

Indicator minerals analytical techniques

Part I: Identification

Marja Lehtonen GTK



Indicator minerals

- Indicate the presence of a mineralization
- Visually and chemically distinct (heavy minerals)
- Chemically and physically resistant
- Target depended
- In Finland conventionally applied to gold and diamond exploration, but also several other targets (base metals, critical metals etc.)
- Sample media usually till
- Several indicator mineral projects carried out at GTK (Hightech metals, NovTecEx, INDIKA, MinExTarget)



Photo: Kari A. Kinnunen

Commonly used indicator minerals (McClenaghan, 2013) (mod).

Commodity / Deposit	Indicator minerals	Chemical composition	Average density (gcm ⁻³)	Typical size range (mm)
Diamond ¹	Cr-pyrope garnet	(Mg,Fe) ₃ (Al,Cr) ₂ (SiO ₄) ₃	3.7	0.25-0.5
	Eclogitic garnet	(Fe ⁺⁺ ,Mg) ₃ Al ₂ (SiO ₄) ₃	4.0	0.25-0.5
	Mg-ilmenite	(Fe ⁺⁺ ,Mg)TiO ₃	4.7	0.25-0.5
	Cr-diopside	CaMg(Fe,Cr)Si ₂ O ₆	3.3	0.25-0.5
	Chromite	(Fe ⁺⁺ , Mg)(Cr,Al) ₂ O ₄	4.8	0.25-0.5
	Forsteritic olivine	(Mg,Fe) ₂ SiO ₄	3.3	0.25-0.5
	Diamond	C	3.5	0.25-0.5
Gold ²	Gold	Au	17.6	0.01-0.25
	Scheelite	CaWO ₄	6.0	0.01-0.25
	Rutile	TiO ₂	4.3	0.01-0.25
	Sulphides		>4.0	0.01-0.25
Magmatic Ni-Cu-PGE ³	Cr-diopside	CaMg(Fe,Cr)Si ₂ O ₆	3.3	0.25-2.0
	Forsteritic olivine	(Mg,Fe) ₂ SiO ₄	3.3	0.25-2.0
	Enstatite	(Mg,Fe) ₂ Si ₂ O ₆	3.2	0.25-2.0
	Chromite	(Fe ⁺⁺ , Mg)(Cr,Al) ₂ O ₄	4.8	0.25-2.0
	Pentlandite	(Fe,Ni) ₉ S ₈	4.8	0.01-0.25
	Pyrrhotite	Fe _(1-x) S (x=0-0.17)	4.6	0.01-0.25
	Chalcopyrite	CuFeS ₂	4.2	0.01-0.25
	Pyrite	FeS ₂	5.0	0.01-0.25
	Platinum group minerals (PGM)		>8.0	0.001-0.1

Continues...

VMS deposits ⁴	Chalcopyrite	CuFeS_2	4.2	0.01-0.25
	Galena	PbS	7.4	0.01-0.25
	Sphalerite	$(\text{Zn,Fe})\text{S}$	4.1	0.01-0.25
	Pyrrhotite	$\text{Fe}_{(1-x)}\text{S}$ ($x=0-0.17$)	4.6	0.01-0.25
	Pyrite	FeS_2	5.0	0.01-0.25
	Gahnite	$(\text{Zn,Fe})\text{Al}_2\text{O}_4$	4.3	0.25-2.0
	Spessartine	$(\text{Mn}^{++},\text{Fe})_3\text{Al}_2(\text{SiO}_4)_3$	4.2	0.25-2.0
	Staurolite	$(\text{Fe}^{++},\text{Mg})_2\text{Al}_9(\text{Si,Al})_4\text{O}_{20}(\text{O,OH})_4$	3.7	0.25-2.0
Pb-Zn deposits ⁵ (Mississippi Valley type)	Galena	PbS	7.4	0.01-2.0
	Sphalerite	$(\text{Zn,Fe})\text{S}$	4.1	0.01-2.0
Porphyry Cu deposits ⁶	Sulphides		> 4.0	0.25-2.0
	Andradite	$\text{Ca}_3\text{Fe}^{+++}_2(\text{SiO}_4)_3$	3.9	0.25-2.0
	Diaspore	$\text{AlO}(\text{OH})$	3.4	0.25-2.0
	Barite	BaSO_4	4.5	0.25-2.0
	Alunite	$\text{KAl}_3(\text{SO}_4)_2(\text{OH})_6$	2.7	0.25-2.0
	Dravite	$\text{NaMg}_3\text{Al}_6(\text{BO}_3)_3\text{Si}_6\text{O}_{18}(\text{OH})_4$	3.1	0.25-2.0
	Apatite	$\text{Ca}_5(\text{PO}_4)_3(\text{OH,F,Cl})$	3.2	0.25-2.0
W-Mo deposits ⁷	Scheelite	CaWO_4	6.0	0.01-0.25
	Wolframite	$(\text{Fe,Mn})\text{WO}_4$	7.3	0.01-0.25
	Sulphides		>4.0	0.01-0.25
	Bi minerals		>6.0	0.01-0.25
"High tech metals" e.g. Nb, Ta, REE	Pyrochlore	$(\text{Na,Ca})_2\text{Nb}_2\text{O}_6(\text{OH,F})$	5.3	0.01-0.25
	Columbite	$\text{Fe}^{++}\text{Nb}_2\text{O}_6$	6.3	0.01-0.25
	Ta-minerals		>8.0	0.01-0.25
	Allanite	$(\text{Ce,Ca,Y})_2(\text{Al,Fe}^{+++})_3(\text{SiO}_4)_3(\text{OH})$	3.75	0.01-0.25

References:

1. McClenaghan & Kjarsgaard (2007) 2. McClenaghan & Cabri (2011) 3 and 6. Averill (2011) 4. Averill (2001) 5. Oviatt et al. (2013) 7. McClenaghan et al. (2013)

Indicator mineral work flow



Sampling



Pre-treatment



Concentration



Separation

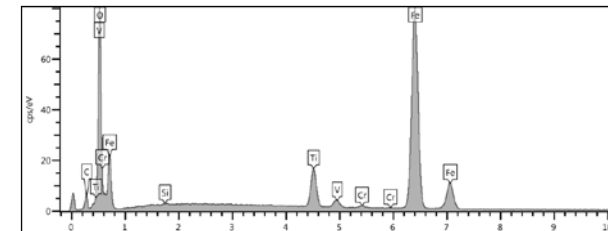
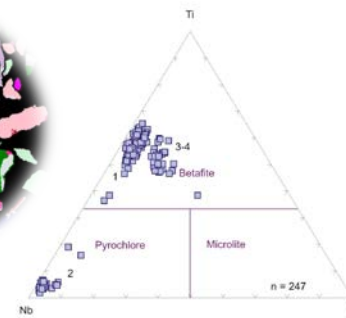


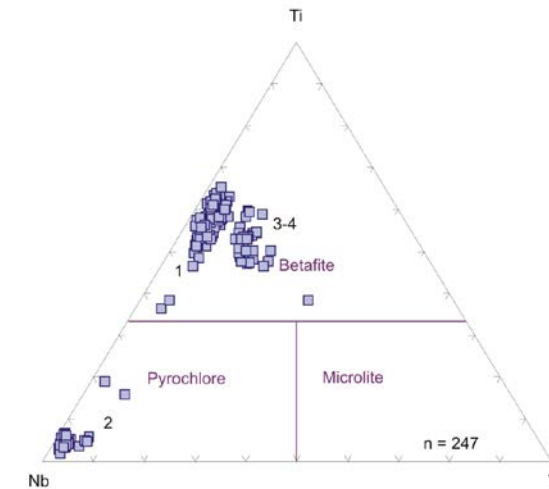
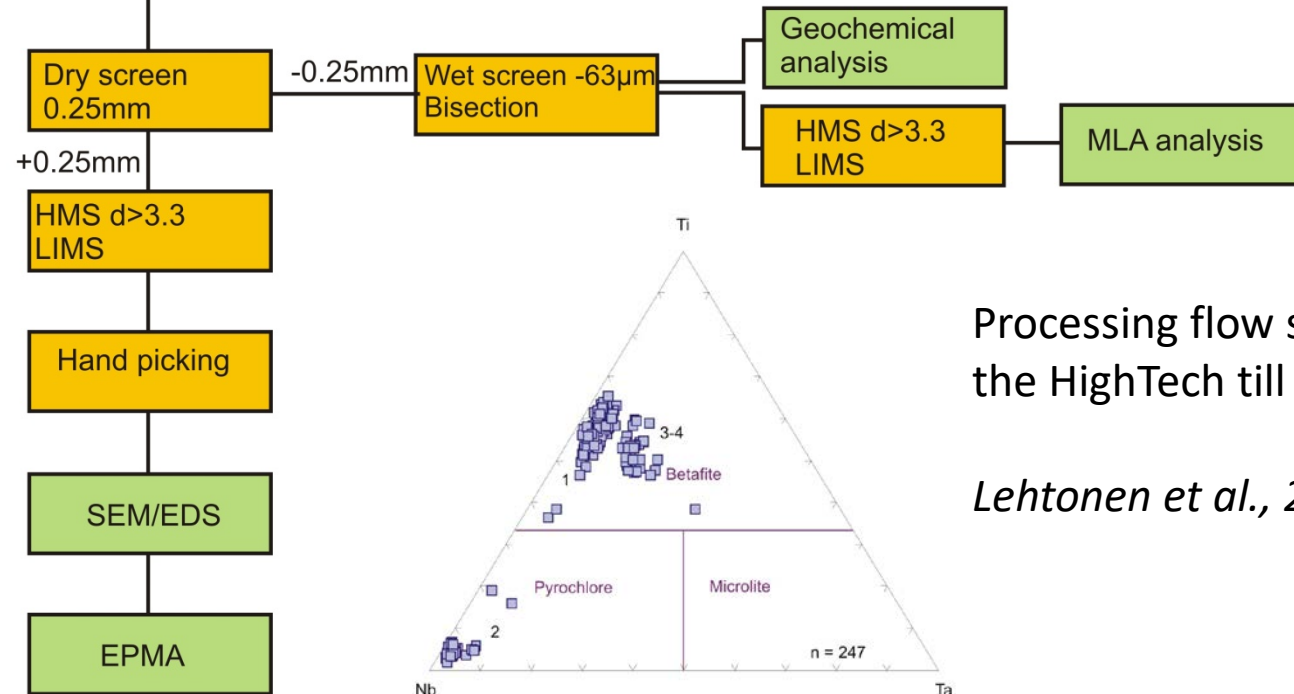
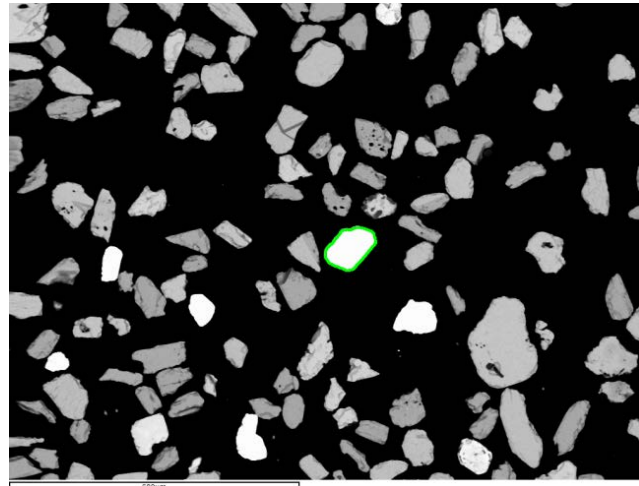
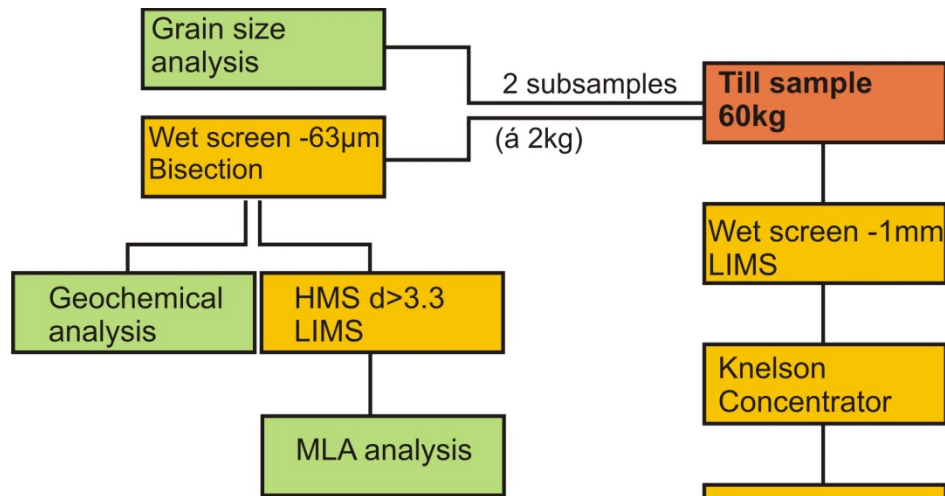
Indicator mineral workflow

Microscopy



Mineral analysis

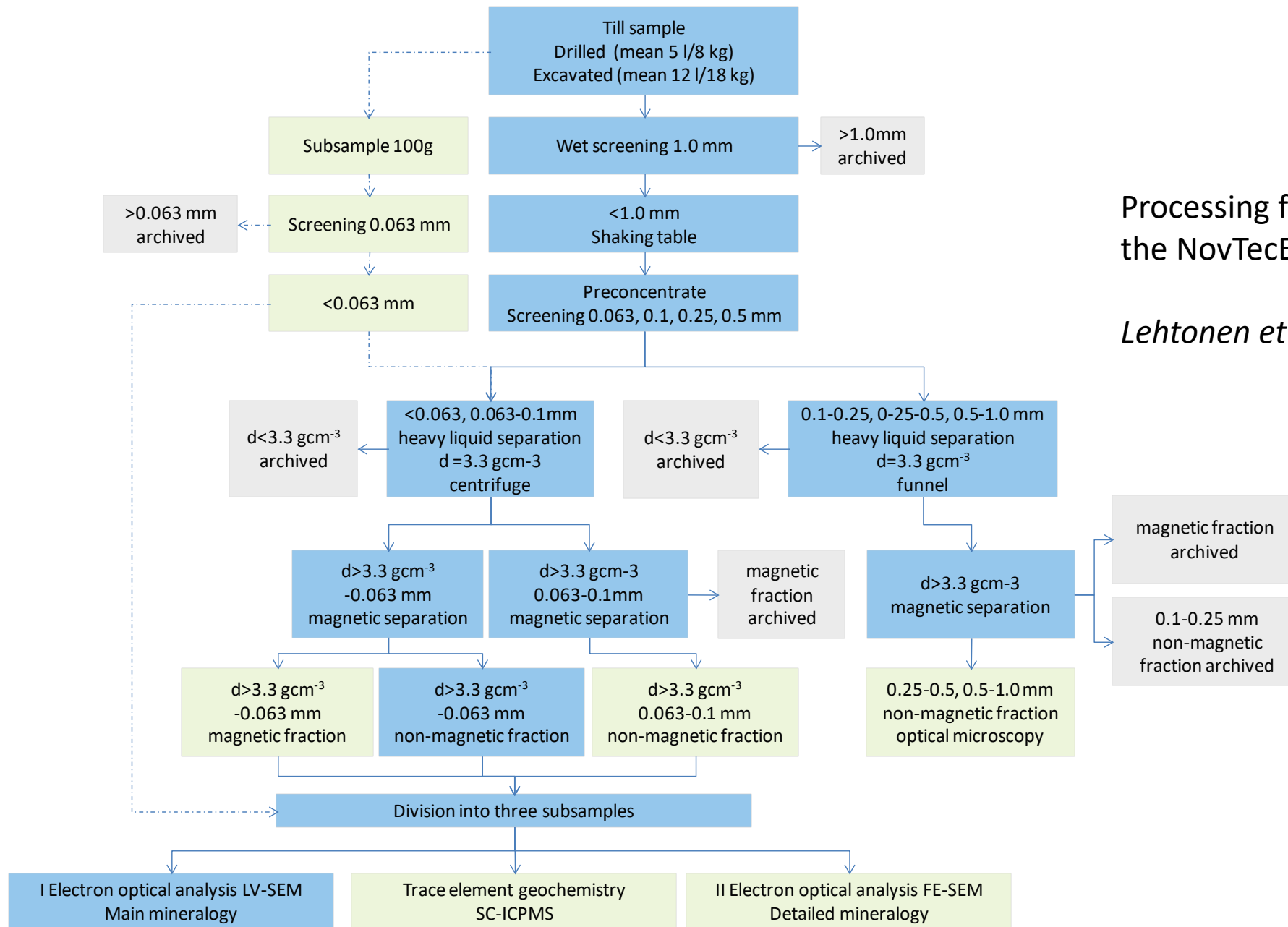




Hogarth (1977)

Processing flow sheet for the HighTech till samples

Lehtonen et al., 2011



Processing flow sheet for the NovTecEx till samples

Lehtonen et al., 2015

Optical Microscopy

Strengths

- Experience and intelligence of the observer

Weaknesses

- Human error: tiredness, loss of focus when observing several indicators simultaneously, results may vary according to the observer
- Some minerals are impossible to identify in grain sizes below $<100\text{-}200\ \mu\text{m}$
- SEM-EDS / EPMA is needed for confirmation
- Requires large original sample sizes to obtain enough sand-sized heavy mineral grains to obtain statistically meaningful results

Automated Mineralogy

Strengths

- Accuracy; very small contents of indicator minerals can be detected
- Optimal grain size range $10\text{-}200\ \mu\text{m}$
- Considerable reduction of the original sample size possible (savings in sample processing, less-invasive sampling methods)
- Can solve host mineral phases of trace elements detected by geochemical analysis (elemental deportment)

Weaknesses

- Tedious sample preparation

GTK processed	Weight < 1 mm table feed (kg)	Microscopy 0.25-1.0 mm: mineral assemblage
RM_POS\$-2012-36.2	10.9	Almandine-ilmenite/staurolite-epidote
RM_POS\$-2012-39.1	18.5	Almandine/epidote-staurolite-kyanite
RM_POS\$-2012-41.2	8.3	Almandine-ilmenite/epidote-staurolite
RM_POS\$-2012-43.3	8.4	Almandine/staurolite-epidote
RM_POS\$-2012-48.2	11	Almandine-ilmenite/staurolite-epidote
RM_POS\$-2012-55.2	7.5	Almandine-ilmenite-hornblende/epidote- staurolite
RM_POS\$-2012-66.1	19.7	Hematite-almandine-ilmenite/staurolite- epidote-kyanite
RM_POS\$-2012-77.2	9.5	Almandine-ilmenite-hematite/epidote- staurolite-kyanite
RM_POS\$-2012-82.1	22.2	Almandine-hematite-ilmenite/epidote- staurolite
RM_POS\$-2012-97.2	11.6	Almandine-hematite/epidote-staurolite

GTK processed	Microscopy 0.25-1.0 mm: accessories								
	Chalcopyrite	Pyrite	Low Cr-diopside	Ruby Corundum	Sapphire Corundum	Mn epidote	Gahnite	Red Rutile	Chromite
RM_POS\$-2012-36.2	1	1	3	0	0	0	0	55	55
RM_POS\$-2012-39.1	0	0	1	0	0	0	0	22	108
RM_POS\$-2012-41.2	1	0	0	0	0	0	0	48	72
RM_POS\$-2012-43.3	0	0	0	0	0	0	31	36	36
RM_POS\$-2012-48.2	0	0	1	0	0	0	1	36	73
RM_POS\$-2012-55.2	0	1	0	0	0	0	0	53	53
RM_POS\$-2012-66.1	0	0	0	0	0	0	0	20	9
RM_POS\$-2012-77.2	0	0	0	0	0	0	0	63	32
RM_POS\$-2012-82.1	0	0	0	0	0	0	0	27	68
RM_POS\$-2012-97.2	0	0	0	0	0	0	0	34	34

Lehtonen et al., 2015 (NovTecEx project)
 Microscopy results from ODM Laboratory

SEM-EDS data**Class**

	RM_POS\$-2012-36.2	
	Grains	% Total grains
Monazite	2 991	1.20
Xenotime	529	0.21
Scheelite		
Bismuth	1	0.00
Gold	4	0.00
Pyrochlore	39	0.02
Columbite	8	0.00
Thorite	4	0.00
Galena	16	0.01
Sperrylite	2	0.00
Total	250 000	

RM_POS\$-2012-43.3

	RM_POS\$-2012-43.3	
	Grains	% Total grains
	21 142	8,81
	64	0.03
	1	0.00
	2	0.00
	1	0.00
	2	0.00
	1	0.00
	7	0.00
Total	240 000	

SC-ICPMS data

	RM_POS\$-2012-36.2	RM_POS\$-2012-43.3
Y ppm	277	2 152
Bi ppm	2	5
W ppm	12	58
Nb ppm	284	416
Au ppb	127	146
Pt ppb	902	608

Picking results (ODM)

	RM_POS\$-2012-36.2	RM_POS\$-2012-43.3
Gold	18	13
Sperrylite	4	

NovtecEx project, Lehtonen et al. (2015)

Particle size vs. Sample preparation

Class	Particle Size	Approximate Number of Heavy Mineral Grains		Approximate No. of Epoxy Blocks per Gram
	µm	Per Gram	Per Epoxy Block	
Very coarse sand	1000-2000	170	125	1.5
Coarse sand	500-1000	1,400	500	3
Medium sand	250-500	11,000	2,000	5.5
fine sand	125-250	88,000	8,000	11
Very fine sand	63-125	700,000	32,000	22
Very coarse silt	32-63	5,600,000	130,000	45
Coarse silt	16-32	45,000,000	500,000	90

Table 1. Variation with particle size in the number of epoxy blocks required for automated analysis of 1 gram of heavy minerals.

Averill & Huneault, 2016

Different size materials have different transportation distances in glacial systems.
Important to evaluate what size fraction to focus.

Grain size distribution of minerals: Example from till heavy mineral concentrates

Grain size mm Density gcm-3	0.1-0.5 mm d>3.3	0.063-0.1 mm d>3.3	<0.063 mm d>3.3
Class	Grains / mass%	Grains / mass%	Grains / mass%
Titanite	655 / 5.5	411 / 3.4	542 / 1.7
Almandine	384 / 3.9	411 / 4.2	514 / 1.9
Ilmenite	1010 / 9.3	1895 / 19.2	7341 / 31.2
Zircon	73 / 0.8	371 / 4.1	1352 / 5.7
Monazite	3 / 0.0	9 / 0.1	82 / 0.4
Gold	0 / 0.0	0 / 0.0	1 / 0.0

Lehtonen et al., 2015 (NovTecEx project)
SEM-EDS Modal mineralogy data

Grain size distribution of minerals: Example from heavy mineral concentrates

	63-100 µm	100-160 µm
Mäkärä		
RM_JOV-2017-4.1*	Zr , Mnz, Ap, (Bdy), (Aln), (Xtm)	Zr, Mnz, (Xtm), (Ap)
RM_JOV-2017-6.2	Zr, Mnz, (Ap)	(Mnz), (Zr)
RM_JOV-2017-8.2*	Zr , Mnz, Xtm, (Aln)	Zr , Mnz, Xtm, (Aln), (Au)
RM_JOV-2017-9.2	Zr, (Mnz)	(Aln), (Mnz)
Sokli		
RM_JOV-2017-11.2*	Zr , Ap, Mnz, Pcl, (Bdy)	Zr , Mnz, Pcl, Ap, (Aln), (Bdy), (Xtm), (Zrc)
RM_JOV-2017-12.2	Zr , Ap, Mnz, Pcl, (Bdy)	Zr , Ap, Mnz, (Pcl), (Bdy)
RM_JOV-2017-13.2*	Zr, Pcl, Bdy, Ap, Mnz , Zrc, (Aln), (Thr), (Bas)	Zr, Pcl, Bdy, Mnz , Ap, Zrc, (Aln)
Rautuvaara		
RM_AMPE-2017-150.4	W-Rt, Mnz, Cbl, (Ap), (Aln), (Xtm), (Zr)	W-Rt, Mnz, Aln, (Ap), (Cbl)
RM_AMPE-2017-150.6	W-Rt, Mnz, (Zr), (Aln), (Xtm), (Cbl)	W-Rt, (Mnz), (Ap), (Cbl)
RM_AMPE-2017-151.2	W-Rt, Mnz, Xtm, Aln, (Ap), (Zr)	W-Rt, Mnz, Ap, (Aln), (Cbl), (Xtm), (Thr), (Zr)
RM_AMPE-2017-151.5	W-Rt, Mnz, (Ap), (Zr), (Xtm), (Cbl), (Bdy), (Aln)	W-Rt, Zr, Mnz, Aln, (Xtm)

Lukkari & Lehtonen 2018 (INDIKA project)

*Frantz 0.3 ja 0.5 A, **bold** = concentration over 5 %, () = trace amount

Aln = allanite, Ap = apatite, Bdy = baddeleyite, Bas = bastnäsite, Cbl = cobaltite, Mnz = monazite, Pcl = pyrochlore, Thr = thorite, W-Rt = W-rutile, Xtm = xenotime, Zrc = zirconite, Zr = zircon

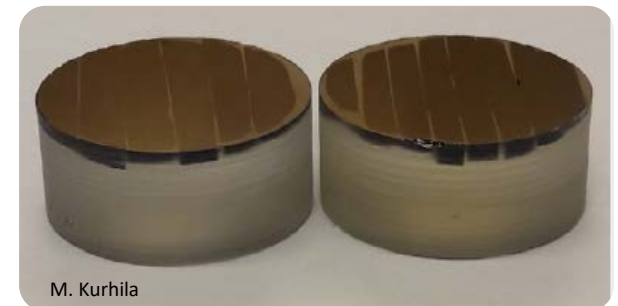
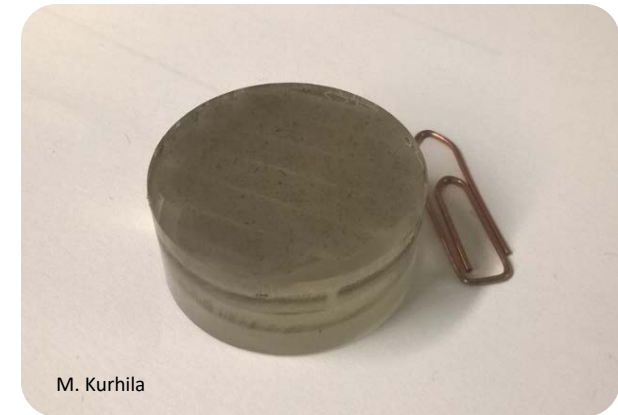
Sample preparation for electron optical studies

- Standard sample types
 - Polished epoxy mounts (\varnothing 25 mm or 30 mm)
 - Polished thin/thick sections (28 x 48 mm)
- Coating by carbon, gold, copper etc. for conductivity to achieve optimal quality of image & analysis
- Grain preparates for morphological studies (semiquantitative analysis)



Sample preparation for electron optical studies

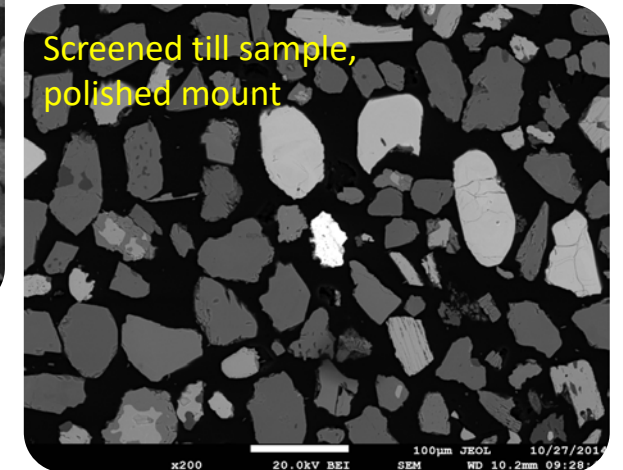
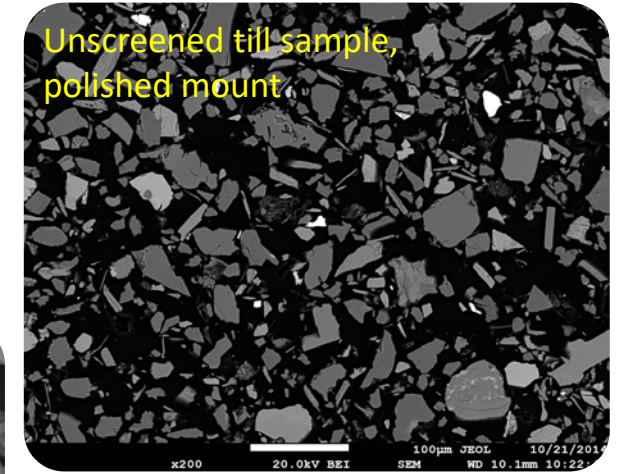
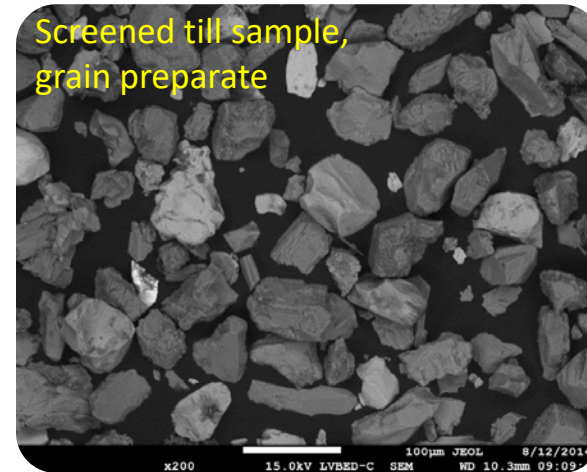
- Vertical polished mounts can be prepared to avoid gravitational differentiation in the epoxy
- Horizontal mount is cut into a few mm thick slices, each turned 90 degrees, glued back together & polished



Vertical polished mounts (30 mm) of mineral powder. Each slice approximately 5 mm.

Sample preparation for electron optical studies


- Optimal set-up
 - Narrow grain size distribution
 - Mineral grains are not touching each other
- Pre-treatment of sample material
 - Sieving
 - Fine grained graphite powder to grain separation



Automated/semi-automated mineralogy (SEM-EDS)

Article

Exploration Potential of Fine-Fraction Heavy Mineral Concentrates from Till Using Automated Mineralogy: A Case Study from the Izok Lake Cu–Zn–Pb–Ag VMS Deposit, Nunavut, Canada

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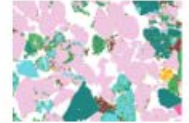
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Well-established method & widely used for indicator mineral surveys.

Feature



Unlocking the applications of automated mineral analysis

Identifying and quantifying the relative abundance of minerals is a fundamental part of many aspects of both pure and applied geology. Historically, quantitative mineralogy could be achieved using optical microscopy and point counting. This is a slow and operator dependent process, and practically impossible to achieve in, for example, very fine grained samples. Over the last decade a range of automated mineralogy technologies have arisen from the global mining industry and are being increasingly used in other branches of geology. These technologies, based on scanning electron microscopy with linked energy dispersive spectrometers, have the potential to revolutionise how we quantify mineralogy. In addition, during measurement, the sample textures are also captured, providing a wealth of valuable data for the geologist. In this article we review the current state of automated mineralogy and highlight the many areas of application for this technology.

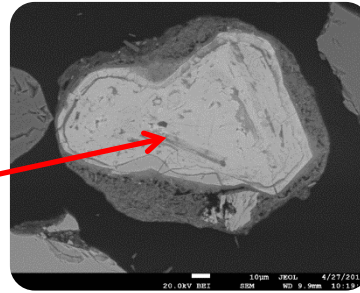
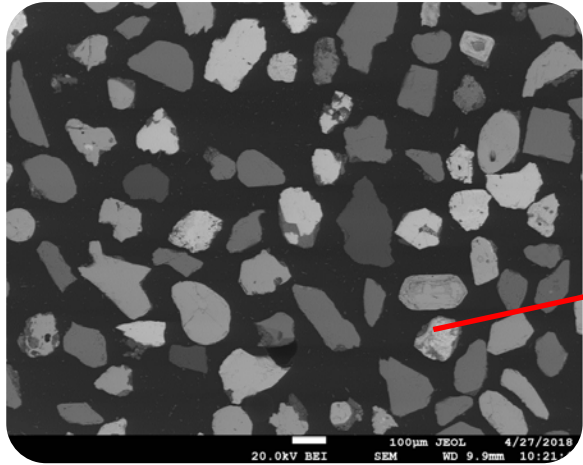
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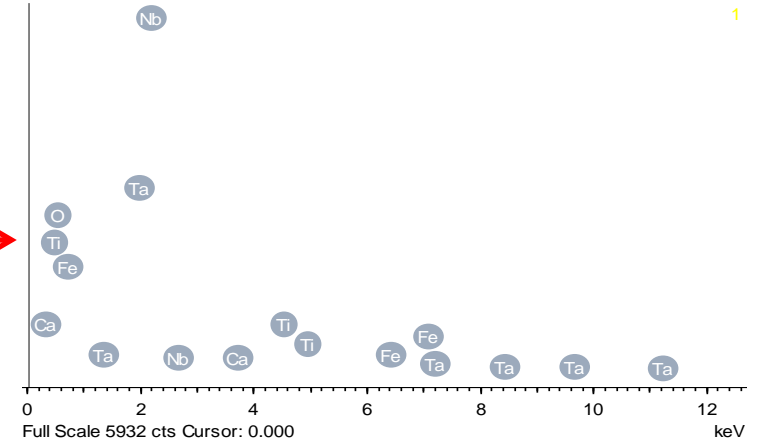
Modal Mineralogy Measurement

BSE image

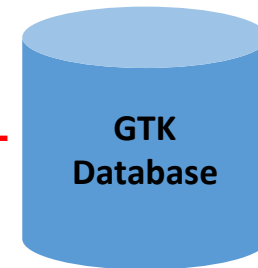


Single grain

EDS spectrum



Chemical composition

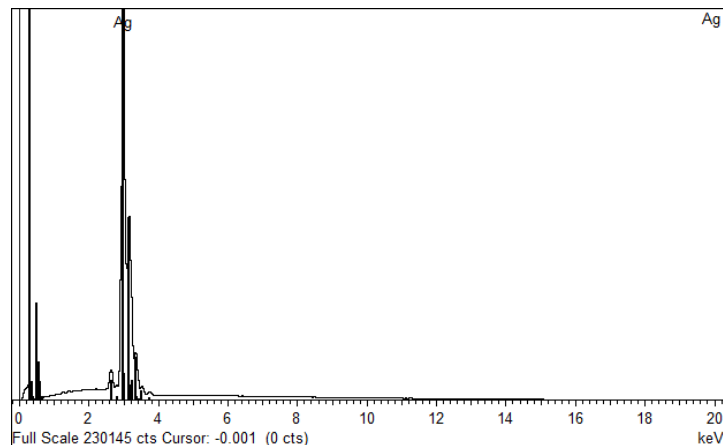
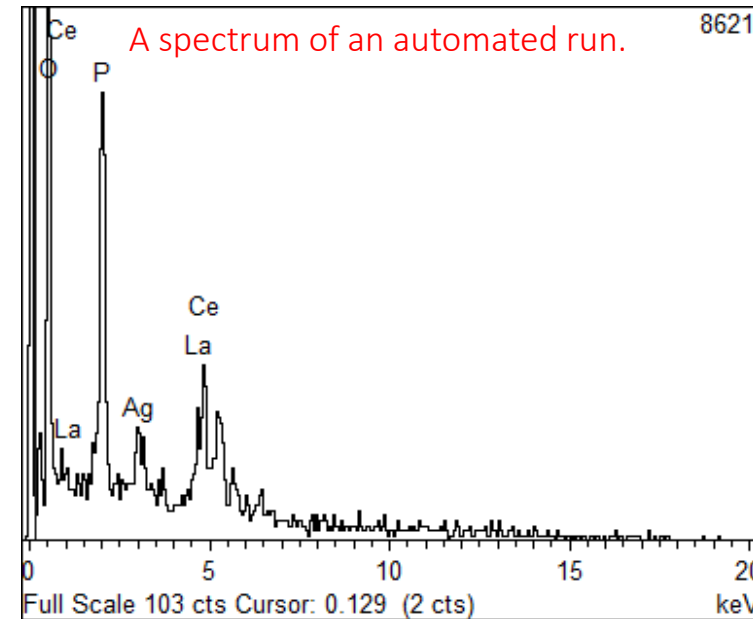
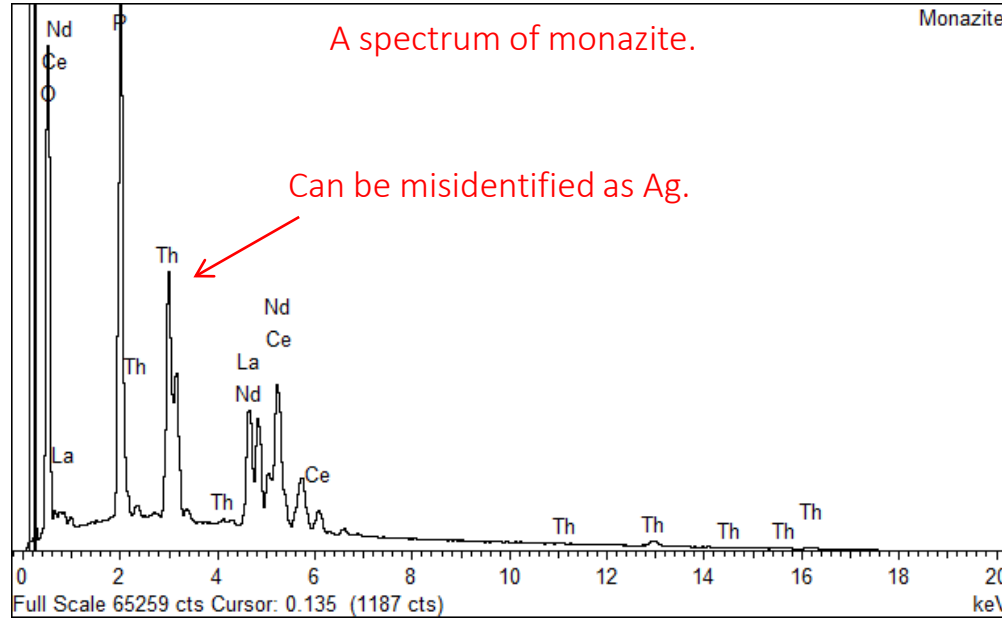


Columbite
(Mg,Fe,Mn)(Nb,Ta)₂O₆

Mineral	Grain count	Vol-%
Monazite	1800	11.70
Fe-oxide	1708	16.28
Zircon	1666	11.45
Baddeleyite	877	6.69
Columbite	32	0.02
.		
.		
Total	9226	100

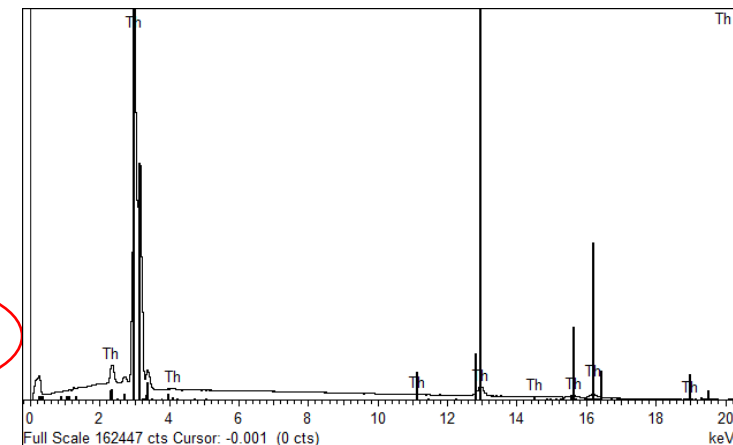


EDS software is not infallible –
It is **crucial** to check the data quality!

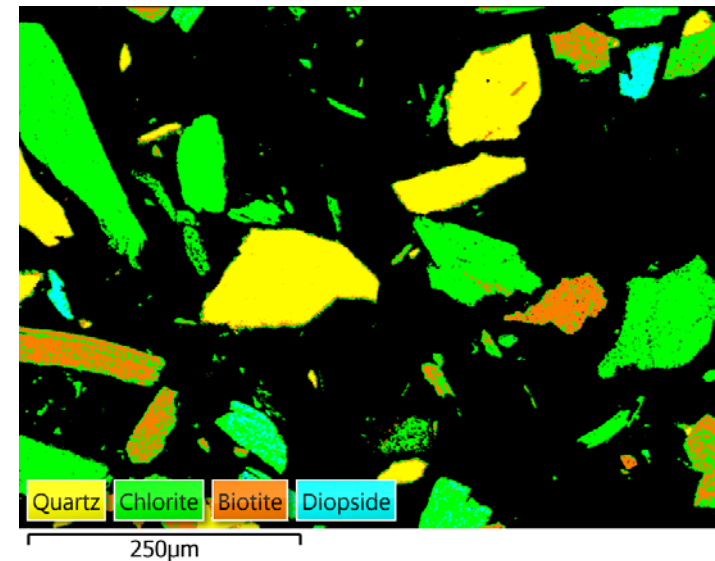
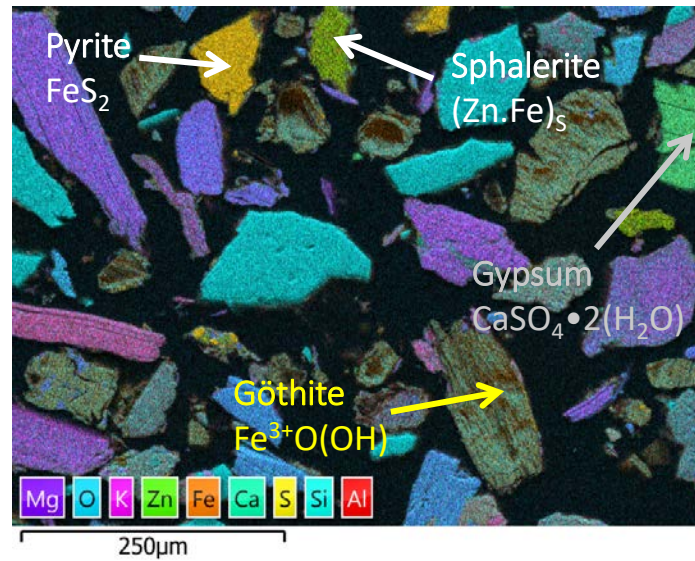
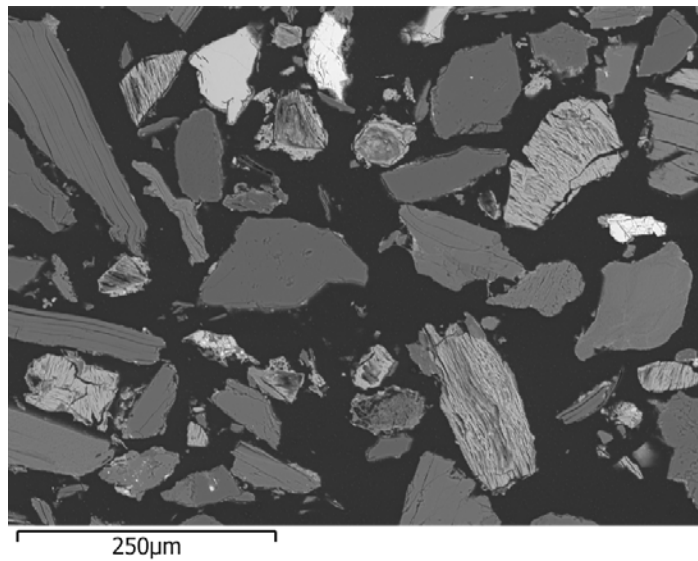


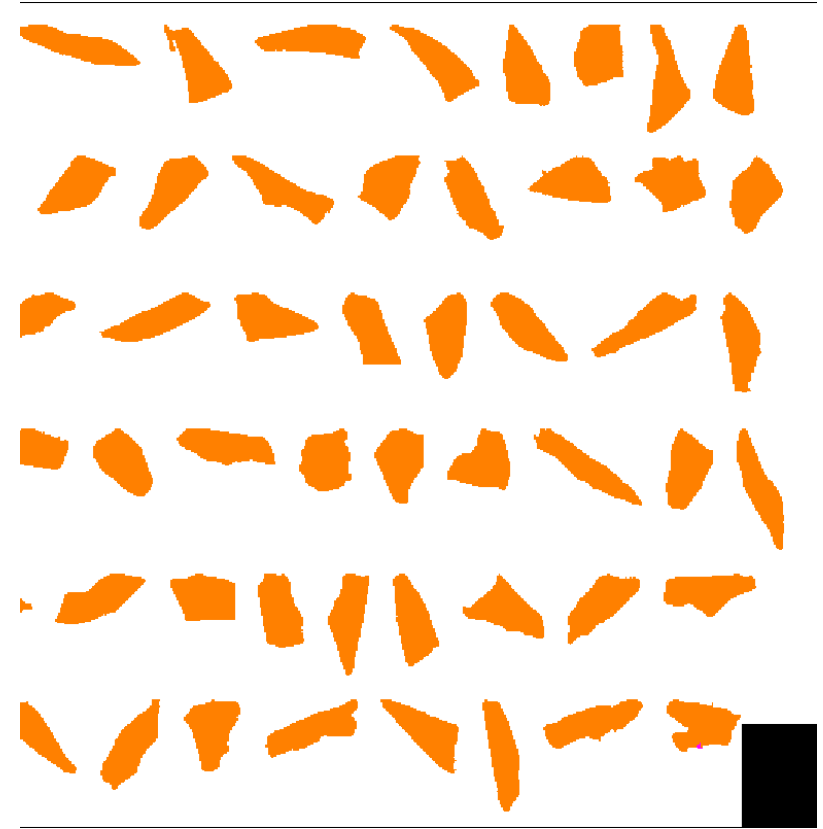
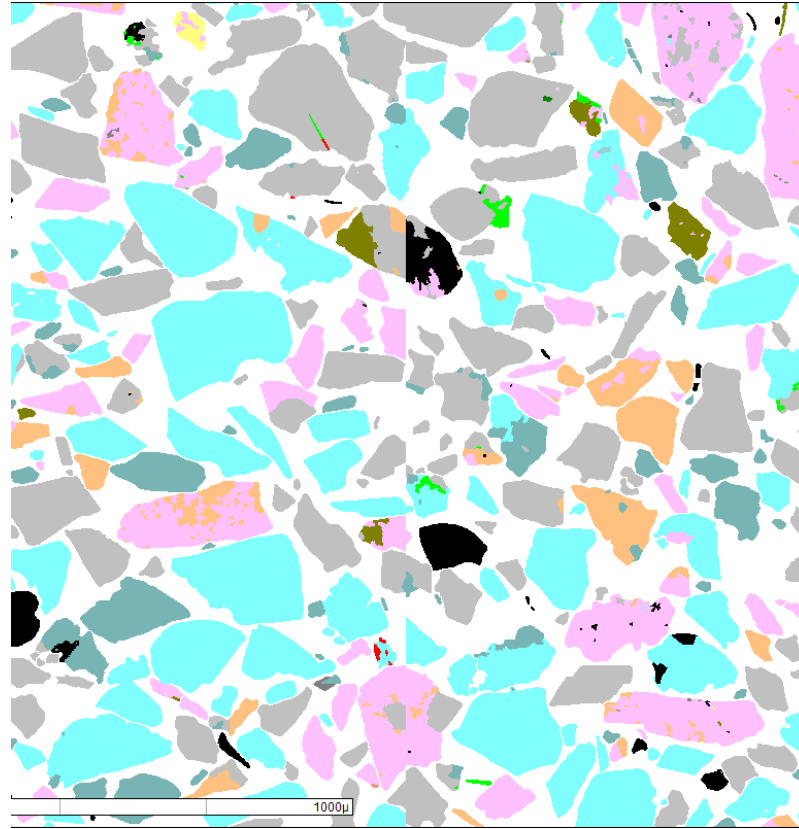
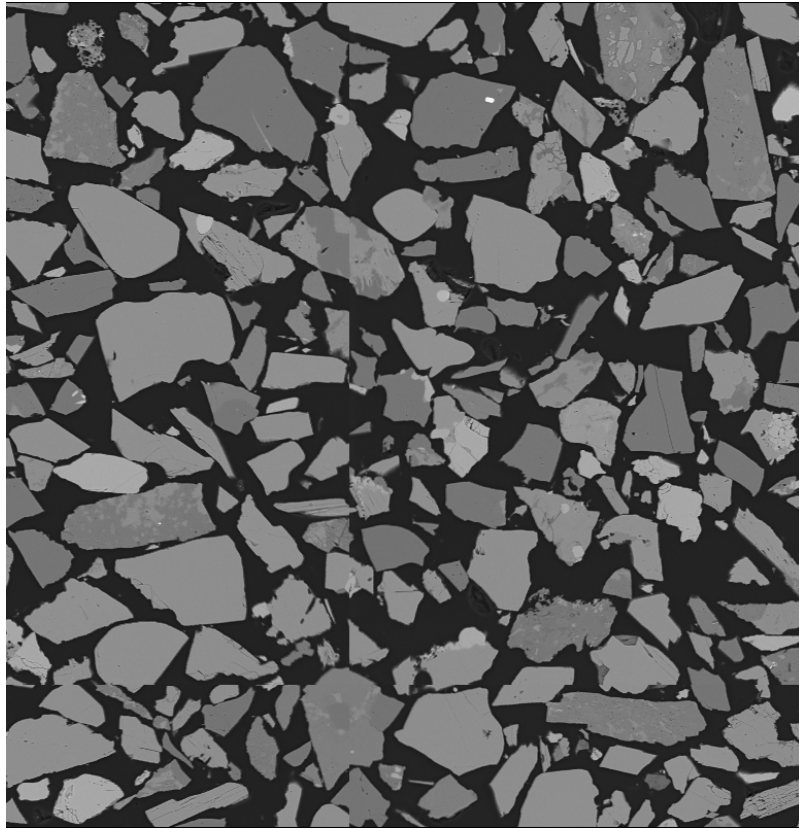
47	107.87
Ag	10.50
Silver	
K α	22.163
L α	2.983

90	232.04
Th	11.72
Thorium	
L α	12.968
M α	2.996



Elemental and Phase Mapping





Phase Mapping & Particle listing

Courtesy: Matti Kurhila

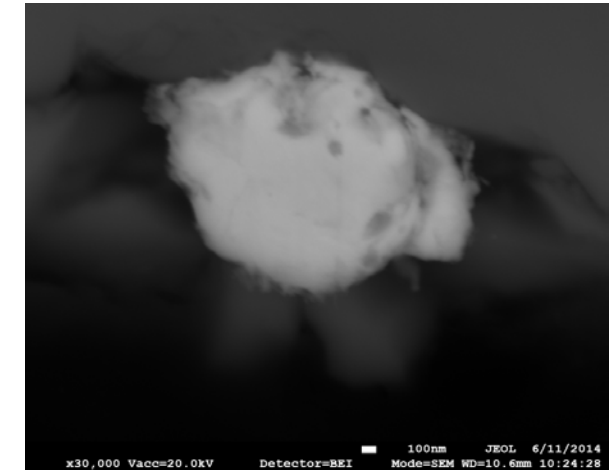
Automated Search for Trace Mineral Phases: BSE Thresholding

A heavy mineral concentrate of till (20-63 μm).

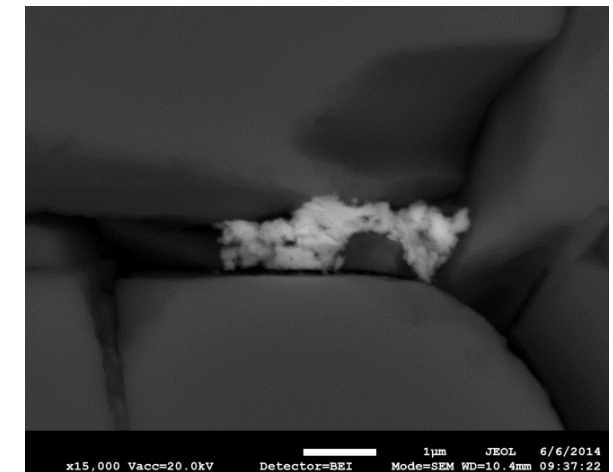
RM_POS\$-2012-36.2

Class	Grains	% analyzed grains	% total grains
Monazite	2 991	83.13	1.20
Xenotime	529	14.70	0.21
Scheelite	0	0.00	0.00
Bismuth	1	0.03	0.00
Gold	4	0.11	0.00
Pyrochlore	39	1.08	0.02
Thorite	8	0.22	0.00
Galena	4	0.11	0.00
Pb-oxide	16	0.44	0.01
Sperrylite	4	0.11	0.00
Uraninite	2	0.06	0.00
Total	3 598	100.00	1.44

Total features (est.) 250 000

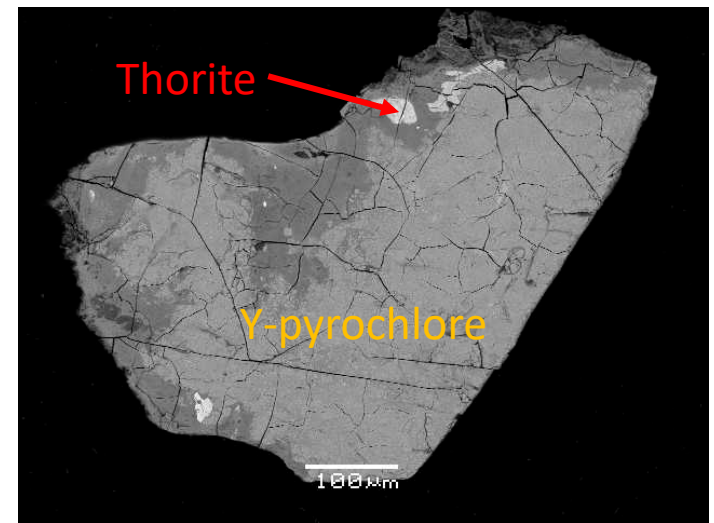
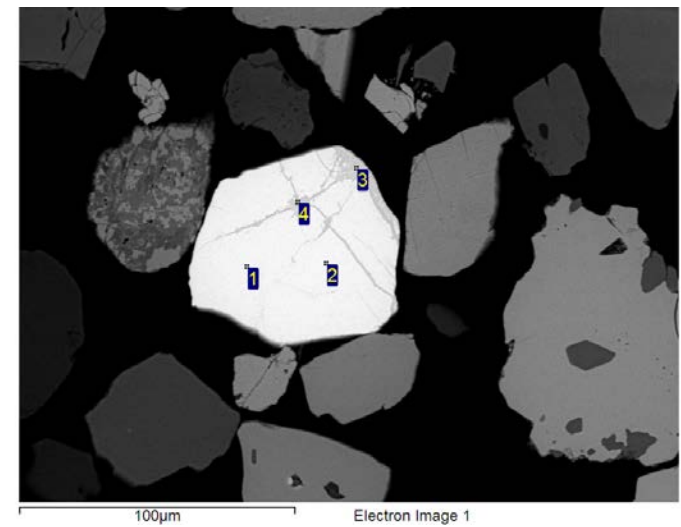


Sperrylite PtAs₂



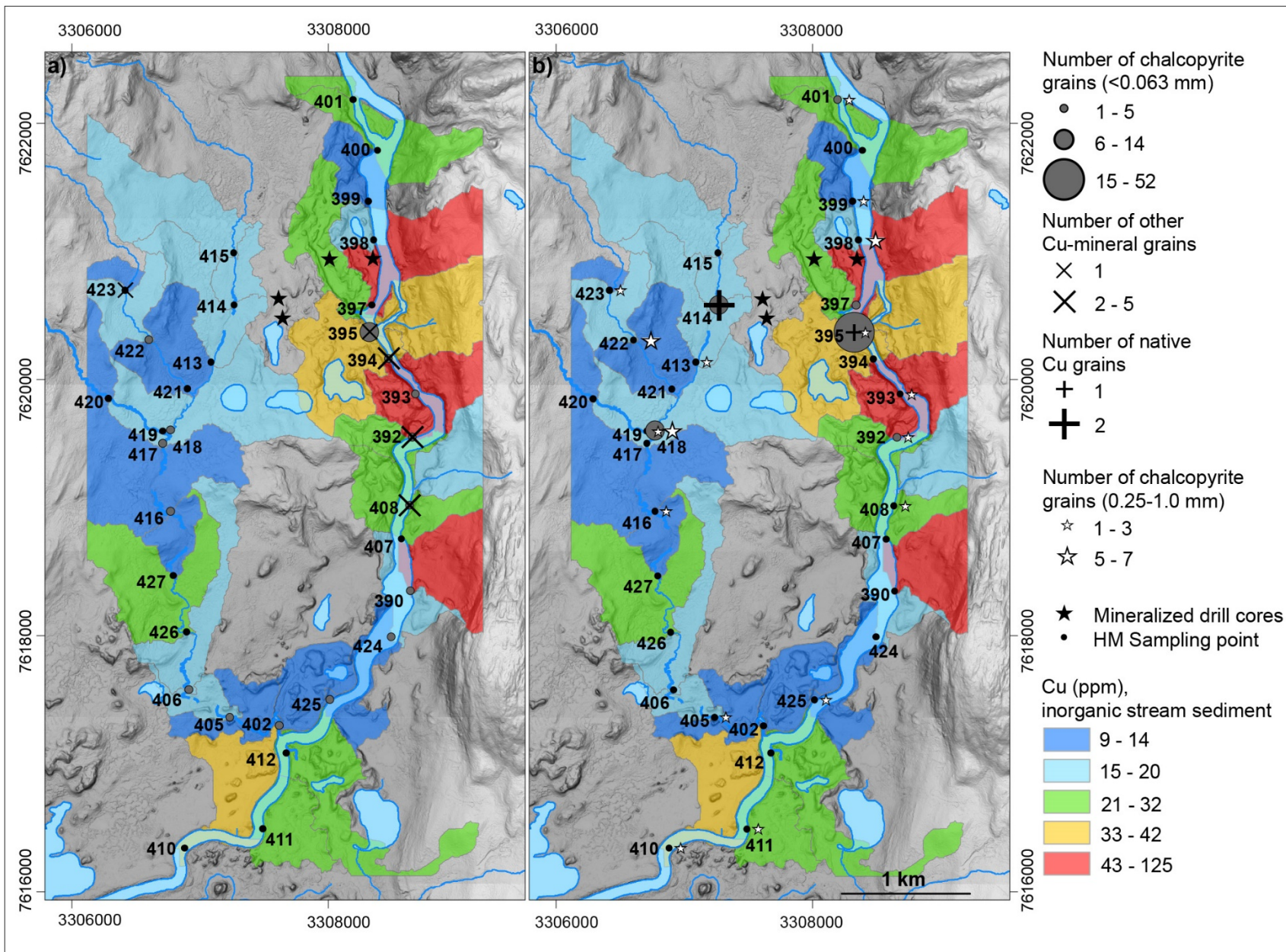
Gold Au

	426U_06MR	426_U_Pin09	426U_10MR	426_U_Kes09	426U_17MR	426_U_Poh09
Sample type	Till	Till precon	Till	Till precon	Till	Till precon
Original sample size kg	2	45	2	45	2	45
Density gcm-3	>3.3	>3.3	>3.3	>3.3	>3.3	>3.3
Grain size µm	-63	-63	-63	-63	-63	-63
Mineral	Grain Count	Grain Count	Grain Count	Grain Count	Grain Count	Grain Count
Yttrio-pyrochlore-(Y)	3	4	2	12	23	24
Other_pyrochlores						
Columbite_tantalite	2	5	1	10		6
Gold		1				1
Bismuth						1
Hedleyite				1		
Scheelite		1	2	11	1	24
Thorite	9	15	41	29	16	25
Baddeleyite	3	28	2	8		4
Monazite	656	3635	1046	1317	560	1289
Bastnasite	5	11	14	27	16	30
Xenotime	73	148	75	65	56	81
Allanite		6	1	20		27
Zirconolite				1		1
Chamosite				1		
Total	751	3854	1184	1502	672	1513
Total grain count (ca)	400 000	800 000	800 000	800 000	600 000	800 000



Lehtonen et al., 2011

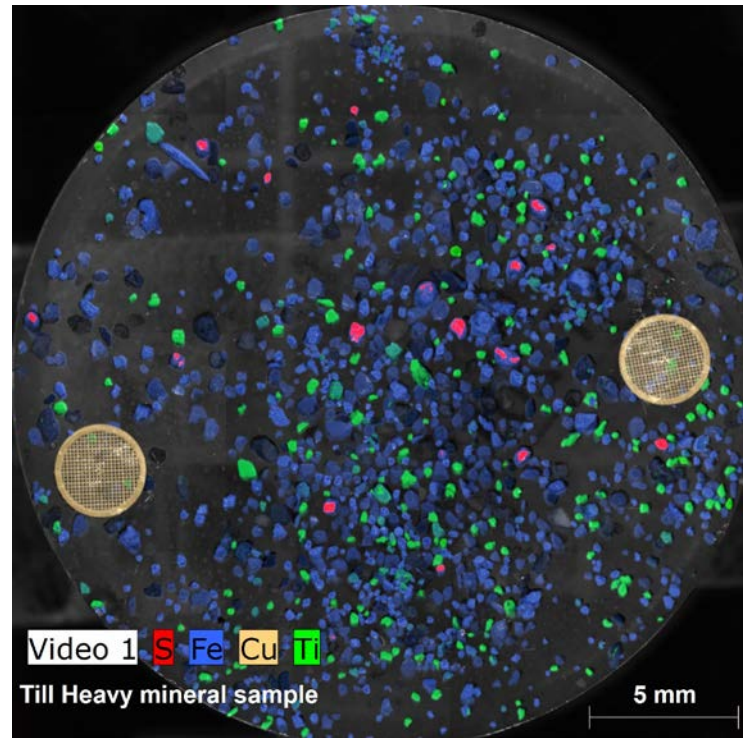
Hand picking of 0.25-0.5 mm						
Yttrio-pyrochlore-(Y)		0		0		66



Hulkki et al., 2018

Micro-XRF Technique

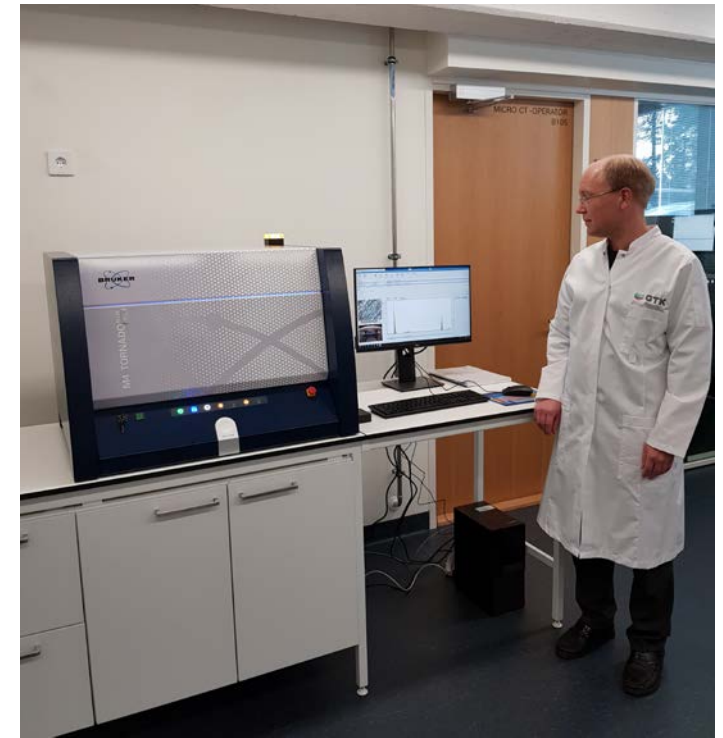
- Recently installed at GTK
- Validation period on-going
- Benefits for indicator mineral work
 - Large sample chamber
 - Fast scanning of elemental distribution maps
 - Possibility to study coarser grain size fractions
 - No (or minimal) sample preparation needed
- Testing on-going for MinExTarget samples



*Micro-XRF map of a MinExTarget heavy mineral concentrate
Beam size 20 μm*

Courtesy: Hugh O'Brien, GTK

Extremely fast scanning techniques also available



Maia Mapper: high definition XRF imaging in the lab

C.G. Ryan,^{a,1} R. Kirkham,^a G.F. Moorhead,^a D. Parry,^a M. Jensen,^a A. Faulks,^a S. Hogan,^a P.A. Dunn,^a R. Dodanwala,^a L.A. Fisher,^a M. Pearce,^a D.P. Siddons,^b A. Kuczewski,^b U. Lundström,^c A. Trolliet^c and N. Gao^d

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^cExcillum AB, Torshamnsgatan 35, 164 40 Kista, Sweden

^dXOS, 15 Tech Valley Drive, East Greenbush, U.S.A.

E-mail: Chris.Ryan@csiro.au

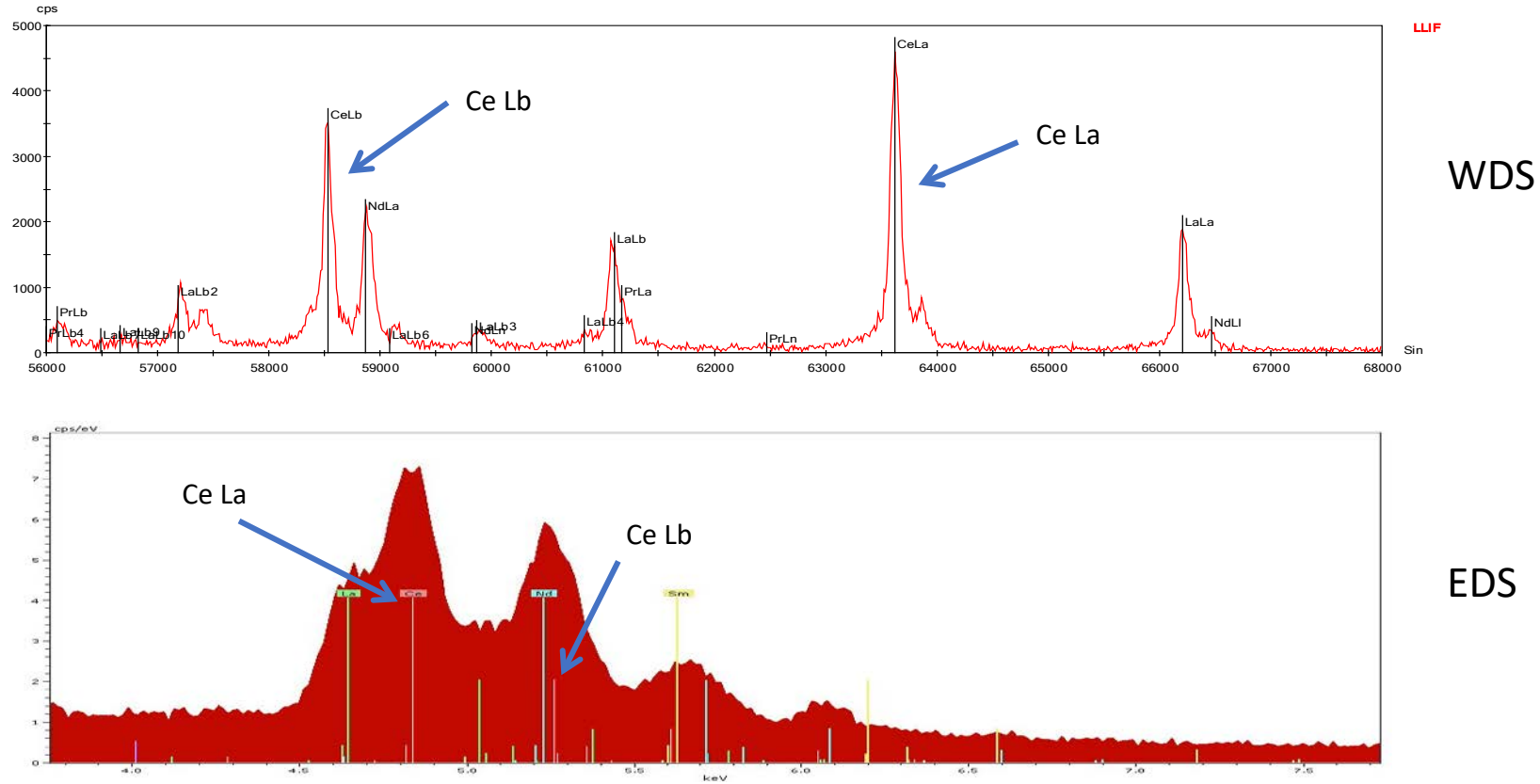
Quantitative chemical analysis of minerals (EPMA)

Quantitative mineral analysis of indicators

- Quantitative chemical analyses of indicator minerals needed for source characterization (fingerprinting)
- Routinely obtained by electron microprobe (WDS based analysis)
 - DL of EDS 0.1-0.5w%; DL of WDS down to a few ppm's but routinely ca 0.05w% (element and matrix depended)

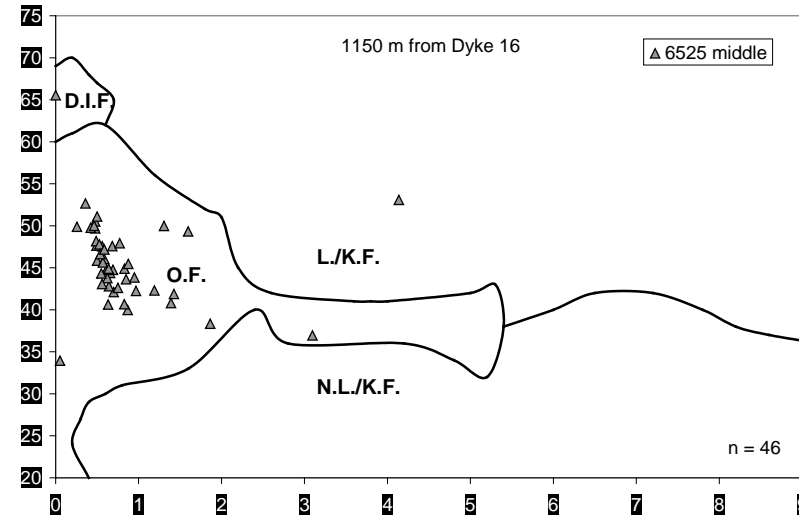
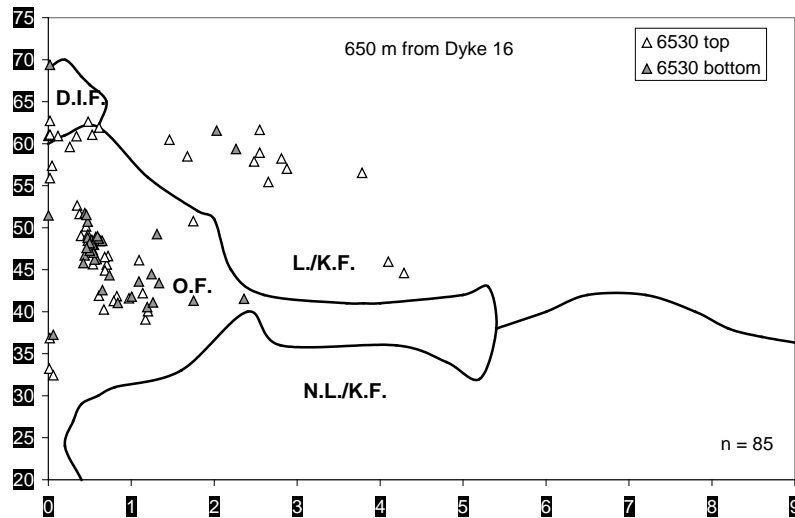
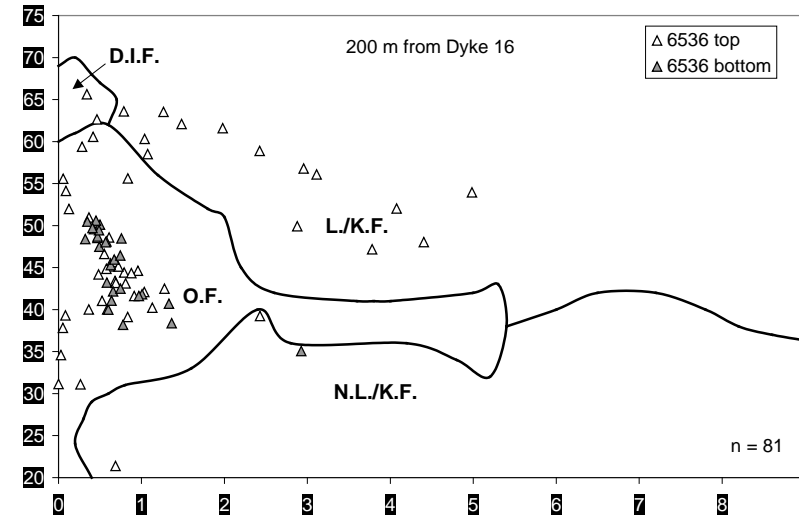
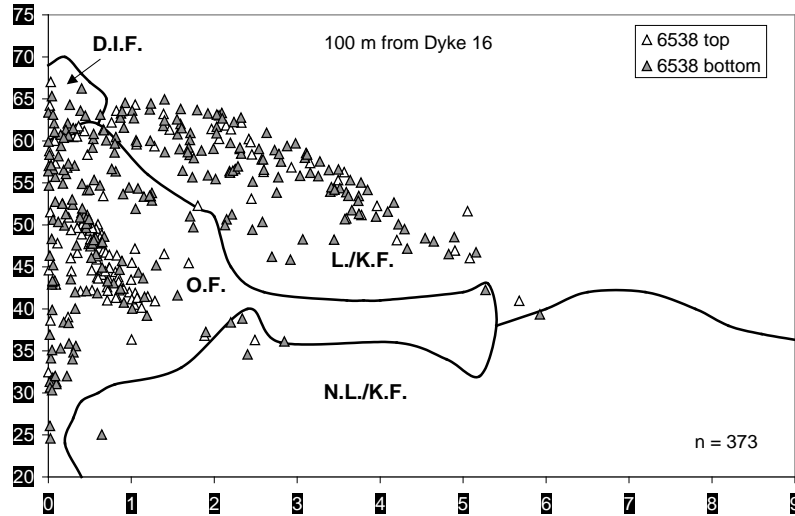


WDS-EDS spectral resolution (Monazite)

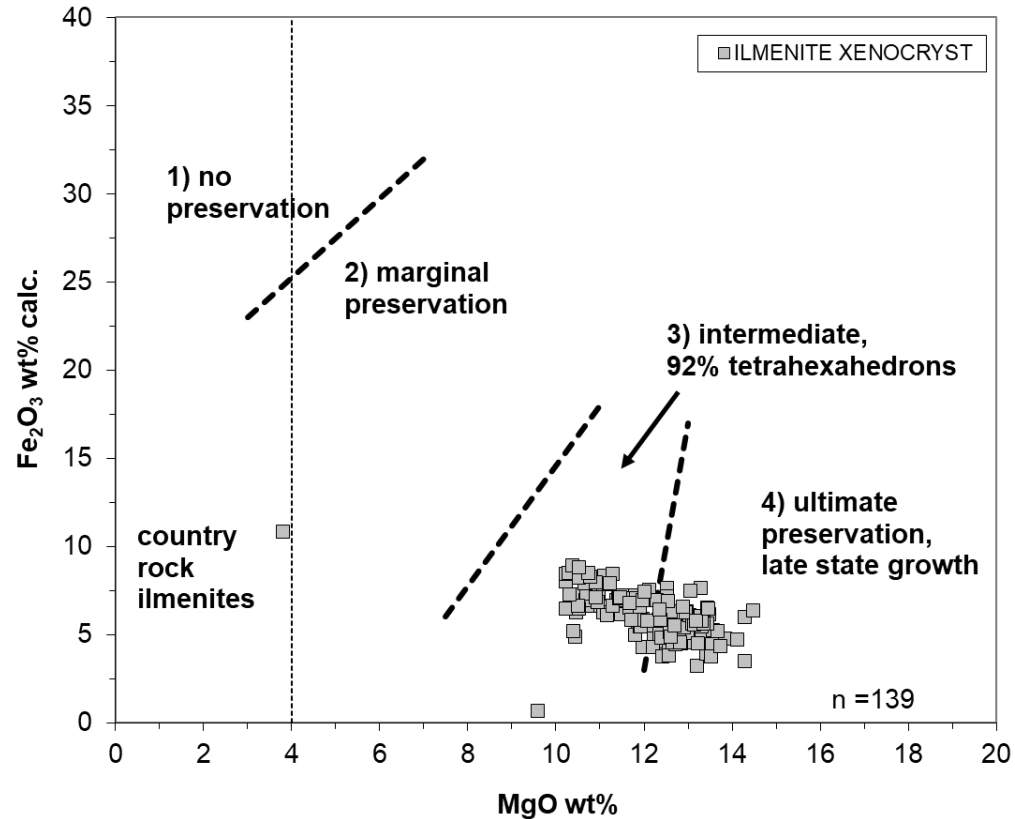


Courtesy: Bo Johanson, GTK

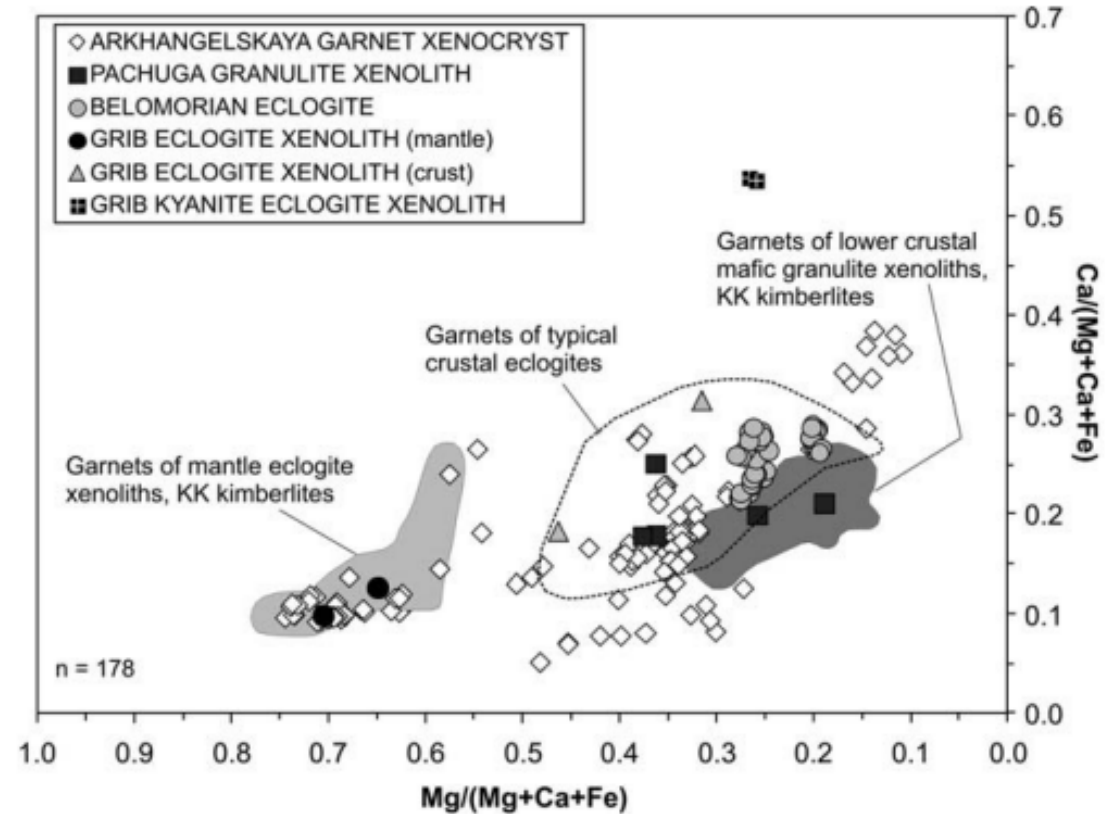
Classification of indicators: Example of kimberlitic / non-kimberlitic chromite



Source characterization based on indicator minerals

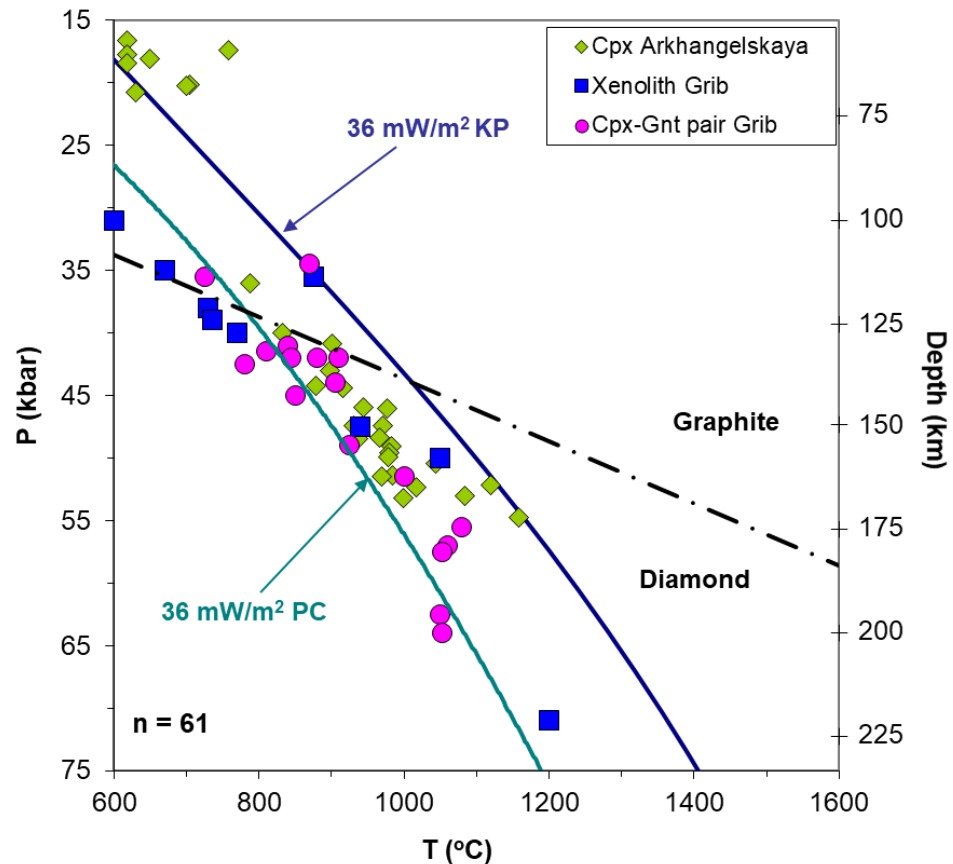


Diamond preservation index by Gurney & Zweistra (1995); Data from Lehtonen et al. (2009)



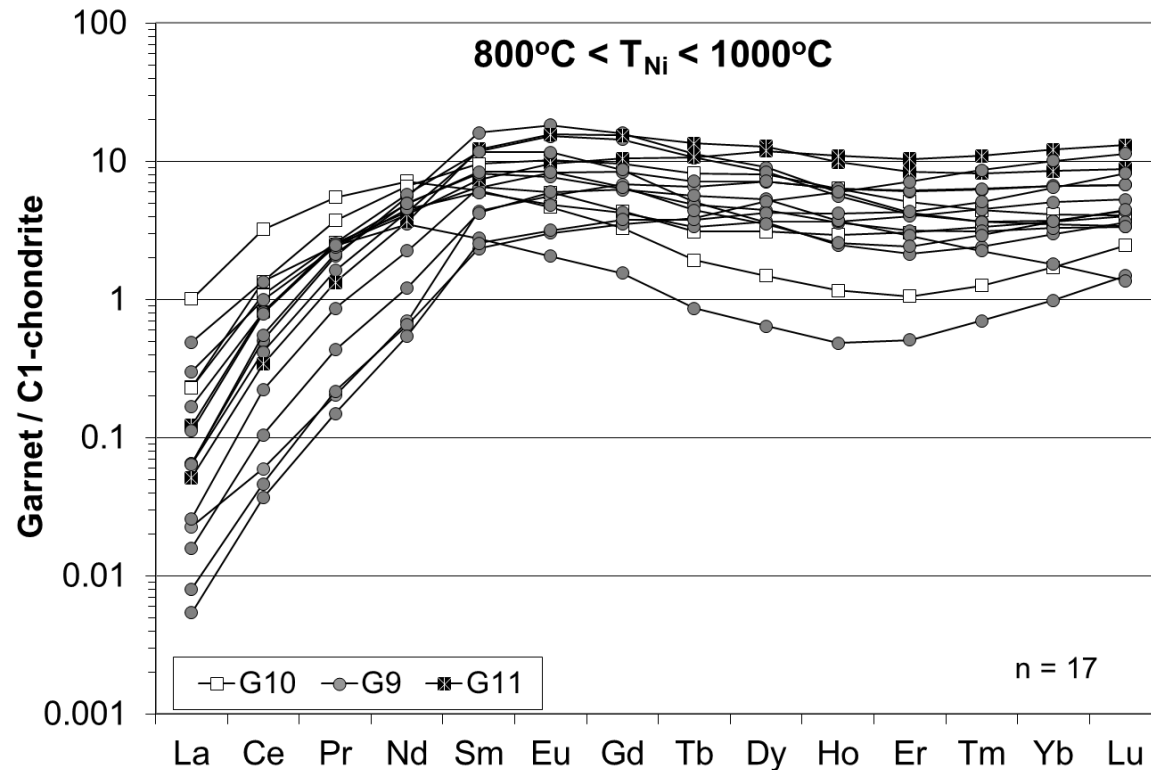
Eclogitic garnet classification; Data from Lehtonen et al. (2009) and references therein

Thermometry and Thermobarometry



p-T data from CPX xenocrysts (Lehtonen et al., 2009); mantle xenoliths (Malkovets et al., 2003; Sablukova et al., 2003); garnet-cpx data from Kostrovitsky et al., 2004; reference geotherms Pollack & Chapman (1977); Kukkonen & Peltonen (1999), diamond – graphite transition Kennedy & Kennedy 1976

Trace element data by LA-SC-ICPMS



Stay tuned for Yann Lahaye's presentation!

C1-Chondrite normalized (McDonough and Sun, 1995) REE profiles of garnet xenocrysts.
The samples are subdivided according to rock type (Grütter et al., 2004). Data from Lehtonen et al. (2009).



Thank you – Kiitos!



Photo: Kari A. Kinnunen

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