Traceability in Continuous Ore Beneficiation Processes using Process Mineralogy Signatures

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ABSTRACT

Traceability is the means to identify and follow real or imaginary lots through a process chain. It gives the opportunity to backtrack through a chain of events or to predict process outcomes given the origin of a lot.

Traceability can be used in different areas, e.g. in the middle of 1990s traceability was a hot subject when different cases of food-carried diseases were exposed, but traceability is also used in other process industries to follow material and products during the processing. There is, however, a lack of traceability in continuous processes to compare with batch processes. The reason is that it is more complicated to identify a lot, and reach good traceability, in a continuous process just because they are contiguous.

In this work, the continuous ore beneficiation process at LKAB (Luossavaara-Kirunavaara AB, Sweden) Malmberget has been investigated in detail. The purpose is to trace the ore through the grinding sections by parameters and signatures like particle mineralogy, mineral associations and particle texture. In order to follow the material through the different process steps, analytical methods like optical microscopy and Particle Texture Analysis (PTA) are used.

In paper I, different traceability methods utilised to achieve traceability in continuous processes are explained. The advantages and disadvantages are presented for each method. In paper II, the relations are shown between the materials that come into the grinding circuits as well as data collected from the PTA analysis is subjected to multivariate data analysis. The combination of automated process mineralogy and multivariate analysis is unique and first presented in this paper. The study in paper III is on an apatite-iron ore deposit at Malmberget, Sweden, and characterises an ore body both mineralogically and texturally in a quantitative manner by using different analytical methods. Paper IV explains how external flows are rerouted into the ordinary flowsheet and what impact they have. Paper V focuses on the same data from a previous paper by using multivariate data analysis. Comparisons between the PTA and QEMSCAN are discussed in paper VI. The differences come into play when the data are further processed. In particular, the liberation analyses seem to be dependent on the algorithms used and their tolerance limits.
To My Loving Wife
ACKNOWLEDGMENTS

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LIST OF APPENDED PAPERS

This thesis is based on the work reported in the following papers:


RELATED PUBLICATIONS (not included in this thesis)


<table>
<thead>
<tr>
<th>Abbreviation</th>
<th>Description</th>
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<tbody>
<tr>
<td>BSE</td>
<td>Backscatter Electron</td>
</tr>
<tr>
<td>EBSD</td>
<td>Electron Backscatter Diffraction</td>
</tr>
<tr>
<td>EBSP</td>
<td>Electron Backscatter Pattern</td>
</tr>
<tr>
<td>EDS</td>
<td>Energy Dispersive System (X-rays detector)</td>
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<tr>
<td>EPMA</td>
<td>Electron Probe Micro-Analyser</td>
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<tr>
<td>IA</td>
<td>Image Analysis</td>
</tr>
<tr>
<td>LKAB</td>
<td>The Swedish mining company Loussavaara Kiirunavaara AB</td>
</tr>
<tr>
<td>LTU</td>
<td>Luleå University of Technology</td>
</tr>
<tr>
<td>OM</td>
<td>Optical microscopy</td>
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<tr>
<td>OPLS</td>
<td>Orthogonal projections to latent structures</td>
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<tr>
<td>OPLS-DA</td>
<td>Orthogonal projections to latent structures- Discriminate Analysis</td>
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<tr>
<td>PCA</td>
<td>Principal Component Analysis</td>
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<td>PLS</td>
<td>Projections to Latent Structures</td>
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<td>Projections to Latent Structures-Discriminate Analysis</td>
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<td>PTA</td>
<td>Particle Texture Analysis</td>
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<td>QEMSCAN</td>
<td>Quantitative Evaluation of Minerals by Scanning Electron Microscopy</td>
</tr>
<tr>
<td>RFID</td>
<td>Radio frequency identification</td>
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<tr>
<td>SEM</td>
<td>Scanning Electron Microscope</td>
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1 INTRODUCTION

1.1 General
In the early 1960s the world population was around three billion people. According to the UNFPA report “State of world population 2008”, there are today over 6.75 billion people in the world and growing. Higher population means greater demands on security and safety on food and water. These things seem obvious, but there is a lot of work and effort to achieve these targets. Small mistakes can lead to major disasters.

In beginning of the 1990s, Mad Cow Disease or Bovine Spongiform Encephalopathy (BSE) was an important topic around Europe. Only in Britain 137,000 cases were reported (Lacey, 1994) making it a disaster for the cattle market with many farmers ruined because of this reason. In May 1999 the chicken dioxin crisis became known and in the beginning of 2001 foot-and-mouth disease created crises in the food industry around the world (Dupuy et al., 2002). At the end of the 1990s, the traceability and tracking of meat became very important and automated systems from farms to slaughterhouses were developed and implemented (Mousavi et al., 2002).

As a consumer of meat, it is important to know where the meat is coming from and for the food companies to be able trace back if something is detected regarding the meat. Traceability makes it possible to find the source of the causes and to be able makes changes fast. An ideal traceability system must be able to track the history of both product and activities that are involved in the process (Kim et al., 1995).

Traceability is today used in several industries, such as pharmacy, food, pulp and paper but also starting in the mining industry.

Mining or extraction of valuable minerals is one of the oldest and most important industries in the history of our time. Minerals have been used for a long time, and still today the market is growing. Iron is the most commonly used metal around the world, and iron ore is the main raw material that is used for steel.

LKAB has since the beginning of the 1900s produced iron ore products from mines in Kiruna and Malmberget and is today one of the world’s leading producers of highly refined iron ore products. The main product is pellets for blast furnaces and direct reduction furnaces. Today there is a higher demand from the customers to obtain a good quality which is one reason why it is important to have good control over the process and the raw material used.
1.2 Scope of the work

LKAB is a mining company that extracts and refines iron ore from deposits located at Malmberget and Kiruna. Because of the greater demand on pellets, the primary product of LKAB, the company invested in a new pelletising plant at Malmberget 2006. With a new pelletising plant, the production increased and raw material from both Kiruna and Malmberget was mixed into the feed. The iron ore from Malmberget and Kiruna behave differently during the refinement process because ore from Malmberget has a coarser grain size, different kind of grain boundaries and different level of contaminants compared to Kiruna ore (Martinsson and Wanhainen, 2000). This means that there might be a risk for quality variations in the final product; this is why traceability becomes an important aspect.

By looking at the mineralogical characters and the chemistry throughout the whole process, useful data are collected in order to achieve traceability in the process.

There are also other factors which are interesting. The mineral shape has been stressed in many papers, but in this case we are interested to find out any systematic changes during the process and detect traceability from information about the mineral grains.

This project is divided into three parts. The participants are three Ph.D. students from different divisions and their supervisors as well as a group of people from LKAB who are involved in the guidance of the project.

Figure 1. The structure of the project and connections among the researchers.
Cecilia Lund from the Division of Ore Geology and Applied Geophysics has collected data from the ore bodies in the mine at Malmberget. Pejman Oghazi from the Division of Mineral Processing has collected data from the concentrator at Malmberget and Björn Kvarnström from the Division of Quality and Environmental Management has collected data from the pelletising plant at Malmberget. The main theme of this project is to achieve traceability from the mine to the final product.

1.3 Outline of the thesis

In this thesis, a short introduction of traceability in batch and continuous processes is done in chapter 2 and the chapters after that explain mineral liberation and process mineralogy in general. In chapter 5 the experimental part is described and how the sample preparation was done. In chapter 6 the different analytical methods are described but also an introduction to using multivariate data analysis where terms such as PCA, PLS and OPLS is explained in detail. Finally, in the last chapters there are results, conclusions and how future work might be brought forward.

2 THE TRACEABILITY IN A PROCESS

2.1 Process in industry

Processes can be divided into two types: batch processes or continuous processes. There are several types of process industries, and it is important to know the variations between different kind of processes regarding how material is mixed and product is behaving in the process (Dennis and Meredith, 2000).

In order to produce a product, the raw material will go through different operations such as mixing, separation, and chemical reaction. In continuous processes, the material will go through the operation that is mentioned above with nominal interruption.
Most of the research and use of traceability is done for batch processes or part production, i.e. food industry or pharmacy production. By using tags or markers it is easy to follow each batch through the process. In continuous processes there are some factors that differ compared to discontinuous processes. It is not easy to tag the material during the process because the material usually changes state and form, but there are also sub-flows and other reflux flows that are mixed with the main flow that make the traceability problematic. It is necessary to have a good overview over the process in order to be able to choose a suitable traceability method for continuous processes. Further information can be found in paper I. In this case the refinement process in mineral industry is used as an example. It is a complicated process where the material will change state and form. The material goes through crushing, grinding and separation.

2.2 Traceability

Traceability can be defined in many ways with the meaning being able trace or track and get information. Traceability can be defined as the ability to trace the history, application or location of an entity by means of recorded identifications (ISO 8402, 1995).

There is not a lot in the literature about traceability. Töyrylä is one author that is often mentioned when the word traceability is used. Töyrylä’s definition of traceability is “the ability to preserve and access the identity and attributes of a physical supply chain’s object” (Töyrylä, 1999). There are others that define traceability; Kvarnström has made an excellent table in his licentiate thesis (Kvarnström, 2008).

According to Golan et al. (2004) the definition of traceability can be broad because most of the time the processes are very complex. Traceability is a tool used in achieving different objectives and can never be complete. In order to have control over all inputs in a certain process, it requires hard work and is very costly. He also mentions that there are three words that are important in reference to traceability: breadth, depth and precision.

<table>
<thead>
<tr>
<th>Breadth: This concerns the quantity of the information that is collected. It is important to collect the key object that provides us with important information.</th>
</tr>
</thead>
<tbody>
<tr>
<td>Depth: How far back or forward can the traceability be used, and how much is necessary?</td>
</tr>
<tr>
<td>Precision: How accurate should the traceability be in a certain process?</td>
</tr>
</tbody>
</table>
Usually it is not difficult to determine the breadth of the traceability if there is a good overview and knowledge of the process and the product. It is more difficult to know how far back the traceability can be used. The precision is an important part of the traceability and by using different analytical methods and instruments, it will help to achieve a good traceability. However, almost in every case it is the time and the economic issue that reconcile the limitation.

3 MINERAL LIBERATION

In order to be able to separate valuable minerals from the gangue or unwanted minerals, it is important to have good liberation. Liberation is accomplished by crushing and grinding (comminution), and according to Wills (2006) the grinding step is the largest energy consumer in the concentrator but also one of the most important steps.

Analysis of mill products gives valuable information about the mineral liberation. It can easily be done by an optical microscopy; a polished section of the product is enough to judge if the mineral grains are liberated or not. A common method is point counting in grain size fractions. Point counting is a slow process and because of the amount of data that is processed, it is a cumbersome method to rely on.

Today with the help of computers good information regarding mineralogy, particle texture, and liberation can be collected quickly and easily. The degree of liberation for the main mineral in a mill product is then established by analysing a polished section. Mineral liberation can be measured by image analysis in two different ways: the area method or the linear intercept method. The area method is based on the areas of the mineral in question and of the host particle in the polished section, and by calculating the percentage of mineral in the particle the liberation can be known.

The linear intercept method is done by measuring the length of the linear intercept across the exposed areas of the mineral of interest and the host particle in a polished section (Petruk, 2000).
4 PROCESS MINERALOGY

In order to understand the fundamentals of mineral processing, it is important to understand and link every single stage in the process chain. It is the mineralogy and the properties of an ore that determine the conditions for the further processing. To meet these challenges an efficient process can be designed and the mineral treatment can be optimised. (Sutherland, et al., 2000). Moen (2006) defines process mineralogy as the mineralogy which is applied to the product in a specific industrial process such as the mineralogy in the concentrator, pelletisation or in other process stages.

It is necessary to identify what kinds of minerals and textures the ore is consisting of due to the fact that the liberation characteristics are intimately related to the mineralogical texture (Lorenzen, et al.1994). A large amount of information about the ore fines needs to be obtained like particle mineralogy, porosity, mineral association, texture, hardness, size distribution, mineral liberation and mineral composition (Donskoi, et al. 2007).

Process mineralogy in this case can be used as a tool that gives detailed information of the mineralogy from different ore bodies which will be linked to mineralogy in the concentrator to improve the mineral processing performance. Traditionally, optical microscopy has been used for the identification and quantification for both mineralogical and texturally properties, but this kind of instrument is consuming a long process time (Petruk 2000). In the last decades there has been a great development in techniques related to image analyses systems based on Scanning Electron Microscopy (SEM) for a more rapid quantitative estimation and description of mineralogy and particle textures (Gottlieb, et al. 2000; Petruk 2000; Gu 2003). QEMSCAN® and MLA both developed in Australia are the better known instruments. SEM-PTA used in this study is a similar instrument developed at NTNU, Trondheim, Norway (Moen 2006).
5 EXPERIMENTAL

During the last decades, mining industries have opened the doors for new instruments and methods to streamline the production. Scanning Electron Microscopy (SEM) has been in the market for a long time and QEMSCAN is one of the better known techniques for mineral characterisation, associations and mineral texture. This SEM-EDS technique also gives information about the chemical assays (Gottlieb et al., 2000). Particle Texture Analysis (PTA) is a system based on the scanning electron microscope and Oxford Inca software (Moen et al., 2006).

Most iron ores contain significant amounts of gangue minerals that need to be eliminated to produce iron concentrates. At Malmberget, the dominant iron mineral is magnetite, but hematite also occurs (Bergman et al., 2001). Gangue minerals are mostly quartz, pyroxene, apatite and feldspar.

5.1 Flowsheet

Figure 2. Flowsheet for grinding section 5 (Tano et al. 2006).
Figure 2 shows a typical flowsheet for concentrating iron ore at Malmberget. The coarse materials at 10-15 mm in size are fed to a wet magnetic cobbing separator (M1). The magnetic concentrate is discharged into a primary ball mill (#1), and the ground product (pulp) is transferred to a primary magnetic separator (M2). The resultant magnetic concentrate is then pumped into a secondary ball mill (#2). A secondary magnetic separation unit (M3) finally upgrades the ground product and the concentrate is used as feed for the tertiary grinding stage (#3) (Tano, 2005).

The new section 6 at Malmberget lacks the wet cobbing stage. Even here ball mill grinding is used in three consecutive steps and with wet low intensity magnetic separators in between. It is important to grind to approximately 68% < 45μm to liberate gangue minerals and to reach the desirable size distribution for the pellet feed.

Table 1. Data from the mills in section 5 and section 6 (data from LKAB, Malmberget R&D 2007).

<table>
<thead>
<tr>
<th>Section 5</th>
<th>Section 6</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Primary mill</strong></td>
<td><strong>Secondary mill</strong></td>
</tr>
<tr>
<td>Model:</td>
<td>Ball mill, Morgårdshammar (CRRK4254)</td>
</tr>
<tr>
<td>Length / EGL:</td>
<td>5400 mm</td>
</tr>
<tr>
<td>Diameter:</td>
<td>3150 mm</td>
</tr>
<tr>
<td>Inner dimension:</td>
<td>2950 x 3300 mm</td>
</tr>
<tr>
<td>Total effect:</td>
<td>1800 kW</td>
</tr>
<tr>
<td>Used effect:</td>
<td>1450 kW</td>
</tr>
<tr>
<td>Revolutions Per Minute:</td>
<td>16.0 rpm</td>
</tr>
<tr>
<td>Critical Revolutions:</td>
<td>21.3 rpm</td>
</tr>
<tr>
<td>Total filling:</td>
<td>40%</td>
</tr>
<tr>
<td>Grinding media:</td>
<td>Steel balls 60 mm</td>
</tr>
<tr>
<td><strong>Secondary mill</strong></td>
<td><strong>Tertiary mill</strong></td>
</tr>
<tr>
<td>Model:</td>
<td>Ball mill, Outokumpu</td>
</tr>
<tr>
<td>Length / EGL:</td>
<td>6500 mm</td>
</tr>
<tr>
<td>Diameter:</td>
<td>4600 mm</td>
</tr>
<tr>
<td>Inner dimension:</td>
<td>4370 x 6300 mm</td>
</tr>
<tr>
<td>Total effect:</td>
<td>2800 kW</td>
</tr>
<tr>
<td>Used effect:</td>
<td>2000 kW</td>
</tr>
<tr>
<td>Revolutions Per Minute:</td>
<td>15.1 rpm</td>
</tr>
<tr>
<td>Critical Revolutions:</td>
<td>20.1 rpm</td>
</tr>
<tr>
<td>Total filling:</td>
<td>26% (max 40%)</td>
</tr>
<tr>
<td>Grinding media:</td>
<td>Steel balls 70 mm</td>
</tr>
</tbody>
</table>

In this work all the focus was on the grinding circuits and the samples were collected from both section 5 and section 6. In Table 1 all the data from the mills from the two grinding sections are shown. In both sections, three mills are used in series. Mills from section 6 are larger compared to section 5 and have a higher capacity.
5.2 Sampling and preparation

In this project, samples were taken in two campaigns from the concentrator at LKAB in Malmberget. In the first campaign, samples were collected from both sections 5 and 6 simultaneously. They were taken before and after each mill with one subsample every 20 minutes for the duration of three hours.

The second sampling campaign was aimed at the reflux process. Here the focus is material flows that are recycled into the main process flow from thickeners and magnetic separator tailings.

The samples were weighted and then filtrated at Malmberget. All the samples were then dried and cut by a Jones splitter into suitable proportions in the laboratory at Luleå University of Technology (LTU). The dry material was sieved with a Ro-Tap shaker down to 75 μm and wet sieved further to 38 μm.

![Figure 3. Polished thin section of the sieved material.](image)

Polished thin sections were made of the sieved fractions at NTNU.
6 CHARACTERISATION AND ANALYTICAL METHODS

6.1 Instruments

6.1.1 Binocular/Optical microscopy

Mineral identification can be made by two different modes. The first stage is the macro-scale by simply using a microscope, acids and a knife (Petruk, 2000).

The second stage contains the micro-scale methods that are possible to be used for mineral identification. Optical microscopy, X-ray diffractometry (XRD), scanning electron microscopy (SEM) and electron microprobe (MP) are some of the equipment that can be mentioned.

Optical microscopy is very important as a first approach. Usually a binocular microscope is used in the first step for larger pieces. The binocular microscope gives us the first information of minerals and the textures. The magnification should not be over 50x.

For a careful examination the polarisation microscope is used. The samples are usually prepared before they are examined. They are polished or polished thin and with reflected polarised light it will be easier to identify minerals, mineral texture, particle shape and measure the grain size. The magnification may be up to 600x.

Figure 4. Example of the microscope, which can be used in optical microscopy (Nikon Eclipse E600).
There are two polarising filters (the polariser and analyser) in the microscope. The polariser is placed below the specimen stage and the analyser sits above the objectives and can be moved in and out of the light path as required. When both the analyser and polariser are in the optical path, their permitted light vibration directions are positioned at right angles to each other. In this configuration, the polariser and analyser are said to be crossed with no light passing through the system and a dark field of view is present in the eyepieces unless the mineral specimen changes the polarisation of the light passing through it. Polarising microscopy can be used both with reflected and transmitted light. Reflected light is useful for the study of opaque materials such as minerals. In this case polished thin sections were optically examined in transmitted and reflected light by a standard petrographical microscopy (Nikon Eclipse E600).

To easily characterise all the different mineral associations and textures, a mineral identification was made of both the silicates and oxides. In this way, minerals are divided into two groups and identified more quickly.

### 6.1.2 Microprobe

An electron microprobe is a microscope that uses electrons to examine a sample. The microprobe is also used to qualitatively or quantitatively determine the chemical composition of a very small particle (1 micron) on a sample surface. It will also give a good overview over the chemical characterisation of the sample.

Mineral analyses were performed on a JOEL JXA-8500F electron microprobe at NTNU, Trondheim, Norway. For the microprobe analyses an accelerating voltage at 15.0 kV, a probe current at 95 μA and a < 1μm beam diameter were used.

Totally over 200 point analyses were made representing both silicates and oxides on the major minerals to cover all different textural and mineral assemblage variations observed in the samples.
6.1.3 Scanning electron microscopy

Scanning electron microscopy (SEM) is close to the microprobe. The SEM produces images of high resolution, which means samples can be examined at a high magnification. The samples have to be conductive and the electrons from the SEM interact with the atoms in the samples that compose the signals that contain information about the surface and the composition (Watt, 1997). Back scatter electrons (BSE) are the electrons that are backscattered from the sample when the primary electron beam hits the target and it is dependent on the atomic number of the phases.

The particle analyses were done on a Hitachi S-4300SE scanning electron microscopy equipped with Oxford Inca Feature software, NTNU, Trondheim, Norway. For the particle analyses, an accelerating voltage of 20.0 kV and a probe current at ~0.5nA was used.
6.1.4 Particle texture analysis

The Particle Texture Analysis (PTA) software is developed at the Norwegian University of Science and Technology (NTNU). By using Back Scattered Electrons (BSE) from the scanning electron microscopy, images are analysed by means of grey levels and every grain of interest is also analysed by X-rays. All analysed grain size fractions are imported to the PTA software where images analyses are done offline to process and evaluate if grains occur as liberated or in composite particles.

Standard queries can be done on the output results in a new database that contains information on the mineral liberation of any mineral, mineral association of any mineral and miniature images of particles of a certain texture category (Moen 2006).

In order to reduce the unclassified group of minerals, an extensive identification of minerals and phases for classification should be done.

6.1.5 QEMSCAN

Each mineral has its own distinctive energy dispersive X-ray spectra, and QEMSCAN is an automated system based on a scanning electron microscope. When the particles are analysed, QEMSCAN uses the raw data from X-ray spectra to compare with a mineral identification library known as the Species Identification Program (SIP). For more information see Lotter et al. (2003). The data for each examined point are evaluated against the list elements that exist in the sample. The system is normally equipped with four x-ray detectors. Some species with similar x-ray spectra and backscatter images are differentiated by their element ratio. Some minerals such as magnetite and hematite with similar x-ray spectra are differentiated with the back scatter images (Gottlieb et al. 2000).
6.2 Multivariate data analysis

Multivariate data analysis (MVDA) is used when numerous data are collected from different systems or measurements from a process. Martin et al. (1996) writes that the bases of MVDA are the projection techniques such as Principal Component Analysis (PCA), Projections to Latent Structures (PLS) and also Orthogonal Projections to Latent Structures (OPLS) that is explained later in this chapter. Hair et al. (1998) argues that MVDA refers to statistical methods that consecutively analyse multiple measurements on an entity. For having better overview of a process, it is necessary to collect data with different variables. By using MVDA many of these variables will be explained and expressed making it easier to understand which variable is the information-rich variable. In order to achieve good results and have better control, numerous industries collect countless amounts of data from their processes where there are many variables and even more observations. A data tool that can extract systematic information in latent variables is therefore needed. One of these tools is SIMCA-P+ (Eriksson et al. 2001) where versions 11.5 and 12.0 have been used.

6.2.1 Principal component analysis

Multivariate data analysis is based on projection methods; it builds a model by using a data matrix X containing N observations (samples). The Principal Component Analysis (PCA) is a multivariate projection method that reduces dimensionality of the data (Kourt et al., 1996) by showing us how the observations are related to each other in a simpler way. PCA and the standard multivariate statistical methods are explained by different researchers and also in many textbooks, e.g. (Kresta et al. 1991, Wold et al. 1987).

As mentioned earlier PCA is a projection method of the original variables onto new ones, orthogonal and arranged according to their eigenvalue. This is done by splitting the matrix X as $X = AB^T + E$ where A represents the score matrix, B represents the loading matrix and E is the residual error (Prats-Montalbán, 2006). The result from PCA can be interpreted by using score plots and loading plots. Score plots are created by observations scores on the principal components (Eriksson et al. 2001). PCA have many advantages with one of them being to show if there are outliers among the collected data and to be able to investigate them closer if it is needed. It is also useful to detect groups and patterns in the data.
6.2.2 Projections to latent structures by means of partial least squares

Projections to Latent Structures (PLS) is another way to analyse how different variables are critical in the model. According to Wold et al. (1999), PLS is an ideal method to relate the two matrices X and Y. By selecting different variables as Y, it will give information how the selected variable(s) is/are related to other variables in the model. PLS will allow for several Y variables at the same time in a model. In particular, PLS modelling works best when the data are comparatively symmetrically distributed, therefore, they are often logarithmically transformed before the analysis. Transformed data are often used when the skewness of a variable is very large, both positively and negatively.

6.2.3 Orthogonal projections to latent structures

Trygg and Wold (2002) mentions that for achieving higher interpretational ability of result from PLS models, Orthogonal projections to Latent Structure (OPLS) can be used. OPLS removes the non-correlated data variation in X to the response set Y, which means remove variability in X that is orthogonal to Y. According to Trygg there are a number of benefits in using OPLS to achieve better models:

- The orthogonal variation can be analysed separately.
- Easier to detect outliers in the scores.
- Decreased of the total number of components.
- The score and loading plots can be interpreted without any relation to Y.

6.2.4 PLS- and OPLS-Discriminate Analysis

PLS Discriminate Analysis, PLS-DA, is another method to sharpen up the separation between the groups or observations, and helps to understand which variables are imperative for the separation of groups. OPLS-DA is an improvement on the PLS-DA to discriminate two or more groups in multivariate analysis (Bylesjö et al., 2006). OPLS-DA concentrates all the discriminatory information into the first component, whereas PLS-DA explains the deviation spread out over all the components (Eriksson et al., 2006). In other words, the predictive OPLS-DA component explains the direction of the difference between different classes in a better and easier to understand way (Westerhuis et al., 2010).
7 RESULTS

In this chapter the results are summarised by presenting the six papers. Initially, the relationship between the papers is described and followed by a list of papers.

7.1 Relationship between the papers

Paper I discusses traceability from a broad perspective, but also how it is possible to achieve traceability in a continuous process. Different methods are described in the paper. Paper II continues to go deeper and look at the concentrator plant and the grinding section. Further, with results from the first paper, paper III shows different attempts to achieve traceability in the mining industry. In paper IV more process data are investigated in detail by looking into flows that are recycled back to the main flow. Paper V develops the result from paper IV by using multivariate analysis tools in a deeper manner. In the last article, results from paper II are compared by using different analytical tools.

Figure 5. Relationship between the papers to each other.
7.2 Summary of paper I


7.2.1 Purpose of the paper

The main purpose of this paper is to explain different traceability methods that may be used to achieve traceability in continuous processes.

7.2.2 Outcome and conclusions

The traceability methods can be divided into two groups, off-line and on-line. Off-line methods aim to measure the time a particle stays in the process section and again when it will enter the next process stage. Different particles/atoms behave differently in the process stage, therefore, the resident time distribution (RTD) must be estimated. It can be measured by adding a tracer substance in the process section. There are some important factors that must be under consideration. One of them is that not all of the particles have the same RTD. Hence, materials with different physical properties behave differently in a process step.

On-line methods require longer preparation and longer implementation times because in each case a specially suited method is used.

In this paper there are three methods discussed:

Material signature where to find a material signature or a “fingerprint” in a process it is important to sample and analyse the process carefully. The signature does not have to be unique to an individual particle; it may be unique for a group of particles. This method is appropriate to use when the material in the process is shifting shape or material with different properties are mixed. There are different methods in which to find a mineral signature with usually optical image analysis being used in the mineral industry.

Process data is another on-line method that can be used whereby comparing different variables from the process and doing advanced analysis on the data, it will help to achieve traceability in the process. It is easy to collect the process data because usually the measurement tools are already installed in the process.
**Traceable unit** is also an on-line method that is usually used to achieve traceability in batch processes. In continuous processes markers can be used in the material flow to achieve the same result, but there are some issues that must be under consideration. In order to have a superior model, it is important that the markers behave as the material in the process and also that they are durable while they go through different process stages. Radio frequency identification (RFID) is one technique that has been discussed. RFID tags have unique identification numbers that can be detected during the process and create a traceable marker. In the mining industry it is not demanding to measure them and is simply done by using a receiver that can measure the tags automatically. On the other hand one problem is the fragility of the tags; they can not be used in grinding or in high temperatures.

It is possible to use a mixture of these methods as well with all being dependent on the existing process.

### 7.3 Summary of paper II


#### 7.3.1 Purpose of the paper

In this paper the grinding sections have been in focus and the data are collected from the old and the new grinding sections. The main task is to find a way to make the traceability easy and practical. One way to reach traceability would be to find a process mineralogical signature or identification. For having a good traceability, we need information from the system. It is important to analyse and look into the variables that have a crucial importance to the process.

#### 7.3.2 Outcome and conclusions

The experimental effort in this paper can be described in three parts: pre-experimental preparation, performing the experiment and analysis. In order to improve the model it was important to reduce the number of variables while the variables of interest were those which describe the morphology, cf. Table 2. By using multivariate tools it was possible to find trends, dominating variables and groups among the collected data. In using this kind of analysis, it is possible to create an understanding of the general differences between the two grinding sections.
Data collected from the PTA analysis were subjected to multivariate analysis. From a statistical overview, a multivariate model explaining the observations was developed. In the result from the analysis in this paper, principal components and score plots were examined to investigate which and how different parameters affect the model during the grinding process.

Figure 6, which is a loading plot, shows the importance of different morphological variables in the X matrix. It explains how the variables contribute to the model that is shown in the score plots, cf. Figs. 7 and 8.

Table 2. List of mineral identifications and parameters which were extracted from PTA and used for analysis. (The morphology parameters are written in *italics*.)

<table>
<thead>
<tr>
<th>Minerals:</th>
<th>Parameters:</th>
</tr>
</thead>
<tbody>
<tr>
<td>Magnetite</td>
<td><em>Area</em>; (Area of whole feature in square microns)</td>
</tr>
<tr>
<td>Ilmenite</td>
<td><em>Length</em>; (Max feret)</td>
</tr>
<tr>
<td>Rutile</td>
<td><em>Breadth</em>; (Min feret)</td>
</tr>
<tr>
<td>Plagioclase</td>
<td><em>Perimeter</em>; (Perimeter of whole feature in microns)</td>
</tr>
<tr>
<td>Quartz</td>
<td><em>Aspectratio</em>; ( \frac{\text{Length}}{\text{Breadth}} )</td>
</tr>
<tr>
<td>Pyroxene/Amphibole(Pyx/Amf)</td>
<td><em>Direction</em>; (Angle)</td>
</tr>
<tr>
<td>Biotite</td>
<td>( \text{Shape} = \frac{\text{Perimeter}^2}{4\pi \times \text{Area}} )</td>
</tr>
<tr>
<td>K-feldspar</td>
<td><em>ECD</em>; ( \sqrt{\frac{4 \times \text{Area}}{\pi}} )</td>
</tr>
<tr>
<td>Enstatite</td>
<td><em>Mean grey level</em>; (Mean image grey level for each particle)</td>
</tr>
<tr>
<td>Apatite</td>
<td></td>
</tr>
<tr>
<td>Titanite</td>
<td></td>
</tr>
<tr>
<td>Calcite</td>
<td></td>
</tr>
<tr>
<td>Sulphides</td>
<td></td>
</tr>
<tr>
<td>Unclassified</td>
<td></td>
</tr>
</tbody>
</table>
The loading plot in Figure 6 shows that size parameters carry most of the information in the first direction while mean grey have a large influence in the third direction. The first direction is length information.

The score plot (Figure 7) shows the relationship among the observations (minerals). This plot can be seen as window into the X space where the objects (particles) are projected on a 2-dimensional hyperplane. Note that the knowledge of the mineral identification is only used to interpret the groups in the score plot by colouring the individual observations (mineral grains).
Figure 7. Score plot with mineral identification for incoming and outgoing material, section 5, primary mill.

In Figure 8, there are several sub-populations to the lower left. These may be interpreted as mixed particles, since they represent relatively smaller, darker objects.

Figure 8. Score plot for incoming and outgoing magnetite, section 5, primary mill.
The score plots did show that there are some sub-populations for the magnetite. It is, of course, interesting to follow a single mineral comprehensively for the length of a grinding circuit, and establish to what extent the number of presumably mixed particles decrease or not.

Looking into these samples from the grinding section is state-of-the-art by using these types of techniques and methods.

### 7.4 Summary of paper III


#### 7.4.1 Purpose of the paper

This paper discusses the possibility to have traceability in the mining industry by parameters and signatures like particle mineralogy, mineral association, texture and mineral liberation. The study is on an apatite-iron ore deposit at Malmberget, Sweden, and characterises an ore body both mineralogical and texturally in a quantitative manner by using analytical methods.

#### 7.4.2 Outcome and conclusions

The textures of the two ore types: ore and ore breccia, were identified by optical microscopy on drill cores. Ore breccia is characterised by magnetite grains with a simple euhedral outline and straight grain boundaries, either as single grains or as aggregates of particles in a matrix of quartz and feldspar.
By studying the process mineralogy of the ore, it is possible to understand the behaviour of it and also have information of what is coming to the concentrator as ore feed. An automated system technique makes it possible to get consistent signatures from both the ore bodies and the ore feed and creates the possibility to achieve traceability between mine and concentrator.

The signatures that have been identified and can possibly create traceability between mine and concentrator as well as through the grinding circuit are the mineral associations of apatite and feldspar to magnetite.

The modal mineralogy on the major ore minerals seems not to be a useful traceability tool since the content of these minerals will change in any upgrading process. However, the modal mineralogy may be used to understand how minerals break into and out of fractions during the grinding process, but also to trace the gangue minerals and other trace minerals in the ore body. This paper will confirm an attempt to find traceability at early stages in ore beneficiation and the results indicate that there is a good possibility to achieve traceability in continuous processes.
7.5 Summary of paper IV


7.5.1 Purpose of the paper

Understanding and optimising the flows that are recycled back to the main flows are of significant concern in process industries. They are re-routed into the ordinary flowsheet since they are thought to be too valuable to be sent to any tailings pond. External flows come from multiple sources, e.g. drainage sumps, spillage thickeners, depleted products etc.

Particle Texture Analysis (PTA) is used to investigate external flows and compare them with existing ordinary flows. It is then possible to pinpoint from a process mineralogy point of view to what extent the external flow in question fits into the ordinary processing flowsheet.

7.5.2 Outcome and conclusion

Modal mineralogy is one way to present the results from the process mineralogy analyses. It is a convenient way to understand the content of different flows. This type of analysis may be used to:

- Identify what sort of material is pumped to the grinding section
- Optimise the process by sending external flows to the best access point in the main flowsheet
- Reduce any overloading in any process step by decreasing unnecessary volumetric flows
This way for collecting total information on how each unit operation is working and what sort of material is recycled to the main flowsheet is very effective. The ideal case is when complete information for each external flow that is added to the main streams exists, and it is then joined to an optimal position in the main flowsheet. However, if the gangue minerals are locked with magnetite they will ultimately end up in the final concentrate since current low-intensity magnetic separators pick any particles with small magnetic volumes. Therefore, mineral association analysis is the most important tool in deciding where to send recycled flows.

Results from analyses show that some recycled flows that are reconnected to the main flow are not connected to the best point. A side effect of the analysis is that it indicates that some flows may be sent to later grinding stages. Thus, the load on the primary mill is decreased, while the retention time in this mill is increased. As a result, the capacity of the grinding section is increased since the primary mills are the bottle-necks of the process.

7.6 Summary of paper V


7.6.1 Purpose of the paper

This article presents the results from a part of a project between Luleå University of Technology (LTU) and the Swedish mining company Luossavaara Kirunavaara AB (LKAB). The focus on the main project is to find a traceability method for a continuous process (Oghazi et al., 2007). Traceability is the ability to access the history of a specific object in the production line or specific material in the process line, which gives the abundance of possibilities for quality control (Flodin et al., 2008).

Earlier in this project samples were analysed from the main flow by using Particle Texture Analysis (PTA) where the mineralogy and morphology were identified (Oghazi et al., 2009). This kind of analysis from the process has shown good results and a general view over different events in the process. Today there is no detailed information about the mineralogy in the recycled flows, which is not optimised from a process technical viewpoint.
In this case, samples were taken from the recycled flows that are sent back to the ordinary or the main flow in a concentrator. The purpose of this study is to understand the difference between the recycled flows that are connected back to the main flow and by doing so it will reduce the production and process disturbances.

7.6.2 Outcome and conclusions

In the first analyses samples #1, 2, 3 and feed to section 5 were combined in the same analysis. The main reason for this grouping was that samples #1, 2 and 3 are very similar to each other and also to observe how they are linked to the raw material that comes into section 5. If they show comparable classification to each other, then they could be reconnected directly to section 5. The data set contains 8 variables and 59,413 observations measured on 4 groups.

PCA will not reveal any connection between the groups, but it will show how the variables are connected to each other. Figure 10 shows a loading (variable) plot for the first and third principal component since these directions give the best visible separation of the variables. Mean grey is the variable that is strongly shifted in the third direction, while morphology parameters are assembled down to the right in the first direction.

![Figure 10: PCA, Samples 1, 2, 3 and Section 5.](SWCA-P+ 12 - 2010-09-21 10:32:10 (UTC+1))
The figure below shows the relationship between the X-variables and the Y-groups as well as the relationships within them. The plot shows how the responses (Y's) vary in relation to each other, which ones provide similar information and their relationship to the terms in the model. According to Eriksson et.al (2001), PLS-DA is like having two PCA models at the same time, one for X and one for Y. In the figure it is shown that feed to section 5 and sample #1 are close to each other and located more on the right side of the graph compared to other samples. The reason for this is in the first direction where mostly Mean grey influences the outcome. From the loadings plot it is seen that variables Section 5 and Sample #1 are affected positively mainly by Mean grey meaning that they contain more magnetite particles.

Figure 11: PLS, Samples 1, 2, 3 and Section 5.
This paper studied the recycled flows and the feed in the concentrator by monitoring the process data with multivariate data analysis such as PCA, PLS-DA and OPLS. This was shown by using the mentioned methods and in some cases also taking the logarithm of the data. There was no specific difference in the result when the data were logarithmically transformed, and further analysis shows that OPLS was the monitoring method that has both strong correlated variables and good separating force between different variables. That said, it must be pointed out that the multivariate methods do not give the absolute quantitative truths, rather rapid qualitative information about the differences and similarities between process flows.

7.7 Summary of paper VI

Oghazi, P., Pålsson, B. (2011). Comparing the mineralogical characterization of iron ore by using QEMSCAN and PTA. 10th International Congress for Applied Mineralogy (ICAM) in Trondheim, August 2011

7.7.1 Purpose of the paper

The PTA and QEMSCAN results are represented by modal mineralogy charts in this paper. Charts of modal mineralogy are showing the percentages of minerals found in the analysed grain-size fraction based on examination of a sample. The purpose is to find any similarities and divergences by using these two analytical methods.

In this paper the same samples are analysed with two different techniques to see if there are any differences between the systems. In this case ores that are fed to the concentrator come from different ore bodies, which are mined and mixed for the beneficiation process. The samples are material from input and output from three ball mills in series from two different grinding sections.

7.7.2 Outcome and conclusions

Comparing section 5 by the analysis method used, the similarities are obvious. The magnetite amount that is detected is almost the same. This can be said for the gangue minerals amount as well. However, there are a few more percent gangues minerals detected for section 5 by the QEMSCAN compared to the PTA. The reason for that could be the limits setup in the secondary SIP file.
Figure 12. Modal mineralogy chart for concentrator section 5 from PTA analysis (in wt per cent)

Figure 13. Modal mineralogy chart for concentrator section 5 from QEMSCAN analysis (in wt per cent)
The differences come into play when the data are further processed, cf. Figure 14. In particular, the liberation analyses seem to be dependent on the algorithms used and their tolerance limits.

![Liberation of Apatite in QEMSCAN and PTA](image)

Figure 14. Liberation of apatite in QEMSCAN and PTA.

A more general difference is that PTA can easily deliver morphological data for further processing. QEMSCAN is a more “locked in” system then PTA. This is good for quantitative industrial research but a disadvantage in more fundamental investigations.

Both systems give almost identical results for the mineralogical identification and mineral content. However, there are a few more percent gangues minerals detected for section 5 in the QEMSCAN compared to the PTA. The differences come into play, when the data are further processed.
In this chapter conclusions are presented as well as recommendations for future work.

There are different methods that can be used to achieve traceability in a process. Therefore, it is important to have a good knowledge from the existing process so that the right traceability method can be applied. During the comminution there are physical changes in particles throughout the grinding circuit, the particle size of the ore is reduced and in the last mill the numbers of particles are very high in the slurry which makes the traceability more complex. In this case, material signature is used to apply traceability in the grinding circuit at LKAB, Malmberget.

Samples from the concentrator are analysed by PTA. It is a very flexible system and gives a good deal of information from each sample. Firstly, it is not often that slurries from a grinding circuit have been examined in such detail. In using a state-of-the-art system to investigate the main and trace minerals in the slurry, useful information is collected. By looking into the particle texture and gathering information about the mineral liberation, mineral association and modal mineralogy, useful data are collected. Multivariate data analysis is then used to treat the data from PTA to find out trends, dominating variables and groups among the collected data. Multivariate analysis compares the data several thousand particles at the same time; this is a state-of-the-art technique when used for process mineralogy in this thesis.

As discussed earlier two different analysis methods were used (PTA and QEMSCAN). Both systems give almost identical results for the mineralogical identification and mineral content. The differences come into play when the data are further processed. This is something that should always be considered in order to avoid any errors in interpreting and comparing with future results.
By comparing MVA-data from grinding sections systematic similarities and differences are found. The result from recycled flows and the feed in the concentrators was monitored by multivariate data analysis such as PCA, PLS-DA and OPLS. These methods give the possibility to automatically reduce insignificant data and emphasis variables that affect the process for more rapid interpretation of the results. There was no specific difference in the result between the models used; however, further analysis shows that OPLS was the monitoring method having both strong correlated variables and good separating power between different variables. Secondly, this kind of investigation gives detailed information from the concentrator that can be useful for other aspects as well, such as optimising the process. Therefore, in order to have complete information from the grinding circuit, it is necessary to have data from all the flows that is connected to the main slurry.

In this thesis the focus has been on the mills and how the material is behaving in the grinding circuit by using a combination of process mineralogy and multivariate data analysis. Although, it must be pointed out that the multivariate methods do not give the absolute quantitative truths, rather rapid qualitative information about the differences and similarities between process flows. Multivariate analysis is a functional tool to interpret differences in process mineralogy signatures and does so in an un-biased way. As discussed earlier it is complicated to achieve traceability in continuous processes. Paper I gives a good description of different traceability methods as well as information for creating traceability in a continuous process.

Moreover, paper I summarizes different traceability methods, and to achieve traceability more than one method should be considered, since there are different sections in a continuous process.

In Paper III an attempt to possibly create traceability between mine and concentrator is complete. The result shows that the focus should be on gangue and trace minerals to create traceability in the Mine-to-Mill sequence.
Results from papers II, V and VI show that using particle analysis and multivariate data analysis is a proper way to present the data from the process. Still there are some limitations regarding analytical speed and interpretation of results. Today it is very common with bulk analysis, but it does not show how individual minerals behave at different stages. In the future it is important to develop a system which handles both bulk analysis and image analysis. With this kind of analysis, any irregularity would be detected in the process line and reduce the quality deviation in the product. Presently, it takes time to get data from PTA because of the queue time of analysis. Another issue is that proprietary liberation algorithms makes the comparison of liberation data between different automated systems difficult. By installing a similar system on site, it would be more efficient to accomplish this type of analysis. Of course, it will take time and effort to have it implemented as a standard process-monitoring tool, but the benefit would be a better coupling between mine, cobbng plant, concentrator and maybe the pelletising plant.
9 REFERENCES


Paper I

METHODS FOR
TRACEABILITY IN CONTINUOUS
PROCESSES
- Experience from an iron ore
refinement process

Kvarnström, B., Oghazi, P. (2008)

Published as:
Methods for traceability in continuous processes–Experience from an iron ore refinement process

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Abstract
Every production process is exposed to disturbances leading to production of defective products. The disturbances are seldom immediately discovered, and need to be traced backwards. Traceability, or the ability to follow a product through the process, is therefore vital since it aids the localisation of the source of the disturbance. Traceability has for a long time been a possibility in part production, but in the continuous process industry it is still problematic. Examples of problems are complex flows, closed systems and large buffers. Hence, the purpose of this paper is to describe methods that can be used to achieve traceability in continuous processes, and give an example of when they may be applied. To identify suitable traceability methods, the literature search was conducted as well as discussions with researchers from the process industry. How the methods work is presented together with their advantages and disadvantages. Furthermore, an example of which traceability methods could be used for achieving traceability in a continuous iron ore refinement process is given. Seeing the diversity of available methods, achieving traceability in continuous processes should be possible.

Keywords: Residence time distribution; Iron ores; Ore mineralogy; RFID

1. Introduction
An announcement of a product recall of about 90,000 locomotive toys was issued in September 2007 by Fisher-Price (US Consumer Product Safety Commission). Product recall announcements like the mentioned one are issued daily by companies around the world and most consumer product stores handle product reclaims daily. This exemplifies that defective products and product deviations are not always identified directly, even if this is desirable. Instead the causes often need to be traced back through the process from customer complaints. The ability to trace a specific product during the process, usually called traceability, is consequently important for identification and elimination of causes of product deviations. Other benefits of traceability are that it minimises the extent of product recalls and ensures lot uniformity in products (Juran and Gryna, 1980). Furthermore, traceability can be used to identify causes of positive changes in product characteristics.

Traceability is common in part production and often easy to achieve, since various kinds of identification markers can be attached to a unit. Moreover, the literature about traceability is dominated by applications in part production. However, creating traceability in continuous processes implies vast challenges: process flows can be parallel, serial and reflux; sub processes can be continuous as well as batch-wise. These challenges imply that other types of traceability methods are needed for creating traceability. To understand more easily how these traceability methods can be applied, the authors consider that an example is appropriate. A suitable continuous process for exemplifying how various methods could be used was found in the iron ore mining industry, and the refinement of iron ore...
to iron ore pellets (the process is described in Section 5).

The reason for choosing the iron ore refinement process is that all the special challenges connected to continuous process industries are present in this process. Therefore, the purpose of the article is to compose and describe different traceability methods that can be used for achieving traceability in continuous processes, and to illustrate how a method for traceability may be selected from the characteristics of a process section. The refinement process of iron ore starts in the mines. However, in this paper only the methods for achieving traceability from the concentrator plants to the final customer are discussed.

2. Research methodology

This article is a result of extensive collaboration between the authors and the Swedish mining company LKAB. This collaboration focused on traceability and traceability methods in continuous processes. As a part of this collaboration, an iterative literature search was conducted aiming at identifying traceability methods. An initial literature search was performed in the databases Compendex, Emerald and ScienceDirect.

Most of the articles found were primarily related to other subject fields than traceability methods in continuous processes. However, from the related articles and discussions among the authors, colleagues and researchers at LKAB, new search strings, such as residence time distributions among the authors, colleagues and researchers at LKAB, were identified. The second literature search together with the initial one led to the identification of the traceability methods described in this paper. The identified methods are described in Section 4, and each description includes a brief explanation of advantages, disadvantages, and examples of application. A conclusion from the literature search is that literature on traceability methods in continuous processes is rare and scattered in a diversity of research fields.

3. Theoretical framework

3.1. Traceability

In this paper three terms for traceability with distinguishing aims will be consistently used: traceability, traceability system, and traceability methods.

Traceability is here defined as “the ability to preserve and access the identity and attributes of a physical supply chain’s objects” (Töyrylä, 1999, p. 38). Traceability is not binary but continuous and always present at some level. This means that it is possible to differentiate between what year, week, or day a product was manufactured.

Traceability system is defined as the system that enables traceability in a process by combining process information with models of material flow in the production process. According to Steele (1995), Töyrylä (1999) and Jansen-Vulers et al. (2003), there are four elements connected to the design of a lot-tracing system:

1. **Physical lot-integrity** – How large a batch of raw material is and how well the integrity of the batch is maintained will determine the resolution or precision of the traceability system. The resolution of a system is the minimum number of units that cannot be individually separated during the process, and for example emanate from the same delivery batch.

2. **Data collection** – Two types of data are needed: process data that records process information, and lot-tracing data that keeps a record of movement and merging of batches.

3. **Product identification** – The linking of product and process data.

4. **Reporting** – Retrieval of data from the system, the actual use of the system.

The most important element is physical lot-integrity, since it determines the maximal resolution of a traceability system. The physical lot-integrity for a process is affected by three elements: lot-mismatching, lot-end-mixing and lot-sequence mixing (Steele, 1995). Lot-mismatching occurs when a new batch is created and the size of the batch does not match the original one, for example when numbers of units simultaneously treated in two process steps differ. Lot-end-mixing arises if lots are processed in repetitive or continuous batches and the organisation fails to retain clear separation between batches, for example when products from parallel process steps with different cycle times are merged. Lot-sequence mixing takes place if the traceability system depends on the first-in–first-out principle and the process fails to pursue this principle, for example when all the rework is conducted at the end of a shift. The physical lot-integrity element is also the element that is primarily affected by the differences in continuous production and part production, since batches are usually not present in a continuous process. The traceability system used for achieving traceability in part production should also be useful for achieving traceability in continuous production, since it is only the methods for creating the models of material flow that differ.

A traceability method, the third term, is defined as a method that can be used for creating models for material flow in process sections. The same traceability method is rarely suitable to use during the process, because of changes in material properties and various operations in process stages. Therefore, suitable traceability methods need to be identified for different process sections, and the material flow, consequently, needs to be modelled step-wise. The traceability methods that are applied in part production processes can seldom be used in continuous processes, due to the mentioned differences.

The relations between the three terms may be described according to the following criteria: models for material flow in process sections are constructed with traceability...
methods. The different material flow models for the process sections are then combined by the traceability system to achieve traceability through the process.

The precision of traceability in a process is therefore dependent on the traceability system, which in turn relies on the models created by the traceability method. A mind-map of how the terms are related and affected by one another is shown in Fig. 1.

3.2. Continuous process

In a continuous process, the products are gradually and with minimal interruptions refined through a series of operations (Fransoo and Rutten, 1994; Dennis and Meredith, 2000). Therefore, there are no natural product lots in a continuous process. The raw material in a continuous process typically comes directly from the natural environment, for instance mines and forests (Fransoo and Rutten, 1994; Hild et al., 2000). In contrast to the raw material in part production, it is usually not processed. Therefore, the raw material in a continuous process is usually afflicted with a larger variation than the raw material in part production (Fransoo and Rutten, 1994; Hild et al., 2000). To minimise the influence of the natural variation in the raw material on the product and avoid interruptions in the production process, large mixing buffers are usually used during the process. Furthermore, reflux flows are common in continuous processes and often necessary for attaining an even and desired output from a process section. The physical characteristics of the material are often changed during the refinement process, which also makes it difficult to define a unit of measure (Fransoo and Rutten, 1994). Finally, the added value in continuous processes is usually quite low (ibid).

4. Description of traceability methods

Traceability systems are usually based on daily observations or mathematical models. The mathematical models are created from measurements of how the material flow is affected by different process parameters. The traceability methods may be classified according to what type of model they create. Traceability methods creating mathematical models are here categorised as off-line methods. In contrast, traceability methods creating models based on daily observations are classified as on-line methods. Off-line methods are used during a shorter period of time, in contrast to on-line methods, which are used repetitively. All on-line methods can be used as off-line methods, but the other way around is seldom possible. Fig. 2 shows a summary of the traceability methods presented in this paper.

All the methods presented are also useful in other types of industries than continuous process industries. However, it is not always necessary to use complicated methods for achieving traceability in continuous processes. An example of such a case is production in a single line with no reflux flows and no buffers.

4.1. Off-line methods (tracer methods)

Off-line methods aim at measuring the time a product/molecule/atom stays in a process section, the residence time, with experiments, and from the collected data creating models of the material flow. But, since some prod-

Fig. 1. A mind-map of how a sufficient level of traceability can be reached in a production process.
and radioactive tracer are the most commonly used methods for studying and investigating the RTD of particles are for studying the material flow. Examples of different methods in the literature studied. These two methods are therefore described below in more detail.

4.1.1. Chemical tracer

One way to identify the RTD in a system is to add a chemical compound to the input and measure the concentration in the effluent flow as a function of time. Numerous chemical compounds are available for estimation of RTD. Furthermore, only an element of the compound may be of interest if the compound dissolves in the material flow. The applied tracer should be a compound that is accurately and easily detectable, has similar physical properties as the studied solid/fluid stream, acts like the studied solid/fluid stream in contact with other surfaces in the process, and, if a fluid is studied, also is completely soluble (Wen and Fan, 1975; Fogler, 2006). In addition, it is important that the tracer does not affect the process or product and it should preferably not be naturally occurring in the process. Before the addition of the tracer, it is necessary to have information about the background variation of the tracer compound or element in the process, the analytical detection limit and the amount of material in the studied process section. This information makes it possible to calculate the minimum amount of compound that should be added.

There are several advantages to the chemical tracer method. It is flexible, since various trace compounds are available. This flexibility implies that the method may be used for processes with gas, fluids, solids and slurry, and in almost any environment. Moreover, special permits are often not required, in contrast to the radioactive tracer method. Hence, the chemical trace method is usually easier to apply than the radioactive tracer method.

Disadvantages include for example that the method quickly becomes costly for larger systems, if combined with expensive tracers and expensive analysis methods, since more samples need to be analysed and an increased amount of tracer needs to be added. The amount of tracer added needs to be increased since the tracer often mixes with the material flow. Hence, when using chemical tracers, the RTD is often estimated stepwise for the material flow in the process. Furthermore, it might be problematic to take representative samples in continuous flows. Such an example is sampling of crude ore in mines (Wills and Napier-Munn, 2006). Using non-representative samples severely affects the reliability of the final model. Finally,
another way to estimate the RTD is to use a radioactive tracer. The radioactive tracer method implies that a part of the actual flow is radioactively charged or that radioactive particles are added to the input. RTD is then estimated by measuring the output, which typically is done continuously with a Geiger counter. The choice of radioactive element depends on the material being traced, the duration of the process, and also interior flow patterns in, for example, reactors. The method is also adaptable to different types of material streams, and can thus be used for analysing flows in most environments.

The major concern with the tracer method is the health hazards linked to the usage and the disposal of radioactive material (Ramaswamy et al., 1995). Consequently, the method usually requires special permits and it is also necessary to ensure that safety regulations are followed. Therefore, the method often demands large resources in the form of time and money.

The radioactive tracer method is often used when other tracer methods are inadequate or when interior flow is of special interest, for example localisation of zones in mixers with poor mixing.

4.2. On-line methods

On-line methods often demand longer preparation and implementation times, compared to off-line methods, since they normally need to be individually suited for the specific process. Therefore, on-line methods are usually more expensive. However, the continuous measurement often results in more accurate estimations of RTD, since the estimation is usually based on more data. Consequently, on-line methods are preferable to use. The final model is also less sensitive to process modifications, since it is possible to continually verify and update the model. The on-line methods described here are the methods that were identified during the literature studies.

4.2.1. Material signature

In many production processes, it is almost impossible to obtain identical raw materials. Such an example is that in a pork chop, the exact amount and combination of chemical elements will depend on the origin, nourishment, soil, birth date and other variables. Hence, almost all pork chops are unique, since they will differ from one another in some of the mentioned factors. Therefore, a way to achieve traceability would be to identify unique signatures or structures in a product. The signature does not need to be unique to individual products, it may instead be unique to a group of products. How small the group must be depends on the demanded precision of the traceability system. Examples of signatures that could be used are fibre length in wood, natural variability in raw material, and variation in chemical composition deriving from differences in background.

4.2.2. Process data

In many production processes, the differences in the raw material result in process data variation. Hence, instead of material signature, variability in process data could be monitored.

Lundqvist and Kubulnieks (1995) created traceability in a paper and pulp production plant using process data. Traceability was possible to create by comparing the appearance time and forms of deviations in kappa number
(a measure indicating the bleach ability of wood pulp) and brightness (a measure of how much light is reflected) at different points in the process. From these comparisons, the RTD for process sections could be estimated, verified, and modelled.

The kappa number and brightness are two product parameters that are continuously measured during the process. Furthermore, the value of kappa number and brightness is often changed by the same absolute value in a process section or not changed at all. If a parameter with similar characteristics as the kappa number and brightness is present in a process, it can be used for achieving traceability.

One advantage of using process data is that the measurement tool usually already exists, and hence no further investments are needed. Furthermore, much data is directly available, since data from the measured points already has been gathered. However, it may be difficult or even impossible to identify suitable variables to use for the estimation. In addition, the variable must display significant alterations over time; otherwise the RTD cannot be estimated by comparing the data for the variables.

4.2.3. Traceable unit

In part production, different batch structures are often used to achieve traceability. Batches do not usually exist in continuous processes, so using batch structures for achieving traceability is difficult. Creating virtual batches by dropping type of marker in the material flow with regular intervals would, however, make it possible to use the batch technique for achieving traceability. The markers then act like imaginary start and end points of the different batches. Recording passage times of the markers along the process would then make it possible to trace a specific batch during the process. To achieve a genuine model, the marker must behave as the material in the material flow. Each marker should preferably be unique, so that the potential mixture in the flow can be modelled. The precision with which a product can be traced is determined by the interval between the markers.

Radio frequency identification (RFID) is one technique that could be applied. The technique offers the possibility to create a traceable marker, by using tags with unique identification numbers. RFID is commonly used for tracking goods in the manufacturing industry. This method is applied today for achieving traceability by some retailers, manufacturers, and health care and pharmaceutical industries (Li et al., 2006). The method has also been used for coal tracking (Lauf); see Wyld (2006) for more on RFID.

One of the strengths of the RFID technique is that every tag is unique and can be measured automatically. Consequently, the residence time in the process can be precisely estimated for each tag. Furthermore, the observed object does not mix with the material flow, and consequently the number of units added to the process flow is not affected by the size of the studied process. Finally, it is feasible to estimate the residence time distribution simultaneously for several process sections, since no sampling is required.

Nevertheless, there are many shortcomings with the RFID technique. One shortcoming is that the RFID technique does not offer the same flexibility for use as other mentioned methods. Examples of attributes that hinder the flexibility are the physical size and fragility of the tags. The fragility results in the tags not being possible to use in process sections with for example grinding or extreme temperatures. Moreover, the RFID technique uses electromagnetic waves for communication between the reader and the tags. Therefore, the technique is improper to use at distances larger than a few metres or when the tags are in direct contact with metals or fluids (Porter et al., 2004). Finally, the technique is sensitive to electromagnetic fields in the surrounding environment.

5. An example from a continuous process

LKAB (Luossavaara-Kiirunavaara AB, Sweden) has since the beginning of the 1900s produced highly refined iron ore products from iron ore mines at Kiruna and Malmberget. The main product is iron ore pellets for blast furnaces and direct reduction furnaces.

In November 2006, LKAB inaugurated a new pelletising plant in Malmberget (hereafter PP2). After the start of the new pellets plant, the production volume increased and therefore the raw material taken from local ores in Malmberget was insufficient to uphold full production at the plant, due to, among other things, concession rights. Therefore, ore from Kiruna mines is also used in the production process. The different ores thus have to be mixed.

The iron oxides in Malmberget have a coarser grain size, different kinds of grain boundaries, different Fe-contents and levels of contaminants compared to those in Kiruna (Geijer, 1930). Hence, the iron oxides from Malmberget and Kiruna behave differently during the refinement process. This implies an increased risk of quality deviations in the final product and therefore more emphasis needs to be focused on traceability aspects, since it is important for the customer that the mineralogical characteristics and chemistry of the final product do not differ considerably over time.

The product affected by the mixing of raw materials is the iron ore pellets (hereafter pellets) produced at Malmberget. As a result, a traceability system is most important for this product. The pellets are produced in a continuous process that is illustrated in Fig. 3 and further described in the following part.

5.1. Production process

The Malmberget mine has more than ten different ore bodies that are currently in production. For each ore body, there are different characteristics such as mineralogical, chemical and textural properties.
From the mine, the material goes to the concentrator plant, which separates the minerals into two parts, tail and product. Before one of the concentrator plants, there is a cobbing step to separate the gangue mineral from the magnetite.

In the two concentrator plants (CP1 and CP2), ball mill grinding is used in three consecutive steps with wet low intensity magnetic separators in between; see Fig. 4 for a flow sheet. It is important to grind to approximately 68% < 45 \mu m to liberate gangue minerals, and to reach the desirable size distribution for the pellets feed. The grinding circuits are the last stage in the comminution; in this stage the particles are reduced by a combination of impact and abrasion (Wills and Napier-Munn, 2006).

Keeping traceability in the grinding sections is problematic, since the particle size decreases resulting in an increase of the number of individual particles. An example of how the particle size distribution is altered during grinding is illustrated in Fig. 5. Moreover, the small size of the material complicates the possibility to achieve traceability in the grinding sections. Another major concern is that each section receives secondary material flows from other sections producing special products and general spillage within each section.

The next step is the pelletising plant. There are two pelletising plants in Malmberget, PP2 and the old PP1 from 1973. A flow chart over the pelletising plants in Malmberget is seen in Fig. 6. Several factors make it difficult to achieve traceability during the pelletising process:

- The surroundings of the material constantly change, from slurry to being dry and finally heated.
- There are several buffers, the result of which is that the flow cannot be characterised by a linear flow (lot-sequence mixing).
- Material from different concentration plants are mixed (lot-sequence mixing).
- The material flow is split into smaller flows and then merged at several places in the production (lot-sequence mixing).
- The material flow differs between the two pelletising plants (lot-end-mixing).
- Some of the process steps entail reflux flows (lot-end-mixing).

After the pelletising plants, the pellets are distributed to the final customers. A flow chart of the distribution chain is seen in Fig. 7. All three elements that may impair the physical lot-integrity are represented in the distribution chain: the transport and storage rooms differ in size (lot-mismatching); there are two possible ways of transportation to customer (lot-sequence mixing); there is no clear separation between batches (lot-end-mixing).

5.2. Application

Traceability can be created in the concentrator plants (Fig. 4) by using the mineralogical signature method. The mineralogical signature method is suitable to use since it can achieve traceability even when the form of the material is changing, as it does during the comminution. Materials from different parts of the process should be sampled regularly and analysed, with regard to the content of mineral at different fractions, mineral liberation and mineral associations. By doing so it should be possible to trace a product in the concentrator plant, since part of the mineralogical signature stays constant during the process.

Samples has been taken from each grinding section and analysed with PTA (Oghazi et al., 2007). The abundance of analysis data makes it necessary to use multivariate tools to identify patterns. By using multivariate analysis it is possible to see correlation between different grinding sections, but no conclusion may yet be drawn (Oghazi et al., 2008). For more details of the multivariate analysis on the PTA data, see Oghazi et al. (2008).

To achieve traceability in PP1 and PP2, several methods have to be used, since no single method is preferable for all the different process stages in the pelletising plants. The process data method is preferable to use for most of the process sections in the pelletising plant, since the material flow can be estimated with existing measurements in the process. However, this method is not feasible for the slurry and mixing tank system (steps one and two in the pelletising plants presented in Fig. 6) and the bin chamber (the

Fig. 4. The grinding section in the concentrator plants (CP1 and CP2) with magnetic separators.

Fig. 5. The typical particle size distribution diagram for the input and output in a grinding section. In the grinding circuit, the $d_{80}$ for the particles is reduced from 4000 \(\mu\text{m}\) to 70 \(\mu\text{m}\) and the variation in particle size is reduced.

Fig. 6. Flow chart for the two pelletising plants at Malmberget.
fourth step in PP2 viewed in Fig. 6). One reason is the large buffer in these process sections, which makes it difficult to estimate the material flow with the existing measurements. The mixing of two materials in the slurry and mixing tank system is another factor that makes the process data method insufficient to use for modelling the material flow.

The material flow in the slurry and mixing tank system can be estimated by using a chemical or radioactive tracer. The chemical tracer method is preferable here, since the material flow can easily be sampled and the size of the system is suitable for using a chemical tracer. Selection of a suitable chemical tracer is the next task. At this stage the material flow is a mixture of solid and fluids, and as previously discussed, the RTD can differ between solids and fluid. However, a previous investigation has shown no significant difference for RTD between solid and fluids in rod and ball mills at LKAB (Andreasson et al. 1985). And since the particles are smaller (same particle size distribution as the material of Mill#3(out) in Fig. 5) in this process section, it is assumable that the particles behave as the fluid. Lithium chloride is suitable as a tracer, because it is soluble in water, does not affect the process or the end product, does not occur in high concentrations, and can easily be analysed.

Estimation of the RTD in the bin chamber can also be made with chemical or radioactive tracer methods. Here too the chemical method is preferable. However, lithium chloride is not suitable as a tracer element since the material is dry. Fluorescent colour can instead be used as a chemical tracer.

The residence time has been measured for the slurry and mixing tank system by chemical tracer substance experiments. In the experiments, lithium chloride was added momentarily to the flow, and the concentration of lithium in the effluent flow was sampled and analysed. Based on the data from the experiments a mathematical model with two tanks was fitted to the data. For more details, see Kvarnström and Bergquist (2008).

In the distribution chain (Fig. 7), the RFID technique, the traceable unit method, may be appropriate to use for achieving traceability. This method is preferable as the residence time in the distribution chain is long and impossible to model by process data, as the material flow is a mixture of batch and continuous flows. Furthermore, the material (the pellets) is only exposed to insignificant external forces and the material is approximately the same size as a tag. The technique could be used to create virtual batches, since it is not realistic to equip each pellet with a tag. The tags would be used as the start and the end points of each batch. Process data can then be linked to a virtual production batch. Tags could be either attached directly to pellets or dropped into the material flow.

The RFID technique has been extensively tested in the distribution chain. The results show that the technique may be used to create traceability in the distribution chain. However, to achieve a sufficient read rate, more than 50%, it is necessary to use RFID tags that are larger than the pellets. In the test, no significant difference in the behaviour between the larger RFID tags and pellets was observed. The tests and the results are further described in Kvarnström and Nordqvist (2008).

6. Conclusion and discussion

Through the literature review and discussions with different researchers, suitable traceability methods for continuous processes were identified; the methods are summarised in Table 1. As illustrated above, there are several methods for creating traceability in continuous processes, and as the example demonstrates, they are applicable at different types of process sections. None of these methods can be seen as a panacea for developing a traceability system, since every method has its own strengths and weaknesses. The methods described in this paper should be seen as examples of methods, and not as a complete list.

Furthermore, the example shows that by applying suitable traceability methods, it should be possible to improve traceability. However, no complete implementation of the methods has been made, and it is therefore still uncertain if traceability can be achieved in the iron ore refinement process. Though the results are promising, there is still work to do before some final conclusions could be made about the possibility to achieve traceability in the iron ore refinement process. For example, the analysis data from the concentrator needs to be further analysed. In
The RTD of the bin chamber also needs to be investigated because the material is profoundly changed, for example, the grinding sections many aspects need to be considered.

Material signature – Flexible – Large amount of data

Process data – Easy to use – Hard to find

Radioactive tracer – Flexible – Health hazards

– No sampling needed – Permits required

– Interior flows can be measured – Based on historical data.

On-line

Traceable unit – High precision – Lower flexibility

– No sampling needed – Cannot be used for fluids

– Could be used in process sections with both batch and continuous flows – Can only be used at shorter distance

Material signature – Flexible – Large amount of data handling

– Informative – Time demanding

– High analyses precision – Costly

Table 1

<table>
<thead>
<tr>
<th>Traceability method</th>
<th>Advantages</th>
<th>Disadvantages</th>
</tr>
</thead>
<tbody>
<tr>
<td>Chemical tracer</td>
<td>Flexible</td>
<td>Dilutes</td>
</tr>
<tr>
<td>– Easy to use</td>
<td>Needs sampling</td>
<td></td>
</tr>
<tr>
<td>– Low-cost</td>
<td>Based on historical data</td>
<td></td>
</tr>
<tr>
<td>Radioactive tracer</td>
<td>Flexible</td>
<td>Health hazards</td>
</tr>
<tr>
<td>– No sampling needed</td>
<td>Permits required</td>
<td></td>
</tr>
<tr>
<td>– Interior flows can be measured</td>
<td>Based on historical data</td>
<td></td>
</tr>
<tr>
<td>Process data</td>
<td>Easy to use</td>
<td>Hard to find</td>
</tr>
<tr>
<td>– Low-cost</td>
<td>Low precision</td>
<td></td>
</tr>
<tr>
<td>– Based on real time data</td>
<td>Initial sampling needed</td>
<td></td>
</tr>
<tr>
<td>Traceable unit</td>
<td>High precision</td>
<td>Lower flexibility</td>
</tr>
<tr>
<td>– No sampling needed</td>
<td>Cannot be used for fluids</td>
<td></td>
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<tr>
<td>– Could be used in process sections with both batch and continuous flows</td>
<td>Can only be used at shorter distance</td>
<td></td>
</tr>
<tr>
<td>Material signature</td>
<td>Flexible</td>
<td>Costly</td>
</tr>
<tr>
<td>– Informative</td>
<td>Time demanding</td>
<td></td>
</tr>
<tr>
<td>– High analyses precision</td>
<td>Costly</td>
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</table>

The possibilities to make rapid and different analyses in aspects such as bulk and particle mineralogical as well as specific mineral search makes the instrument useful in the search for different signatures.

The usually low product value added in continuous processes is a factor that complicates the design of traceability system and selection of traceability methods. However, the authors’ firm belief is that traceability can be achieved profitably even in continuous processes. Nevertheless, resources are always an important aspect that has to be kept in mind when choosing among different traceability methods and designing a traceability system.

The importance of traceability has been continuously increasing as a response to wishes to optimise the production process and new regulations. The importance of traceability in production processes can therefore not be stressed enough.

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References


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Paper II
Applying traceability to
grinding circuits by using
Particle Texture Analysis (PTA)

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Applying traceability to grinding circuits by using Particle Texture Analysis (PTA)

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Abstract

LKAB has started a new pelletizing plant at Malmberget, where the raw material will be a mix of ores from Kiruna and Malmberget. The new plant necessitated an investment in a new grinding section in the concentrator. As usual, the new section has larger mills. It also lacks the wet cabling stage present in the old sections.

Comparing the analysis data from the new grinding section with the old grinding sections it shows that they give similar results. There are slight variations; the older mills produce a steeper final particle size distribution. Also, it appears that the new mills are more efficient, since they have higher calculated grindability indices.

To better understand the differences between the sections, and the process implications of the new grinding section, a combination of Particle Texture Analysis (PTA) and the statistical method multivariate data analysis (MVDA) is used. It shows that it is possible to identify and follow systematic changes in the particle morphology of the mill products. Also, that there are differences in process mineralogical aspect between the old and new grading sections.

1. Introduction

LKAB has since the beginning of the 1900s produced iron ore from mines in Kiruna and Malmberget and is today one of the world’s leading producers of highly refined iron ore products. The main product is pellets, for blast furnaces and direct reduction furnaces. Today there is a higher demand from the customers and to obtain a good quality it is important to have good control over the process and the raw material used.

LKAB started the new pelletization plant at Malmberget (MIC3) in November 2006. The raw material will be a mix from Kiruna and Malmberget, i.e., different ores having different Fe-content and levels of contaminants (Martinsson and Wanhainen, 2000).

That is why the traceability of the continuous process is one of the crucial factors for future development of granular product(s). Traceability gives the advantage to have a better control over the material through the process and there can be adjustments taken, if needed. It can show “the current” values of different parameters and how much we have to adjust to achieve the goals. In food and pharmacy industries it is very common to use different traceability tools but in the mining industry, which is mostly a continuous process, traceability is an untouched area.

As mentioned earlier, traceability is common in part or batch production and often relatively easy to achieve, since different kinds of identification markers can be attached to a unit or different parameter can be measured at different process stages. In continuous processes on the other hand the main part of the collected data relate to process variables that are frequently measured, while product data are limited and infrequently measured (Hild et al., 2000).

The literature dealing with traceability is dominated by applications from parts production. However, creating traceability in continuous processes implies vast challenges: process flows can be parallel, serial and circular; sub-processes can be continuous as well as batch-wise; have large buffers or no interruptions in product handling. The purpose of this paper is to compile and describe how process mineralogy might be used for achieving traceability in continuous processes. In this case the grinding sections are in focus, and multivariate data analysis is used to interpret the mineralogy and textures of the minerals in this section.

2. Material

Most iron ores contain significant amounts of gangue minerals that need to be eliminated to produce iron concentrates. At Malmberget, the dominant iron mineral is magnetite but also hematite occurs. Gangue minerals are mostly quartz, pyroxene, apatite, and feldspar (Geijer, 1930).

Material from the mine is sent to the concentrator plant which separates the minerals into two parts, tail and product (Kvarnström and Oghazi, 2008).
The samples that are used in these studies are from the concentrator’s, old Section 5 and the new Section 6.

2.1. Flowsheet

Fig. 1 shows a typical flowsheet for concentrating iron ore at LKAB. The coarse material at 10–15 mm in size is fed to a wet magnetic cobbing separator (M1). The magnetic concentrate is discharged into a primary ball mill (#1), and the ground product (pulp) is transferred to a primary magnetic separator (M2). The resultant magnetic concentrate is then pumped into a secondary ball mill (#2). A secondary magnetic separation unit (M3) finally upgrades the ground product and the concentrate is used as feed for the tertiary grinding stage (#3) (Tano et al., 2005).

The flowsheet for the new grinding Section 6 resemble the one for Section 5, the only difference is that there is no wet cobbing stage before the primary mill. Here, ball mill grinding is used in three consecutive steps with wet low intensity magnetic separators in between. It is important to grind to, approximate 68% < 45 μm to liberate gangue minerals, and to reach the desirable size distribution for the pellets feed. In the result part there is a complete data of how each mill performs (Table 1).

3. Experimental

3.1. Sampling

Feed samples were taken manually when the material was in motion at a point of free fall, by making a cut at right angle through the falling stream. The other samples were taken as manual pulp samples with pear shaped scoop throughout the circuit before and after each grinding stage. The samples were weighted and then filtrated at Malmberget. In the laboratory at LTU the samples were dried and then cut by a Jones splitter into suitable proportions. The dry material was sieved with a Rot-Tap shaker down to 75 μm and wet sieved further to 38 μm.

3.2. Particle Texture Analysis

It is important that the liberation data for minerals in a sample come from a sieve fraction. Tests and comparisons have shown that measured liberation of specific size particles in unsieved samples are not the same as the sieved sample. For unsieved samples, the result is not correct (Petruk, 2000).

To have a good view over the different samples, Particle Texture Analysis (PTA) was done at NTNU, Trondheim, Norway. The PTA data system is based on the Oxford Inca Feature software and an existing scanning electron microscope (Moen, 2006). Using Back-Scattered Electrons (BSE) the images are analysed by means of grey level and every grain of interest is analysed with X-rays. With the Inca data information the images will be processed and calibrated (grey-scale and binary images); and the grains will be identified and evaluated if they are liberated or in composite particles and which minerals occur in composite particles. When all particles are analysed, the data will be imported to the PTA software. The PTA software gives plots and thumbnail images regarding mineral liberation, mineral association analysis and intergrowth analysis.

3.3. Multivariate data analysis

For achieving good control and having better overview of the process data it is necessary to collect data with many variables and many properties from the process. By using multivariate data analysis (MVDA) these variables will be explained and expressed and condensed into a few latent variables or principal components so it will be easier to understand the importance and contribution of each variable. A general description of MVDA and its methods applied to chemistry was first done by Wold et al. (1984).

Multivariate data analysis is based on projection methods. Principal Component Analysis (PCA) is a projection method of the original variables onto new ones, orthogonal and arranged according to their eigenvalue. This is done on all the data in a matrix X, typically containing the observations as rows and variable data in col-

Table 1

<table>
<thead>
<tr>
<th>Section</th>
<th>Feed (% &lt;45 μm)</th>
<th>Disch. (% &lt;45 μm)</th>
<th>d80 in (μm)</th>
<th>d80 out (μm)</th>
<th>Power (kW)</th>
<th>Feed (t/h)</th>
<th>Grindability (kg &lt;45 μm/kW h)</th>
<th>Apparent-work index Wapp (kW h/ton)</th>
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<td>Section 6</td>
<td>06KV001</td>
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<td>23.0</td>
<td>3 250</td>
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<td>1918</td>
<td>350</td>
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<td>63.0</td>
<td>125</td>
<td>77</td>
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<tr>
<td>Section 5</td>
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<td>3.0</td>
<td>19.0</td>
<td>4 780</td>
<td>315</td>
<td>1461</td>
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<td>2573</td>
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<td></td>
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<td>60.0</td>
<td>140</td>
<td>74</td>
<td>2463</td>
<td>268</td>
<td>23.9</td>
</tr>
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</table>
The data matrix $X$ is decomposed into and approximated by a lower dimensional matrix product $T^TP$, plus a matrix of residuals $E$, accordingly.

$$X = T^TP + E$$

$T$ is a matrix of scores that summarises the $X$-variables, and $P$ is a matrix of loadings showing the influence of the variables. The dimensions in $T^TP$ are called the principal components of $X$. A component is considered significant if its normalised eigenvalue is larger than a cut-off value. The eigenvectors constitute the dominating directions, or latent variables, in $X$ space. By plotting the score vectors of $T$ against each other, one gets the positions of the observations projected onto a hyper plane defined by the principal components. A plot of the loadings vectors against each other gives the length, and thus the strength, of the variables projected on the same hyper plane. This means that a score plot contains the pattern of the observations in $X$ space, while the loadings plot shows the strength of the variables responsible for the pattern. So, by using PCA it will help to make a data reduction and elucidation among the all the collected data, and it will also detect any outliers. A modern description of data analysis by MVDA methods may be found in, e.g., Eriksson et al. (2006).

Other MVDA techniques are PLS (projection to latent structures), SIMCA (soft independent modelling of class analogies), and MSPC (multivariate statistical process control) are not used here, since this contribution is centred on pattern recognition.

The MVDA is run with the software program SIMCA-P+, version 11.5 (Eriksson et al., 2006).

4. Result

4.1. Size analysis

The size distributions of particles in the material for both sections (Sections 5 and 6) are shown in Figs. 2 and 3, respectively.

The particle size analyses of the feed and the products in the grinding process show the reduction of particle size ($d_{80}$) from 4700 µm in the feed to 70 µm in the final product.

By comparing Sections 5 and 6 there are slight variations in the results, the finer feed to Section 5 is due to the wet cobbing preceding the primary mill. The older mills in this section produce a steeper final particle size distribution. It is, however, too early to say if this is linked to mill size, or if it is the result of a better, worn in, graded charge.

The particle size analysis from Section 6 gives similar results compared with the results of Section 5. The grinding ratio in the primary mill is dramatically higher than the secondary and tertiary mills, which is due to the well known fact that the larger particles grind easily. Size distribution analyses from the output of the primary mill and the input to the secondary mill indicate that the LIMS mainly eliminates smaller gangue minerals fractions. This may explain why the feed to the secondary mill is coarser than the discharge from the primary mill.

![Fig. 2. Particle size distribution, Section 5.](image1)

![Fig. 3. Particle size distribution, Section 6.](image2)
4.2. Mill efficiency

There are two major ways to easily compare operating mills: grindability and apparent work index (Tano et al., 2005). Grindability index ($G_i$) showing the produced amount of material finer than 45 µm is calculated according to

$$G_i = \frac{\left(\frac{S_{45 \mu m}}{S_{45 \mu m}}\right)_{D} - \left(\frac{S_{45 \mu m}}{S_{45 \mu m}}\right)_{F}}{1000 + F} \times 100$$

where $S_{45 \mu m}^D$ and $S_{45 \mu m}^F$ is the percentage of material finer than 45 µm in discharge and feed, respectively. $F$ is the amount of feed (tonne/h) and $P$ is the mill power (kW).

The apparent-Work index ($W_{app}$) is shown as

$$W_{app} = \frac{P}{10 \times F} \times 100$$

where $P$ is mill power (kW), $F$ is the amount of feed (tonne/h) and $d_{80}$ is the 80% passing size (µm) for discharge and feed, respectively.

The result gives that all mills in the new Section 6 has a better grindability compared to the old section (cf. Table 1). However, for the coarse end of the particle size ranges it is not that clear, since there is no consistent difference in apparent work index between the sections.

4.3. Particle Texture Analysis (PTA)

The PTA gives information on how different minerals are distributed in different fractions. The analysis shows that the magnetite content decreases with fraction size, cf. Fig. 4. This is due to the sugar grain structure of the Malmberget magnetite, which easily breaks along grain boundaries. Feldspar and pyrox/amphibole are more evenly distributed over the size fractions, while apatite occurs largely below 100 µm.

The PTA also shows how minerals are liberated in different fractions. In this case magnetite is overall well liberated in the fractions examined, but there is also some other minerals that are associated with magnetite. In the largest fraction, associated minerals are plagioclase and ilmenite.

With PTA it is also possible to have a good overview over the mineral liberation. It is calculated by the area method, an area of mineral in interest is measured and also the host particle in the polished section, and calculating the percent of mineral in the particle. However, the liberation result shows that the liberation was 90% or better in all cases.

4.4. Multivariate analysis

All the data from the PTA were collected and arranged in different Excel files, and then imported into the software SIMCA. Each particle was an observation in a data file with variables according to Table 2. A typical PTA data for one sample contained 7000–10,000 observations/particles. PCA-models were created to check for pattern in the data. Here, the score and loading plots are used. They give important information about variables that are

<table>
<thead>
<tr>
<th>Minerals</th>
<th>Parameters</th>
</tr>
</thead>
<tbody>
<tr>
<td>Magnetite</td>
<td>Area (area of whole feature in square microns)</td>
</tr>
<tr>
<td>Ilmenite</td>
<td>Length (max frt)</td>
</tr>
<tr>
<td>Rutile</td>
<td>Breadth (min frt)</td>
</tr>
<tr>
<td>Plagioclase</td>
<td>Perimeter (parameter of whole feature in microns)</td>
</tr>
<tr>
<td>Quartz</td>
<td>Aspectratio; $\sqrt{\frac{p}{q}}$</td>
</tr>
<tr>
<td>Pyrox/amphibole (Pyx/Amf)</td>
<td>Direction (angle)</td>
</tr>
<tr>
<td>Biotite</td>
<td>Shape; $\sqrt{\frac{p}{q}}$</td>
</tr>
<tr>
<td>Anf</td>
<td>ECD; $\sqrt{\frac{p}{q}}$</td>
</tr>
<tr>
<td>Enstatite</td>
<td>Mean grey level (mean image grey level for each particle)</td>
</tr>
<tr>
<td>Apatite</td>
<td>% Element (wt%)</td>
</tr>
<tr>
<td>Trinitite</td>
<td>Calcite</td>
</tr>
<tr>
<td>Sulphides</td>
<td>Unclassified</td>
</tr>
</tbody>
</table>

Table 3

<table>
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<tr>
<th>Components</th>
<th>$R^2$</th>
<th>$R^2$ (cum)</th>
<th>Eigenvalues</th>
<th>$Q^2$</th>
<th>Limit</th>
<th>$Q^2$ (cum)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
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<td>0.174</td>
<td>11.3</td>
<td>0.112</td>
<td>0.0153</td>
<td>0.112</td>
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<tr>
<td>2</td>
<td>0.157</td>
<td>0.33</td>
<td>10.2</td>
<td>0.161</td>
<td>0.0155</td>
<td>0.256</td>
</tr>
</tbody>
</table>
**Fig. 5.** Score plot coloured according to mineral identifications for feed material to Section 5 (38 μm fraction).

**Fig. 6.** Loading plot for the PCA model, feed material to Section 5.

**Fig. 7.** Score plot for the PCA model of feed and discharge material, Section 5.
responsible for the pattern seen among the observation and how they are related to each other.

4.4.1. Overview

The first analysis is run to get an overview in two dimensions (principal components) based on all identifications and parameters in Table 2. Here in Table 3, R²X (cum) is the cumulative sum of squares for the variation of all data explained by the extracted components. Q² (cum) is the total variation of the data that can be predicted by the same components, as estimated from cross-validation. Table 3 shows that the model has poor explanation and predictability.

The score plot shows the relationship among the observations (minerals). This plot can be seen as window into the X space, where the objects (particles) are projected on a two-dimensional hyperplane in the 65 variable spaces. In Fig. 5 it is shown that there is a separation between the magnetite and the gangue minerals in the second direction. There is also a separation between the gangue minerals which put them in different groups.

Table 4
Statistical summary for the developed model for Section 5. PCA-X observations (N = 20,083, Variables (K) = 8 (X = 8, Y = 0)).

<table>
<thead>
<tr>
<th>Components</th>
<th>R²X</th>
<th>R²X (cum)</th>
<th>Eigenvalues</th>
<th>Q²</th>
<th>Limit</th>
<th>Q² (cum)</th>
</tr>
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<tbody>
<tr>
<td>1</td>
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<td>0.627</td>
<td>5.02</td>
<td>0.581</td>
<td>0.111</td>
<td>0.581</td>
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<tr>
<td>2</td>
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<td></td>
<td>1.46</td>
<td>0.109</td>
<td>0.125</td>
<td>0.562</td>
</tr>
<tr>
<td>3</td>
<td>0.117</td>
<td>0.927</td>
<td></td>
<td>-0.168</td>
<td>0.143</td>
<td>0.589</td>
</tr>
<tr>
<td>4</td>
<td>0.055</td>
<td>0.982</td>
<td>0.439</td>
<td>0.693</td>
<td>0.167</td>
<td>0.874</td>
</tr>
</tbody>
</table>

Table 5
Statistical summary for the developed model for Section 6. PCA-X observations (N = 16,050, variables (K) = 8 (X = 8, Y = 0)).

<table>
<thead>
<tr>
<th>Components</th>
<th>R²X</th>
<th>R²X (cum)</th>
<th>Eigenvalues</th>
<th>Q²</th>
<th>Limit</th>
<th>Q² (cum)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
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<td>0.623</td>
<td>4.98</td>
<td>0.575</td>
<td>0.111</td>
<td>0.575</td>
</tr>
<tr>
<td>2</td>
<td>0.185</td>
<td>0.808</td>
<td>1.48</td>
<td>0.127</td>
<td>0.125</td>
<td>0.620</td>
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<tr>
<td>3</td>
<td>0.113</td>
<td>0.92</td>
<td>0.902</td>
<td>-0.223</td>
<td>0.143</td>
<td>0.592</td>
</tr>
<tr>
<td>4</td>
<td>0.0579</td>
<td>0.978</td>
<td>0.464</td>
<td>0.666</td>
<td>0.167</td>
<td>0.864</td>
</tr>
</tbody>
</table>

Fig. 8. Loading plot for the PCA model of feed and discharge materials, Section 5.

Fig. 9. Score plot with mineral identification for feed and discharge to the primary mill, Section 5 (38 µm).
In the first PCA overview the material is clearly spread out in the first direction. Fig. 6, which is a loading plot, show the importance of different variables in the X matrix. It explains how different variables contribute to the model which is shown in the score plot. As it show in Fig. 6 gangue and magnetite are positioned in opposite direction, this explain why the gangue and magnetite are so well separated in the score plot (Fig. 5). On the top right side of the loading plot the morphology variables are gathered. It is these variables which need to be investigated more.

4.4.2. Comparison of feed and discharge to a primary mill

The first analysis aimed to compare the feed and discharge for each mill. By excluding all variables except the morphology ones (those in upper right 'red ring in Fig. 6), the model was improved. Table 4 shows that the improved model contains four principal components and it fits the model very good with the predictability of 87%.

The loading plot in Fig. 8 shows that size parameters carry most of the information in the first direction while mean grey have a large influence in the third direction. The first direction is length information.

With the same model and the score plot instead coloured according to mineral classification it is possible to see how the minerals differentiate, cf. Fig. 9. The main parameter pulling magnetite away appears to be Mean grey. This is what to be expected, since magnetite appears brighter in the Back-Scatter Electron images.

However if the same plot is reduced to leave only magnetite information, sub-populations of magnetite emerges, cf. Fig. 10. To this moment it is not entirely clear what causes the split on sub-populations. The lower value of Mean grey and shorter length indicates that the two minor groups may constitute of mixed particles.

The loading plot for the primary mill Section 6 is very similar to the loading plot from Section 5, and therefore not shown. In Table 5 it shows that the model has four principal components and is very
Fig. 12. Score plot for apatite in Section 5.

Fig. 13. Score plot for feldspar in Section 5.

Fig. 14. Score plot for apatite in Section 6.
similar to Table 4. The score plot in Fig. 11 proves that the pattern found is systematic, since its general appearance is the same as for Fig. 7. PC direction 1 carries length/size information, while PC3 is mostly Mean grey.

4.4.3. Gangue mineral changes in the sections
To understand the beneficiation process, it is necessary to investigate the gangue minerals more carefully. Usually iron ores occur in formations with siliceous rocks, and in particular minerals such as apatite and feldspar. The results of a PCA for the whole Section 5 are shown in Figs. 12 and 13 for apatite and feldspar respectively. Here, data from feed and discharge of each mill in Section 5 is analysed together, creating a data set with approximately 58,000 observations/particles.

Most of the particles are distributed along the first direction; it is the morphology parameters that are found in the first direction in the same way as in Fig. 8. In the score plot some particles are outside the confidence interval and are marked with a red circle, by investigating this group with a contribution plot (not shown) it is found that these particles are larger than average apatite particles.

Fig. 13 shows that almost all feldspar are absent from the system after the first magnetic separator. It indicates that there are no mixed feldspar particles in Section 5. The apatite in Section 6 has a similar pattern compared with the same mineral in Section 5, cf. Fig. 14.

It is obvious that there is more apatite in Section 6 even after the last mill compared to Section 5. This may be attributed to the absence of a cobbing stage before Section 6.

The plot for feldspar in Section 6 once again shows similarity with the one for feldspar in Section 5, and is not shown here. During the run of the investigation, it was a consistent experience that the principal components in the statistical analysis did not change with product and/or grinding section.

5. Conclusion
The combination of PTA and MVDA seems to be a promising development. Further refinements would be to use MVDA models to discriminate between “good” and “bad” feed materials using SIMCA classification techniques.

By comparing the MVDA for the sections and different minerals it can be shown how the cobbing stage affect the circuit. By comparing the feldspar and apatite results it is obvious that the apatite continue to exist during the whole circuit, while most of the feldspar is gone after the first separation stage. It seems that the free particles disappear while mixed particles continue to exist in the circuit.

For Sections 5 and 6, a direct similarity is found between the two sections but also some divergences, which may depend on the cobbing stage or that the grinding mill differs from each section.

It would be interesting to further investigate the material from both sections by using optical microscopy. Optical analysis will better identify the intergrown minerals.

The main benefit of multivariate analysis on particle texture data is that it simultaneously compares several thousand particles. This type of investigation is state of the art in this area and it can provide unambiguous information about how each process step influences the material.

Acknowledgements
Financial support from LKAB Research and Development is gratefully acknowledged. We would also like to thank Johan Bucht for valuable ideas regarding the multivariate analysis.

References
Paper III
Applying traceability in a
Mine-to-Mill context by using
Particle Texture Analysis

Published as:
It is possible to have traceability in the mining industry, by parameters and signatures like particle mineralogy, mineral association, texture and mineral liberation. The study is on an apatite-iron ore deposit at Malmberget, Sweden, and characterizes an ore body both mineralogically and texturally in a quantitative manner by using analytical methods like optical microscopy, microprobe (EMPA) and an automatic SEM based system, Particle Texture Analysis (PTA). The mineralogy was evaluated by PTA and characterized by modal mineralogy, mineral liberation and mineral associations. Magnetite has a simple outline and straight grain boundaries and the gangue minerals have a finer particle size with a more complicated texture. The PTA analysis also shows that apatite is associated to magnetite as mixed particles, while smaller grains of magnetite are inclusions in feldspar. Result from particle texture analysis shows that there is a connection which link to the mine-to-mill context, and it may be used to create traceability. The link is the associations of the main ore mineral magnetite, nor the modal mineralogy. Instead, it is the mineral associations of contaminating minerals (apatite and feldspar) that appear to be most promising since they survive from mine to mill.

The modal mineralogy may be used to understand how contaminated minerals break into or out of particles size fraction during grinding.

General

The present paper is divided into two steps. The first step is to characterise magnetite ore bodies at Malmberget mine by identifying significant mineralogical and textual signatures. In the second step the focus is to investigate the raw material that comes from the mine and continues to the concentrator. The main goal is to identify common signatures from different analytical tools used to examine liberation, texture and mineral associations, but also to find a method to connect signatures from mine to mill and possibly products to see if there are any systematic relations in the value chain.

The key factor of having a good and even production is to have a good control over the process and the raw material. As a result traceability has become important in any process, since it offers the opportunity to tie process data throughout the process to a certain product/batch. Therefore, traceability offers the possibility to understand how the origin of variation and learn how to adjust the process to avoid production of low quality products. However, in mineral processing the raw material consist of ores from one or several bodies with varying properties among and within the bodies.

Customers expect products (pellets) with good quality, and this requires a detailed knowledge of the processes from mine to customer. To achieve this it is important to have traceability of ore fed to the plants and control over the production process but it also demands a good knowledge of the raw material. Production at Malmberget continues from several ore bodies (Martinsson and Virkkunen, 2004) that are mixed in the mine and this means there will be plant feeds having different Fe-content and levels of contaminants. The most undesirable contaminants are phosphorous and potassium. They are found in the gangue minerals apatite and feldspar respectively. To achieve a good feed to the pellet plants the contaminants must be forced down to very low levels, < 0.020% P and < 0.1% K. Therefore, the ability to trace contaminating mineral phases throughout the value chain is of almost importance.

Traceability can be explained in several ways (Töyrylä, 1999) and also traceability can be achieved by different methods (Kvarnström and Oghazi, 2008).

Traceability is much easier to apply in batch processes than in continuous processes. In food and pharmacy industries it is very common to use different traceability tools (Moussavi et al., 2002) but in the mining industry, which is mostly a continuous process, traceability is a difficult task. In continuous processes there are several factors that makes the traceability complicated, there are reflux flows and mixing of streams that makes it problematic to achieve a high level of traceability (Kvarnström, 2008).

Geological setting

In the province of Norrbotten, Sweden, LKAB is producing 90 % of Europe’s iron ore from two large underground mines, Kiruna and Malmberget (Fig.1). These two apatite-iron ore deposits have a similar origin and were formed by magmatic-hydrothermal processes at 1.89-1.88 Ga (Bergman et al., 2001). However, there are major differences in character between them, due to later overprinting by metamorphosis, deformation and granitic intrusions, which are stronger for the Malmberget deposit (Martinsson and Virkkunen, 2004).
known in the Malmberget ore deposit, occupying an area of 2.5 x 5 km. The Malmberget deposit was probably from the beginning a more or less continuous tabular ore lens which was exposed to at least two phases of folding and metamorphism. Today they occupy a large-scale fold structure where the individual ore bodies stretch parallel to the fold axis, which plunge 40°-50° towards SSW (Bergman et al., 2001).

Mineralogy

The iron ore minerals are both magnetite (Fe3O4) and hematite (Fe2O3) but the magnetite is more common of the two. Due to the strong metamorphic recrystallization of the area, the minerals are recrystallised, coarse grained, and elongated in the direction of the lineation of the rocks.

Two types of ore were identified and used in this study, called ore breccia and ore. The ore breccia is bordering the massive ore, but occurs also partly as inclusions in the massive ore. The ore breccia is largely consisting of magnetite but do have gangue minerals like quartz, amphibole, pyroxene, apatite, biotite and feldspars in different proportion occurring as breccia in the wall rocks. The ore is more massive magnetite that contains gangue minerals like amphiboles and apatite in lesser amounts.

Concentrator

After blasting and underground primary crushing, the ore is hoisted to the cobbing plant where dry magnetic separators make the first separation and send the magnetic product to the concentrator, where wet grinding mills and wet low-intensity magnetic separators are used. There are several sections in the Malmberget concentrator, in this study only section 6 is reported on. In section 6 the coarse material at 10-15 mm in size enters a primary ball mill, and after the first grinding mill the product (pulp) is fed to a magnetic separator. Its magnetic concentrate then flows to a secondary ball mill and from there to another magnetic separation unit, for more information about the flow sheet see Tano et al., (2006).

In other older sections, ball mill grinding is also in three consecutive steps with wet low intensity magnetic separators in between, but with a primary wet magnetic cobbing separator before the first mill.

The particle size distribution of ore feed from Malmberget (Fig.2) shows a characteristic hump around 1 mm where there also is a noticeable drop in the Fe content for coarser grain sizes.

Figure 2. Particle size distribution for section 6. The “hump” is marked with red circle in the figure.

Process mineralogy as a tool

Optical microscopy has traditionally been the instrument used for the identification and quantification for both mineralogical and textural properties, but this is a time consuming process (Petruk, 2000). A number of different techniques using image analyses system based on SEM techniques have been developed during the last decades for a more rapid and quantitative estimation and description of mineralogy and particle textures (Jones and Gavrilovic, 1970; Gottlieb et al., 2000; Petruk, 2000; Gu, 2003). GEMSCAN® and MLA both developed in Australia, are the better known instruments. SEM-PTA (particle texture analyses) used in this study is a similar technique developed at NTNU, Trondheim, Norway (Moen, 2006).

In mining it is the mineralogy and the properties of an ore that determines the conditions for the further processing. Moen (2006) defines process mineralogy as the mineralogy which is applied to the product in specific industrial process such as the mineralogy in concentrator, pelletization or in other process stages.

An automated system produces the quantitative information of the mineralogy in three general forms; modal information, textural information and liberation information (Sutherland and Gottlieb, 1991). Process mineralogy in the present case is used as a tool that gives detailed information of the mineralogy from different ore bodies to be linked to the mineralogy in the concentrator for improving the mineral processing performance. These mineralogical data are specific in such a way it will be possible to express mineralogy as individual signatures.

EXPERIMENTAL

Sampling and preparation

The ore samples are from both drill cores and pulp streams. The mine is represented by samples from drill cores taken from various parts of the Fabian ore body (see Table 1). Samples from section 5 and 6 in the concentrator were taken before and after each grinding mill.

The drill core samples were crushed in a Retsch jaw crusher before they were analysed and the samples from the concentrator were weighted and then filtered at Malmberget. All the samples were then dried and cut by a Jones splitter into suitable proportions in the laboratory at Luleå University of Technology (LUT). The dry material was sieved with a Ro-tap shaker down to 75 μm and sieved further to 38 μm. For the ore studies, the fractions 75 and 150 μm were used, while the concentrator feed was investigated at 38, 75, 150, 300 μm. Polished thin sections were made of all the samples. To be able to characterise original textures, samples of intact drill cores were also used in this study.

Analyses methods

Polished thin sections from the ore were optically examined in transmitted and reflected light on a standard petrographic microscope (Nikon Eclipse E600).

Mineral analyses were performed on a JOEL JXA-8500F electron microprobe at NTNU, Trondheim, Norway. For the microprobe analyses an accelerating voltage at 15.0 kV, a probe current at 95 μA and a < 1μm beam diameter were used.

Particle texture analysis (PTA)

The Particle Texture Analysis (PTA) system is developed at the Norwegian University of Science and Technology (NTNU) (Moen, 2006). The scanning electron microscopy produces Back Scatter Electron (BSE) images that can be analysed on the basis of grey levels and every grain of interest is also analysed by X-rays. All analysed particle size fractions were imported to the PTA software, where images analyses are done offline to process and evaluate if grains occur as liberated or in composite particles.

Standard queries can be done on the output results in a new database that contains information on the mineral liberation of any mineral, mineral association of any mineral and miniature images of particles of a certain texture category.

RESULTS AND DISCUSSION

Ore

The textures of the two ore types ore and ore breccia were identified by optical microscopy on drill cores (Table1). Ore breccia is characterised by magnetite grains with a simple euhedral outline and straight grain boundaries, either as single grains or as aggregates of particles in a matrix of quartz and feldspar (Fig.3). Smaller euhedral magnetite grains are locked as inclusions in and between feldspar grains. This matrix has a subgranular texture and the grains shows anhedral granular outline with complicated grain boundaries.

In the ore the texture of magnetite is dominated by grains having simple euhedral outlines with straight grain boundaries. The grains are
of different size with coarser grains often elongated in the direction of lineation occurring in a finer grained matrix of both magnetite and smaller amount of apatite (Fig.3).

Figure 3. Photomicrographs showing mineral textures and mineral associations from ore and ore breccia. A) Ore breccia, smaller euhedral magnetite grains are locked as inclusions in and in between gangue minerals. B) Ore, coarser magnetite grains often elongated in a direction of lineation with apatite as interstitial grains.

The modal mineralogy shows the percentages of minerals found in the analysed fraction based on examination of a sample from PTA (Fig.4). For ore and ore breccia similar results between the 150 μm and 75 μm size fractions are found. Magnetite decreases in the finer fraction and instead different groups of gangue minerals increase. The two particle fractions in the ore samples have the same minerals represented but in different proportion. Note that feldspar is nearly absent in ore. In ore breccia more minerals are present and the amount of minerals like feldspar and amphibole/pyroxene increases pronouncedly in the finer fraction. The degree of liberation of magnetite is high for both ore types (80-90%) and increases slightly for the finer fractions (Fig. 5).

Figure 4. The modal mineralogy of ore and ore breccia. The mineral classes follow the same order as the graph.

Magnetite was analysed with respect to its mineral associations with the finding that each ore type had one major mineral association with magnetite; apatite for the ore and feldspar for the ore breccia. In the ore the binary magnetite-apatite association occurs as typically mixed grain and the sizes of the two minerals are often a smaller grain of apatite which is attached to a larger grain of magnetite in an equal proportion.

The ore breccia has more mineral associations as binary and complex magnetite-bearing particles compared to ore. Feldspar with magnetite is the most frequent association. However, quartz and amphibole are often included both as binary association but also as complex magnetite-bearing particles. Magnetite occurs as small inclusions in feldspars and amphibole (Fig.3). As a fingerprint of the ores it is interesting to look at the mineral association for apatite and feldspar and examine the material that continuous to the concentrator with the same method. Figure 7 shows the apatite association in ore. There are too few apatite particles found in ore breccia to create a reliable statistic, and it is not shown. Most of the apatite is liberated; however, at 150μm most of the apatite is associated to magnetite while at 75 μm apatite is also associated to other minerals.

The interesting result here is that in ore the feldspar is associated to more minerals, mostly magnetite at finer particle sizes. In the case of ore breccia almost half of the feldspar is associated to other minerals at 150μm. It means that there are more mixed grains in coarser particles in ore breccia while the opposite is found in ore. This might be interpreted as a result of the metamorphoses in the way that the ore has got more of re-crystallisation, creating cleaner and coarser magnetite grains, while at the same time pushing the contaminating elements into separate gangue mineral phases.
amphibole and magnetite is quite similar for both ore types and makes it difficult to use as a signature.

The ores are hoisted to the surface operations, where they are treated in a dry cobbing process to remove coarse gangue minerals. This process uses a combination of screening, crushing and dry LIMS. These operations are not selective in the -1 mm region; therefore, it is possible to have a coupling between the mineralogy for ore and mill feed in this range.

The mineral associations for magnetite only reveal that magnetite is liberated to 90% or even more for the 300 μm fraction. They will not add any signature value and is not shown here.

Instead a summation of the mineral content is more illustrative, where for coarser particle classes there is a higher content of magnetite, which corresponds to the observation for the material in the mine.

Most of the magnetite is liberated but at coarser size magnetite is more associated to other minerals. The difference between the fractions is small. Closer look with an optical microscopy shows that the magnetite and apatite occur as mixed grain in the samples. Overall the results also show that the liberation is very high in ore, ore breccia and the feed to the concentrator.

Since the mineral association for magnetite did not carry much information, the interest is instead focused on the associations of the contaminating minerals, i.e., apatite and feldspar. Examining the material that comes to the concentrator (Fig.9) shows a strong binary association of apatite and magnetite. Some similarities are found between the minerals that are associated to apatite, in both cases almost 80 percent of apatite is liberated and there are more binary groups in 75μm then in 150μm. The same observations were made for the ore from the mine (Fig.7).

Within the mills, different phenomena seem to be at play. In mill #1, the grinding lower the apparent grade of gangue minerals in the 38μm fraction due to breakage of coarse liberated magnetite grains. In mill #2, the grinding on the other hand increases the gangue mineral grade. This is probably the result of breakage of large composite particles. The same patterns were observed for mill #1 and #2 in section 5.

CONCLUSIONS

To characterise an ore deposit, mineralogical and textural information is a critical factor during the exploration stage. Also, by studying the process mineralogy of the ore it is possible to understand the behaviour of it and also having information of what is coming to the concentrator as ore feed. An automated system technique makes it possible to get consistent signatures from both the ore bodies and the ore feed and creates the possibility to achieve traceability between mine and concentrator.

The signatures that have been identified and can possibly create

Figure 8. Mineral association to feldspar.

Figure 9. Mineral association to Apatite.

Figure 10. Mineral association to Feldspar.

Figure 11. The modal mineralogy for grinding section 6.

Within the mills, different phenomena seem to be at play. In mill #1, the grinding lower the apparent grade of gangue minerals in the 38μm fraction due to breakage of coarse liberated magnetite grains. In mill #2, the grinding on the other hand increases the gangue mineral grade. This is probably the result of breakage of large composite particles. The same patterns were observed for mill #1 and #2 in section 5.

CONCLUSIONS

To characterise an ore deposit, mineralogical and textural information is a critical factor during the exploration stage. Also, by studying the process mineralogy of the ore it is possible to understand the behaviour of it and also having information of what is coming to the concentrator as ore feed. An automated system technique makes it possible to get consistent signatures from both the ore bodies and the ore feed and creates the possibility to achieve traceability between mine and concentrator.

The signatures that have been identified and can possibly create
traceability between mine and concentrator, and also through the grinding circuit, are the mineral associations of apatite and feldspar to magnetite.

The modal mineralogy, in itself seems not to be a traceability tool, since the content of minerals will change in any upgrading process. However, the modal mineralogy may be used to understand how minerals break into and out of fractions during the grinding process.

The next logical step is to simultaneously investigate all the PTA results in an un-biased way. However, because of the huge amount of data it necessitates the use of tools such as multivariate data analysis capable of extracting hidden connections. The methods of multivariate analysis are very efficient, since all data are converted to figures that show the connection and relation between different parameters and objects. A few samples from the concentrator have already been investigated by these methods see (Oghazi et al., 2009), but it is important to use this kind of information with the data from different ore bodies and see if there are any more connecting signatures. 

ACKNOWLEDGEMENT

The authors are grateful for the financial support by LKAB and HLRC. They are also grateful for the assistance during sampling at LKAB, Malmberget. We would also like to thank Prof. Terje Malvik and Dr. Kari Moen, NTNU, Norway for supervising and helping with the process mineralogy analyses.

REFERENCES

Paper IV
Classifying best access point for return of external flows into flowsheets

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Classifying Best Access Points for Return of External Flows into Flowsheets

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Abstract: External flows are process streams that come from auxiliary processes or events. They are re-routed into the ordinary flowsheet since they are thought to be too valuable to be sent to any tailings pond. External flows come from multiple sources, e.g. drainage sumps, spillage thickeners, depleted products etc. Therefore, external flows may fit or not fit into an existing flowsheet depending on several factors like, flow rate frequency, dilution ratio variation, chemical and mineralogical composition, particle size or particle morphology.

By using Particle Texture Analysis to investigate external flows and compare them with existing ordinary flows it is possible to pinpoint from a process mineralogy via point to what extent the external flow fits into the ordinary processing flowsheet. Results from this information category helps to reach a higher quality of process knowledge and control for every step in the concentrator.

Results show that some recycled flows reconnected to the main flow are not connected to the best point. A side effect of the analysis is that some flows may be sent to later grinding stages. Thus, decreasing the load on the primary mill, and increasing the retention time.

Keywords: Process optimisation, mineral processing, process mineralogy, recycled flows, Particle texture analysis.

1. INTRODUCTION

Selection of the best process for a plant is a vital task for a process engineer. In doing so it is important to have full knowledge of the variables that affect decisions such as mineralogical and process data or simply to know what sort of material flows into the plant. In a processing plant there are numerous processes and flows that are connected to each other so that a minimum of the valuable component that is being processed goes to waste. What is often the case in mineral processing is that flows are lumped together and it is important to check when this is the best solution for the process. In this context external flows are those that come from auxiliary processes and are re-routed into the ordinary flowsheet. Detailed knowledge of these flows moving around in the flowsheet is often missing.

The purpose of this study is to try to understand the difference between the external flows that are connected back to the ordinary flow. It is important to know what is sent back to the main flow. An investigation of the material that comes into the concentrator is important to reduce irregularity in the process. The topics are formulated into two research questions:

- What are the specific characterization methods that can be used to classify different flows?
- Is it possible to pinpoint any decisive mineralogical factor for re-routed flows that are to be connected to the main process flow?

The study was carried out at the LKAB (Luossavaara-Kiirunavaara AB) Malmberget concentrator. In the concentrator, ball mill grinding is used consecutive in three following steps and with wet low intensity magnetic separators in between. It is important to grind to, approximate 68% < 45µm to liberate gangue minerals, and to reach the desirable size distribution for the pellets feed. For more details of the process cf. [1].

2. THEORETICAL FRAMEWORK

The ore is coming from different ore bodies with and behave. This means that there could be a risk for quality variation in the final product, this is why traceability become a important aspect so the mineralogical description does not differ when the raw materials are mixed. By collecting information and knowledge from the flows, they can be operated to the adjusted part of the process.

Firstly it is imperative to describe the mineralogy of the material that is coming into the concentrator. The iron ore minerals are both magnetite and hematite, but (in Swedish operations) magnetite is the more common of the two. The important gangue minerals of the ores are: apatite and feldspar (microcline) since they are quality issues for the produced iron ore pellets. (can quantitative information be included?) Furthermore, the final circuit concentrate should have a total gangue content < 1.5%.
Secondly, to understand the fundamentals of the mineral processing it is important to understand and link every single stage in the process chain. It is the mineralogy and the properties of an ore that determine the conditions for the further processing. To meet these challenges an efficient process can be designed and the mineral treatment may be optimized. Process mineralogy is a useful tool for flowsheet development and selection of the optimal process for the specific plant. Process mineralogy explains the characterization of minerals according to how they behave during specific treatment, and gives us useful information such as grain size, mineral association, liberation and trace element content that affect the process.

All mineral companies struggle to have as high recovery as possible and to do so information that characterizes the minerals is vital. In the present case, Particle Texture Analysis (PTA) is used for getting information on the process mineralogy. By using Back Scattered Electron (BSE) from the scanning electron microscopy, images are analyzed by means of grey levels and every grain of interest is analyzed by X-rays. All analyzed grain size fractions are imported to the PTA software, there images analyses are done offline to process and evaluate if grains occur as liberated or in composite particles.

3. EXPERIMENTAL

It is important to realize that the raw material for the concentrator comes from several ore bodies that are mixed in the process and this means there will be ores having different Fe-content and levels of contaminants. The ores are treated in three major parallel grinding sections. All the spillage from the concentrator is reprocessed in section of pre-concentration again.

Samples from nine different points were taken in the concentrator's section for external flows. Most samples are from flows that are to be recycled into the main process flow from thickeners and magnetic separators. In Table 1 all the sample points are presented. Sample points 1-8 are chosen to cover all the recycled material types that are going back to the main stream. Short explanation can be found in Table 1 below. Different amount of material were...
taken from the bulk and it is important that the samples are representative. Samples from points 1 to 9 were taken manually from the dressing plant. They were taken before in 20 minutes interval. Sample point 9 is a reference sample from the final magnetic separator in section 6 that gives information on the material that leaves the concentrator.

All the samples were dried and cut by a Jones splitter into suitable proportions in the laboratory at Luleå University of Technology (LTU). The dry material was sieved with a Ro-Tap shaker down to 75 μm and wet sieved further to 38 μm. Samples 38-53μm were sent to NTNU, Norway for particle texture analysis.

4. RESULT

The modal mineralogy from Fig. (2) shows that in the underflow from thickener number two (sample #1) there is mostly magnetite while in thickeners four and five (sample #2 and #3) there is around 25-30% magnetite and the rest is gangue minerals.

The material that is sent to thickeners four and five is the tailings from the wet cobbing separators that contain mostly gangue minerals. The modal mineralogy for samples #2 and #3 looks almost similar to each other, the reason is that the feed material for both thickeners is the same and is split between the thickeners. The d50 is also approximately the same for both thickeners.

Sample #4 (wet LIMS concentrate) in Fig. (3) shows the same result (high magnetite content) as sample #1. Therefore, these two streams would be better sent directly to the tertiary mill in the main grinding section. It is unnecessary to grind them more because of their high content of magnetite, and they are also much finer compared to samples # 2 and #3.

Table 1. Sample Points with Explanation and Information

<table>
<thead>
<tr>
<th>Samples Point</th>
<th>Sample Explanation</th>
<th>d80 (μm)</th>
<th>%&lt;45μm</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>Underflow thickener #2 Sample contains a mix from section 1-3, mostly hematite with a large variation.</td>
<td>120 μm</td>
<td>22 %</td>
</tr>
<tr>
<td>2</td>
<td>Underflow thickener #4 Contains waste from wet cobbing plant section 4-5, also tails from magnetic separators section 3 and section 6.</td>
<td>210 μm</td>
<td>18 %</td>
</tr>
<tr>
<td>3</td>
<td>Underflow thickener #5 Thickener #5 parallel to #4</td>
<td>210 μm</td>
<td>22 %</td>
</tr>
<tr>
<td>4</td>
<td>Concentrate from magnetic separator. Feed is spillage from secondary mill section 1.</td>
<td>110 μm</td>
<td>11 %</td>
</tr>
<tr>
<td>5</td>
<td>Concentrate from magnetic separator. Coarse particles from classifier.</td>
<td>380 μm</td>
<td>14 %</td>
</tr>
<tr>
<td>6</td>
<td>Concentrate from magnetic separator. Fine particles from classifier.</td>
<td>111 μm</td>
<td>32 %</td>
</tr>
<tr>
<td>7</td>
<td>Concentrate from magnetic separator. Fine particles from classifier.</td>
<td>105 μm</td>
<td>37 %</td>
</tr>
<tr>
<td>8</td>
<td>Concentrate from magnetic separator. Feed is combined flows material collected from the re-routed flows.</td>
<td>211 μm</td>
<td>22 %</td>
</tr>
<tr>
<td>9</td>
<td>Concentrate from magnetic separator, from tertiary mill in grinding section 6. Used as reference.</td>
<td>75 μm</td>
<td>23 %</td>
</tr>
<tr>
<td>Feed Mill #1 section 5</td>
<td>Primary mill feed. Used as reference.</td>
<td>4780 μm</td>
<td>3 %</td>
</tr>
</tbody>
</table>

Fig. (2). Modal mineralogy from samples 1-3, taken from thickener underflows.
Fig. (3) shows the modal mineralogy from the concentrates of magnetic separators that are used in the recirculation circuit except for sample #9 which is taken from the final magnetic separator after the tertiary grinding stage. Sample #9 is the reference here, representing the desired product that all other samples are compared with. The concentrate from the concentrator (in this case sample #9) goes to the next process step, which is the pelletising plant. As seen in Fig. (3), the gangue content in sample #5 is almost double compared to sample #6 or #7 while the last mentioned samples are almost identical. Materials in sample #5 are the coarse discharge from two spiral classifiers. The overflow from the same spiral classifiers that contains fine particles and enters the magnetic separators represented by samples #6 and #7. In Table 1 can be read that the d80 for sample #5 is much coarser compared to sample #6 and #7 and should so be. Fig. (3) illustrate that there are more gangue minerals in the coarse stream from the classifiers which is logical.

All recycled flows go through a magnetic separator (sample #8) and from this the material is pumped to the primary mill in section 5. By comparing the existing feed entering the primary mill with the material in sample #8 that is sent to the primary mill it shows that sample #8 contains over 96% magnetite and a particle size distribution with a d50 around 200 μm, but the feed to the primary mill has 86% magnetite and a d50 is around 4800 μm. Therefore, sample #8 does not match with the feed to the primary mill. To obtain a better process the concentrate (sample #8) should bypass the primary mill and preferably be sent to the secondary mill since the material properties are similar. Today’s reason for not sending it to the tertiary mill is that there is one coarse intermittent flow entering before sample point #8. Could this coarse flow be treated separately sample #8 might be sent directly to the secondary mill.

Today this material (sample #8) is sent to the primary mill, but it is too fine for the primary mill. It will only create an unnecessary volumetric load and reduce the retention time in the primary mill. The modal mineralogy in Fig. (3) shows that there is mostly magnetite in sample #9 (final concentrate); however, it also contains a few particles of gangue minerals. One of the advantages with modal mineralogy is the possibility to exactly know how much of a certain mineral that exists in the sample. Counting the particles in the final concentrate gives that of totally 4513 particles there were 31 gangue mineral particles and the rest of it was magnetite. Thus, the material that leaves the concentrator contains 99 percent magnetite.

Fig. (4) illustrates the mineral associations to quartz and feldspar in the thickener underflows. Most of the quartz and feldspar are liberated. However, around 10 percent of them are associated to magnetite. This means that these mixed particles need some regrinding, otherwise they will increase the SiO2 content of the final pellets.

The association of minerals to apatite is shown in Fig. (5). Sample #1 was shown earlier (Fig. 2) to contain mostly magnetite, but there were small portions of other minerals as well. Fig. (5) demonstrates that none of the magnetite in sample #1 is associated to apatite.

The similarities between sample #1 and sample #4 are shown once again in Fig. (5 and 6). This result further substantiates the recommendation to bypass the primary mill and send these streams to the secondary or tertiary mill in the grinding section. For samples #4-#9 there were not much quartz and feldspar associations in the samples. Therefore, the analyses are not presented in this paper. However, there were some (4%) in sample #8, but that most likely comes
Minerals associated to Quartz and Feldspar

Fig. (4). Mineral association to Quartz and Feldspar in thickener underflows.

Minerals Associated to Apatite

Fig. (5). Mineral association to Apatite in thickeners.

Minerals associated to apatite

Fig. (6). Mineral association to Apatite in concentrate from magnetic separators.
from samples #2 and #3 (underflow thickeners) or the coarse intermittent flow entering just before point #8.

Analysis from samples #5-#8 shows that there are some apatites mainly associated to magnetite. On the other hand for sample #8 it is clear that over 25% of the apatite is associated to magnetite and this is particularly high compared to other samples from the recirculation section. It can be explained by the coarse intermittent particle stream which enters the magnetic separator at this position. There were few apatite grains in sample #9, which is the final concentrate.

5. CONCLUSIONS AND DISCUSSION

Modal mineralogy is one way to present the results from the process mineralogy analyses. It is a convenient way to understand the content of different flows. This type of analysis may be used to:

- Identify what sort of material is pumped to the grinding section
- Optimise the process by sending external flows to the best access point in the main flowsheet
- Reduce any overloading in any process step by decreasing unnecessary volumetric flows

This way for collecting total information on how each unit operation is working and what sort of material is recycled to the main flowsheet is very effective. The ideal case is when complete information for each external flow that is added to the main streams exists and it is then joined to an optimal position in the main flowsheet. In this case circulating flows are today collected in a mixer, and later going throw a magnetic separator before they are sent to the primary mill. As mention before underflow thickener #2 or sample #1 is not returned to the best process point and it puts an unnecessary load on some stages in the main flowsheet. The result from modal mineralogy shows that there were some gangue minerals in the streams from the thickeners and also some in intermediate streams. Most of them are gone after the re-treatment section. Based on gangue content and particle size only this combined flow should bypass the primary mill. However, if the rapidity of the analysis was better maybe this method would be more common in the daily process.

Minerals association analyses on these samples are a more informative and important way to pinpoint how non-liberated gangue minerals occur in these streams. If the gangue minerals are locked with magnetite they will ultimately end up in the final concentrate since current low-intensity magnetic separators pick any particles with small magnetic volumes. Therefore, mineral association analysis is the most important tool in deciding where to send recycled flows. However, to avoid data overload it is better to first use particle size and modal mineralogy data to make a first selection of possible connection points. In a second step the mineral association may be used to confirm the selection or do an incremental change.

6. FUTURE WORK

The next step in the development work will be to use the data from the PTA and investigate it with Multivariate Data Analysis to see in detail to what degree the particle morphology is affecting the process. Even here, a comparison of main stream flows with external flows to check for relationships will be done, but with the added benefit of an unbiased analysis.

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REFERENCES

Paper V
Using process mineralogy signatures and multivariate statistics to classify flows in a concentrator

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USING PROCESS MINERALOGY SIGNATURES AND MULTIVARIATE STATISTICS TO CLASSIFY FLOWS IN A CONCENTRATOR.

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ABSTRACT
The constantly growing demand from customers for higher product quality implies that the ability to monitor and adjust the final product is crucial. This is fairly easily done in batch-processes, where a unique identifier can be attached to each process lot. In continuous processes other identifiers must be used, and in the case presented here signatures coming from process mineralogy come into play. In addition, it is important to have complete information about the flows that are passing through the process, and how they are connected. Here is demonstrated how process mineralogy results from Particle Texture Analysis (PTA) can be used to classify flows in a concentrator.

Loads of information is produced from each PTA run on a sample, the problem with these data is that they are noisy and highly co-linear, and it is hard to extract the systematic information. By using multivariate statistic analysis it is possible to get a data reduction that will convert and show the values that have a vital role in a process. The research in this paper makes frequent use of PTA-data in multivariate analysis and orthogonal projections to latent structures (OPLS) in particular.

The techniques PCA, PLS-DA and OPLS-DA are used on the same data and the results show that OPLS-DA shows a distinguishing ability that simplifies interpretation of process mineralogy data.

Results also show that by only using the morphological parameters, it will expedite the processing of conclusive interpretations. Rapidly obtaining understandings of huge datasets will save time for process management.

Introduction
Here is presented results from a part of a project between Luleå University of Technology, (LTU) and the Swedish mining company Luossavaara Kirunavaara AB (LKAB). The focus is to find a traceability method for a continuous process (Oghazi et al., 2009). Traceability is the ability to access the history of a specific object in the production line or specific material in the process line, which gives an abundance of possibilities for quality control (Flodin et al., 2008).

Earlier in the project samples were analysed from the concentrator’s main flows by using Particle Texture Analysis (PTA) where mineralogical and morphological characteristics were identified (Oghazi et al., 2009). This kind of process analysis has shown good results and gives a general view over different events in the process.
In the case presented here, samples were taken from the concentrator’s recycled flows that are sent back into the ordinary or main flow. The purpose of this study is to understand the difference between the recycled flows that are connected back to the main flow and by doing so hopefully reduce production and process disturbances. Currently there is no detailed information about the process mineralogy in the recycled flows, which are not optimised from a process technical viewpoint. Comprehensive information about the process and the sampling points can be found in Oghazi et al. (2011).

Results from the PTA include massive data for each sample described by several variables. A typical sample for PTA is a particle size fraction mounted in epoxy. It may contain between 5000 to 15000 individual grains that each gets different variable values. One way out of this information over-load is to use multivariate statistics to extract the systematic information in and between samples. Using multivariate analysis by Principal Component Analysis (PCA), Projections to Latent Structures (PLS) or Orthogonal Projections to Latent Structures (OPLS) (Wold et al., 1993) gives an opportunity of interpretation of the data that are helpful to understand the relation between flows in the process. In this contribution, OPLS treated process mineralogy data is discussed and multivariate data analysis is used to find relations between different flows in the process.

Multivariate statistics on process data has been used earlier for process control (Martin 1996). Multivariate analysis of data has also been used for process investigation by numbers of researchers (Kresta at al. 1991; Wise et al. 1990). However, to investigate morphology parameters by using multivariate statistical methods to find relations between process flows is new.

Multivariate analysis
Multivariate analysis covers a set of techniques dedicated to the analysis of data sets with more than one variable. The different analytical techniques used in this investigation are introduced below.

PCA Principal Component Analysis
The main idea of Principal Component Analysis (PCA) is to reduce the quantity of a data in a data set where there are a large number of interconnected (co-linear) variables, while retaining as much as possible of the variation present in the data set. It is a method to find a correlation from the transformed dataset by using a new coordinate system and put the largest variance on the first component, the second largest on the second component and so on (Eriksson et al., 2001).

Wold et al. (1987) and later Prats-Montalbán et al. (2006) have a simplified version of explaining PCA, where PCA is a projection method of original variables onto new ones, called latent variables, which are orthogonal and arranged according to their eigenvalue. A matrix $X$ with the original data set is expressed as:

$$ X = AB^T + E $$

Where $A$ is the score matrix containing object information, $B$ the loading matrix with variable information and $E$ the error (or residual) matrix.
**PLS and OPLS**

PLS stand for Partial Least Squares alternatively Projections to Latent Structures, and is used to understand the variables in the input process $X$ that influence the output variables in the resulting process $Y$. By using statistical regression, PLS modeling finds a relation between the variables in the two groups (Wold et., al. 1999).

Orthogonal projections to latent structures or OPLS is a modification of PLS that is designed to handle variations in $X$ that are orthogonal to $Y$. OPLS is helping to improve the transparency and interpretability for the model (Eriksson et al., 2001). Simply speaking, OPLS provides a way to remove systematic variations in $X$ that are not correlated to $Y$. By removing the non-correlating variations the interpretational ability of the models also improves (Trygg et al., 2002). As Trygg explained, removing variability in $X$ that is orthogonal to $Y$, will not only give a better predictability but also the interpretational ability of the models will improve. Alternatively, OPLS may be used to filter out a strong primary influence, so weaker secondary variations can be interpreted freely.

Trygg and Wold have described that there are three criteria in the orthogonal correction method that should be met to use OPLS:

- Component should involve a large systematic variation in $X$.
- Component must be predictive by $X$.
- Component must be orthogonal to $Y$.

According to Trygg and Wold the greatest advantage with OPLS is the simplification which makes the interpretation much easier. They mention three advantages:

- How the correlated variation is separated from the non-correlated orthogonal variation.
- To be able to analyze the non-correlated variation and how to identify with them.
- The number of PLS components might be reduced to a single component.

**PLS-DA and OPLS-DA**

The add-on DA stands for Discriminant Analysis. It is a way to try to distinguish between classes in a data set. In classical DA, an indicator variable is used to indicate presumed class membership that is to be confirmed or rejected. This is also called classification by guided hard learning, since it includes a prior knowledge imposed on the analysis (Ugland and Massart, 1995)

In the analysis presented here, the knowledge that a sample comes from a certain process flow is the basis for assigning a class attribute. The software used, SIMCA-P+ ver 11.5 and 12.0 (Umetrics, 2009), assigns an indicator (discriminant) variable to each sample (or process flow) to be treated as a single $Y$ variable or in a $Y$ block.
Materials and method

Samples were taken from the recycled flows at the LKAB Malmberget concentrator, and compared to the main grinding section flows. A recent description of the concentrator after addition of a new grinding circuit #6 was given by Tano et al., (2006). More than twenty ore bodies are known in the Malmberget ore field, occupying an area of 2.5 x 5 km (Lund, 2009). The iron ore minerals are both magnetite (Fe₃O₄) and hematite (Fe₂O₃) but the magnetite is more common of the two. Eight sample points were chosen from the recycle section (Figure 1 and Table 1) and samples were collected over five rounds. Sub-samples from different rounds were combined to a point sample that was filtered at the site. Samples were dried and sieved at LTU, for further analysis with Particle Texture Analysis (PTA) at NTNU, Norway. The samples selected for the analyses presented here, are the 38-75 μm fraction from each point sample. For more detailed information about the sample points, cf. Oghazi et al. (2011).

Figure 1. Scheme over sample points in the section for pre-concentration and dewatering of recycle flows.
These samples are to be compared to the concentrator’s main sections for analysing/checking how external/recycle flows should be sent to the best theoretical access point in the main flowsheet.

Table 1. Sample points in the recycle flows section.

<table>
<thead>
<tr>
<th>Sample Points</th>
<th>Sample explanation</th>
<th>d_{50} (μm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>Underflow thickener # 2</td>
<td>120</td>
</tr>
<tr>
<td></td>
<td>Sample contains a mix from section 1-3, mostly hematite with a large variation</td>
<td></td>
</tr>
<tr>
<td>2</td>
<td>Underflow thickener # 4</td>
<td>210</td>
</tr>
<tr>
<td></td>
<td>Contains waste from wet cobbing plant sections 4-5, also tails from magnetic separators section 3 and section 6.</td>
<td></td>
</tr>
<tr>
<td>3</td>
<td>Underflow thickener # 5</td>
<td>210</td>
</tr>
<tr>
<td></td>
<td>Thickener #5 parallel to #4</td>
<td></td>
</tr>
<tr>
<td>4</td>
<td>Concentrate from magnetic separator. Feed is spillage from secondary mill section 1.</td>
<td>110</td>
</tr>
<tr>
<td>5</td>
<td>Concentrate from magnetic separator. Coarse particles from classifier.</td>
<td>380</td>
</tr>
<tr>
<td>6</td>
<td>Concentrate from magnetic separator. Fine particles from classifier.</td>
<td>111</td>
</tr>
<tr>
<td>7</td>
<td>Concentrate from magnetic separator. Fine particles from classifier.</td>
<td>105</td>
</tr>
<tr>
<td>8</td>
<td>Concentrate from magnetic separator, feed is combined flows material collected from the re-routed flows.</td>
<td>211</td>
</tr>
</tbody>
</table>

In this study, PTA was used to give morphology information such as shape, area, length, breadth, perimeter and mean image grey level for an individual feature or grain. The parameters, which are used in the data set, are listed in Table 2.

Table 2. List of parameters which were extracted from PTA and used for analysis.

<table>
<thead>
<tr>
<th>Parameters:</th>
</tr>
</thead>
<tbody>
<tr>
<td>Area: (Area of whole feature in square microns)</td>
</tr>
<tr>
<td>Length: (Max feret)</td>
</tr>
<tr>
<td>Breadth: (Min feret)</td>
</tr>
<tr>
<td>Perimeter: (Perimeter of whole feature in microns)</td>
</tr>
<tr>
<td>Aspect-ratio: (Length/Breadth)</td>
</tr>
<tr>
<td>Shape: (Perimeter^2/4π*Area)</td>
</tr>
<tr>
<td>ECD: (\sqrt{\frac{4\times Area}{\pi}})</td>
</tr>
<tr>
<td>Mean grey level: (Mean image grey level for each particle)</td>
</tr>
</tbody>
</table>
**Statistical analysis**

The samples from the recycling flows and raw material that comes into concentrator main sections #5 and #6 were collected and analysed with PTA. The raw PTA-data for a few samples at a time are then combined in several common data sets to check if any correlation can be established by multivariate analysis.

Score plots, maps with the objects (mineral grains) projected on hyper planes are not very useful here, due to the large number of objects. Instead, loading plots are used to find relation among the variables; in the present case what type of multivariate model gives the best divergence or correlation among the variables.

**Result and Discussion**

**Patterns**

In the first analysis, sample #1, #2, #3 and feed to main section #5 are combined in the same data set. The reason for this grouping is that sample #1, #2, #3 are very similar to each other and it is relevant to observe how they compare to the raw ore that comes into main section #5. If they show comparable classification to each other then they may be reconnected directly to feed end of section #5. This dataset contains 8 variables, 59413 observations measured over four groups (samples).

PCA in itself does not reveal any direct connection between the groups/samples (since they are not included in loading plots) but it shows how the variables are related to each other. The loading plot in Figure 2 shows the variations in the first and third Principal Component (PC) directions. The use of the first and third PC direction is since they give the best spread of the data points and the variables. *Mean grey* is the variable that is strongly shifted in the third direction, while morphology parameters representing size information are assembled down to the right in the first direction. It should be noted that *Mean grey* is a measure of the brightness of a particle in a BSE image, and thus indirect information about magnetite content.

![PCA loading plot, Sample 1, 2, 3 and Section 5.](image)
This spread of observations and variables has been shown to be a common characteristic for this type of samples (Oghazi et al., 2009). And since the changes in the multivariate patterns appear to be systematically related to changes in process flows, it is natural to try to distinguish among samples by multivariate discriminant analysis.

**Relations**

The PLS-DA loading plot in Figure 3 shows the relation between the \( X \)-variables and the \( Y \)-groups (samples), and the relations within them. The plot shows how the responses (\( Y \)'s) vary in relation to each other, which ones provide similar information and their relationship to the terms in the model. According to Eriksson et.al. (2006) PLS-DA is like to have two PCA models at the same time, one for \( X \) and one for \( Y \). In the figure it is shown that feed to main section #5 and sample #1 is close to each other and more located on the right side of the graph compared to the other samples. The reason for that is found in first direction - *Mean grey*. Or in other words, indicator variables Section #5 and sample #1 are co-located (showing similarity) and affected positively mainly by *Mean grey*. This is an indication that both are higher in magnetite then the other samples since they appear brighter in BSE pictures, which the data set is based on.

![Figure 3. PLS-DA loading plot, Sample 1,2,3 and Section 5.](image)

Figure 3. PLS-DA loading plot, Sample 1,2,3 and Section 5.

▲ Morphology parameter, ■ Sample discriminant variable.

Most of the variables do show high skewness. Therefore, a logarithmic transformation of the variables were tried, bit it did not lead to improved models, and is not shown here.
To separate these groups better OPLS modelling was used, cf. Figure 4. It shows more clearly the similarity between sample #1 and the feed for main section #5. Sample #1 is positioned above the feed to section #5 and it is likely for the reason that sample #1 contains slightly finer particles and more magnetite. Sample #2 and #3 deviate from rest of the samples, probably since they contain more gangue minerals.

![OPLS-DA loading plot, Sample 1, 2, 3 and Section 5](image)

To show that there is consistency in the analysis, OPLS-DA was also used to compare the feed to main section #6 with samples #1-3, cf. Figure 5. Similar pattern is seen here, but unlike the plot in Figure 4 the feed to section #6 is not that close to sample #1. The explanation for this behavior is that the feed to section #6 contains more gangue minerals compared to the feed to section #5. In both sections three mills are used in series. The mills in section #6 are larger and have higher capacity compared with section #5. However, the probable reason for the difference is that section #6 lacks the wet cobbing stage resulting in more gangue minerals are fed to the section, while in section #5 the feed has less gangue minerals.
To further analyse the samples from the recycle section its samples #5-7 are compared with the feed to section #5, cf. Figure 6. Here sample #5 is a concentrate from a magnetic separator, where the feed comes from discharge of spiral classifier sands, which contain more gangue minerals compared to other sampling points in the same graph, which is the reason why sample #5 is low in Figure 6. Furthermore, samples #6 and #7 have similar modal mineralogy (not surprising since they both represent classifier fines), but sample #6 contains more of finer particles, indicating that the classifiers are not evenly loaded.
Another way of looking at the data is to compare the feed to the main sections with the total recycle flow (sample #8) from the re-treatment section, cf. Figure 7. The two sections are obviously different. Section #6 appears to have slightly coarser particles compared to section #5, which seems to have more elongated particles. Sample #8 is located to the right in the first direction, and this means that it contains smaller particles than the two feeds, and at the same time it has a correlation with Mean grey meaning that it contains more magnetite. So, the total recycle flow is dissimilar to the main grinding section feeds, to where it is currently returned. A better return point would be as feed to the secondary grinding steps.
Conclusions
This paper has studied the recycled flows in relation to the feed to the main grinding sections in a concentrator, by investigating process mineralogy data with multivariate data analysis such as PCA, PLS-DA and OPLS-DA. These methods give the possibility to automatically reduce insignificant data and emphasise variables that affect the process for more rapid interpretation of the results. The best method seems to be OPLS-DA since it was the monitoring method that has both strong correlated variables and fair separation between different indicator variables. That said, it must be pointed out that the multivariate methods do not give the absolute quantitative truths, rather rapid qualitative informations about the differences and similarities between process flows.

The process conclusions from these analyses show that:

- Materials from supposedly similar sampling points may deviate.
- The result shows that the total recycle flow is so different from feeds to sections #5 and #6 that it should not be combined with them.
- Multivariate analysis is a functional tool to interpret differences in process mineralogy signatures, and doing so in un-biased way.

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References


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COMPARING THE MINERALOGICAL CHARACTERIZATION OF IRON ORE BY USING QEMSCAN AND PTA

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Abstract
Automatic scanning systems can be used to detect valuable minerals in polished sections. These instruments have now reached a maturity level for the actual analysis, but since they generate a lot of information the bottleneck is now the interpretation of the data flow to get quantitative information. For example, today most industries are interested to use automated scanning system to achieve better products or to simplify the process by having better knowledge of the material that they are using in production. In this case two methods: Particle Texture Analysis (PTA) and QEMSCAN are checked for differences. Measured properties were modal mineralogy, mineral associations and mineral liberations in several samples over the same size fraction 38-53 μm. The difference is the largest for the degree of liberations, probably since this measure is sensitive for the computational assumptions made.

Keywords: Automatic scanning, QEMSCAN, Particle Texture Analysis, Liberation

1 INTRODUCTION
The economy is a crucial factor in beneficiation of the ore. Another important factor is the quality of the product. Today high quality is a necessity for ore beneficiation companies. As a consequence detailed information from the raw material is required. According to Andersen et al. [1] it is important for profitability and optimisation to have a good understanding in relating mineralogy texture and particle characterization with each other by automated methods.

The context of mineralogy gives information about the ore and if it is beneficial to extract it, and where microscopic analysis uncover the mineralogy for us regarding the efficiency of processing the ore [2]. Petruk [3] explains how applied mineralogy can be used in different steps in a mining company. He also mentions different techniques that are used for determining the mineral characterization. According to Petruk, applied mineralogy is the key for an engineer in matter of selecting the type of process technique for concentrating a mineral.
During the last decades, mining industries have opened the doors for new instruments and methods to streamline the production. Scanning Electron Microscopy (SEM) has been used for a long time and QEMSCAN is one of the better known techniques for mineral characterization, associations and mineral texture. This SEM-EDS technique gives also information about, among other things, the chemical assays [4]. Particle Texture Analysis (PTA) is another system based on scanning electron microscope different from the Oxford Inca software [5].

All these techniques are used to identify minerals and mineral compositions. In the present paper, identical samples are analysed with the two difference techniques to see if there are any differences between the systems. In this case it is very interesting to investigate mineral associations and liberation for gangue minerals and the magnetite. This result can be obtained by the systems and show if there are some mixed magnetite particles that are gangue containing and may affect the processing or create future process problems.

2 ANALYTICAL SYSTEMS

2.1 Particle Texture Analysis

The Particle Texture Analysis (PTA) was developed at the Norwegian University of Science and Technology (NTNU). By using Back Scattered Electron (BSE) from the scanning electron microscopy, images are analysed by means of grey levels. Simply the polished section is scanned under the beam and every grain that is overlapping or has similar grey-levels is also analysed by X-rays. All analysed grain size fractions are imported to the PTA software, where images analyses are done offline to process and evaluate if grains occur as liberated or in composite particles. For more detailed description see Moen [5]. The equipment that was used/applied for the PTA is listed in Tables 1 and 2.

2.2 QEMSCAN

There are some articles that explain QEMSCAN very well, that is Butcher [6] and Gottlieb [4]. Each mineral has its own distinctive energy dispersive X-ray spectra, and QEMSCAN is an automated system based on a scanning electron microscope. When the particles are analysed, QEMSCAN uses the raw data from X-ray spectra to compare with a mineral identification library known as the Species Identification Program (SIP), for more information see Lotter [7]. The data for each examined point are evaluated against the list elements that exist in the sample. The system is normally equipped with four x-ray detectors, some species with similar x-ray spectra and backscatter images are differentiated by their element ratio. Some minerals such as magnetite and hematite with similar x-ray spectra are differentiated with the back scatter images [4]. These analyses provide us with information from mineral liberation, modal mineralogy, and also mineral association. The equipment that was used/applied for QEMSCAN is listed in the Tables 3 and 4.

2.3 Experimental and material overview

For this investigation representative samples were collected at the LKAB concentrator at Malmberget. Ores that are fed to the concentrator come from different ore bodies, which are mined and mixed for the beneficiation process. The samples are material from input and output to three ball mills in series from two parallel grinding sections. Flowsheets for the grindings section were reported by Oghazi et al. [8]. The samples were weighted and then filtered at Malmberget. All the samples were then dried and cut by a Jones splitter into suitable proportions in the laboratory at Luleå University of Technology (LTU). The dry material was sieved with a Ro-Tap shaker down to 75 μm and wet sieved further to 38 μm. Polished thin sections were made of the sieved fractions at NTNU.
3 RESULT AND DISCUSSION

The PTA and QEMSCAN results are represented by modal mineralogy charts in Figures 1 to 4. Charts of modal mineralogy are showing the percentages of minerals found in the analysed grain-size fraction based on examination of a sample. There is no large difference between the sections, although it is shown that the magnetite content in section 5 is slightly higher than in section 6. Comparing section 5 by the analytical method used, the similarities are very obvious. The magnetite amount that is detected is almost the same, and the gangue minerals amount also. However, there are some few more percent gangues minerals detected for section 5 in the QEMSCAN compared to the PTA. Reason for that could be the limits setup in the secondary SIP file. The sip-file is the term used in QEMSCAN terminology to describe the relationship between mineral data and analysed data. This relationship is used to convert the electron detectors’ raw output to mineral output. In this correlation it is important to be correct to ensure that the right mineral data output is produced [9].

For section 6 the overall representation is the same as for the previous section with both methods, the gangue minerals detection in QEMSCAN is more clearly presented compared to section 5. By comparing the incoming material to the primary mill in section 6, QEMSCAN detect almost 73-percent magnetite and 27-percent gangue minerals (counting the unclassified to the gangue minerals). While in PTA same sample show roughly 79-percent magnetite and the rest were gangue minerals. Another interesting point is the amount of feldspar and pyroxene that is detected in the primary mill for section 6. For both the input and output it is a notable difference between the two analysing system. In QEMSCAN the amount of these minerals are larger compared to the PTA analysis. Once again the divergences were found in section 6 between the systems, while for section 5 the result were almost identical. As mentioned before due to the setups in the SIP file and also grey-scale values of the image can contribute to the shown result.

Minerals that are associated to apatite as detected by QEMSCAN and PTA are shown in Table 5. Note, that the magnetite association is almost identical, while K-feldspar is more detected in PTA compared to QEMSCAN.

Liberation of apatite differs considerably between the instruments as shown in Figure 5. It appears that for QEMSCAN the liberation analysis are more detailed compared to PTA. In PTA, back scattered images are segmented by using grey-levels; an off-line software is used to create the liberation analysis. However, in QEMSCAN back scatter images is also used but it is equipped with up to four x-ray detectors to identifies the minerals at each point by collecting an energy dispersive x-ray spectrum that is analysed to give the chemical composition in every point [4], [6]. This might be the reason for the perception that QEMSCAN gives more mixed grains in the liberation analyses. Another possibility is that the algorithms used differ to some degree.

4 CONCLUSIONS

QEMSCAN is used today in commercial research; QEMSCAN gives detailed quantification information of the process material. It also provides a detailed particle mapping which is an advantage, furthermore the system is rapid. Another advantage with QEMSCAN is that the result can visibly been shown very clear how the gangue population is present, how this population is associated and even if there are some inclusion in the magnetite or vice versa. One benefit of the PTA is the extraction of good information about particle size, mixed particles and liberation.
In a QEMSCAN a SEM and four EDS detectors is used, although an advanced software package for identification and quantification of the minerals and phases must be accessible. So, a disadvantage with this system is the investment cost for the QEMSCAN.

Both systems give almost identical results for the mineralogical identification and mineral content. The differences come into play, when the data is further processing. In particular the liberation analyses seem to be dependent on the algorithms used, and their tolerance limits.

Also, not mentioned earlier, is that PTA can easily deliver morphological data output for further processing. QEMSCAN is a more “locked in” system than PTA. This is good for quantitative industrial research, but a disadvantage in more fundamental investigations.

There are still some development works left for the PTA, such as the software but also the integration with Oxford Inca Feature to get a better analysis. Still the PTA is young compared to QEMSCAN, and hopefully can be even more developed in the coming years.

5 REFERENCES


Table 1: Equipment for PTA analysis.

<table>
<thead>
<tr>
<th>Equipment</th>
<th>Type</th>
</tr>
</thead>
<tbody>
<tr>
<td>Field Emission SEM</td>
<td>Hitachi S-4300 SE</td>
</tr>
<tr>
<td>EDS detector</td>
<td>Oxford light element detector</td>
</tr>
<tr>
<td>Acquisition software</td>
<td>Inca Feature</td>
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### Table 2: The setting for the PTA analysis.

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Setting</th>
</tr>
</thead>
<tbody>
<tr>
<td>Working distance</td>
<td>15 mm</td>
</tr>
<tr>
<td>Accelerating voltage</td>
<td>20 kV</td>
</tr>
<tr>
<td>Gun Brightness</td>
<td>1.9 kV</td>
</tr>
<tr>
<td>Beam current</td>
<td>0.25-0.75 nA</td>
</tr>
</tbody>
</table>

### Table 3: Equipment for QEMSCAN analysis.

<table>
<thead>
<tr>
<th>Equipment</th>
<th>Type</th>
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<tbody>
<tr>
<td>QEMSCAN</td>
<td>E340</td>
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<tr>
<td>EDS detector</td>
<td>Silicon Drift Energy Dispersive x-ray Detectors</td>
</tr>
<tr>
<td>Acquisition software</td>
<td>iDiscover</td>
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### Table 4: The setting for the QEMSCAN analysis.

<table>
<thead>
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<tr>
<td>Working distance</td>
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<tr>
<td>Accelerating voltage</td>
<td>20 kV</td>
</tr>
<tr>
<td>Beam current</td>
<td>5 nA</td>
</tr>
</tbody>
</table>

### Table 5: Apatite association in QEMSCAN and PTA.

<table>
<thead>
<tr>
<th>Apatite association</th>
<th>QEMSCAN 06 Mill#1 (in)</th>
<th>PTA 06 Mill#1 (in)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Unclassified</td>
<td>2.34</td>
<td>4.5</td>
</tr>
<tr>
<td>Titanite</td>
<td>0.08</td>
<td>0</td>
</tr>
<tr>
<td>Ilmenite</td>
<td>0.03</td>
<td>0.7</td>
</tr>
<tr>
<td>Pyroxene</td>
<td>0.61</td>
<td>0</td>
</tr>
<tr>
<td>Calcite</td>
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<td>0</td>
</tr>
<tr>
<td>Biotite</td>
<td>0.16</td>
<td>0</td>
</tr>
<tr>
<td>Chlorite</td>
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<td>0</td>
</tr>
<tr>
<td>K feldspar</td>
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<tr>
<td>Feldspar</td>
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<tr>
<td>Quartz</td>
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<td>0</td>
</tr>
<tr>
<td>Magnetite</td>
<td>3.99</td>
<td>4.1</td>
</tr>
</tbody>
</table>
Figure 1: Modal mineralogy chart for concentrator section 5 from PTA analysis.

Figure 2: Modal mineralogy chart for concentrator section 5 from QEMSCAN analysis.
Figure 3: Modal mineralogy chart for section 6 in concentrator from PTA analysis.
Figure 4: Modal mineralogy chart for section 6 in concentrator from QEMSCAN analysis.

Figure 5: Liberation of apatite in QEMSCAN and PTA.