

# Application of the portable X-Ray Diffraction (pXRD) analyser in surficial geological exploration

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**D**evelopment of on-site analysis techniques for geochemical and mineralogical research in mineral exploration has been intensive in the recent years. Portable X-ray fluorescence analysers (pXRF) have become one of the key instruments in the geochemical field analyses for several type of ore types and sample materials, as pointed out for example by Fisher *et al.* (2014), Gazley *et al.* (2014), Hall *et al.* (2014), Sarala (2016) and Sarala *et al.* (2015). Hyperspectral techniques have also been developed in mineralogical identification of different rock types or glacial till (Clark 1999, Middleton *et al.* 2011, Turner *et al.* 2014) and there are portable, hand-held analysers available for on-site use.

X-ray diffraction (XRD) is a conventional method for mineralogical research. It is based on crystal constructive interference of monochromatic X-rays and a crystalline sample following the Bragg's Law. Each crystalline material has a characteristic atomic structure which is seen as a diffraction of X-rays in a unique and characteristic way. XRD is good particularly for the detection of small mineral grains, such as clay and a fine fraction of till. XRD analyses are done in laboratory after pre-processing of samples (drying, pulverizing and briquetting). However, the portable XRD analyser (pXRD) has been available for on-

site analysis only for a short time. The first commercial pXRD that use a charge-coupled device (CCD) based camera was launched at the beginning of this century. This device was developed together with the project "CheMin XRD/XRF" for Mars research and was funded by NASA (Sarrazin *et al.* 2005, Blake *et al.* 2012).

Different on-site analysers and advanced automated indicator mineral techniques in critical mineral exploration are studied in the project "Automated indicator mineral identification methods for the critical mineral exploration" (Indika) which was initiated in 2016 (Geological Survey of Finland 2017). The project aims to develop a research concept for critical minerals in glaciated terrain including (1) surficial sediment and weathered bedrock sample pre-processing and indicator mineral concentration techniques, (2) on-site mineralogical analysis and/or identification techniques and (3) automated indicator mineral identification using laboratory methods. The project is led by the Geological Survey of Finland (GTK) and research partners are the Lapland University of Applied Sciences and the Oulu Mining School (OMS) at the University of Oulu. The project was funded by the European Regional Develop Funding (ERDF), professional associations and industry sector companies.

In the Indika project, Olympus Terra pXRD was tested at the first time in Finland. pXRD was used for testing critical mineral identification in two study areas in northern Finland (Fig. 1); in the Mäkärä area (Au-REE occurrence), northern Sodankylä, and in the Sokli area (P-REE deposit), northern Savukoski. Both study areas include known mineralizations for the critical metals. Here we introduce the analyser, describe the test arrangements and field concentration techniques (spiral and Knelson concentrators and micro panning) used in the field and show the first

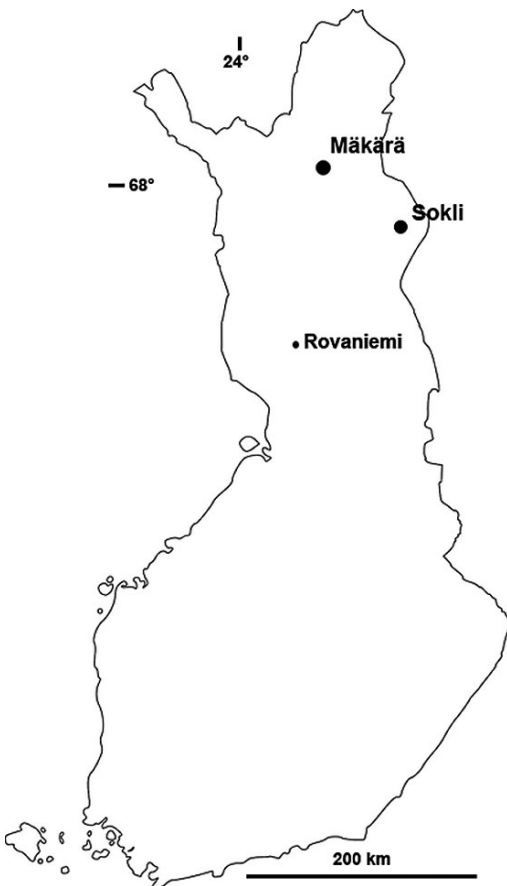


Figure 1. Location of the study areas in northern Finland.

Kuva 1. Tutkimusalueiden sijainti Pohjois-Suomessa.

results on the usability of pXRD analyser in on-site analyses using different materials (weathered bedrock and till). The results are compared to laboratory XRD results and Field Emission Scanning Electron Microscope coupled with Energy Dispersive X-ray Spectroscopy (FE-SEM+EDS) results.

## Study areas and methods

### Mäkärä, Vuotso

The Mäkärä study site has been explored for several decades, lately since 2009 (Sarapää and Sarala 2013). It is included in the Tana Belt in northern Finland that is one of the REE and Au potential regions in northern Europe. It has prominent lanthanum (La) and yttrium (Y) anomalies in regional surficial and lithochemical datasets. High Y indicates the enrichment of heavy REE in the bedrock (Sarapää *et al.* 2013, Sarapää and Sarala 2013). Deep pre-glacial weathering (saprolite-type) and Au content in the weathered bedrock and glacial till have a strong positive correlation with the positive electromagnetic anomalies. The highest La and Y contents in till correlate well with the maxima of the radiation datasets (caused by thorium). The wall rocks are composed of hornblende and arkosic gneisses. Exploration studies revealed a couple of kilometres long Au-hematite-quartz vein system with the best sections containing 3 ppm Au and 0.04–0.1 % REE on average. Those REE contents are correlative with the ionic adsorption clays in China. Typical REE minerals are monazite, rhabdophane, xenotime and kaolinite, but also allanite, bästnasite, euxenite, columbite and zircon were observed. Main minerals are hematite and quartz as well as Fe-hornblende, albite and muscovite, and as minor minerals chlorite, ilmenite and several types of garnets (Lehtonen *et al.* 2011, Sarapää and Sarala 2013).

## Sokli, Savukoski

The Sokli carbonatite intrusion is located in eastern Finnish Lapland and is a part of the Kola alkaline province, which is Devonian, ca. 360–380 Ma old (Kramm *et al.* 1993). The carbonatite intrusion is a funnel-shaped, multistage pluton 5 km in diameter. It consists of a magmatic carbonatite core and is surrounded by metacarbonatite and a wide fenite aureole (Vartiainen 1980, 2001). The intrusion hosts a huge phosphate ore deposit presently owned by Yara Finland Ltd. Furthermore, the deposit is enriched in niobium (Nb), tantalum (Ta), zirconium (Zr), uranium (U) and REE (Korsakova *et al.* 2012). In addition, GTK has done exploration work in Jammi, the southern Sokli area (4 km south of the core of the Sokli carbonatite complex) concentrating on the fenite aureole and late-stage carbonatite dikes that cross-cut the fenite (Sarapää *et al.* 2013). The bedrock of the Jammi area consists of Archean mafic volcanic rocks and tonalitic gneisses which were intensively fenitized. The main REE-rich carbonate minerals recognized in the Jammi carbonatite veins are ancylite and bastnäsite. They are strongly enriched in light REE, P, fluorine (F), strontium (Sr) and barium (Ba). Apatite occurs as large and elongated grains, closely associated with baryte and monazite. In addition, rhabdophane, francolite, pyrochlore, baddeleyite and titanium-zinc-strontium bearing minerals have been observed as well as goethite, calcite, tremolite and phlogopite (Sarapää *et al.* 2013).

## Materials and sampling

For testing pXRD, indicator mineral sample material was produced in the field from the Mäkärä and Sokli sites during 2017. The sample material was collected from the pre-existing test trenches and pits and new, tractor excavated test pits. At the Mäkärä site, the first

sampling line using old trenches was placed over the known Au-hematite-quartz vein to collect a sample (sampling interval ca. 5 metres) from the weathered bedrock surface. In addition, till samples were collected on the distal side, i.e. in down-ice direction of the mineralized bedrock. New test pits were used to collect new weathered bedrock and till samples along the known mineralized structure in the bedrock. There were a couple of observations of the white and green clay veins in weathered bedrock in the test trenches and those materials were also sampled for detailed study and to test pXRD. At the Sokli site, new samples were collected from the old test quarries and their piles at the centre of carbonatite massif. Particular interest was placed on the areas having enriched Nb contents in the weathered ore body. Furthermore, a few till samples were gathered using hand-made test pits in the southern fenite zone in the places, where there are known, elevated REE contents in till based on the GTK's earlier sampling programs.

The sample size for the indicator mineral samples was twelve litres (i.e. 20–25 kg) and 2–3 duplicates were collected for each sample. Both weathered bedrock surface and till (C-horizon) samples were collected in the field and after removing bigger stones (ca. > 2 cm) the samples were placed in plastic bags. Sampling points were mainly chosen based on pre-existing knowledge of the elevated critical minerals/metal contents in the weathered bedrock and till using old and new tractor excavated test trenches, and new hand-made test pits. Coordinates, field observations and sample information were collected directly in the field using a field computer and the GTK's geodatabase interface.

## Field concentration

For the field sample processing, one sample

bag (12 l) per each sampling points was homogenized and divided into two parts. Before pre-processing, the natural samples were analysed using the pXRF analyser. Then both subsamples were wet sieved < 2 mm size fraction using iron sieves. The first subsample was concentrated by a spiral concentrator (also called a 'Gold hound') and the second subsample using a Knelson concentrator (Fig. 2). The concentrator was a two-phase process where in the first phase the heaviest fraction was concentrated and with the second phase for separating the mid-heavy fraction. However, all the fractions included also a lighter mineral fraction which was removed by micro panning. The micro-panned samples were then dried using Bunsen burner before sieving into the size fractions of under and over 150 µm.

## Portable XRD

The Olympus Terra portable XRD/XRF instrument (pXRD; Fig. 3) has been developed for field use. There are no moving parts in the system. The basic difference between the portable instrument and the one used in laboratory is that the sample is moving as suspension, not the parts of the instrument. Thus no sample preparation like powdering is required for the analysis on the portable instrument. Phase identification is based on comparing the diffraction pattern of the sample with the database of XRD mineral patterns. XRF data can be used as a complementary tool in mineral identification, especially when it comes to rare minerals or minor phases.

The instrument weighs 14.5 kg and it has a 1024×256 pixel 2D Peltier-cooled Charge-



Figure 2. (a) Spiral concentrator, also called the "Gold hound". (b) Knelson separator. Photos: P. Sarala (a) and H. Koskinen (b)

Kuva 2. (a) Spiraalikonsentraattori, joka tunnetaan myös nimellä "kultakoira", (b) Knelson-konsentraattori. Valokuvat: P. Sarala (a) ja H. Koskinen (b).



Figure 3. Olympus Terra portable XRD/XRF instrument wirelessly connected to field laptop. Photo: H. Koskinen

Kuva 3. Olympus Terra kannettava XRD/XRF-analysaattori kytkettynä langattomasti maastotietokoneeseen. Kuva: H. Koskinen.

Coupled Device (CCD). It operates with Co radiation within a temperature range from  $-10\text{ }^{\circ}\text{C}$  to  $35\text{ }^{\circ}\text{C}$ . There are four batteries with field autonomy of four hours. The instrument has a patented Vibrating Sample Holder (VHS) to obtain random orientation of mineral crystallites. The sample particles should be less than  $150\text{ }\mu\text{m}$  and the minimum sample size is 15 mg. The data collection range of 2 is from  $5$  to  $55^{\circ}$ , with increment of  $0.25^{\circ}$ .

The sample material should be sieved  $< 150\text{ }\mu\text{m}$  size fraction before placement in the sample holder. That fraction was not powdered before analysis. The oversize grains should be crushed, otherwise they cause a bad diffraction pattern. In the research, the material was divided into sizes under and over

$150\text{ }\mu\text{m}$  when different grain size fraction was analysed. The material should still be coarse enough to move properly in the vibrating sample holder. The powdered particles may stick to each other and to the sample holder window.

One variable to test was the data collection time. The longer the time, the more reflections will be produced and the better the signal-noise ratio. However, the time for analysing one sample is limited in the field circumstances, so the aim was to define a data collection time producing an accurate enough pattern. Burkett *et al.* (2015) have tested pXRD with data collection times of 5, 10, 20 and 40 minutes. The results indicated that even 5 minutes was enough for a qualitative analysis of all phases

whereas the accuracy of the quantitative analyses concerning the minor mineral phases got significantly better the longer the data collection time was. In this trial, the analysing time varied from 10 to 20 minutes.

The data obtained with Olympus Terra pXRD was interpreted with the X Powder software based on the Reference Intensity Ratio (RIR) method and using the AMCSDB database (American Mineralogist Crystal Structure Database) consisting of minerals only.

Reference analyses were done in the GTK's mineralogical laboratory using Bruker XRD instrument and *diffrac.eva* software and in the Centre of Microscopy and Nanotechnology at the University of Oulu, using Rigaku SmartLab XRD and PDXL2 software. For the laboratory XRD, the samples (under and over 150 µm size fraction) were powdered before analyses.

## Test results

By following the sample pre-processing and analytical procedure described in the method section, about 25 samples were analysed using pXRD. The results show that common rock forming minerals like albite, amphiboles, feldspars, magnetite, muscovite and quartz are detected using pXRD. In addition, typical heavy and/or indicator minerals for both study areas can also be identified both in till and weathered bedrock samples. At Mäkärä, minerals like almandine, hematite, goethite, monazite, xenotime and zircon were observed, as well as chlorite, illite, kaolinite and vermiculite in some clay-rich weathered bedrock samples. At Sokli, typical indicator minerals were apatite, biotite, chlorite, columbite, hematite, ilmenite, magnetite and pyrope. Some indications were also made of baddeleyite, pyrochlore, rhabdophane, xenotime and zircon.

Comparison to the laboratory XRD analyses has been started and so far we have only preliminary results. Here we introduce the analytical results only for one sample from Mäkärä and one from Sokli.

The first sample was a Knelson-separated, micro panned, < 150 µm fraction of hematite-rich till from Mäkärä. Minerals identified with pXRD and X Powder software included hematite, magnetite, albite, microcline, ilmenite, richterite and quartz. Most of the minerals were the same also when identifying with the laboratory XRD analysed in the GTK's laboratory in Espoo, except richterite that was suggested as magnesio-hornblende and talc that was not identified by pXRD. Zircon was strongly suggested by X Powder. Ilmenite did not come out as strong although its peak was clear in the lab-based analysis and also the FE-SEM results show a 10 % proportion of ilmenite. Muscovite came out strongly with X Powder but not at all in the lab analysis and only a small amount on FE-SEM. Kyanite, topaz and xenotime were minerals only suggested by X Powder. The results from the different instruments are in Table 1.

The over 150 µm fraction of the same sample was analysed with Rigaku SmartLab XRD and PDXL2 in Oulu University. The sample was powdered. The result of the analysis suggested 50 % of hematite, 17 % magnetite, 13 % quartz, 16 % hornblende, 3 % cordierite and 3 % almandine. X Powder did not suggest almandine at any point. The amount of hematite was estimated at 6 % and magnetite at 3 % whereas the proportion of amphibole was as high as 40 %. X Powder also suggested both albite and plagioclase which did not come out in the laboratory XRD analysis.

The second example was from the Sokli study area. The till sample was Knelson-

Table 1. Comparison between pXRD, lab based XRD and FE-SEM, till sample (< 150 µm) from Mäkärä.

*Taulukko 1. Mäkärän moreeninäytteen (< 150 µm) kannettavan XRD:n, laboratorio-XRD:n ja FE-SEM-tulosten vertailu.*

8.1. Knel1 < 150 µm	pXRD %	Lab XRD	FE-SEM %
Hematite	3	+	Hem + Mgt = 23
Magnetite	2	+	
Quartz	6	+	13
Albite	11	+	15
Amphibole	28	+	16
Muscovite	14	-	1
Ilmenite	1	+	10
Plagioclase	13	+	5
Kyanite	13	-	-
Topaz	5	-	-
Xenotime	1	-	-
Monazite	1	-	0.18
Zircon	2	-	1
Talc	-	+	2
Almandine	-	-	2

separated, micro panned, < 150 µm fraction from the centre of Sokli carbonatite massif. The pXRD and X Powder identified minerals like apatite, magnetite, chromite, some ilmenite and a large amount of amphibole. Apatite was on the list but not among the first suggestions, even though the FE-SEM results confirm the biggest percentage for it. Chromite came out strongly but FE-SEM did not indicate it. Baddeleyite and richterite were on the top of the suggestions list. Also, pyrochlore and rhabdophane were identified which are supposed minerals for the Sokli type deposit, but those were not identified by the laboratory XRD and FE-SEM. The laboratory XRD in GTK lab gave some different minerals such as vermiculite and Mg-hornblende. Clay minerals like vermiculite were not suggested by pXRD and FE-SEM. No chlorite was found in either of the XRD analyses, only with FE-SEM. The results for the Sokli sample are in Table 2.

## Observations during the testing

The accuracy and reliability of the results are the most important factors when evaluating the usefulness of the device. According to a previous study, pXRD can detect, for example, hydrothermally modified mineral fissures, which is important for exploration, as it is possible to track the conditions of the formation of the ore deposits or the geothermal system. However, it is not possible to reliably identify minerals that account for only a few percent of the sample. Also, poorly crystallized and amorphous materials, typically in secondary mining waste, for example, produce poor diffraction results.

There are many reasons for the different results between the analysis methods. Among them are the database used, the different techniques that the methods are based on, and the different sample preparation protocols.

Table 2. Comparison between pXRD, lab based XRD and FE-SEM, till sample (< 150 µm) from Sokli.

12.1. Knel1 < 150 µm	pXRD %	Lab XRD	FE-SEM %
Hematite	2	-	Hem + Mgt = 41
Magnetite	7	+	
Amphibole	46	+	1
Ilmenite	2	+	7
Apatite	12	+	40
Chromite	8	+	-
Pyrope	7	-	-
Baddeleyite	1.5	-	0.08
Monazite	-	-	0.6
Zircon	1	-	0.3
Pyrochlore	2.5	-	0.5
Chlorite	-	-	4
Vermiculite	-	+	-
Columbite	2	-	-
Rhabdophane	0.6	-	-

With the pXRD, the diffractograms were sometimes not really clear. There were lots of peaks where could not say whether it is a real peak or background. The variables affecting the diffractogram are the sample material and its amorphous or metamictic content, grain size, the concentration and the sample preparation, and also, whether it is powdered or not. With the background subtraction option you can lower the amorphous percentage in the quantitative statistics of the diffractogram but that does not help much with the

problems related to the sample. In Figs 4 and 5, the difference between raw data and background subtracted data can be seen.

Numerous peak-like occurrences lead to a long list of mineral suggestions by the XPowder program. The interpretation depends on the program user, so there is a strong human aspect involved. Moreover, the standardization of the interpretation is not simple since the elemental content in one mineral changes even if the mineral name stays the same. In the natural samples not all the minerals identified

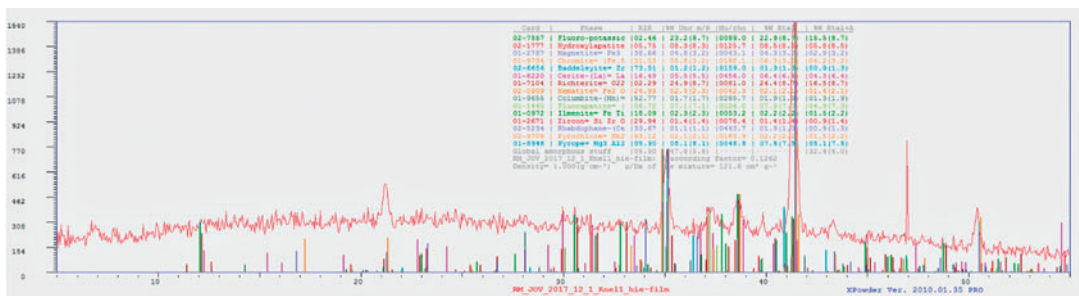


Figure 4. The diffractogram without background subtraction. The Sokli sample, analyzing time 15 min. *Kuva 4. Soklin moreeninäytteen XRD-difraktogrammi ilman taustakorjausta; analysointi-aika 15 min.*





Figure 5. The same diffractogram as in Fig. 4, but with the background subtraction.

Kuva 5. Sama diffraktogrammi kuin kuvassa 4, mutta taustakorjattuna.

having the same name produce similar peaks and thus the different mineral cards from the database are suggested. A readily defined standard mineral assemblage would not necessarily fit into the peaks of a particular sample.

The rare minerals and the ones with very small proportion of the sample do not come out clearly with the XRD technique. It helps if the list of presumable minerals are known, for example on the basis of XRF measurements or former research, but in any case the results are not reliable enough without some complementary methods. For example, according to the XRF results the sample from Sokli presented in Table 2 contains considerable amounts of Y and Sr, but no minerals containing these elements were identified with the other methods except pXRD/XPowder (rhabdophane containing Y).

Some of the rare minerals tend to be metamictic, having weakly bonded cation and anion groups, for example zircon, thorite, allanite, gadolinite, pyrochlore, euxenite, Nb-tantal, etc. Those are hard to identify with XRD because their crystalline structure is destroyed. Recrystallizing in as high temperature as 1000° C would transform them into another minerals. Thus the original mineral could become possible to track (Lima-De-Faria 1964).

The experience from testing the pXRD

shows that the most of the main minerals are identified with the method. The minor minerals, for example those containing REE, can be found by concentrating the samples carefully. Using XRF results and knowledge of the mineralogy of the sample location also helps in mineral identification. However, the percentages given by XPowder software are not always reliable. Interpreting the diffractograms requires some experience and mineralogical knowledge – the program does not give fixed results but only suggestions based on a mineral diffractogram database. When it comes to the field usability of the pXRD, it takes some time to prepare the samples and analyse them. This is why the method is suitable for locations with some mineralogical data available, so that you can choose a moderate amount of samples for analysis. However, the experience with pXRD was encouraging and the analyser has a lot of potential for example as an on-site analysis in mineral exploration and mineralogical work using different materials.

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## Tiivistelmä:

### *Kannettavan röntgendiffraktio-analysaattorin (pXRD) sovelta- minen maaperägeologiseen malminetsintään*

Nykyaikaiset kenttäanalysointilaitteet, kuten kannettava XRD (pXRD) ja XRF (pXRF), antavat hyvän mahdollisuuden maa- ja kallioperän geokemialliseen ja mineralogiseen tutkimukseen suoraan kentällä yhdessä kenttäkonsentroitimenetelmien kanssa. Näitä tukevat modernit elektroniopiset analyysitekniikat, joilla mineraaleja voidaan tunnistaa automatisoidusti. Näillä menetelmillä malminetsintäprosessia voidaan nopeuttaa ja säästetään analyysi- ja näytteenkäsittelykustannuksissa. Tässä kirjoituksessa kerrotaan uudesta kannettavasta XRD-analysointilaitteesta ja annetaan ensimmäisiä tietoja laitteen toimivuudesta kriittisten mineraalien etsintätutkimuksessa, jota toteutetaan menossa olevassa Indika-projektissa (EAKR-projekti). Testattava laite on Olympuksen Terra pXRD, jota on käytetty kahdella Pohjois-Suomessa sijaitsevalla etsintäkohteella: Mäkälässä, Vuotsossa (Au-REE etsintäkohde) ja Soklissa, Savukoskella (P-REE-esiintymä). Ensimmäiset pXRD-tulokset moreenista ja rapakalliosta osoittavat, että analysointilaitteella voidaan tunnistaa kohtalaisen hyvin sekä päämineraaleja että indikaattorimineraaleja, jotka ovat tyypillisiä testauksen kohteena olleille mineralisaatioille. Lisäksi tulok-

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sille on nähtävissä korrelaatioita sekä laboratorio-XRD että FE-SEM-tuloksissa. Tulosten valossa kannettava XRD vaikuttaa käyttökelpoiselta menetelmältä ns. on-site mineralogiiseen tutkimukseen ja malminetsintään.

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