector filter with flow rate of 1 ml / min. The metal content of the filters were measured with handheld XRF straight after the filtration from the wet filters. The experiment was repeated three times for each metal concentration and altogether for six different metals including Cu, Zn, Ni, Pb, U and Mn. The metal concentrations of different spiked samples were measured with ICP-MS before the filtration.

The XRF results measured from the wet filters correlate linearly with the real metal concentrations of the water samples (Fig. 1B). The results indicate that the developed method is capable of measuring broad metal concentrations ranges between 10 ppb to 10 ppm with reasonable reliability in field conditions in less than 15 min per sample. The developed method can be used for on-site hydrogeochemical investigations.

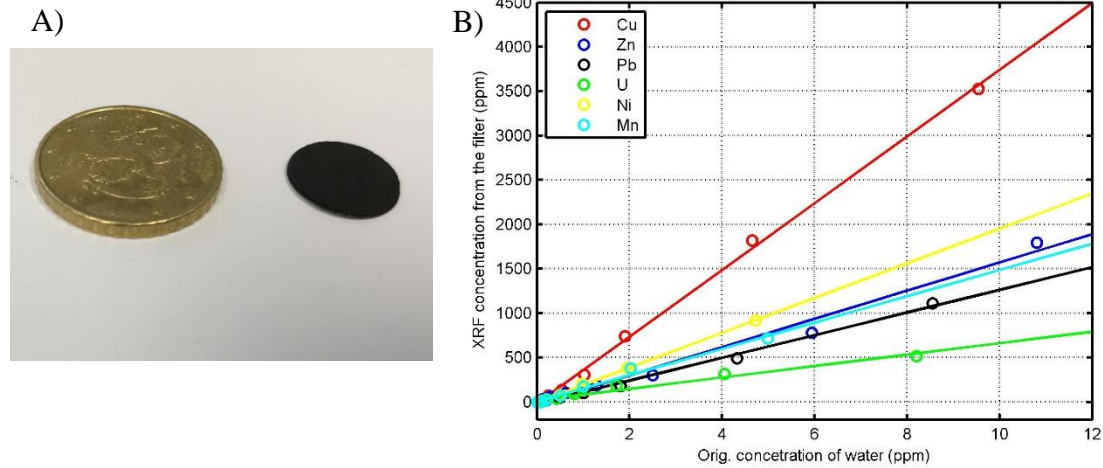


Fig. 1. A) Metal collecting filter made from our nanomaterial compared to ten euro cent coin. B) Metal contents of wet filters measured with a handheld X-ray fluorescence device correlate linearly with original metal concentration of water samples used in filtration.

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## **Applying pXRF analyzer for the soil geochemical baseline studies**

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Portable X-ray fluorescence analyzers (pXRF analyzers) have been used successfully for ore prospecting (e.g. Sarala et al., 2015) as well as for soil contamination and remediation purposes (e.g. Jarva 2016) since the 1990's. However, applying a pXRF for baseline studies is a less studied topic. Although the pXRF works well in soil of high concentrations it is not known how well the method describes soil baseline level concentrations. Baseline concentration refers to naturally occurring concentrations of harmful substances in soil and elevated concentrations occurring in a large area in the surroundings of the suspected contaminated site (Jarva, 2016). The upper limit of geochemical baseline variation can be used in estimating the regional or local baseline concentration for different elements.

Geochemical environmental studies focus mostly on the occurrence of harmful substances in soil and bedrock. The Finnish Government Decree on the assessment of soil contamination and remediation needs (VNa 214/2007) has given threshold and guideline values for As, Cd, Co, Cr, Cu, Hg, Ni, Pb, Sb, V and Zn concentrations in soil. In addition, the Decree gives a possibility to use the regional baseline concentrations for the assessment of soil contamination and remediation needs. The Government Decree (214/2007) refers to both the natural geological background concentrations of elements and the diffuse anthropogenic input with the term "geochemical baseline".

Geochemical mapping and environmental research projects of Geological Survey of Finland (GTK) have tested the functionality and suitability of the Innov-X Systems Olympus Delta 6000 Premium pXRF analyzer for the soil geochemical baseline studies. In this study the total number of samples measured using Soil and Mining+ modes of the analyzer was 1067, number of measurements 1600 and the samples were from 13 different sample batches. The data used in this study were collected in the following projects: the geochemical mapping projects in the towns of Tampere, Heinola and Lahti in 2013 – 2014; Kittilä municipality in 2015; the geochemical baseline mapping of manmade soil in the Helsinki metropolitan area 2013; the ASROCKS project in Tampere, Pirkanmaa and Kanta-Häme region 2011-2014; the Pirkanmaa POSKI project in the Pirkanmaa region in 2015; and the NovTecEx project in Savukoski municipality in 2015. Each sample batch was analysed separately. Measurements were made from soil, bedrock and rock aggregate samples, but only results from soil samples (N=860) are analysed here. The results of the element concentrations measured with the pXRF analyzer were compared with laboratory analyses and the effects of the sample pre-treatment for the pXRF analyzer's results examined.

In Finland, where the soil baseline concentrations of As and some other harmful metals are relatively low the use of the pXRF analyzer for geochemical baseline mapping depends greatly on the elements to be examined as well as the location of the target area. Cr, Cu, Pb, Z and V concentrations in Finnish soils are usually at the level which the Innov-X Systems' Olympus Delta 6000 Premium Analyzers can detect reliably. By contrast, Sb, Cd and Hg as well as Ni baseline concentrations in Finnish soils are too low for the pXRF analyzer to detect. The As concentrations measured in this study are above the pXRF analyzer's detection limits only in the samples taken from the arsenic provinces where the As concentrations in soil and bedrock are known to be naturally high (Hatakka et al. 2016). The

measured Co concentrations are usually above the pXRF analyzer's detection limit but the Co concentrations in this research seem to follow the Fe concentrations very closely. Thus, the pXRF analyzer's Co measuring results should be accepted only with reservations.

In comparison to the soil baseline concentration levels in south and north Europe, the pXRF measuring results are feasible for soil geochemical baseline mapping of Cr, Cu, Pb, Z and V. In addition, in southern Europe the soil baseline concentrations of As and Ni are at the level where the pXRF analyzer results are workable. The pXRF analyzer is not usable for geochemical soil baseline mapping for Sb, Cd and Hg in Europe because of its excessively high detection limits for these elements.

Table 1. Comparison of pXRF measurement results medians for separate sample batches used in this study with pXRF detection limits for several elements reported by manufacturer, median values in Finnish till according to Koljonen 1992, maximum baseline variation values in northern and southern Europe for several elements.

<sup>1</sup> The detection limits for the studied elements using Soil- and Mining + -measurement modes of the Innov-X Systems'n Olympus Delta 6000 Premium –analyzer according to the manufacturer (Railo 2012).

<sup>2</sup> The median concentrations of some harmful elements in the Finnish till (< 0.06 mm grain size, total leach, Koljonen 1992).

<sup>3</sup> The values of upper limits of baseline variation in topsoils (FOREGS dataset, Salminen et al., 2005) in northern and southern Europe based on Tukey Inner Fence (TIF) values.

Element	pXRF / Soil-mode, this study Median min – max mg/kg	Aqua regia leach, +ICP-MS / ICP-OES this study Median min – max mg/kg	pXRF detection limit (manufacturer) mg/kg <sup>1</sup>	Median in Finnish till mg/kg <sup>2</sup>	TIF FOREGS Northern Europe mg/kg <sup>3</sup>	TIF FOREGS Southern Europe mg/kg <sup>3</sup>
N	860	860	-	1057	267-269	561-571
Antimony (Sb)	<DL	<0.05 – 0.34	15–20	0.3	1.3	5.4
Arsenic (As)	<DL – 12.8	0.67 – 15.7	1–3	2.6	-/14.3	27.5/66
Mercury (Hg)	<DL	0.014 – 0.028	2–4	-	0.098	0.346
Cadmium (Cd)	<DL	0.02 – 0.15	12–15	-	0.4	1.8
Cobalt (Co)	5.3 – 23.0	4.1 – 11.6	10–20	14.0	20	66
Chromium (Cr)	42.0 – 115	22.4 – 46.5	5–10	60	109	148
Copper (Cu)	6.5 – 37.0	5.2 – 27.7	5–7	20	47/46	99/108
Lead (Pb)	<DL – 29.0	2.5 – 13.2	2–4	-	22	32
Nickel (Ni)	<DL	10.4 – 23.6	10–20	24	50	159
Vanadium (V)	44.5 – 82.0	25.0 – 58.0	7–15	79	185	147
Zinc (Zn)	27.0 – 81.0	13.8 – 86.5	3–5	57	133	233

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## Vesinäytteenottoa multikopterilla

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Ympäristötutkimuksissa ja -seurannassa vesinäytteenotolla on merkittävä rooli. Vesinäytteenotto ja -mittaukset tehdään tavanomaisesti altaiden tai virtavesien rannoilta, laitureilta, silloilta tai veneestä käsin esimerkiksi Limonoksella tai kuppimaisella näytteenottomella. Eli käytännössä sieltä, minne kussakin paikassa on mahdollista mennä.

Varsinkin kaivosympäristöissä näytteenottopaikat voivat sijaita vaarallisissa ja vaikeakulkuisissa paikoissa. Louhosten reunat voivat olla vaarallisen jyrkkiä, herkkiä sortumille ja toimivilla kaivoksilla liikkuu myös suuria työkoneita, joista on hyvin rajoittunut näkökenttä ympäristöön. Kaivos- ja prosessialtailla voi myös veden laatu ja ominaisuudet vaihdella altaan eri osissa ja syvyyksillä.

Nämä asiat olivat lähtökohtana Drominä-projektin suunnittelussa. Haluttiin kehittää järjestelmiä, joilla parannetaan muun muassa näytteenottajien työturvallisuutta, voitaisi saada edustavampia näytteitä, voitaisi ohjata näytteenottoa sitä edeltävien mittausten perusteella ja joilla voitaisi nopeuttaa näytteenottoa ja mittauksia.

Projektissa suunniteltiin ja toteutettiin kaksi multikopterijärjestelmää. Niin kutsuttu kansalaismalli, jolla kuka tahansa voisi tarkkailla veden laatua esimerkiksi sähkönsäätävyyksillä ja ammattilaismalli, jolla voidaan tehdä mittauksia ja näytteenottoa (Kuva 1.).

Drominä-projektin rahoittajana ovat Euroopan aluekehitysrahasto (EAKR), yritykset ja toteuttajat. Projektin toteuttajaosapuolina ovat Oulun yliopiston Mittaustekniikan yksikkö, Suomen ympäristökeskus (Oulu) ja Geologian tutkimuskeskus.



Euroopan unioni  
Euroopan aluekehitysrahasto  
Euroopan sosiaalirahasto



SAVO-KARJALAN  
YMPÄRISTÖTUTKIMUS OY

Vipuvoimaa

EU:lta  
2014–2020





Kuva 1. DJI Matrice 600 matkalla vesinäytteenottoon Kittilän Pahtavuomassa (kuva Tatu Lahtinen/GTK).

□

## Biochar as dry cover material and greening solution in mine waste areas

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Several mineral waste cover structures and landscaping methods of waste areas will be tested and developed in Finnish Lapland to find effective new solutions that will minimize the costs for the environment. The study will be realized in a three-year project “Utilization of Biochar in dry cover material and landscaping of mine waste areas” (Biopeitto) which is mainly funded by European Regional Development Fund. Project started in June 2017.

Glacial till with variable thickness is the most common material exploited in covering mine waste areas in Finland. However, landscaping is challenging due to extreme grow conditions and heavy erosion in tailings areas and waste rock piles.

Testing biochar, mine wastes and other rest materials as a component of mineral waste cover in tailings and waste rock pile solutions is important both environmentally and as a part of circular economy. Biochar is a natural product that can be produced from several organic raw materials but also for example from wood-based construction waste and composted municipal waste. This supports circular economy and opens new possibilities to increase the local livelihoods. Biochar’s capacity to improve the technical properties of cover materials, such as infiltration capacity, oxygen diffusion and metal absorption will be tested and the optimal mix ratios with the till will be evaluated. Biochar addition could decrease the need of natural till.

The pilot tests are made in laboratory conditions and *in situ* tailings area. In laboratory test ten columns are filled with different cover structure materials (Fig. 1) and watered in a six-month cycle. The infiltrated waters are analysed in the laboratory and also using tXRF (water XRF) method. Measured parameters include also pH, EC, redox, temperature and oxygen.



Fig. 1. Cover structure material testing in the laboratory.



The *in situ* testing is going on in a tailings area at the Rautuvaara Mine in Kolari, northern Finland. The cover tests aim at finding the best ways to improve the sustainability of cover material in water management, carbon balance and nutrient economy, and preventing erosion. The tests include six lysimeters with different cover thickness and composition or no cover at all and measuring sensors (oxygen, moisture, electric conductivity, temperature) (Fig 2.). The infiltration waters will be analysed in a laboratory and pH, electric conductivity, redox, temperature and oxygen content of the water samples are also determined.

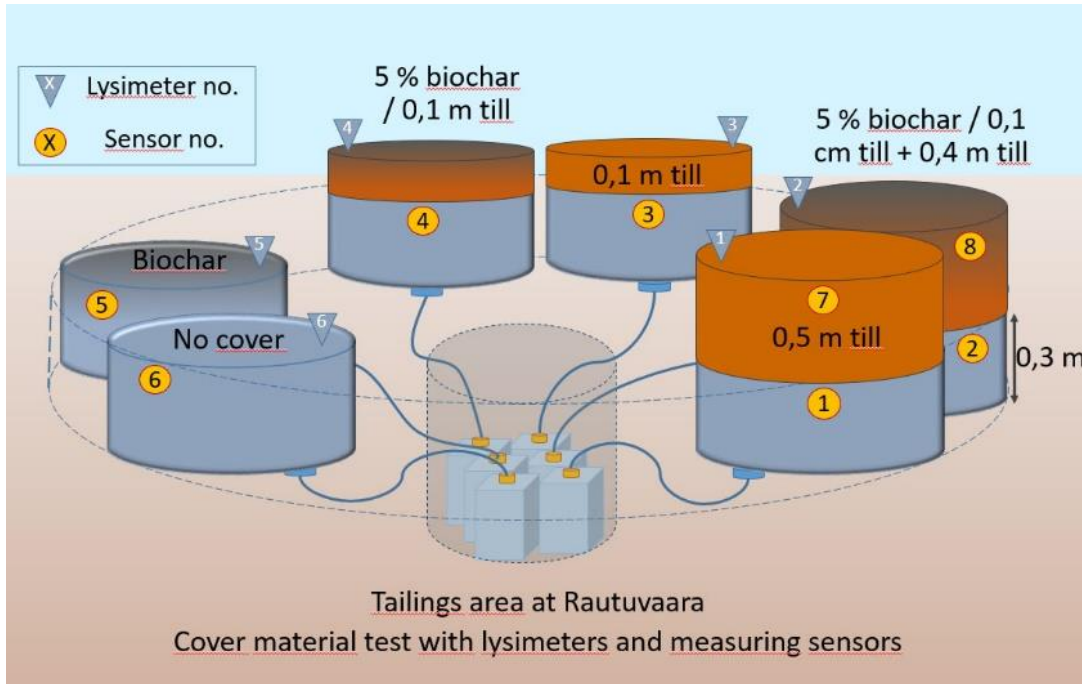


Fig. 2. Lysimeter test at Rautuvaara with different cover structures.

For the landscaping, the best combination of till, biochar and green liquid dredge will be tested in a greenhouse and laboratory conditions. The success of plants in variable cover conditions *in situ* at Rautuvaara is observed (Fig. 3).



Fig. 3. Greening test at Rautuvaara with different cover structures.

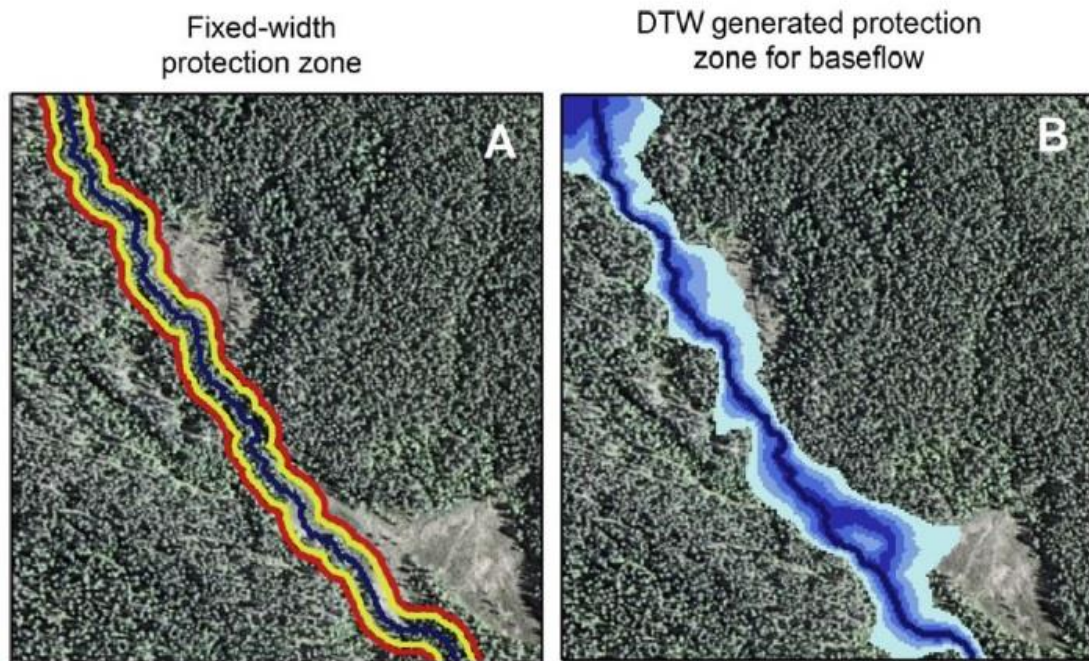
## Maaperän mikrobien vaikutus purojen rantavyöhykkeisiin

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Jokivarsien metsät ja puot ovat biodiversiteetin kannalta tärkeitä ekosysteemejä. Erityisesti sellaiset jokivarsialueet, joilta purkautuu pohjavettä ovat ekologisesti ja biokemiallisesti merkittäviä alueita (Gundersen et al. 2010). Nämä alueet ovat kuitenkin vaarantuneet mm. metsätalouden takia. Hakkuut aiheuttavat alueen lajimäärien laskua ja muutoksia, liettymistä, sekä hydrologisia muutoksia (Bengtsson et al. 2000). Tästä johtuen jokivarsialueiden metsäteollisuuteen tulisi kiinnittää tarkemmin huomiota ja pyrkiä kehittämään alueiden puskurointivyöhykkeitä (Castelle et al. 1994).

Tähän asti jokivarsialueiden hakkuukartoitukset on tehty silmämääräisesti alueella etukäteissuunnittelun sijasta. Tällöin ekologisesti merkittävät kosteat alueet saattavat jäädä tunnistamatta esimerkiksi lumipeitteen takia (Ågren et al. 2014). Kosteat alueet ja vesien virtausreitit voidaan laskea digitaalisilla mallinnusmenetelmillä sillä oletuksella, että topografia kontrolloi veden liikettä ja, että pohjaveden pinta noudattelee maanpintaa (Rodhe and Seibert 1999). LiDAR-aineiston ja topografisen kosteusindexin avulla saadaan määritettyä aluespesifinen suojavyöhyke, joka tarjoaa yksityiskohtaista tietoa kosteudesta ja veden virtausreiteistä. Aluespesifisissä suojavyöhykkeissä korostuvat kosteat pohjavesien purkautumisalueet, kun taas ekologisesti vähemmän merkittävät kuivat alueet ovat kapeampia (kuva 1).



Kuva 1. Lineaarinen suojavyöhyke ja kosteusindexiin perustuva suojavyöhyke.

Postdoc-tutkija Mari Tolkkisen tutkimuksessa selvitetään ovatko Pohjois-Suomen jokivarsimetsät biodiversiteetin hotspotteja ja vaikuttaako metsätalous eri tavalla jokivarsimetsien kuiviin ja kosteisiin alueisiin. Pudasjärven alueelle asetettiin 6 eri puron varrelle teepusseja, joista 3 kuukauden päästä asettamisesta lasketaan TBI:n (tea bag index) mukainen hajotusnopeus. Alueelta on otettu myös maakairanäytteitä, joista analysoidaan

kokonaishiili, ravinteet, sekä määritetään mikrobien DNA. Lisäksi tutkitaan alueen maaperän koostumusta ja sen vaikutusta mm. veden virtaukseen ja mikrobiyhteisöihin. Näiden tutkimusten avulla pyritään selvittämään vastaukset edellä mainittuihin tutkimuskysymyksiin.

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## The portable hyperspectral measurement device Halo and how to use it in exploration.

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Hyperspectral mapping in exploration is used for identifying alteration patterns that can't or is hard to objectively record by visual observations by geologist. The method is non-destructive and can be done on all geological material as long as it is dry. Interpretation of results can be done by automated comparison to spectral libraries of minerals or by manually investigate each spectrum

The instrument described here is a Malvern Panalytical's ASD TerraSpec® Halo, a portable spectrometer instrument. The Instrument can record spectra within the NIR and SWIR range 350-2500 nm. It has an inbuilt library of mineral spectra and can refer captured data with this library to identify the minerals in the sample. The unit also has an inbuilt GPS that can geotag the captured data. The weight of the instrument is 2,5kg.

Data can be exported from the device in simple csv files that includes how well spectra have matched with the internal library. Complete spectra can also be exported as graphs and can for example be imported in the TSG™ (The Spectral Geologist) software for further analysis. The advantage of using is TSG™ is that an intuitive QAQC check can be made of the data like aspectral data caused of very dark samples or noise/artificats in the data. A combination of using TSG™ and ioGAS™ will allow a simple visualization of spatial distribution of different alteration assemblages (Halley 2016).

The three main objectives for capturing spectral data in exploration is: 1. Map mineral occurrence, 2. Map changes in minerals proportions and 3. Map minerals composition and crystallinity. By using these three parameters together with other data like core sample assays it is possible to identify controls on the mineralization or vectors towards mineralized and altered zones.



Fig. 1. ASD TerraSpec Halo Mineral Identifier.

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## Applying heatmap plots in interpreting urban geochemical data in the city of Oulu

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The Geological Survey of Finland (GTK) has carried out soil geochemical baseline mapping since early 2000's. Since 2009, a great number of mapping has been conducted in urban areas (e.g. Jarva 2016). In 2016, GTK made the urban soil geochemical baseline mapping in the city of Oulu, Northern Finland. Total number of 260 soil samples were taken from various land use types from non-contaminated areas. The samples presented different soil parent materials ranging from relatively undisturbed natural sediments to completely man-made soil with variable textures. The sampling depth was 0 – 10 cm under any grass cover. The samples were dried in <40°C and sieved to <2 mm grain size. Concentrations of more than 40 elements were analysed using ICP-OES and ICP-MS measurements after aqua regia extraction. The used methodology followed the EuroGeoSurveys URGE protocol (Demetriades and Birke 2015a, 2015b). Quality control was based on project standard samples, international reference samples inserted in the analytical batches, field duplicates and analytical replicates (Tarvainen and Eklund 2017).

Soil geochemical results are a typical example of compositional data (CoDa) characterized by the fact that they are part of a whole and do not vary independently. Multi-element geochemical dataset should only be analysed in multivariate space. Method is based on the estimated strength of the relationship between any pair of variables and the calculation of correlation coefficients of the geochemical data. All resulting correlations can be represented in a correlation matrix, which can be conveniently visualised by a heatmap (Filzmoser et al., 2010). Summarizing the correlations in a heatmap gives a powerful tool for bivariate data analysis.

Heatmap is a colour coding of the correlations, with different colours for positive and negative correlations. Each cell of the correlation matrix is represented by a square in the heatmap. Like the correlation matrix, the heatmap is also symmetric around the main diagonal. Re-arrangement by clustering the correlation matrix, using hierarchical clustering with distances based on the correlations make the relationships between variables easier to understand and analyse. The same clustering of elements will be on a heatmap. Using symmetrical coordinates the relations between any two variables in the two datasets can be compared and interpreted (Reimann et al., 2017).

Out of the 40 elements analysed, altogether 25 elements (Al, As, Ba, Bi, C, Ca, Cd, Co, Cr, Cu, Fe, K, Mg, Mn, Mo, Ni, P, Pb, S, Se, Sr, Ti, U, V, Zn) delivered results where the majority of concentrations were above their respective detection limits. Data was further analysed with R software using R Studio program.

Heatmap figures were formulated for four soil groups derived from samples taken in the city of Oulu: natural fine-grained sediments, natural coarse-grained sediments, man-made minerogenic soil material and man-made soil material rich in organic matter. Natural fine-grained sediments revealed three strong groups of elements. The first group is related to mica minerals with relatively high concentration of aqua regia extractable Al, K, Mg and Fe. Chromium, C, U, Ti, Ni and Co are strongly bound to this mica group. The second group is dominated by organic matter (C, S). Lead, Bi, Cd, Ba and Zn and partly Cu are bound to organic matter in natural fine-grained sediments (Fig .1). Arsenic and Mo formed the third group that has strong linkage to the mica group. Coarse-grained natural sediments show

similar main groups related as the fine-grained sediments to the dark mica minerals and to soil organic matter.

Similar grouping of elements were found for minerogenic man-made soil material as well. However, in addition to the mica and soil organic matter groups, calcium, P and Sr formed a distinct group that is most probably related to the abundance of apatite mineral or to the application of lime and fertilizers in green urban areas. Man-made organic fillings did not form any distinct groups as the other soil materials. Most of the elements were grouped with soil organic matter (C and S). However, barium, Pb, Sr, Cd, Zn, P, Cu and Bi composed a separate group with unknown origin in man-made soil material.

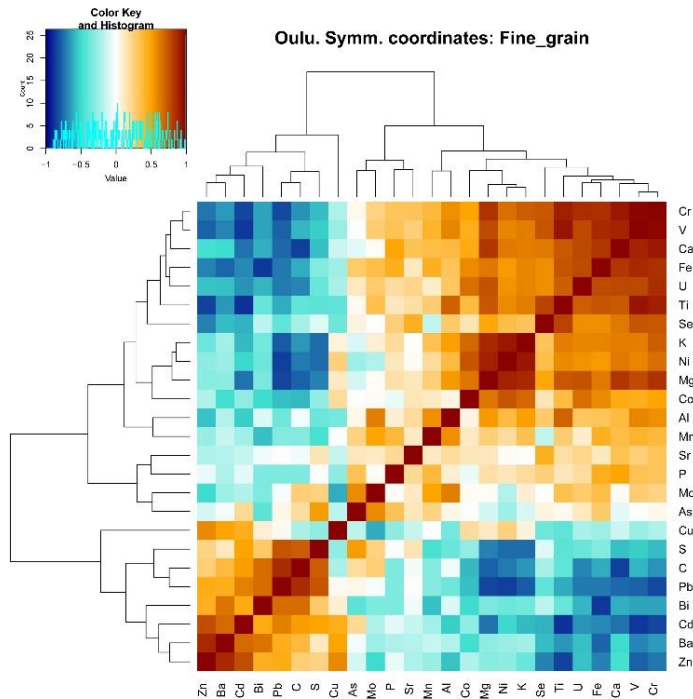


Fig. 1. Heatmap of correlation coefficients based on symmetric coordinates for the natural fine-grained sediments in the city of Oulu. Elements along the axes are sorted according to the results of a cluster analysis. Cluster of elements related to the mica minerals are shown in the upper right corner. Cluster of elements bound to soil organic matter are seen in the lower left corner. The heatmap calculations are based on R functions produced by Peter Filzmoser.

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## **Upscaling deep buried geochemical exploration techniques into European business – UpDeep**

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An aim of the UpDeep project is to develop geochemical expertise in deep buried exploration methodology and anchoring the knowledge accrued as a local surface geochemical business into Europe. Partial and weak leach techniques for soil and biogeochemistry together with a light-weight sampling have solid status in grass roots exploration in other continents, but have as yet gained only minor ground in Europe. Anthropogenic impacts and lack of continuous research are identified as the main factors contributing to their low acceptance. The driving-forces for these extremely low-environmental-impact sampling techniques are increased public environmental awareness and prolonged processes to gain land access permits. UpDeep focuses on the scientific evidencing of surficial geochemical techniques in mineral exploration as a collaboration of the research institutions GTK, BRGM, GEUS, VUT and VTT, and a geo-consulting company GeoPool to promote the use of surface geochemistry across Europe. UpDeep has utilized existing soil and biogeochemical data from Finland and will acquire new datasets over known mineralizations in France and Greenland. The business feasibility and market study will be conducted by Spinverse.

We tackle several technical aspects of surface geochemistry including efficient sampling protocols and sample media selection, the production of surface geochemical standard reference material bank, and compositional data analysis. A web based data analysis and delivery platform will be constructed to facilitate a quick delivery of results and continuous interaction in data interpretation between the geochemical consultant and an exploration company. An educational component in producing learning materials and proving classes for the exploration industry should increase awareness and understanding about the suitability of surface geochemical sampling media in different environments. The general business model will be modular to suit the needs of customers with backgrounds in surface geochemical exploration. UpDeep is a three-year project (2017-2020, <http://projects.gtk.fi/updeep/>) funded by the European Information and Technology Raw Materials.

## Indikaattorimineraalit kriittisten metallien etsinnässä - Indika-projekti

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Nyky-yhteiskunnassa tarvittavien metallien ja mineraalien kirjo kasvaa koko ajan ja lähes koko jaksollinen alkuainejärjestelmä on tarkastelun alla. Kierrätyksestä huolimatta tarvetta on koko ajan löytää uusia metalli- ja mineraaliesiintymiä ja myös etsintäalueet laajenevat entisestään. Kysyntää on yhteiskunnan infrastruktuurin rakentamistarpeesta johtuen eteenkin perusmetalleille, mutta näiden lisäksi etsinnän kohteeksi ovat nousseet erityisesti ns. kriittiset mineraalit, joita on EU:n mineraalistrategiassa nostettu agendalle parisen kymmentä. Tavoite on varmistaa korkean teknologian teollisuuden raaka-ainesaantia vähentämällä riippuvuutta EU:n ulkopuolisesta tuonnista. Tarve ko. mineraaleille ja metalleille on koko ajan suurempi ja omavarainen raaka-ainesaanti on merkittävä kilpailutekijä eurooppalaiselle teknologiateollisuudelle. Suomi ja erityisesti Pohjois-Suomi ovat tässä avainasemassa kallioperämme malmipotentialista johtuen.

Kriittisiä mineraaleja ovat: 1) mineraalit, joita käytetään sellaisenaan teollisuuden raaka-aineina (esim. lannoitteissa käytettävät boraatti ja fosfaattimineraalit sekä grafiitti ja magnesiitti) ja 2) mineraalit, joista saadaan erotettua teollisuuden tarvitsemia alkuaineita ja metalleja, kuten harvinaisia maametalleja (REE), antimonia, kobolttia, magnesiumia, niobia ja platinaryhmän metalleja (PGM) (taulukko 1). Jälkimmäisiä käytetään mm. elektroniikkateollisuudessa, älytuotteissa, katalysaattoreissa ja akuissa. Kriittisten mineraalien omavaraisuuden nosto EU-alueella on lähitulevaisuudessa entistä merkittävämpää.

Taulukko 1. EU:n mineraalistrategiassa kriittisiksi luokitellut mineraalit ja metallit. Lähde EU.

<b>2017 CRMs (27)</b>			
Antimony	Fluorspar	LREEs	Phosphorus
Baryte	Gallium	Magnesium	Scandium
Beryllium	Germanium	Natural graphite	Silicon metal
Bismuth	Hafnium	Natural rubber	Tantalum
Borate	Helium	Niobium	Tungsten
Cobalt	HREEs	PGMs	Vanadium
Coking coal	Indium	Phosphate rock	

Kriittisten mineraalien etsintämenetelmien kehittämiseksi käynnistettiin kesällä 2016 projekti: Indikaattorimineraalien automatisoitu tunnistaminen kriittisten mineraalien etsinnässä (Indika). Etsintämenetelmien kehittämisessä on hyödynnetty uusinta analysointi- ja mineraalien tunnistusteknologiaa ja käytännön kenttätöillä on selvitetty uusien automatisoitujen ja on-site-indikaattorimineraalitutkimusmenetelmien soveltuvuutta kriittisten mineraalien ja metallien etsinnässä. Tavoitteena on ollut kehittää ennen kaikkea tutkimuskonsepti indikaattorimineraaleille sisältäen maaperänäytteiden esikäsittelyn, on-



site-menetelmätestauksen ja laboratoriotutkimuksen, mutta samalla arvioida myös yksittäisten menetelmien ja/tai analysaattorien soveltuvuutta indikaattorimineraali-tutkimuksessa.

Projektissa hyödynnetään ja voimistetaan olemassa olevaa Pohjois-Suomen tutkimus- ja osaamisverkostoa. Projekti on Euroopan aluekehitysrahaston (EAKR) rahoittama ja sen vetovastuu on Geologian tutkimuskeskuksella (GTK). Tutkimuspartnereina ovat Lapin ammattikorkeakoulu (Lapin AMK) ja Oulun yliopiston kaivannaisalan tutkimusyksikkö (OMS). Tutkimusta ovat lisäksi rahoittaneet Suomen kaivosyrittäjät, Lapin kullankaivajain liitto ja Hitachi High-Tech Analytical Science (entinen Oxford Instruments).

Indikassa on tutkittu erilaisia menetelmiä maaperänäytteiden indikaattorimineraalien erottamiseksi, analysoimiseksi ja mineraalien tunnistamiseksi sekä suoraan kentällä että laboratorio-olosuhteissa. Kenttämenetelmiä ovat olleet konsentroiintiin käytettävät Knelson-konsentroiinti ja spiraalirikastus käyttäen ns. kultakoiraa ja mikrovaskausta sekä konsentraattien koostumuksen analysointiin käytetty kannettava XRF (pXRF) ja mineraalien tunnistamiseen kannettavaa XRD-analysointia (pXRD)(Kuva 1.). Lisäksi on selvitetty hyperspektrianalyysiin toimivuutta sekä *in situ* -näytteille että konsentraateille. Laboratoriossa on tehty lisäksi tärypöytää ja raskasnestettä käyttäen raskaimman ja keskiraskaan mineraalifraktion separointia sekä mineraalien automatisoitua tai semi-automatisoitua tunnistamista FE-SEM- ja labraXRD-menetemillä. Lisäksi kokeiltiin myös hydroseparointia. Tutkimuksissa on myös tehty vertailevaa tutkimusta eri raekokojen vaikutuksesta indikaattorimineraalien esiintymiseen näytteissä. Myös pXRF-datan on-line tiedonsiirto on ollut yhtenä kehittämiskohteena digitaalisuuden ja datan nopeamman hyödyntämisen edistämiseksi. Testikohteina ovat olleet tunnetut esiintymät Mäkälässä, Vuotsossa (Au-REE) ja Soklissa, Savukoskella (P-REE) sekä uusi etsintäkohde Mustikkakankaalla, Vuolijoella (zirkoni-REE) ja rikastushiekka-allas Rautuvaarassa, Kolarissa (REE-potentiaali).



Kuva 1. Indika-projektissa testattuja kenttämenetelmiä moreeninäytteiden konsentroidmiseksi ja analysoimiseksi.

Projektin päätuloksena on uusi ja käytännössä testattu sekä dokumentoitu indikaattorimineraalinäytteiden esikäsittely- ja tutkimusproseduuri, jota voidaan soveltaa uusien kriittisten mineraalien malmipotentialisten alueiden ja esiintymien etsinnässä sekä vanhojen esiintymien tai kaivosympäristöissä olevien potentiaalisten selvittämisessä.

Keskeisiä osa-alueita ovat olleet:

- 1) erilaiset näytteiden käsittely- ja rikastusmenetelmät kriittisten mineraalien ja metallien indikaattorimineraalien erottamiselle muusta näyteaineksesta,
- 2) tutkimustiedon tuottaminen ns. on-site analyysimenetelmien soveltuvuudesta kentällä/maastossa tehtävään mineraalinäytteiden analysointiin,
- 3) automatisoitujen mineraalien analysointi- ja tunnistamismenetelmien tutkimusproseduurin kehittäminen,
- 4) pilotoitu tutkimus menetelmäkehityksen tuloksista neljällä testikohteella.

Proseduuri sisältää uusia toimintamalleja maaperänäytteen esikäsittelyyn ja konsentroimiseen sekä automatisoituun mineraalitunnistukseen hyödyntäen edistyneitä analyysimenetelmiä ja digitaalisena tuotettujen tulostietojen on-line siirtoa ja hallintaa. Tulokset ovat hyödynnettävissä erityisesti uusien kriittisten mineraalien esiintymiä etsittäessä ja ne tukevat kiertotalouden jalkauttamista kaivannaisalalla esimerkiksi selvittämällä mahdollisuuksia vanhojen sivukivi- ja rikastushiekkakasojen uusiokäytölle. Selkeitä hyötyjä ovat mm.:

-projekti on lisännyt pohjoisten toimijoiden (GTK, Oulun yliopiston Kaivannaisalan tiedekunta, Lapin AMK ja yhteistyöyritykset) osaamista ja yhteistyötä sekä vuorovaikutusta sidosryhmiin

-se antaa uusia mahdollisuuksia alan nykyisen yritystoiminnan kehittämiseen ja uuden luomiseen. Mm. tätä varten projektin etenemisestä ja tuloksista on tiedotettu aktiivisesti projektin aikana sekä järjestetty työpajoja, joihin alan toimijat ovat voineet osallistua.

- kriittisten mineraalien etsinnässä uusien malmiesiintymien etsinnän lisäksi on olemassa suuri potentiaali vanhoilla kaivosalueilla rikastushiekka- ja sivukivikasoissa. Näiden hyöty- ja uusiokäyttö ovat olleet viime vuosina runsaan keskustelun kohteena ja niiden hyödyntämismahdollisuuksille haetaan ratkaisuja kiertotalouden näkökulmasta.

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## Metallisen kullan esiintyminen moreenissa Iso-Kuotkon alueella

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Iso-Kuotko sijaitsee noin 70 km koilliseen Kittilän keskustasta ja noin 13 km Kiistalassa sijaitsevasta Kittilän (Suurikuusikon) kultakaivoksesta pohjoiseen. Alueen kallioperä kuuluu Keski-Lapin vihreäkivivyöhykkeeseen. Kuotkon alueella vallitsevat pääkivilajeina ovat mafiset laavat ja tynnylaavat. Pyroklastisia liuskeita esiintyy välikerroksina ja felsisiä juoniyksiköitä mineralisaatioiden yhteydessä tai läheisyydessä (Kortelainen, 2012). Kuotkolle ominaista on voimakas tai intensiivinen kemiallinen muuttuminen; albiittituminen, karbonaattituminen sekä etenkin massamaisia tai raitaisia albiittikiviä leikkaaviin Qz-Do juoniin liittyvä kiisuuntuminen (Kortelainen, 2012). Alueen isäntäkivissä on selkeästi esillä hydroterminen muuttuminen ja kivissä esiintyy vaihtelevin määrin arseeni-, magneetti-, ja rikkikiisua. Geologian tutkimuskeskuksen (GTK) aiempien tutkimusten yhteenvedon perusteella jopa 80 % Kuotkon kullasta on vapaata metallista kultaa ja 20 % piiloutuu arseeni- ja rikkikiisujen hiloihin (Härkönen ym., 2000). Kuotkon kiisut ovat pääasiallisesti magneetti-, rikki- ja arseenikiisua. Kiisupitoiset juonet ovat kultapitoisia ja Kuotkon alueelle tyypillisiä (Härkönen ym., 2000). Kullan pitoisuudet ovat epätasaisia, mutta jopa 20-30 ppm pitoisuuksia esiintyy esim. Kati-esiintymän yhteydessä olevissa kapeissa juonissa (Kortelainen, 2012). Kittilän ryhmän mafisten vulkaniittien yhteydessä ja Kuotkon alueella esiintyy erottuvia tektonisia suurrakenteita, hirtovyöhykkeitä, ruhjeita ja ylityöntöpintoja (Kortelainen, 2012).



Kuva 1. Iso-Kuotkon RM-tutkimukset 2018.

Iso-Kuotkon kaivospiirillä tehtiin kesän 2018 aikana raskasmineraalitutkimusta, jota varten suunniteltiin 82 pistenäytettä ja 14 uutta tutkimusojaa (Kuva 1). Kerättyjen näytteiden koko oli 10 litraa moreenia tai moreenin sekaista rapakalliota. Tutkimusojista kerättiin noin 10 näytettä per oja. Tulokset pistenäytteistä ovat olleet hyvin vaihtelevia. Parhaasta näytteestä löytyi yli 2300 kultaraetta, kun taas joistakin vain yksittäisiä kultarakeita keskiarvon ollessa n. 70 raeetta/näyte. Paras ja huonoin näyte on jätetty keskiarvosta pois.

Kultarakeiden lukumäärät on saatu Knelson-konsentroidin sekä mikrovaskauksen tuloksena (taulukko 1).

Taulukko 1. Kultarakeiden määrä osassa tutkituista näytteistä.

Sample	Gold grains	Type	Remarks
FIEXC101756 (RM 34)	41	MR	rich in hematite, few brown garnet grains, iron oxide glow as blue under uv-light- scheelite, plenty qz, amphibole and mica grains
FIEXC101757 (RM 35)	5	MR	rich in fine to coarse grain hematite and magnetite, chlorite rich pebbles
FIEXC101759 (RM 64)	20	MR	15 scheelite grains, half of fraction is mafic volcanite, plenty of felsic rock grains, mostly feldspar
FIEXC101758 (RM 72)	3	MR	rich in silt sized iron oxides, hematite and magnetite, pebbles rich in chlorite, few felsic rounded feldspar pebbles
FIEXC101760 (RM 73)	163	MR	much fine gold under 0,03mm, granular and plated, clear vein quartz, hydrothermally altered mafic volcanite, magnetite and hematite mostly silt size grains

Osasta näytteissä tavattiin selkeästi pyörityneitä kultarakeita, mikä viittaa kullan kulkeutumiseen jäätiköiden ja virtaavan veden vaikutuksesta. Pääasiallisesti rakeet ovat kuitenkin särmikkäitä ja pitkänomaisia. Kultarakeiden määrä maa-aineksessa on korkeimmillaan ylärinteillä, missä rapakallio on aivan pinnassa ja kultarakeet ovat paikallista alkuperää. Kuotkon tutkimusalueen pohjois- ja eteläosissa esiintyy pieniä määriä pyörityneitä kultarakeita, mutta suurin osa kullasta on kuitenkin teräväsärmäisiä, paikallisia tai vain lyhyen matkaa kulkeutuneita. Pohjoisen graniittialueen vaikutus näkyy selvästi lisääntyneenä graniittisena materiaalina sekä hiekkojen runsaana esiintymisenä, eteläisissä osissa moreenin kivet ovat pyörityneempiä ja erillisiä hiekkakerroksia ei ole. Varsinaisista geokemian näytteistä vasta pieni osa on kirjoitushetkellä lähetetty analyysiin näytteenoton vielä jatkuessa. Tulokset varmentuvat viimeistään marras-tammikuun välillä. RM-tutkimus on osoittanut soveltuvansa paikalliseen tutkimukseen.

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## **Geochemistry and U-Pb zircon age of the Pha Aok Granite, SE Cambodia**

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The Pha Aok Granite is a coarse grained, light grey to white-yellow granite intrusion that crops out as two large hills near the town of Kiri Vongs, Takeo Province, SE Cambodia. The intrusion has a sub-rounded shape with a diameter of approximately 10km. The granite is quarried commercially for dimension stone, while pegmatites are exploited on an artisanal scale for gemstones, primarily aquamarine. The major mineral phases are K-feldspar (as microcline), plagioclase, quartz, and biotite, with accessory apatite and zircon. Muscovite occurs mainly as a secondary alteration product within feldspars; however occasional primary muscovite is also present.

Major and trace element concentrations were determined at the University of Stellenbosch using a combined XRF/LA-ICP-MS procedure. The granite is rich in silica (73-77% SiO<sub>2</sub>) and alkalis (avg. 4.74% K<sub>2</sub>O; 3.62% Na<sub>2</sub>O) and extremely depleted in iron (avg. 1.14% FeOT) and magnesium (avg. 0.03% MgO). Chondrite normalised REE patterns show a slight LREE enrichment, flattening out in the MREE to HREE with a strong negative Eu anomaly. Spider diagrams show negative Ta-Nb-Ti spikes as well as relative depletion in both Ba and Sr.

Zircons were separated from two samples and were analysed for U-Pb compositions using LA-ICP-MS at the Geological Survey of Finland, Espoo. The first sample resulted in a consistent, concordant population from which an age of  $76.4 \pm 0.6$  Ma (n=13) was calculated. The second sample proved more problematic, but a concordia age of  $73.8 \pm 0.8$  may be calculated from 9 data points. However, these 9 data points fall into two distinct groups: one of which gives  $75.4 \pm 1.1$  Ma (n=4) while the other gives  $72.5 \pm 0.9$  Ma (n=5). No geological reason for the age discrepancy between the two samples can be determined, so the result from the first sample is considered to be more robust and representative of the intrusion age. The "older" group from the second sample is within error of the result from the first sample, while the "younger" group could have been subjected to alteration, perhaps from a long-lived magmatic-hydrothermal system after the intrusion event.