Sustainable, Decarbonised Co-Extraction Of Vanadium And Titanium Minerals From Europe's Low-Grade Vanadium-Bearing Titanomagnetite Deposits

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Analytical protocol

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Table of contents

<u>Appendices/ links to external sources</u>:

I. Minutes _WP1 Technical discussion at the Kick-off meeting
II. Complete guidelines on Standard Operating Procedure (SOP) (AVANTiS teams: AVANTiS_SOP_March_2024.pdf)
III. AVANTiS_Material_database_01_03_24 (AVANTiS teams: AVANTiS_materials_database_01_03_24.xlsx
IV. Guideline for AVANTiS Material DATABASSE_13_3_24 (AVANTiS teams: GUIDELINES FOR AVANTiS MATERIAL DATABASE_13_3_24.pdf
V. Certified Reference Sample data (https://amis.co.za/wp-content/uploads/AMIS0567-Certificate.pdf)
VI. In situ mineralogical analysis protocol
VII. Electron microprobe analyses data

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1. Introduction

Given the variability and complex nature of V-Ti-Fe-(P) bearing oxide ores, it's imperative to grasp their mineralogical characteristics before initiating any processing trials. Understanding the mineralogy and geochemical composition of both the ores and the accompanying gangue minerals is essential in assessing their influence on the recovery of titanium, vanadium, iron, and phosphorus from these complex ore deposits (see Figure 1).

AVANTiS analytical protocol refers to a systematic procedure to analyze the project samples using cutting-edge analytical techniques and interpret the geochemical, mineralogical, and metallurgical data. The protocol outlines the step-by-step processes and guidelines for sampling, sample handling, and conducting analyses or investigations to ensure results' accuracy, reliability, and reproducibility. By following established protocols and procedures, AVANTiS-WP1 experts can standardize their methods, minimize errors, and enhance the quality of their data.

The project experts have developed a Standard Operating Procedure (SOP) as a crucial component of the analytical protocol. This SOP delineates geological sampling procedures, ensuring the highest quality of project sample material and yielding optimal scientific outcomes. In AVANTiS studies, collecting representative samples is a crucial step for mineralogical investigations, geochemical analysis, and mineral processing operations. Careful handling of geological samples is essential to maintain safety standards, ensure accurate analyses, prevent contamination, and uphold ethical principles in research. Regular equipment calibration ensures dependable analytical outcomes, ensuring quality assurance. Adherence to the standard procedures is key to maintaining accurate and reliable results. Effective sample tracking through proper labeling and documentation is facilitated by the project's experts, who have developed material databases and guideline documents for this purpose.

Conventional XRF, XRD, SEM, EPMA, and advanced techniques (XCT, µXRF, LA-ICP-MS, automated mineralogy, Raman) characterize the complex geochemistry, mineralogy and texture of V- and Ti-bearing oxide minerals and associated valuable critical raw materials. Validating analytical techniques across different laboratories is crucial for reliable and consistent results in the AVANTiS-WP1 team's analytical protocol. This process confirms the accuracy and precision of the analytical method regardless of its location. Experts conducted inter-laboratory studies to ensure accurate analytical techniques in processing AVANTiS geological samples. In these studies, three laboratories (GTK, AGH, NTNU) analyzed the same samples using the same method. The method's robustness and reproducibility are evaluated by comparing the results obtained.

When performing geochemical analyses (in-house or out-sourcing), it is crucial to ensure the accuracy and reliability of the results. One standard practice to achieve this is by making use of certified reference materials (CRMs). CRMs are substances with precisely known concentrations of specific elements or compounds. For consistent and reliable measurements, geochemical laboratories servicing the AVANTiS project should use CRMs as standards.

Another important step in verifying analytical results is to include BLANK samples into the analytical series. BLANK samples should be similar in mineralogy and appearance as the target material, but preferably not contain (any) of the target element(s) or very low content of the elements of interest. BLANK samples are used to reveal any contamination of samples or in the analytical equipment during the preparation of sample batches.

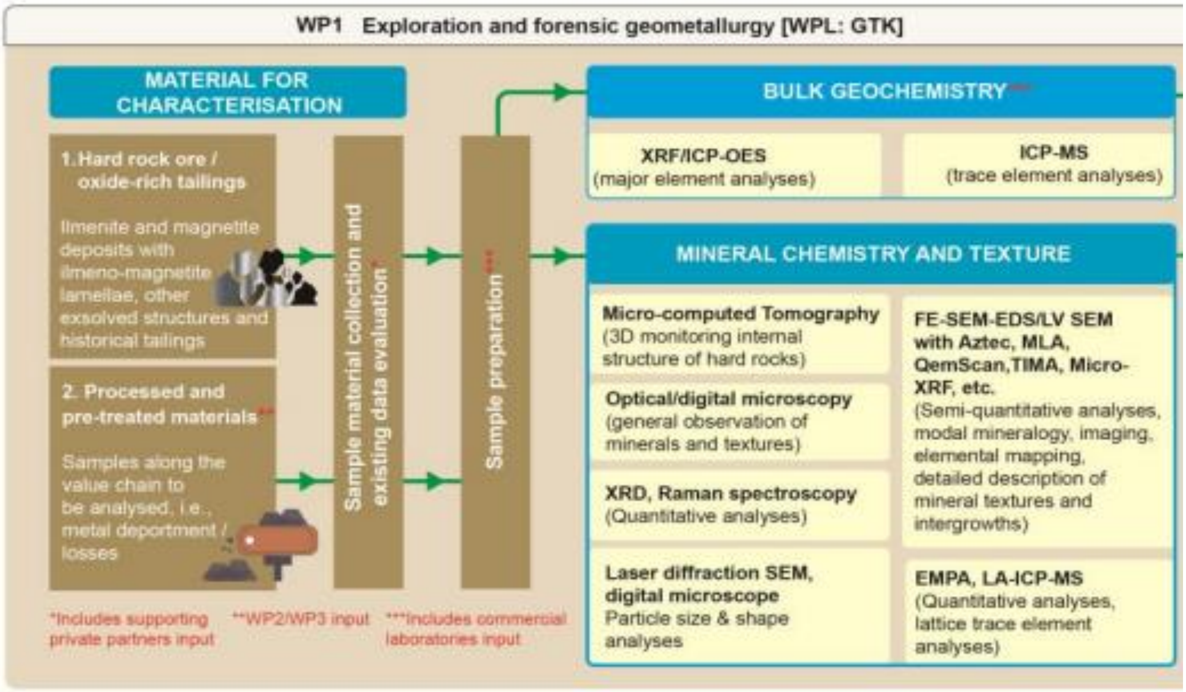

Figure 1. AVANTiS's forensic geometallurgy starting protocol and the included characterization techniques

In the process of developing guidelines on the project sampling procedures, and validating analytical techniques in different laboratories, the following steps are agreed upon by AVANTiS WP1 experts (See Appendix I).

1. Preparing Standard Operating Procedure (SOP) (GTK)
2. Preparing material database (KUL and GTK) and guideline document for material database (GTK and KUL)
3. Use Certified Reference Material (CRM) for validating geochemical analyses (GTK)
4. Checking mineralogical analysis techniques for accuracies using own certified standards (GTK, AGH, NTNU, KUL)

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2. Development of Standard Operating Procedure

A separate document (26pp) on a Standard Operating Procedure (SOP) is attached to this report as Appendix II and saved in the AVANTiS team folder.

While adhering to a proper analytical protocol is crucial, achieving optimal results also hinges on implementing standardized sampling and sample handling procedures. This document outlines the geological sampling procedures that will be utilized in the AVANTiS Project to ensure the highest quality samples and optimal scientific results. The geoscientists who are responsible for collecting geological data during the AVANTiS Project must develop their field sampling plans and make sure that proper sampling, sample handling, preservation, quality control, and record-keeping procedures are followed to minimize errors and deficiencies. This Standard Operating Procedure (SOP) has been developed based on best practices, industry standards, organizational policies, and regulatory requirements, and serves as an essential tool for quality assurance, compliance, and continuous improvement.

According to SOP, the material database and guideline document are supportive documents related to the sample material flow of the project. Both documents are attached to this report as Appendix III and IV, and saved in the AVANTiS team folder.

3. Development of analytical protocol

3.1 Certified Reference Material

When validating geochemical analyses, the Certificated Reference Materials (CRM) should be chosen based on the element(s) under examination with concentrations closely matching the samples to be analyzed using the same methods. Discrepancies between the certified values of the CRM and the measured values can indicate potential issues with the analytical procedure, calibration, or instrument performance. By regularly incorporating CRMs into their analyses, researchers can track the performance of their methods, identify any sources of error, and ensure the quality of their geochemical data.

3.1.1 CRM for AVANTiS use

According to Certificated Reference Materials (CRM) for AVANTiS, the project's expert has scouted out CRM from African Mineral Standards (AMIS; https://amis.co.za/) and OREAS (https://www.oreas.com/). According to project purposes, the best available candidate was AMIS 0567 standard (see https://amis.co.za/product/fe-21-98-ti-2-98-v-3763-ppm-vanadium-bushveld-south-africa/).

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The standard represents titaniferous and magnetite-rich vanadium mineralization sourced from the Vametco Mine, Rustenburg Suite, situated in the upper zone of the Bushveld Igneous Complex. Following the Standard Operating Procedure (SOP), the AVANTiS project elected to distribute 20 Certified Reference Material (CRM) bags, each containing 30 grams, to every project partner (KUL, Proxis, GTK, NTNU, AGH). Dr. Jukka Konnunaho from GTK has ensured the delivery of these bags to the respective partners.

In regards to CRM certificate (see https://amis.co.za/wp-content/uploads/AMIS0567-Certificate.pdf), CRM has been assayed several methods such as Multi-acid digestion with ICP (i.e., near total), Fusion digestion with ICP (i.e., total) and XRF. XRF is suitable for Ti, V, Fe, and P characterization of oxide ores and multi-acid digestion to fusion for trace elements and REE characterization. Selected methods are also available from several commercial laboratories such as ALS (see procedure: https://www.alsglobal.com/en/geochemistry/geochemistry-fee-schedules).

The recommended procedure for the use of this CRM as a control standard in laboratory quality control is to develop a Shewhart chart (see SOP), where a mean value and corresponding 1, 2, and 3 standard deviations are derived from replicate measurements of the CRM.

3.2 Validating mineralogical analyses (EMPA)

The standard operating procedure (SOP) encompasses analytical methods and mineralogical analyses (Pages 12-13), while a comprehensive procedure for in-situ mineralogical analyses is additionally provided in Appendix VI. To ensure consistency across laboratories in validating analytics, the Electron Microprobe Analysis (EMPA) technique was specifically chosen to generate comparable results across all partner laboratories using identical samples. EMPA facilities at GTK, NTNU, and AGH laboratories participated in the procedure. By establishing common standards and protocols, AVANTiS-WP1 experts can ensure that their analytical methods are reliable and produce consistent results, regardless of where they are implemented.

3.2.1 Validation sample and methodology

The validation sample selected is a V-Fe-Ti oxide-rich gabbro sourced from the Mustavaara deposit within the Koillismaa intrusion in NE Finland, provided by GTK. It consists of two polished thin sections, sample 2024000175 representing the middle ore layer, and sample 2024000176 representing the lower ore layer of the deposit with the oxides in the form of ilmenomagnetite grains with distinct ilmenite exsolutions. Designated sections containing ilmenite and magnetite grains were assessed in both samples, with the additional inclusion of titanite measurements (exclusive to sample 2024000175) conducted by the laboratories mentioned above.

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To ensure that measurements in the different laboratories are within the selected grains as close as possible to each other, coordinate reference grids were attached to the thin sections and coordinates of the individual measurement points shared between the laboratories. Each laboratory adheres to its unique analysis protocol regarding reference standard usage, background measurement, measurement dwell time, and selection of ZAF correction method. Nevertheless, to ensure comparability and minimize potential systematic errors, all three laboratories reached a consensus on utilizing a beam current of 20nA, an accelerating voltage of 20kV, and a beam diameter of 1 µm. The instruments used for the validation are JEOL iHP200F Hyperprobe Field Emission Gun-Electron Probe Microanalyzer (FEG-EPMA, GTK), JEOL JXA-8230 Superprobe EPMA (AGH), and JEOL JXA-8530F Plus FEG-EPMA (NTNU).

3.2.2 Evaluating EMPA Results and Comparisons

Variations among the results from individual laboratories are observed for the elements of interest Ti, V, and Fe (Fig 2), with discrepancies falling within certain error margins. Specifically, for major concentrations (> 10 wt%) of TiO_2 (ilmenite, titanite) and FeO (ilmenite, magnetite), the error margin is approximately 6 rel%. For minor concentrations (1 – 10 wt%) of V_2O_5 (magnetite), TiO_2 (magnetite), and FeO (titanite), the error margin ranges from 10 rel% to 30 rel%, while for trace concentrations (< 1 wt%) of V_2O_5 (ilmenite, titanite), the error margin extends up to 20 rel%. EPMA analysis results of all three laboratories are presented in Table 1 in the appendix (Appendix VII).

Note: Rel% is a relative percentage, it is just the difference/error between the values, i.e. the difference between 50 and 52 is about 4 rel%.

These accuracy differences between laboratories are to be expected, particularly with major and trace element concentrations, due to i.e., differences in standard materials used, software packages for quantification corrections, and background calculations. The important element V has been quantified in all laboratories within a very good agreement in trace to minor level concentrations. The significantly high difference in minor element concentrations of 30 – 40 rel% can be explained by the heterogeneous nature of the minerals in these samples and exsolution lamellae in the case of TiO_2 or an inaccurately referenced standard in one of the laboratories. Apart from this difference, the general trend of all analyses performed shows a comparable consistency and a good agreement within typical EPMA accuracy uncertainties.

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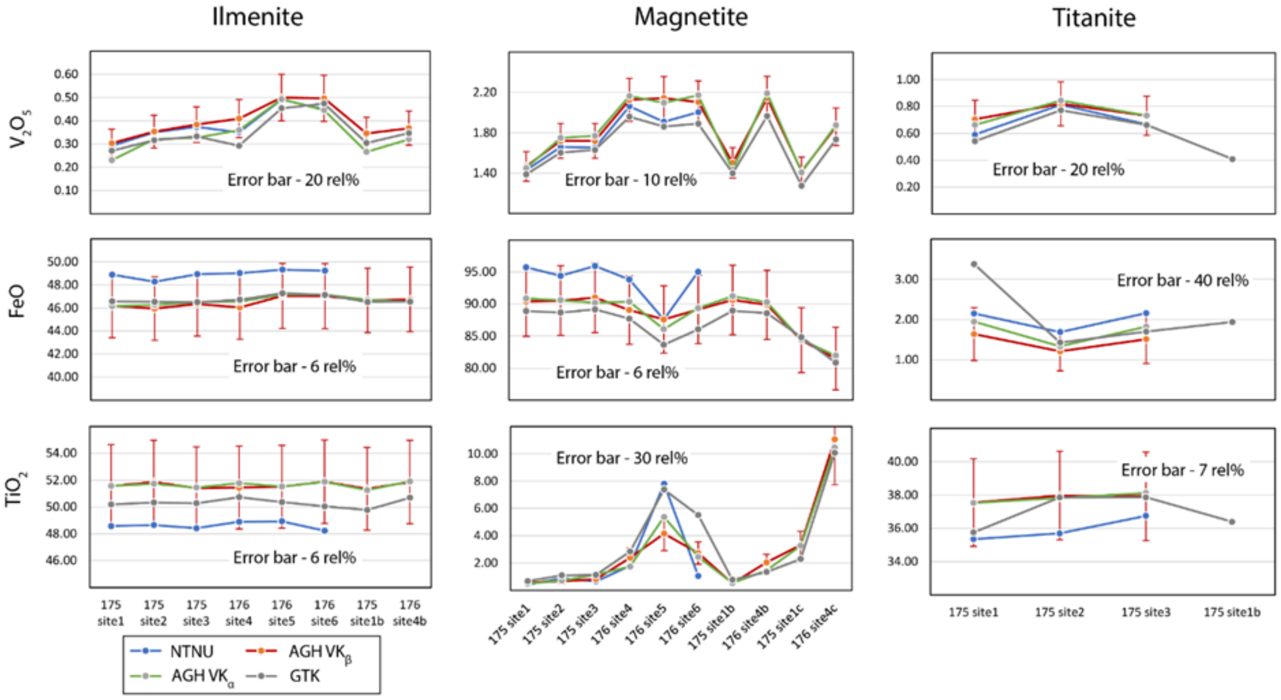

Figure 2. *Comparison of elements of importance as measured by the three different laboratories (GTK, AGH, NTNU) with relative error bars indicating most of the analyses results being within an acceptable error considering EPMA uncertainties between laboratories and sample heterogeneity, in particular Ti content from magnetite lamellae. GTK and NTNU measured the V Kβ-line. AGH results are shown for both, measurements of V Kα-line with Ti overlap correction and V Kβ-line.*

4. Implementation

Executing the AVANTIiS project's sampling and sample handling strategy, along with adhering to the analytical protocol, encompasses several crucial steps outlined in this report as SOP, EPMA validation, and the utilization of CRM to uphold accuracy, consistency, and reliability of data. Moreover, efficient data management regarding the project's sample material flow is deemed essential for prompt handling and assessment.

The project has established clear goals for the sampling and analysis and has developed a sampling strategy that takes into account various factors such as spatial distribution, sample size, representativeness, and sample type including surface and drill cores. The samples are collected using appropriate techniques and tools. Additionally, the project has developed a material flow database that is currently in use. This database ensures unbiased labeling, sequential sample numbering, and proper documentation of all samples collected (*see Excel-based sample material flow sheet and related pdf guide document*).

The project is committed to ensuring the accuracy and precision of data through rigorous quality control measures. These measures encompass duplicate sampling, blank samples, and the use of certified reference materials (CRMs) to validate whole-rock analytical results.

Analytical methods are carefully chosen in alignment with the AVANTiS objectives and the characteristics of the geological samples. The analytical protocol incorporates a blend of common techniques alongside advanced state-of-the-art methods, as illustrated in Figure 1.

Geochemical analyses, whether conducted in-house or outsourced, adhere to the protocols to maintain accuracy and reproducibility. The CRM provided is employed during analyses at accredited laboratories. These laboratories follow strict procedures for sample preparation, analysis, and data interpretation, ensuring reliability and consistency in results.

The protocol encompasses thorough data interpretation of analytical results within the framework of the research objectives. Documentation of sampling, analytical methods, procedures, results, and interpretations is integral. Data will be carefully recorded and made readily accessible as needed, fostering transparency and facilitating further analysis.

All partners must adhere strictly to every step outlined in the SOP and any protocols associated with it. Any deviation from these established procedures must be documented thoroughly, with a clear explanation provided for the deviation.

CONCLUSION

The AVANTiS project prioritizes scientific rigor and data integrity in sampling and analysis, ensuring accuracy through SOP guidelines, analytics validation, and CRM use. Quality control measures like duplicate sampling and certified reference materials boost confidence in results. Transparent documentation and data management aid in thorough recording and accessibility, fostering trust and facilitating analysis. Overall, AVANTiS upholds high scientific standards, contributing to geological understanding and exploration methods.

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Appendix I. Minutes _WP1 Technical discussion at the Kick-off meeting

EU HORIZON EUROPE AVANTIS

19th January 2024, Leuven

WP1 - Parallel technical discussion

Participants:

Philippe Muchez, Thomas Van Gerve (KUL)
Jukka Konnunaho, Tuomo Karinen, Janne Hokka, Tegist Chernet (GTK)
Kurt Aasly, Bendik Frantzen (NTNU)
Jakub Ryznar, Marcin Loska (Proxis)
Konstantinos Komnitsas (TUC)
Władysław Zygo (AGH)
Marte Tøgersen, Åsa Barstad (Titania AS)

<u>Topics discussed and action points agreed:</u>

1. On the process of validating analytical techniques and procedure in four laboratories (T1.2, T1.3)
    a. Agreed to use Certified Reference Material (CRM) for validating geochemical analyses:
        i. CRM will be purchased from AMIS (African Mineral Standards) that is a leading international ISO17034 accredited manufacturer and supplier of a wide range of matrix matched Certified Reference Material.
        ii. GTK (Jukka) will order the CRM from AMIS and distribute for the laboratories (GTK, NTNU, KUL, AGH)

    b. Agreed on checking mineralogical analysis techniques for accuracies using own standards:
        i. Select two polished sections or thin sections from Mustavaara by GTK,
        ii. Use Electron Microprobe Analyses (EMPA)
        iii. Delineate reference points/grains/areas where all partners will make analyses from the same sections,
        iv. Evaluate the results.

    c. Agreed to prepare Standard Operating Procedures (SOP)
        i. Define geological sampling procedure aimed at ensuring the best quality of AVANTIS project samples for optimal scientific results,
        ii. Responsibilities related to sample handling/storage and shipment to minimizing uncertainties in the data,

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   iii. Instruction steps for geochemical analyses to take into consideration reproducibility (different instruments – XRF, ICP-MS, Titrimetric, LOI) and precision (replicate samples).
   iv. Mineralogical analyses procedure (µXRF, FE-SEM, SEM-MLA, etc.,) using Mustavaara selected sections by all laboratories to set up analytical procedures to follow.
   v. All WP partners will draft the SOP; GTK will take the initiative.

2. Agreed on existing data survey and evaluation methods and data documentation system _ Jukka/Tuomo (T1.1)

3. Agreed that each WP1 partners scheduling sampling campaign together with the corresponding private partners.

To be agreed soon:
Sample number flow sheet /excel chart under development (Philippe/Jukka)

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Operating Procedure (SOP) for the AVANTIS Project: Geological Sample collection, Handling and Quality Control

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Contents

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Introduction

Purpose of the Standard Operating Procedures (SOP)

This document defines geological sampling procedures aimed at ensuring the best quality of AVANTIS Project samples for optimal scientific results. The geoscientists responsible for geological data collection in the AVANTIS Project must prepare their own field sampling plans and ensure that sampling, sample handling, preservation, quality control, and record-keeping are followed to minimize errors and deficiencies.

Scope of Geological Sampling

The common denominator among selected deposits in AVANTIS is the complex, low-grade V-bearing titanomagnetite material. This is examined through innovative selective blasting, fragmentation, and preconcentration technologies, all supported by a tailored geometallurgical protocol.

The sampling procedures, including any relevant equipment, should align with the overarching purpose of sampling. This alignment extends to the objectives of sample analysis and/or testing, as well as the subsequent decisions or designs that will be informed by the derived data.

Quality Assurance and Quality Control (QA/QC)

The main objective of the QA/QC program is to minimize errors introduced in sampling, sample preparation, and sample assaying procedures. It is a continuous process providing information necessary to identify and correct defects in the shortest amount of time possible.

- Quality Assurance (“QA”) is put in place to prevent problems, while Quality Control (“QC”) aims to detect them in the event that they may occur.

QC procedures are necessary to monitor contamination, precision, accuracy and bias and typically involve using specially prepared standards of known grade and sample duplicates to achieve this.

- The use of duplicates check precision and possibly bias.

- The use of a set of standards (CRMs) check accuracy.
- Insertion of blanks into the samples scheme to check for possible contamination in sample preparation at the laboratory.

Typically, a robust QA/QC program is one that is active, ongoing and is reviewed throughout the data collection process, enabling corrective action to be taken during the sampling campaign or at the laboratory. In each case study within the AVANTIS Project, the sampling campaign is relatively compact. This essentially entails conducting the sampling in a single effort, with the subsequent monitoring of results taking place upon their receipt.

All QC check samples sent for analysis to the external commercial laboratory should be blind, meaning that the laboratory should not be able to differentiate a check sample from a regular submission. Therefore, a blind numbering system needs to be assigned for samples submitted to laboratory.

The external QC samples should not be confused with laboratories internal QC checks or standards used to monitor the performance of analytical laboratory equipment. The internal laboratories QC checks reported by the analytical laboratory as a measure of its own sampling precision and accuracy should never be considered as part of formal QA/QC program (Rossi & Deutsch 2014).

If any QC samples fail, it indicates that the entire batch or a selected portion—such as five samples on both sides, including the control sample—should be re-assayed. This involves communication with the laboratory management.

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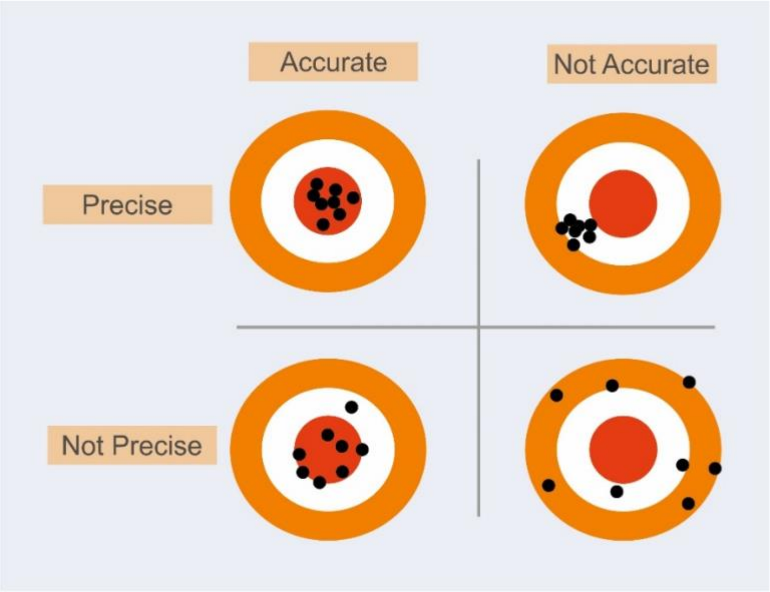

Figure 1. The classic analogy used to describe the differences between precision, accuracy and bias is throwing darts at a dartboard. If the arrows are clustered within the bull's-eye, they show good accuracy and precision whereas if they are clustered away from the bull's eye they show poor accuracy but good precision and high bias. In reality, samples invariably incur a degree of imprecision and inaccuracy and by using proper sampling practices it can be ensured that the errors are as small as possible (Modified from Coombes 2008).

Definitions and Terminology

- **Chain of Custody**: security of samples
- **Contamination**: introduction of any substance to a geological sample that is not in the original in situ location of that sample.
- **CRM**: Certified Reference Material (standard). CRM's are used to monitor the accuracy of analyses reported by mineral testing laboratories. A systematic difference from the expected CRM result indicates a bias within or between assay batches. Standard samples may be purchased commercially or may be prepared internally and it is recommended to submit standards that span the practical range of likely assay values.
- **BLANKS**: or samples without mineralization are submitted with each batch of samples sent to the laboratory. The blank material is collected from a location known to be devoid of any mineralization or purchased from a reputed supplier. Results from these samples indicate any contamination introduced during the sample preparation or analysis procedures.

- **Field Duplicate (Check)**: are samples collected, prepared and assayed in an identical manner to an original sample collected and submitted to provide a measure of the total variance introduced by the entire sampling and assaying process (precision).
- **Coarse Reject Duplicate (CRD)**: are collected by taking a second split after crushing, before the pulverizing stage. Coarse duplicate has the error of sample size reduction in the preparation lab and the error of analyses.
- **Pulp Duplicate (Check)**: These samples are the identical pulp samples collected at the final stage of sample reduction. Generally, Check samples are the identical pulp samples of previously analyzed sample. The duplicate results are compared to the originals to monitor analytical precision as well as any potential bias in the process caused by improper cutting of the sample, homogeneity, washing during the cutting or loss of fines during preparation.
- **Quarter Core Sample**: The remaining core (half-core) after splitting is re-split (quarter-core) and submitted as a duplicate sample to the laboratory.
- **Umpire Check Samples**: Umpire assays check the analytical precision of the laboratory relative to an umpire laboratory (Check assaying through an umpire laboratory does not determine which laboratory is more accurate). Pulp samples from the primary laboratory are retrieved and submitted as a batch to the umpire laboratory for analysis. The selection of the umpire laboratory is done after ensuring that it uses an assaying technique identical to the principal laboratory.
- **ACCURACY**: nearness to truth
- **BIAS**: average departure from truth.
- **PRECISION**: reproducibility of values.
- **RANDOM ERROR**: symmetric with respected to mean.
- **STATISTICAL ANALYSIS**: methods for dealing with error.
- **Lower Limit of Detection (LOD)**: this is the lowest concentration at which an instrument can detect a signal above background. The LOD is obtained by measuring the signal in a blank so as to determine the background signal.

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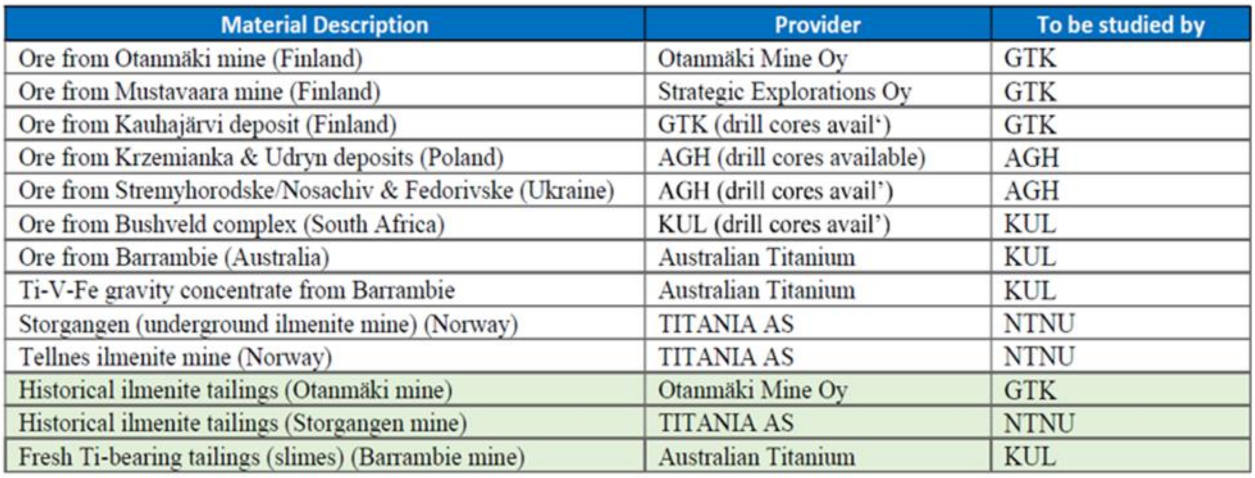
Assuming random and normally distributed errors of the background signal (XB) with a standard deviation SB, the LOD = XB+SB (3σ; Potts 1987; Jenner 1996).

- **Limit of Quantification (LOQ)**: is the concentration above the background where a result is considered quantifiable and trusted. Given a background with a concentration of XB and a standard deviation of SB, the LOQ is 10SB above the background (i.e., LOQ = XB+10SB ≈ 3.3 LOD) (Potts 1987; Jenner 1996).
- **Upper Limit of Detection (ULD)**: is the highest concentration that an instrument can reliably quantify.

Responsibilities

All geoscientists responsible for geological data collection as part of the AVANTIS project should understand the responsibilities related to field sampling, sample storage, and shipment. This understanding is crucial to ensure that these processes are conducted properly, thereby minimizing uncertainties in the data.



Material Description	Provider	To be studied by
Ore from Otanmäki mine (Finland)	Otanmäki Mine Oy	GTK
Ore from Mustavaara mine (Finland)	Strategic Explorations Oy	GTK
Ore from Kauhajärvi deposit (Finland)	GTK (drill cores avail‘)	GTK
Ore from Krzemianka & Udryn deposits (Poland)	AGH (drill cores available)	AGH
Ore from Stremyhorodske/Nosachiv & Fedorivske (Ukraine)	AGH (drill cores avail’)	AGH
Ore from Bushveld complex (South Africa)	KUL (drill cores avail’)	KUL
Ore from Barrambie (Australia)	Australian Titanium	KUL
Ti-V-Fe gravity concentrate from Barrambie	Australian Titanium	KUL
Storgangen (underground ilmenite mine) (Norway)	TITANIA AS	NTNU
Tellnes ilmenite mine (Norway)	TITANIA AS	NTNU
Historical ilmenite tailings (Otanmäki mine)	Otanmäki Mine Oy	GTK
Historical ilmenite tailings (Storgangen mine)	TITANIA AS	NTNU
Fresh Ti-bearing tailings (slimes) (Barrambie mine)	Australian Titanium	KUL

Table 1. AVANTIS-project case studies cover more than 10 deposits and 3 mine tailings.

Initiating data control at the point of sample collection is crucial. As highlighted by Dominy et al. (2019), the most significant source of uncertainty arises from sample representativity. This encompasses high error ratings in sample mass representativity, moderate error ratings in spatial distribution and the number of samples, and low error ratings in the collection and handling of samples.

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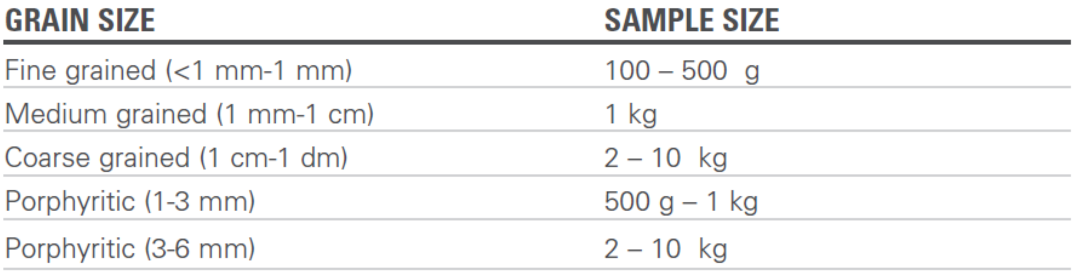
Here are a few critical parameters to consider when sampling:

1. Optimum Sample Size:
    - What is the optimum sample size and is it representative of the material that it came from? Was the host material homogeneous at the scale at which the sample was taken?
    - The size of the sample taken is dependent on the:
        - Grain size of the material to be tested.
        - Mineralogical composition with respect to the element of interest.
        - The distribution of the minerals that contain these target elements.
        - The concentration of the target elements in the least abundant minerals in the samples.
2. Sample Location
3. Sampling Methodology



GRAIN SIZE	SAMPLE SIZE
Fine grained (<1 mm-1 mm)	100 – 500 g
Medium grained (1 mm-1 cm)	1 kg
Coarse grained (1 cm-1 dm)	2 – 10 kg
Porphyritic (1-3 mm)	500 g – 1 kg
Porphyritic (3-6 mm)	2 – 10 kg

Figure 2. An approximate sample size required for rocks of given grain size (SGS, 2014).

Safety Precautions

Prioritize safety! Always adhere to the safety protocols established by your entity and the company that owns the premises where you are conducting the sampling. This includes wearing the necessary personal protective equipment (PPE), being aware of emergency procedures, and maintaining situational awareness to mitigate potential risks during the sampling process. Your commitment to safety not only protects you

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but also contributes to the overall well-being of the team and the integrity of the sampling environment.

Personal Protective Equipment (PPE)

When conducting sampling activities, ensure the use of proper protective gear, including safety glasses and gloves, to safeguard against potential hazards. Avoid wearing any jewelry on your hands, as it can introduce contaminants to the sample. Additionally, wear appropriate clothing and footwear to minimize the risk of personal injury and maintain a clean working environment.

Sampling Equipment and Tools

Hard Rock samples and Unconsolidated materials

Utilize a fit-for-purpose tool during sampling, such as a rock hammer, mini drill, or diamond saw, depending on the geological context. In the case of unconsolidated materials like tailings, opt for tools like a sandblasted shovel or a plastic scoop for effective sampling. If available, consider employing heavy machinery such as excavators or drill rigs to enhance efficiency and accuracy in sample collection. Always ensure that the chosen tools are suitable for the specific material being sampled and adhere to safety guidelines to mitigate risks during the sampling process.

Documentation and Record-Keeping

All information regarding the materials collected within the AVANTIS Project should be documented in the Excel spreadsheet named 'AVANTIS_materials_database_7_2_24' and saved to the AVANTIS team channel.

Field Notebooks and Data Sheets

A good documentation of the sampling process and geological features is an integral part of robust data collection and subsequent reporting.

Record-keeping can be done either using a field notebooks or electronic data sheets.

The minimum data documentation should include the following:

- Location (country, municipality, site)
- Sample code
- Sample type (rock, drill core, chips, tailing, pre-concentrates)

- Sample site coordinates (X, Y, Z) including datum specifications
- Date and time
- Responsible geologist/field assistant
- Sample size (>100 mm, 100-50 mm, 50-1 mm, <1 cm)

Geological features (i.e.: lithology, alteration, ore minerals, percentage of sulfides, ore type or domain, grain size, moisture content (e.g., wet, moist, dry)).

- Comments

Photographs

Document the sampling process thoroughly by capturing photographs of the sampling site and the specific rock or material being sampled. Always incorporate a scale or measuring tape in the photographs to provide a visual reference for size and proportion. This documentation serves as a crucial complement to written records, offering a comprehensive overview of the sampling context. Additionally, ensure that the photographs are clear, well-lit, and properly labelled, contributing to the integrity and transparency of the entire geological sampling procedure.

GPS Devices

In the field, the use of GPS devices is crucial for accurately documenting sample locations. Whether utilizing handheld GPS units or Differential GPS systems for enhanced precision, it is essential to adhere to a standardized procedure to minimize errors.

Sampling Procedures

Sample strategy

The sample strategy depends on the sample matrix, such as solid rock, rock chips, or tailings. A minimum sample size of 100 kilograms is mandated by the pilot plants (WP2; UOULU and Mintec). The crucial aspect in AVANTIS sampling is to obtain a bulk sample that is representative of the complexity of the ore being studied.

In the optimal case, the sample material represents homogeneous ore, which corresponds to a large spatially constrained ore domain/type. However, sampling may also consist of a composite sample representing multiple ore lenses from a defined area to extract the necessary amount of material.

Each sample type includes specific aspects that should be considered before sampling.

Sample Type 1 (drill core/rock chips):

Drill core samples enable the collection of material from the sub-surface, specifically targeting distinct sections of the mine or ore domain. To obtain a sufficient amount of sample material, it may be necessary to sample material from multiple drill holes, a process that can be time-consuming. The sample strategy is contingent on factors such as the diameter of the drill core, deposit type, and the style of mineralization.

Previously sampled drill core samples stored as rock chips (“laboratory coarse rejects”) may also be considered. Typically, the coarse rejects are less than 6 mm (>70% passing 6mm).

Sample Type 2 (surface hard rock):

Outcropping surface ore can be sampled using tools such as a rock hammer, a mini drill, or a diamond saw. The rock hammer (grab sample) and mini drill provide point samples, which can be taken in a closely spaced grid. In contrast, surface channel sampling with a diamond saw is a linear method that allows continuous sampling from a specific area. It is important to note the weathered surface, if oxidized, should be removed and excluded from the selected sample lot. Collect samples in plastic bags or cotton bags with proper labeling, and securely seal the bags, for instance, using a cable tie. Rock samples with sharp edges may break the plastic bags in transportation, therefore, it is recommended to rounded them with a rock hammer before bagging.

Sample Type 3 (tailings/pre-concentrates):

Tailings or pre-concentrated material consists of unconsolidated particles with a specific grain size. Sampling can be done by quickly scooping the material into sample bags, but for tailings it is recommended to assess the site before proceeding with sampling.

1. Create a section profile using a shovel to determine the moisture content. If the material is dry, continue digging until the sample becomes more saturated. Attempt to identify the boundary between the unsaturated and saturated zones, if possible, and measure the depth of this boundary.

2. Draw a profile log, documenting each layer's thickness and grain size. Include the thickness of any potential surface layer and characterize it.
3. Take a photograph with a measuring tape in the frame. Create multiple section profiles to ensure a representative view of the tailings. Compare the logs and design the sample strategy based on the results. This can involve approximately 3-5 sample locations, where the sample is a composite taken from the same depth (e.g., 0.5-1.0 m), or from one representative profile and one unit.
4. Collect samples in plastic bags with proper labeling, and securely seal the bags, for instance, using a cable tie. Avoid taking a wet sample which increase the transportation costs.

Sample Handling and Preservation

Labeling and Identification

All samples are labeled with a unique sample code. This is done to avoid confusion and to trace the sample back to the original source material. This is because in the AVANTIS Project, the aim is to document the entire chain and capture information with a focus on the project's goals in terms of best practices.

It is advisable to use a permanent marker to mark the sample bag. Additionally, for added safety measures, insert a waterproofed sample ticket inside the bag or use for example a mini-grip plastic bag for the tickets.

QC samples are randomly placed within the sample stream before dispatching the sample batches to the laboratory. When utilizing a commercial accredited laboratory, ensuring the anonymity of QC samples is crucial. One approach is to use a blind number code, inserted into the sample bag sent to the laboratory. This necessitates meticulous documentation in your sample template to establish a clear link between the project code and the newly generated blind code.

The placement of QC samples should encompass the entire batch, ensuring uniform treatment of all materials. Depending on batch size, distribute approximately equal amounts of CRMs and blanks throughout the sample stream. Field duplicate samples can be collected on-site or split at the sampling facility before submission to the laboratory. When using laboratory subsamples such as coarse and pulp duplicate samples (see Definitions and Terminology) communication is essential and should be included in the sample sheet provided to the laboratory (e.g., 20240001CD, 20240001PD).

Packaging and Transportation

Collect samples in plastic bags with proper labeling, and securely seal the bags, for instance, using a cable tie. Rock samples with sharp edges may break the plastic bags in transportation, therefore, it is recommended to rounded them with a rock hammer before bagging. Avoid taking a wet sample which increase the transportation costs.

Samples should be securely packed for transportation to minimize the risk of contamination. Ensure that all relevant information is clearly indicated on the waybill issued by the carrier. The waybill outlines details and provides instructions related to the shipment of the cargo consignment.

Chain of Custody

Adhere to a robust Chain of Custody process throughout the collection, handling, transfer, and storage of geological samples, starting from the moment of collection until the final analysis. This process includes systematic record-keeping of every individual who has had possession or control of the samples. Once the geologist has ensured the quality of the fieldwork and proper submission of samples to the laboratory, the laboratory takes on the responsibilities of quality control and quality assurance.

All of this also needs to be documented in the 'material database' Excel spreadsheet used in the AVANTIS project. This will be stored on the AVANTIS project's TEAMS platform.

Sample Preparation and Analytical Methods

Samples can be prepared and analyzed either by a commercial accredited laboratory (e.g., ALS, SGS, Actlabs) or in-house laboratory. The following sample preparations and analytical packages are recommended to be used. However, the sample preparation has to measure up various sample materials and univocal sample preparation is difficult to establish. Please note that if an in-house laboratory is used, sample preparation can be purchased from a commercial provider.

Sample Preparation Procedures

The samples are prepared in the laboratory using the same or similar packages:

Hard rock samples

- Step 1 a) (for sample material is larger than ?? mm): Coarse crushing of rock chips or drill core/chip samples to more than 70 % finer than 6 mm, if necessary.
- Step 1 b): Crush <6 mm sample material to more than 70 % finer than 2 mm
- Step 3: Split the sample material using a riffle spliter to one 250 g duplicate (coarse duplicate)
- Step 4: Pulverise the coarse duplicate, by using e.g. disc mill, to more than 85% finer than 75 microns.

Tailing samples

- For fine-grained tailings, drying ovens are recommended to remove excessive moisture. Do not exceed 50 °C to avoid the decomposition of sulfides and other minerals sensitive to heat.
- Measure particle size and follow procedures for hard rock samples according to particle size.

Analytical Methods and Codes

The samples are analyzed in the laboratory using the same or similar packages (See ALS procedure 2024):

- XRF for oxide ores by ME_XRF21n+LOI
- Trace elements and REE by ME-MS61L^{tm}+MS61L-REE^{tm}
- Ferrous iron titration by FE-VOL05
- Total carbon and sulphur by ME-IR08

For in-house analyses, LOI should be carried out for 1 hr at 1000 °C. Samples should be inserted in the furnace at T<300 °C.

Mineralogical analyses

Depending on the analysis technique, some samples may require preparation such as grinding, polishing, or thin sectioning considering minimizing sample disturbance for in-situ analyses.

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- Mineralogical analyses include facilities such as XRD, SEM/FE-SEM, EMPA, and MicroXRF, (LIBS, XCT, Raman, LA-ICPMS).
- Ensure the instrument is configured for in-situ analysis, which may involve calibration, and selecting appropriate measurement modes and settings.

Quality Control (QC) Measures for mineralogical samples

In-situ mineralogical analysis refers to examining and identifying minerals within their natural environment. The general protocol for conducting in-situ mineralogical analyses for AVANTIS will be as follows:

Data collection:

- Perform in-situ measurements on the samples using the chosen analytical technique(s).
- Record all relevant data, including images, spectra, peak intensities, elemental compositions, and any other observations.

Data analysis:

- Interpret the collected data to identify minerals present in the samples.
- Compare spectra or elemental compositions with reference databases or known standards to aid in mineral identification.
- Consider the geological context and any additional information available to refine mineral identification and understand the significance of findings.

Documentation, reporting, and dissemination:

- Document all findings, including sample descriptions, analytical results, interpretations, and conclusions according to the project objectives.
- Prepare reports, presentations, or publications summarizing the in-situ mineralogical analyses and their implications.
- Provide required data for related tasks within WP1 and to other work packages (WP2, WP3, WP4, WP5) within the project.

Quality control:

- Implement quality control measures to ensure the reliability and accuracy of results. This may include using standards for calibration, conducting replicate measurements, and verifying instrument performance.

Quality Control (QC) Measures for chemical analyses

The Project will insert external quality control samples (Certified Reference Materials (CRM), blanks and duplicates) into the sample stream in random positions before sending the sample batches to the laboratory. The price for QC samples are expected to be the same as for normal samples.

The batch should contain sufficient number of samples to allow the insertion of control samples (this is not always possible to achieve in small sampling campaigns). At the same time, the sample batch cannot be too large to become too difficult to manage, evaluate, or re-assay.

The QA/QC program should cover the following:

- sampling conditions in the field;
- sample preparation;
- analytical accuracy and precision; and
- correctness of the laboratory reports and transfer of the information to the database (s).

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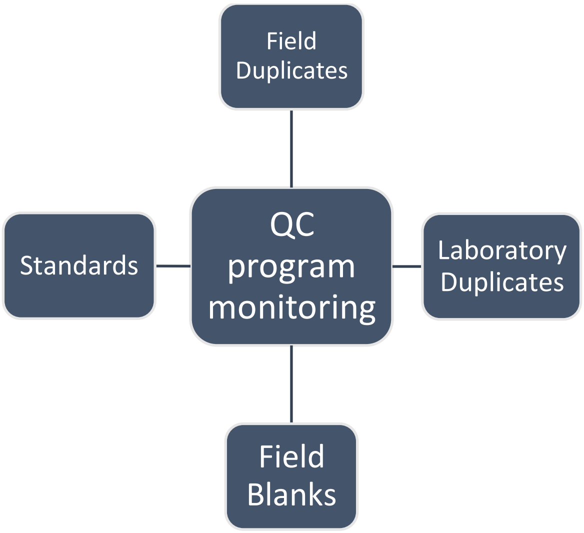
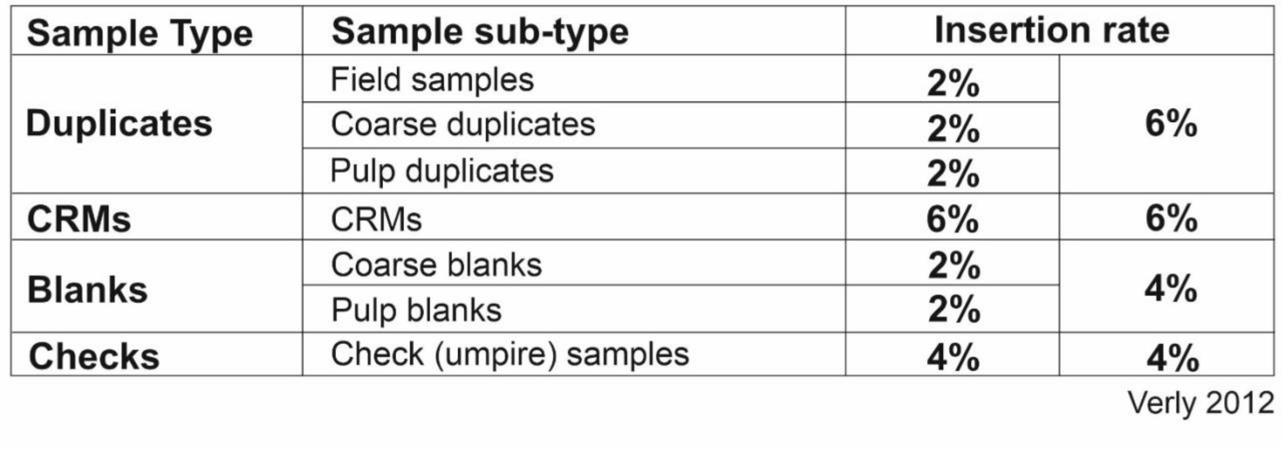


Figure 3. Quality Control Program.

Abzolov (2008) recommends insertion of 3-5% of the standard/s in each sample batch to identify bias. QC program should always be planned according to the style of mineralization and deposit type under investigation.

Table 2. Recommended insertion rates for QC samples (Verly 2012).



Sample Type	Sample sub-type	Insertion rate	
Duplicates	Field samples	**2%**	**6%**
	Coarse duplicates	**2%**	
	Pulp duplicates	**2%**	
CRMs	CRMs	**6%**	**6%**
Blanks	Coarse blanks	**2%**	**4%**
	Pulp blanks	**2%**	
Checks	Check (umpire) samples	**4%**	**4%**

Verly 2012

Sample failure and rejection criteria

When analyzing QC results it is important to know the sample rejection criteria and mechanism for fixing rejected (failed) samples.

If any check samples fail, it indicates that the entire batch or a selected portion—such as five samples on both sides, including the control sample—should be re-assayed.

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Conduct quality control measures immediately upon receiving the final assay results. In the event of sample failure, promptly contact the laboratory's supply contact. If commercial laboratories are employed, ensure that the cost of resampling is discussed during the contractual process. If you have a valid laboratory agreement, verify the agreed-upon resampling procedures before submitting samples.

The rejection criteria are described below.

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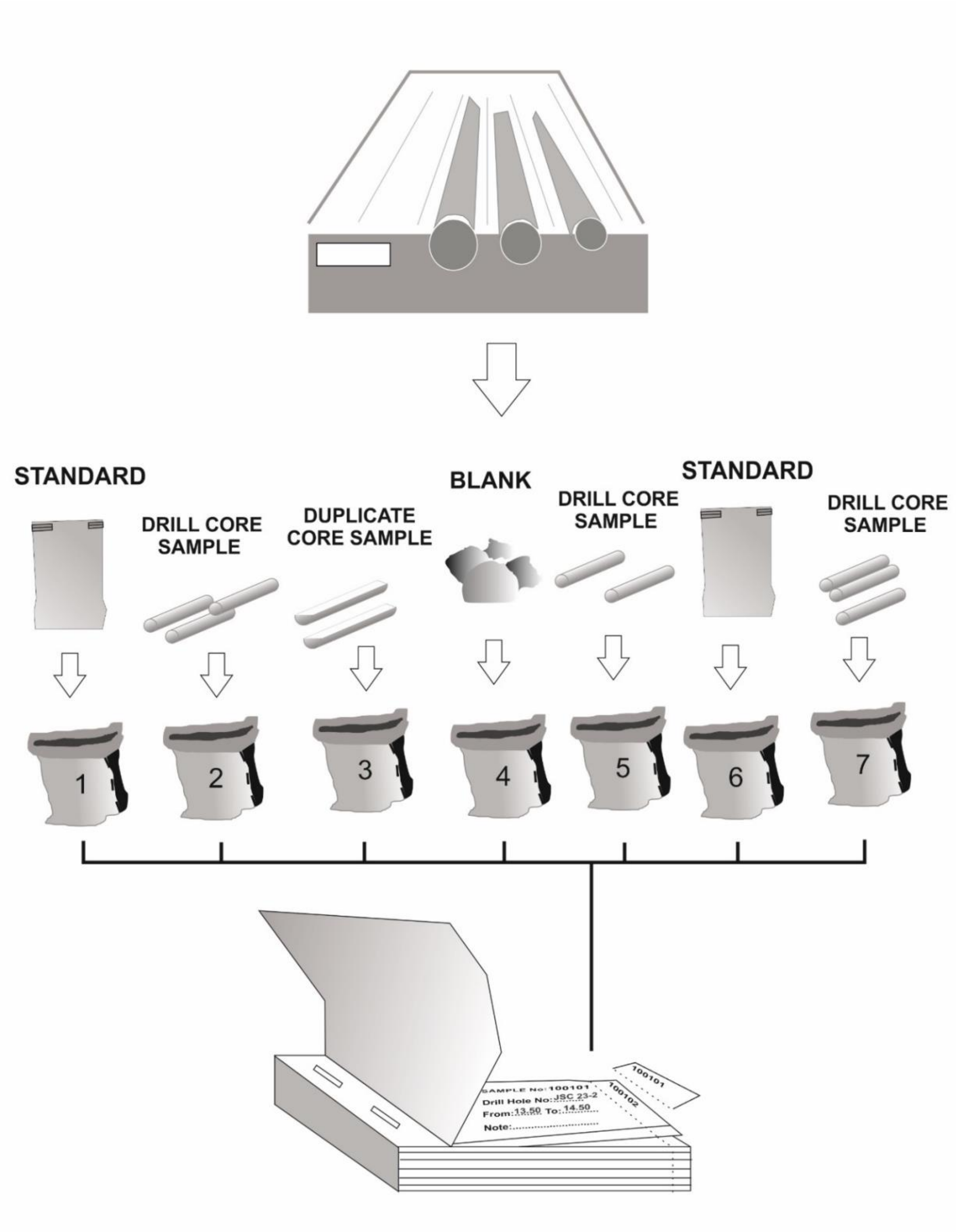

Figure 4. QC sample injection during the sample preparation.

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Certified Reference Material (CRM)

A certified reference material AMIS0567 is used (certificate: AMIS0567-Certificate.pdf) for geochemical assaying. The AMIS0567 is a matrix matched Certified Reference Material, fit for use as control sample in routine assay laboratory quality control when inserted within runs of test samples and measured in parallel to test samples.

The material originates from Vametco Mine. Vanadium mineralization in the region occurs within titaniferous magnetite-rich layers within the Rustenburg Suite in the upper zone of the Bushveld Igneous Complex. The magnetite layers are typically concordant, continuous along strike and down-dip, but can vary considerably in thickness and grade (of magnetite, vanadium and titanium). Three main seams are present – the Upper, Intermediate and Lower.

The recommended procedure for the use of this CRM as a control standard in laboratory quality control is to develop a Shewhart chart, where a mean value and corresponding 1, 2 and 3 standard deviations are derived from replicate measurements of the CRM (see appendix X (AMIS0567 Certificate, Appendix. 4) or link AMIS0567-Certificate.pdf).

Shewhart Control Chart

Accurate data is lying within two standard deviations of the mean with samples randomly distributed above and below the certified mean value. Some variation is always expected; however, we want to ensure that the returned value as well as the variability of the returned values is reasonable.

The rules concerning the interpretation of these charts are based on the confidence levels built into the 2sd and 2sd control limits.

- No data should fall outside the control limits (UCL and LCL) and if so, must be investigated. The lines at ±2sd (standard deviation) above and below the mean are known as the Upper and Lower Warning Limits (UWL and LWL). Those at ±3sd are the Upper and Lower Control Limits (UCL and LCL).
- No more than 5 % (1 in 20) of the data should fall outside the warning limits (UWL and LWL).
- Two successive data outside of the UWL and/or LWL may signify a loss of control and must be investigated.

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- More than five successive data on one side of the mean may signify a bias trend and must be investigated.
- A sudden increase in the variation of values about the mean signifies a loss of precision.

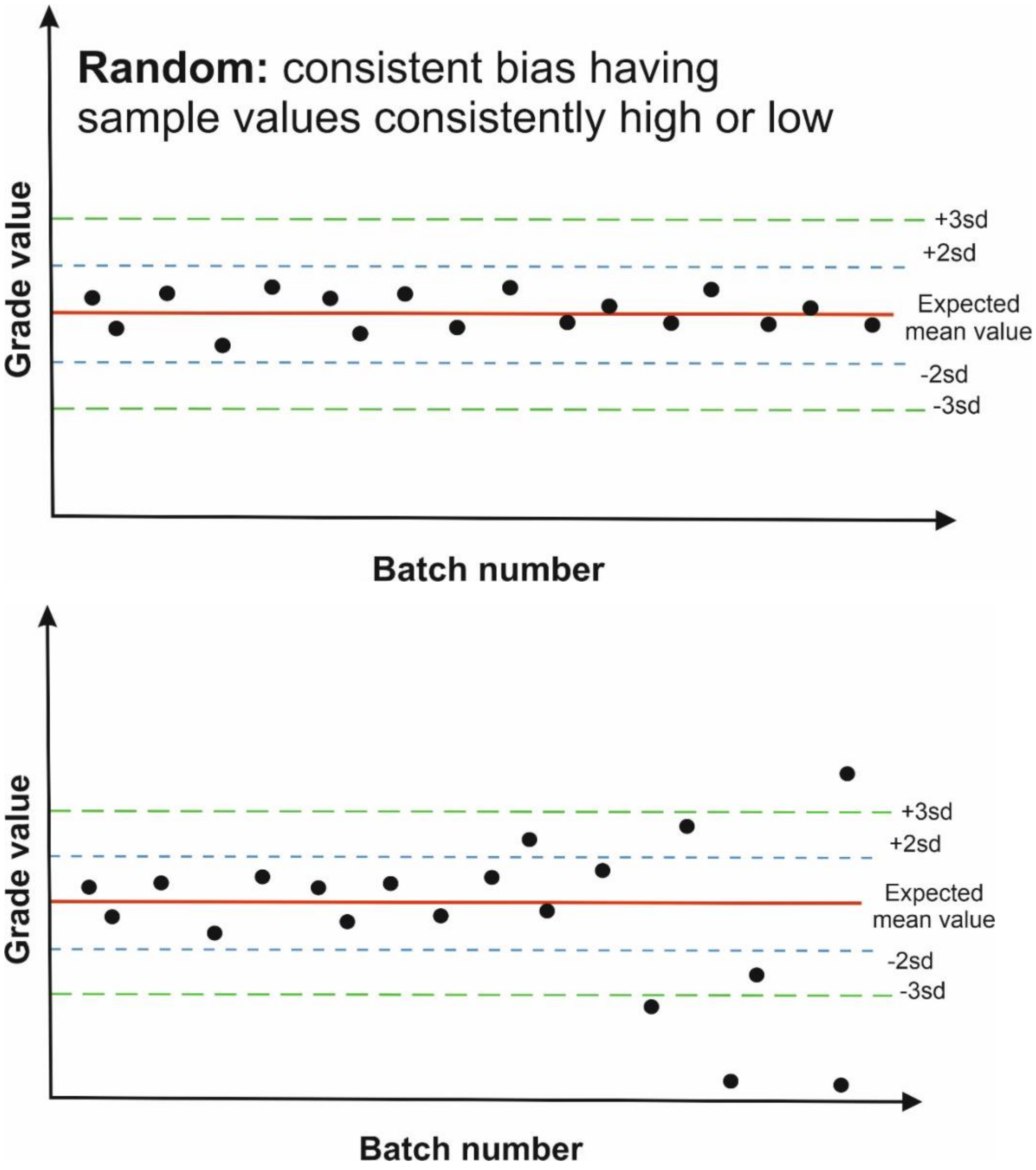

Figure 5. The standard values should plot a random and there should be no consistent bias or pattern (non-random behavior). Any discernable patterns should be a warning to interrogate the batch of data further. The two standard deviations (2s) is calculated as for example: uc x 2 = 0.23 x 2=0.46%.

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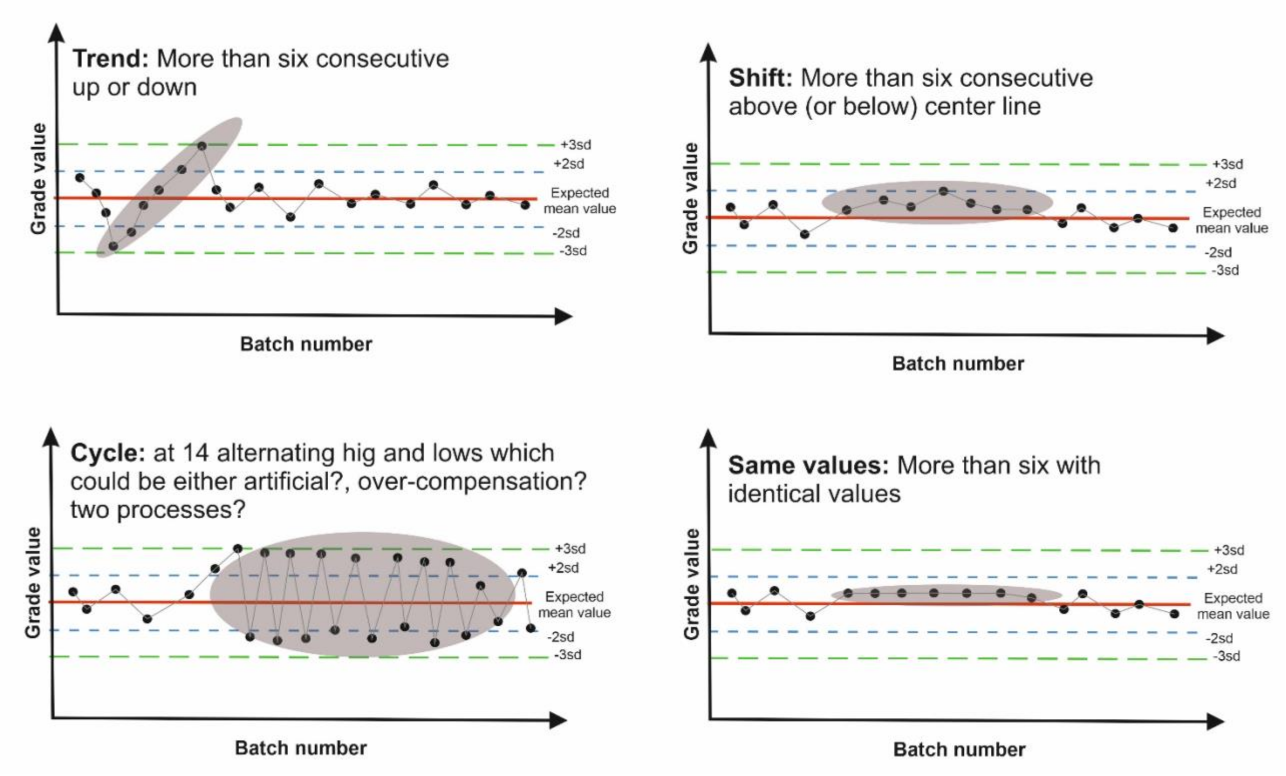

Figure 6. Random outliers indicate a potential data transcription error during sample preparation or database management. Alternatively, they may suggest random errors. Definite analytical bias indicating potentially poor calibration of instrumentation or a change in instrumentation, sampling, or preparation methodology. A rapid decrease in the magnitude of errors during the QA/QC program illustrates potential data tampering or different sample handling and data treatment. Systematic decrease (or increase) in values indicates potential drift and/or degradation of the reference material during the course of the QA/QC program.

Alternatively, if repeated assays of the CRMs are not available in a single analytical batch (which often contains only one or two CRM samples), preventing the use of the abovementioned statistical test analyses of CRMs, accuracy estimation can be achieved through the following statistical test:

$$|X - \mu| \leq 2\, \sigma_C$$

Where X is assayed value of the CRM; σC certified within-laboratory standard deviation of the CRM; µ certified mean of a given standard sample.

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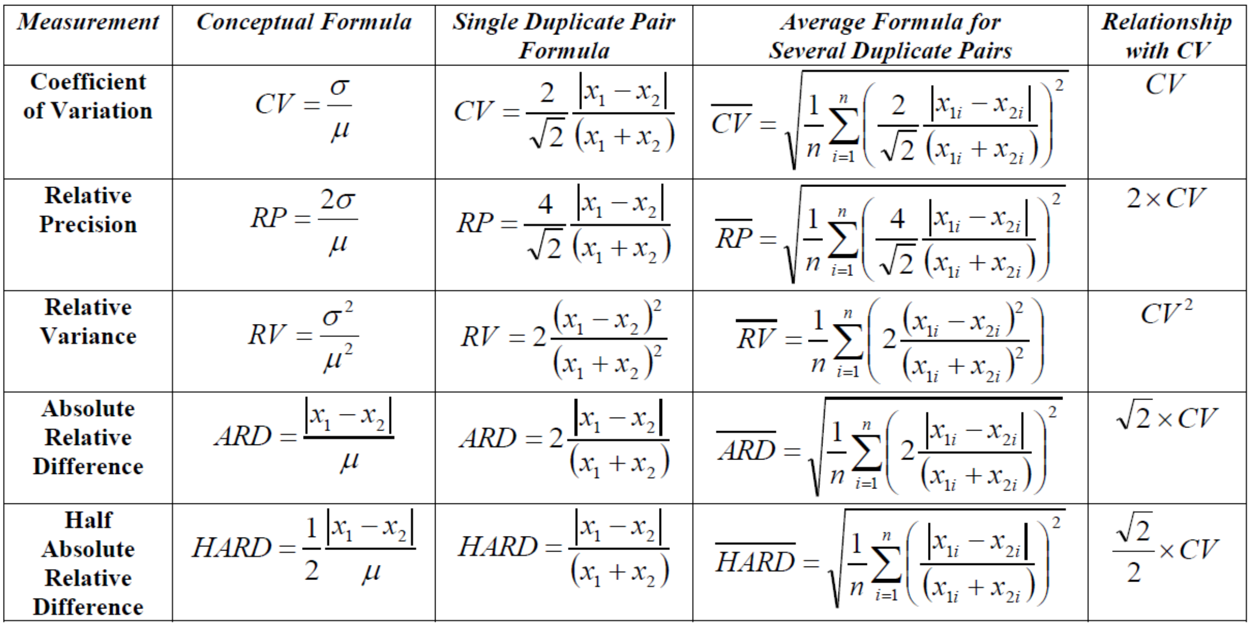
Duplicate Sampling

Duplicate sampling measures the precision which is an estimate of the reproducibility of the sampling and analytical system. Precision can be estimated using various methods (Table 2).

A common method used is the Youden plot (X-Y scatterplot) which allows visual assessment of original versus duplicate sample pairs through the closeness to the 1:1 line.

Other methods used to monitor the precision from duplicate sample pairs are for example percent relative difference method (%RD), relative standard deviation (RSD) or quantile-quantile plots.

Table 3. Various methods of estimating precision and their common factors.



Measurement	*Conceptual Formula*	*Single Duplicate Pair Formula*	*Average Formula for Several Duplicate Pairs*	*Relationship with CV*
Coefficient of Variation	$CV = \frac{\sigma}{\mu}$	$CV = \frac{2}{\sqrt{2}} \frac{\lvert x_1 - x_2 \rvert}{(x_1 + x_2)}$	$\overline{CV} = \sqrt{\frac{1}{n} \sum_{i=1}^{n} \left(\frac{2}{\sqrt{2}} \frac{\lvert x_{1i} - x_{2i} \rvert}{(x_{1i} + x_{2i})} \right)^2}$	CV
Relative Precision	$RP = \frac{2\sigma}{\mu}$	$RP = \frac{4}{\sqrt{2}} \frac{\lvert x_1 - x_2 \rvert}{(x_1 + x_2)}$	$\overline{RP} = \sqrt{\frac{1}{n} \sum_{i=1}^{n} \left(\frac{4}{\sqrt{2}} \frac{\lvert x_{1i} - x_{2i} \rvert}{(x_{1i} + x_{2i})} \right)^2}$	$2 \times CV$
Relative Variance	$RV = \frac{\sigma^2}{\mu^2}$	$RV = 2 \frac{(x_1 - x_2)^2}{(x_1 + x_2)^2}$	$\overline{RV} = \frac{1}{n} \sum_{i=1}^{n} \left(2 \frac{(x_{1i} - x_{2i})^2}{(x_{1i} + x_{2i})^2} \right)$	CV^2
Absolute Relative Difference	$ARD = \frac{\lvert x_1 - x_2 \rvert}{\mu}$	$ARD = 2 \frac{\lvert x_1 - x_2 \rvert}{(x_1 + x_2)}$	$\overline{ARD} = \sqrt{\frac{1}{n} \sum_{i=1}^{n} \left(2 \frac{\lvert x_{1i} - x_{2i} \rvert}{(x_{1i} + x_{2i})} \right)^2}$	$\sqrt{2} \times CV$
Half Absolute Relative Difference	$HARD = \frac{1}{2} \frac{\lvert x_1 - x_2 \rvert}{\mu}$	$HARD = \frac{\lvert x_1 - x_2 \rvert}{(x_1 + x_2)}$	$\overline{HARD} = \sqrt{\frac{1}{n} \sum_{i=1}^{n} \left(\frac{\lvert x_{1i} - x_{2i} \rvert}{(x_{1i} + x_{2i})} \right)^2}$	$\frac{\sqrt{2}}{2} \times CV$

*From Stanley and Lawie (E&MG, 2007)

Percent Relative Difference

- Percent relative difference (%RD) is calculated from the replicate analyses of the reference materials using:

$$\%RD = 100x \frac{\mu_i - STD_i}{STD_i}$$

where μ_i = mean value of element i in the standard over a number of analytical runs; and STD_i = 'known' or 'certified' value of i in the standard or reference material.

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In general %RD values of:

- 0–3% are considered to have excellent accuracy,
- values from 3–7% are considered to have very good accuracy;
- 7–10% have good accuracy;
- values above 10% are not accurate (Jenner 1996).

Accuracy, like precision, decreases as values approach the detection limit of an instrument, and is influenced by the element and type of material (e.g., Au has a nugget behavior and can be highly inaccurate).

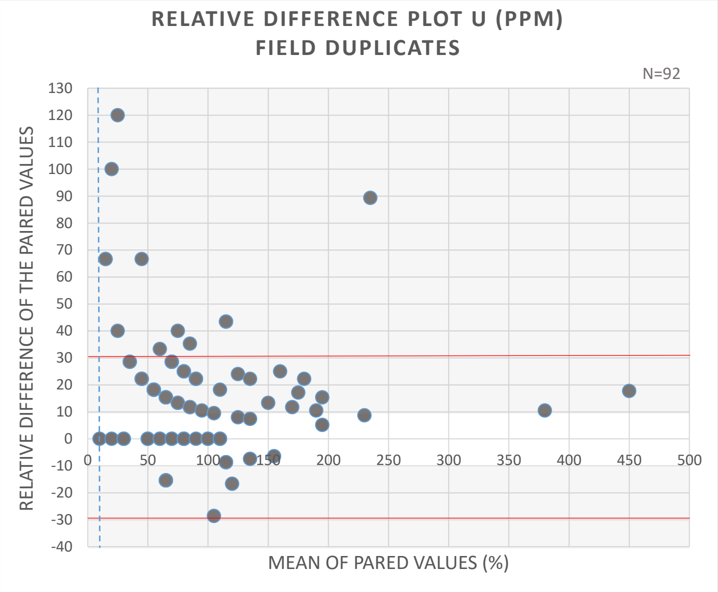

Figure 7. Example of relative difference plot.

Relative Standard Deviation (RSD)

A variation on this display is to plot the Relative Standard Deviations (RSDs) of the duplicate pairs rather than their absolute differences on the y-axis.

A Relative Standard Deviations (RSDs) of the duplicate pairs rather than their absolute differences on the y-axis

RSD is calculated as follows:

$$\%RSD_i = 100x\frac{S_i}{\mu_i}$$

where $\%RSD_i$= percent relative standard deviation for element i; S_i = standard deviation of the mean from the series of analytical runs for element i; and μ_i = mean value of element i over a series of analytical runs.

%RSD varies as the sample approaches the LOD and ULD, and is dependent on the material in question (e.g. whole rock versus nuggety gold), if an element is above the LOQ, then %RSD between

- 0 and 3% is excellent,
- between 3 and 7% is very good to good,
- 7–10% RSD is good,
- and >10% is not precise (Jenner 1996).

Notably, %RSD will be greater, and precision poorer, as one approaches and goes below the LOQ towards the LOD.

X-Y Scatterplots

- 1st step in handling duplicate data is to plot the original analysis vs the duplicate analysis.
- Look for “fliers” that may indicate sample mis-ordering or nugget effect.

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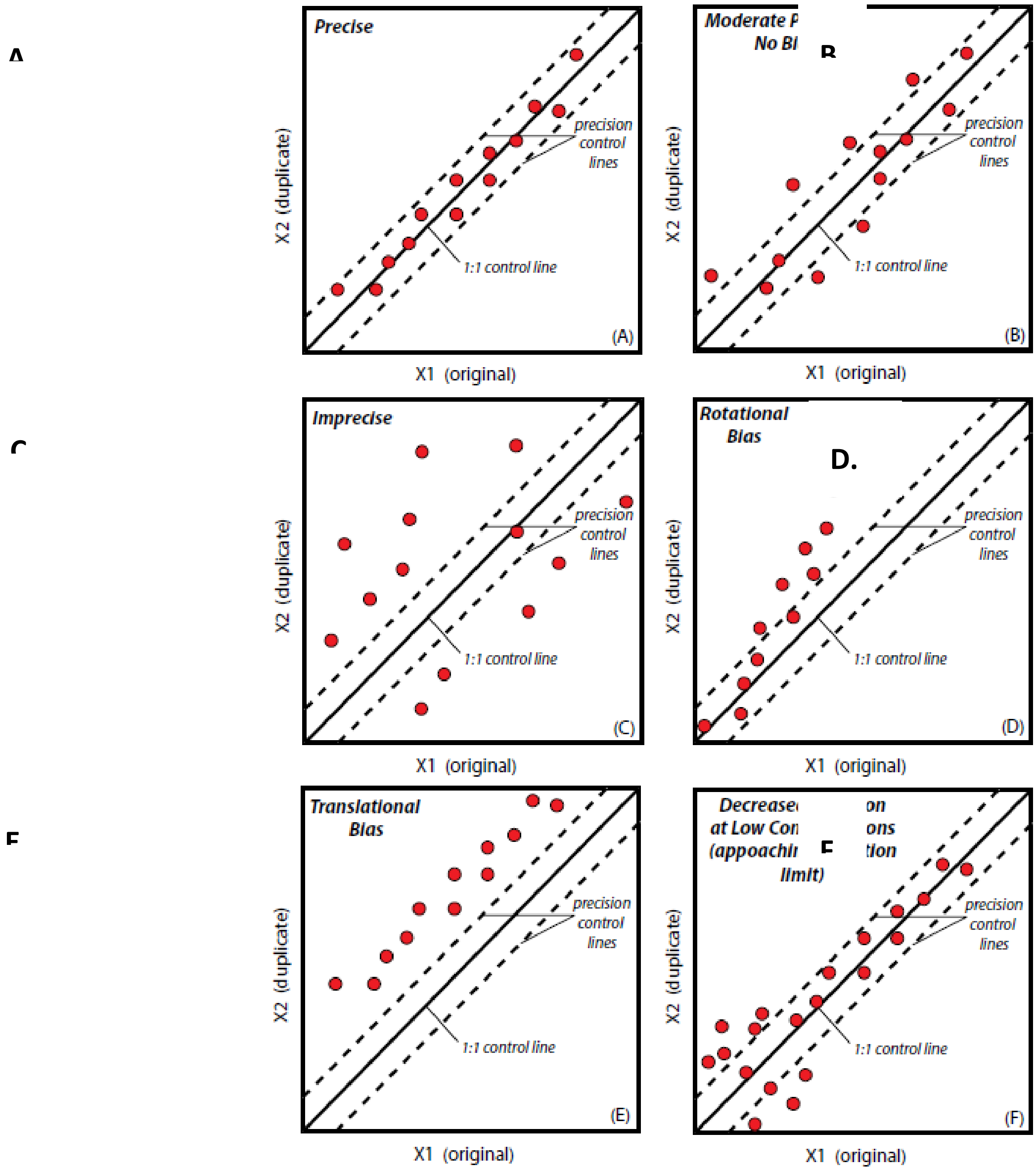

Figure 8. A) Precise data lying within the control lines. B) Moderately precise data with scatter suggesting no bias. C) Very imprecise data with a shot-gun distribution. D) Data with a rotational bias. E) Data with a translational bias (i.e., multiplier effect). F) Decreasing precision with concentration (hetereoscedastic effect) (Piercey, 2014).

Blank Sampling

Contamination can occur at any stage during the sample preparation and analytical process, including contamination due to poor cleaning of crushing and pulverizing equipment, from unclean acids during sample preparation, or memory effects on instrumentation where the instruments are not sufficiently flushed with solution between analyses.

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- Barren coarse material ("a blank") is submitted with samples for crushing and pulverizing to determine if there has been contamination or sample cross-contamination in preparation.
- The purpose is to check laboratory contamination and to verify correct handling of the samples.
- Blanks should be matrix-matched and go through the same sample preparation as original samples.
- It is preferred that blank have the same matrix and experience the same sample preparation as original samples.
- It is preferred that blanks have the same mineralogy or at least color and result in a pulp with the same characteristics as the main samples so it is not obvious to the laboratory.

If in-house blanks are utilized, it is advisable to conduct geochemical assaying to confirm the absence of any grades of interest. If time constraints prohibit this, select blank material from a location sufficiently distant from the ore zone.

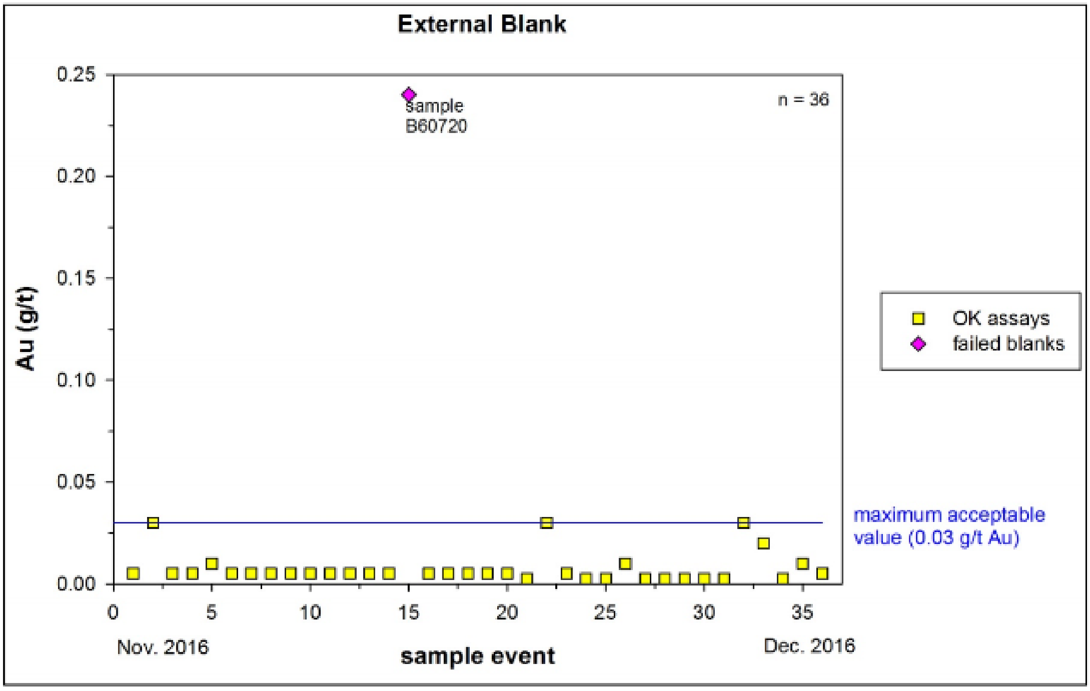

Figure 9. An example of blank diagram (Lingman Lake 2017).

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Data Analysis and Reporting

The sampling process and data analysis of QC results should be reported transparently and distributed to all project partners.

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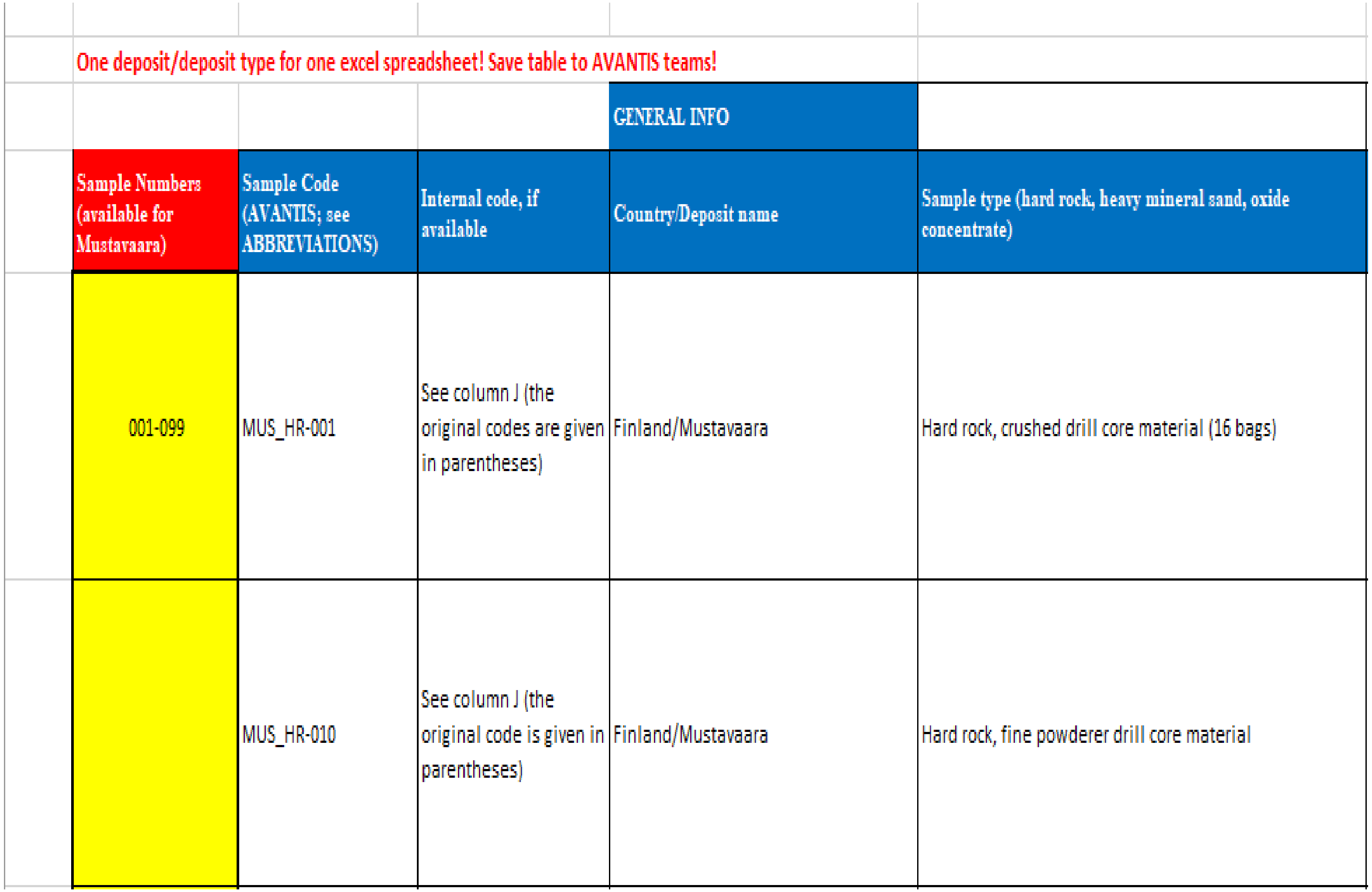
Appendix III. AVANTiS Material database



One deposit/deposit type for one excel spreadsheet! Save table to AVANTIS teams!

			GENERAL INFO	
Sample Numbers (available for Mustavaara)	Sample Code (AVANTIS; see ABBREVIATIONS)	Internal code, if available	Country/Deposit name	Sample type (hard rock, heavy mineral sand, oxide concentrate)
001-099	MUS_HR-001	See column J (the original codes are given in parentheses)	Finland/Mustavaara	Hard rock, crushed drill core material (16 bags)
	MUS_HR-010	See column J (the original code is given in parentheses)	Finland/Mustavaara	Hard rock, fine powderer drill core material

Link: AVANTiS teams (AVANTIS_Material_database_01_03_24)

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Appendix IV Guideline for AVANTiS Material DATABASE

GUIDELINES FOR AVANTiS MATERIAL DATABASE

(EXCEL SPREADSHEET)

Jukka Konnunaho

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Contents

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INTRODUCTION

Within the AVANTiS project it is crucial to keep record for the project’s sample flow and associated data, such as assays and thin sections. In accordance with this requirements, the AVANTiS project has tailored an excel spreadsheet for this purpose (available at:

Avantis_materials_database_01_03_24.xlsx or AVANTiS teams path: (General\Materials)). Copy the primary database* to your computer and fill it during the sampling process. Afterwards, save the database into a suitable folder into the AVANTiS teams. Indicate the database location to your copartners and the sample receiver.

The database has mainly been designed for WP1 purposes but is also adaptable to other WPs. This document serves as a short guideline for the database. See also AVANTiS SOP document!

Use one database for one deposit/deposit type!
The database row 5 and 6 as an example (Mustavaara), E7-E21 reminder for countries and deposits *Delete unnecessary numbers (i.e., number inside of yellow cells). They are only reminders!

AVANTiS SAMPLE CODE SYSTEM

The key element of the database is the individual sample code assigned to each sample, which is established in the AVANTiS project. For each deposit, WP1 has decided to allocate a range of **99 sample number** (the number range can be found on the first page of the database (see row B at AVANTIS_SAMPLE_SHEET)).

Another crucial element is the **individual code assigned to each deposit / deposit type** (the code can be found on the fourth sub-page of database (ABBREVIATIONS)).

Together, these elements constitute the individual sample code for each sample, such as the first sample of Mustavaara and Tellnes deposit:

MUS_HR-001 = MUSTAVAARA HARD ROCK-001

TEL_HR-400 = TELLNES HARD ROCK-400

It is crucial to assign an individual sample code to each sample in the project. WP1 will use an accepted numbering system (99/deposit or deposit type). **Other WPs may also utilize these numbers, but they must ensure availability of numbers from the database**. This means that every user **update coding** during sampling.

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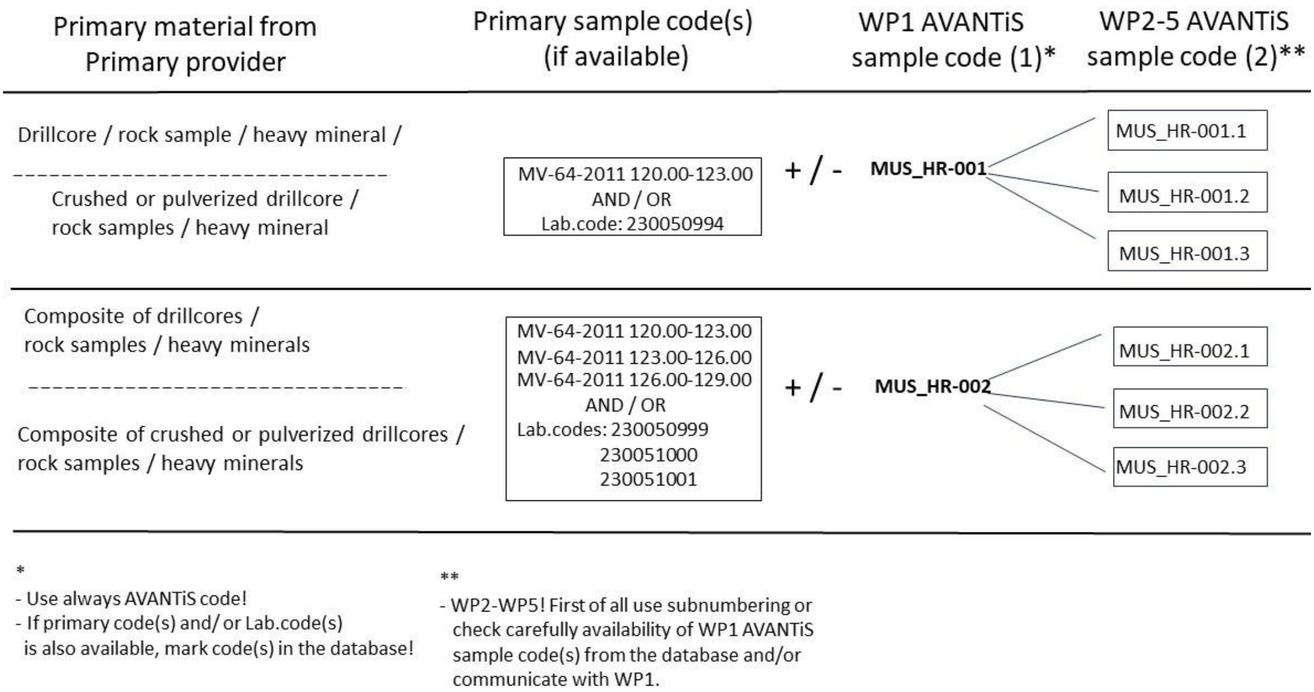
Additionally, **other WPs may use sub-numbers** such as MUS_HR-001.1, 001.2 or TEL_HR-400.1, 400.2. This numbering maintains a link to the original samples, for example, in the case of processing tests from primary sample MUS_HR-001 or TEL_HR-400. However, we would like to highlight good communication with WP's related to numbering and filling of database on time. See also figure 1 for sampling and numbering.

Thin section(s), historical (if available) or new, should be listed in the database (RELATED THIN SECTION). Use the laboratory's internal code for thin sections. **If you want to use AVANTiS code, check the availability of codes from the database.**



*
- Use always AVANTiS code!
- If primary code(s) and/ or Lab.code(s) is also available, mark code(s) in the database!

**
- WP2-WP5! First of all use subnumbering or check carefully availability of WP1 AVANTiS sample code(s) from the database and/or communicate with WP1.

Figure 1. AVANTiS sample flow chart from primary material from primary provider (Mining company) to WP1 and various purposes of WP2-WP5.

DATABASE STRUCTURE

The database consists of four sub-pages: 1) AVANTIS_SAMPLE_SHEET, 2) RELATED ASSAYS, 3) RELATED THIN SECTIONS and 4) ABBREVIATIONS. **Sub-page 1** is primarily designed for sample numbering and coding, information related to samples, delivery details, sampling process and the quantity of delivered sample material. **Sub-page 2** summarizes historical and new assay data related to the described sample material on sub-page 1 or new sample material. **Sub-page 3** summarizes information related to thin sections and **sub-page 4** provides general information related to abbreviations used.

There is **no strict guide on how to fill the database cells and columns, but sample numbering and sample code are mandatory** and accepted by AVANTiS. However, all essential information is better than nothing.

SUB-PAGE – AVANTIS_SAMPLE_SHEET

The **first sub-page (AVANTIS_SAMPLE_SHEET) includes 36 columns** (B4- AK4) with color symbols: red (3 column), blue (7 column), olivine green (14 column), dark green (7 column), pink (4 column), and blue (1 column). The list below summarizes their purpose:

Column B4 in red and yellow: The sample number range for the deposit/deposit type. When you begin filling out the database, delete unnecessary numbers (i.e., numbers inside the yellow cells). They are only reminders!

Column C4 in red: Sample code, which is an abbreviation of the deposit/deposit type + running number See chapter 2 above. **Ensure that the same code is not used twice!**

Column D4 in red: Fill this cell if there is an internal code and/or historical code available or provide additional information related to the sample code.

Column E4 in blue: Country and deposit name of the sample material.

Column F4 in blue: The sample type (hard rock, heavy mineral sand, concentrate…).

Column G4 in blue: Description of sample material (name of ore layer, ore type/texture, fraction size of processed sample…).

Column H4 in blue: Specify sample source and type (drill core, hand sample, concentrate…). Classify sample source if possible (e.g., in the case of composite sample, list all drill core codes).

Column I4 in blue: Composite sample (yes/no).

Column J4 in blue: If sample source is a drill core, mark drill core code and interval (from-to) here. In the case of composite sample from separate drill core, all drill cores + intervals have to be listed here.

Column K4 in blue: Sample location (coordinates) and coordinate system. In the case of composite sample from separate drill cores, list all drill cores here. Replace the coordinate system (Euref FIN 35-TM on heading), if a different coordinate system is used.

****Column L4 in olivine green:*** Sample material primary provider here (e.g., mining company).

****Column M4 in olivine green:*** Shipping date from primary provider to secondary provider 1.

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Column N4 in olivine green: Receiving date (secondary provider 1).

Column O4 in olivine green: Contact person info (email, phone number) of person responsible for shipping (primary provider).

Column P4 in olivine green: Information of secondary provider 1 (organization, city, project, WP, Task).

Column Q4 in olivine green: Shipping date from secondary provider 1 to secondary provider 2.

Column R4 in olivine green: Receiving date when secondary provider 2 has got shipment.

Column S4 in olivine green: Contact person info (email, phone number) of person responsible for shipping (provider 1).

Column T4 in olivine green: Information of secondary provider 2 (organization, city, project, WP, Task).

Column U4 in olivine green: Shipping date from secondary provider 2 to secondary provider 3.

Column V4 in olivine green: Receiving date when secondary provider 3 has got shipment.

Column W4 in olivine green: Contact person info (email, phone number) of person responsible for shipping (provider 2).

***Based on L4-W4, material flow covers 3 shipping steps (primary provider -> secondary provide 1 -> secondary provider 2 -> secondary provider 3)**

Column X4 in olivine green: This is essential for WP3 sampling, for example. They will process WP1 primary material. Provide the code and refer to other cells in the the database.

Column Y4 in olivine green: Material required by WP, Task and anyone else?

Column Z4 in dark green: Where in process was sample taken and how was material produced? How was sampling done; representative for? Etc.

Column AA4 in dark green: State of material (dry/wet); crushed to 1 cm; dried? Dried at which temperature? Washed? Sieved?

Column AB4 in dark green: Risk related to sample materials?

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Column AC4 in dark green: Photo(s) of sample material, sampling etc. **Please, save photos into the AVANTIS teams (give path)?**

Column AD4 in dark green: Sampling made by?

Column AE4 in dark green: Characterization of sample available digitally in AVANTIS teams? Indicate, if historical assays are available and saved into the AVANTIS teams (give path).

Column AF4 in dark green: Other relevant info.

Column AG4-AJ4 in pink: Quantity of sample material/co-partner?

Column AK4 in blue: Total weight of material.

SUB-PAGE – RELATED ASSAY

The **second sub-page (RELATED ASSAYS) includes 17 columns** (B6- R6) with color symbols: yellow (17 column). The list below summarizes their purpose:

Column B6: Sample code of AVANTiS. See chapter 2 above and figure 1.

Column C6: Blind sample code for new assays, if available.

Column D6: Sample code historical, if available.

Column E6: Indicate if historical assay data available for AVANTiS project.

Column F6: Indicate if this is a new assay sample for AVANTiS project.

Column G6: Who has ordered the historical samples (company name and person, if available)

Column H6: Specify the path for assay data within the AVANTiS teams. If you indicate that historical data is available, it must be saved into AVANTiS teams!

Column I6: Number of samples? Composite sample can be one sample, but it consists of several individual samples!

Column J6: Sample type (hard rock, heavy mineral sand, concentrate…) and additional info (crushed drill core or pulverized drill core…).

Column K6: Sample source. If sample source is drill core, mark drill core code and interval (fromto) here.

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Column L6: Assay type (whole rock etc.).

Column M6: Describe assay method if possible (leaching, fusion, analyze technique) or describe purpose of method (REE, XRF…).

Column N6: Assay year (historical if available and new for AVANTiS).

Column O6: Laboratory name (historical if available and new for AVANTiS).

Column P6: Method code (historical if available and new for AVANTiS).

Column Q6: QA/QC utilization in sampling and assay process (historical if available and new for AVANTiS).

Column R6: All other information related to assays.

SUB-PAGE – RELATED THIN SECTIONS

The second sub-page (RELATED ASSAYS) includes 17 columns (B5- S5) with color symbols: red (17 column). The list below summarizes their purpose:
Column B5: New or historical thin section (AVANTiS new / Historical). **You can separate all available thin section to own rows!**

Column C5: If thin sections are available for AVANTiS purposes (Yes/No).

Column D5: Who has ordered thin sections (Name or Not known).

Column E5: Source of thin section. Give as detailed information as possible.

Column F5: Number of available thin section(s).

Column G5: Mark AVANTiS (laboratory code) or historical code here.

Column H5: Thin section type.

Column I5: Manufacturer

Column J5: Year of manufacture

Column K5: If description has been made, it has to save in the AVANTiS teams (give path).

Column L5: List of assayed minerals **if assays are available (see column M5)**!

Column M5: Assay results available for AVANTiS purposes (Yes/No).

Column N6: Assay method and machine type.

Column O6: Assay year.

Column P6: Laboratory name and country.

Column Q6: Location of assay data (path).

Column R6: Other relevant info.

SUB-PAGE ABBREVIATIONS

See chapter 2 and 3 above.

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Appendix V Certified Reference Sample data

(https://amis.co.za/wp-content/uploads/AMIS0567-Certificate.pdf)

AMIS_Documents **Revision No: 001**

Revision Date:30.11.2017

Doc: ADOC_074
Originator: Quality Specialist
Approver: Managing Director
Issued By: Quality Specialist

Certificate

AMIS0567

Certified Reference Material

Vanadium, Lower Seam, Bushveld Complex, South Africa

Certificate of Analysis

AMIS

(A Division of Torre Analytical Services (Pty) Limited)
(Reg. No. 1989/000201/07)
A: 11 Avalon Road, West Lake View Ext 11, Modderfontein, South Africa
P: PO Box 856, Isando, 1600, Gauteng, South Africa
T: +27 (0) 11 923-0800
W: www.amis.co.za

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Appendix VI In situ mineralogical analysis protocol

In-situ mineralogical study procedure

10.5.2024

In-situ mineralogical analysis refers to examining and identifying minerals within their natural environment. The general protocol for conducting in-situ mineralogical analyses for AVANTIS will be as follows:

1. **Sample collection and labeling:**
    a. As instructed in the SOP document

2. **Sample preparation:**
    a. Depending on the analysis technique, some samples may require preparation such as grinding, polishing, or thin sectioning considering minimizing sample disturbance for in-situ analyses.

3. **Instruments and instrument setups:**
    a. Mineralogical analyses include facilities such as XRD, SEM/FE-SEM, EMPA, and MicroXRF, (LIBS, XCT, Raman, LA-ICPMS).
    b. Ensure the instrument is configured for in-situ analysis, which may involve calibration, and selecting appropriate measurement modes and settings.

4. **Data collection:**
    a. Perform in-situ measurements on the samples using the chosen analytical technique(s).
    b. Record all relevant data, including images, spectra, peak intensities, elemental compositions, and any other observations.

5. **Data analysis:**
    a. Interpret the collected data to identify minerals present in the samples.
    b. Compare spectra or elemental compositions with reference databases or known standards to aid in mineral identification.
    c. Consider the geological context and any additional information available to refine mineral identification and understand the significance of findings.

6. **Documentation, reporting, and dissemination:**
    a. Document all findings, including sample descriptions, analytical results, interpretations, and conclusions according to the project objectives.
    b. Prepare reports, presentations, or publications summarizing the in-situ mineralogical analyses and their implications.
    c. Provide required data for related tasks within WP1 and to other work packages (WP2, WP3, WP4, WP5) within the project.

7. **Quality control:**
    a. Implement quality control measures to ensure the reliability and accuracy of results. This may include using standards for calibration, conducting replicate measurements, and verifying instrument performance.

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Appendix VII Electron microprobe analyses data

Table 1. EPMA results of validation samples 2024000175 and 2024000176 by the laboratories GTK, NTNU, and AGH.

EPMA average results utilising different settings GTK with 20kV accelerating voltage and 20nA beam current. V measured on Kβ-line.

beam size (if other than 1 my)																		***20my***	***20my***			
mineral	***ilmenite***								***magnetite***										***titanite***			
background correction*	***MAN****						***off-peak***		***MAN****						***off-peak***		***off-peak***		***MAN****			***off-peak***
No. of analyses	***7***	***6***	***6***	***6***	***6***	***6***	***3***	***3***	***5***	***5***	***5***	***5***	***5***	***5***	***3***	***3***	***2***	***2***	***3***	***4***	***4***	***2***
	175 site1	***175 site2***	***175 site3***	***176 site4***	***176 site5***	***176 site6***	***175 site1b***	***176 site4b***	***175 site1***	***175 site2***	***175 site3***	***176 site4***	***176 site5***	***176 site6***	***175 site1b***	***176 site4b***	***175 site1c***	***176 site4c***	***175 site1***	***175 site2***	***175 site3***	***175 site1b***
Nb2O5 GTK	0.01	0.02	0.00	0.00	0.01	0.00	0.01	0.01	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.01	0.02
Ta2O5 GTK	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.02	0.00	0.00	0.00	0.01
WO3 GTK	0.00	0.00	0.00	0.00	0.00	0.00	0.02	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.02	0.02	0.01	0.00	0.00	0.00	0.03
SiO2 GTK	0.00	0.00	0.00	0.03	0.01	0.00	0.01	0.01	0.47	0.42	0.16	0.02	0.01	0.02	0.25	0.12	1.59	0.03	29.79	30.34	30.55	30.20
TiO2 GTK	50.19	50.33	50.27	50.73	50.36	50.04	49.77	50.69	0.68	1.10	1.13	2.85	7.38	5.51	0.78	1.35	2.30	10.07	35.76	37.86	37.87	36.39
SnO2 GTK	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.01
ZnO GTK	0.00	0.01	0.00	0.00	0.00	0.00	0.03	0.01	0.00	0.00	0.00	0.00	0.00	0.00	0.01	0.01	0.02	0.01	0.00	0.00	0.00	0.01
Al2O3 GTK	0.00	0.00	0.00	0.00	0.01	0.00	0.05	0.08	0.12	0.02	0.04	0.00	0.02	0.00	0.14	0.12	0.45	0.06	2.31	1.04	1.12	1.61
V2O5[#] GTK	0.27	0.32	0.33	0.29	0.45	0.47	0.30	0.35	1.39	1.60	1.63	1.96	1.86	1.89	1.40	1.97	1.27	1.73	0.54	0.77	0.66	0.41
Cr2O3 GTK	0.00	0.00	0.00	0.01	0.01	0.01	0.03	0.00	0.02	0.03	0.03	0.08	0.07	0.08	0.03	0.08	0.03	0.08	0.00	0.01	0.01	0.00
FeO GTK	46.57	46.53	46.50	46.72	47.28	47.14	46.52	46.55	88.91	88.66	89.16	87.71	83.65	86.05	88.95	88.56	84.84	80.83	3.38	1.43	1.70	1.94
MnO GTK	1.96	1.91	1.93	1.48	1.33	1.36	1.90	1.49	0.02	0.02	0.04	0.08	0.22	0.14	0.01	0.03	0.06	0.28	0.06	0.03	0.04	0.03
MgO GTK	0.12	0.13	0.14	0.00	0.02	0.02	0.13	0.00	0.11	0.03	0.07	0.04	0.03	0.04	0.08	0.06	0.31	0.01	1.17	0.06	0.09	0.33
CaO GTK	0.09	0.05	0.11	0.03	0.01	0.02	0.03	0.02	0.28	0.39	0.19	0.01	0.00	0.04	0.36	0.01	0.96	0.00	24.78	26.63	26.36	25.06
TOTAL GTK	99.21	99.29	99.28	99.28	99.49	99.06	98.79	99.22	91.99	92.27	92.44	92.73	93.25	93.78	92.01	92.34	91.86	93.12	97.79	98.18	98.41	96.04

***** *Background correction calculated based on mean atomic number (MAN) correction method using Probefor EPMA-software.***

[#] ***V measured on Kβ-line.***

EPMA average results from NTNU with 20kV accelerating voltage and 20nA beam current. V measured on Kβ-line.

beam size (if other than 1 my)																		***20my***	***20my***			
mineral	***ilmenite***								***magnetite***										***titanite***			
background correction*	***MAN***						***off-peak***		***MAN***						***off-peak***		***off-peak***		***MAN***			***off-peak***
No. of analyses	***10***	***6***	***6***	***9***	***6***	***6***			***10***	***5***	***5***	***10***	***5***	***6***					***6***	***4***	***4***	
	175 site1	***175 site2***	***175 site3***	***176 site4***	***176 site5***	***176 site6***	***175 site1b***	***176 site4b***	***175 site1***	***175 site2***	***175 site3***	***176 site4***	***176 site5***	***176 site6***	***175 site1b***	***176 site4b***	***175 site1c***	***176 site4c***	***175 site1***	***175 site2***	***175 site3***	***175 site1b***
Nb2O5 NTNU	0.00	0.00	0.00	0.00	0.00	0.00			0.00	0.00	0.00	0.00	0.00	0.00					0.00	0.00	0.00	
Ta2O5 NTNU	0.02	0.01	0.02	0.01	0.01	0.02			0.00	0.01	0.01	0.01	0.01	0.02					0.02	0.00	0.01	
WO3 NTNU	0.01	0.02	0.01	0.01	0.00	0.00			0.01	0.01	0.00	0.01	0.01	0.01					0.01	0.01	0.01	
SiO2 NTNU	0.09	0.09	0.08	0.08	0.07	0.08			0.14	0.23	0.09	0.10	0.10	0.10					31.15	31.16	30.81	
TiO2 NTNU	48.57	48.65	48.41	48.89	48.93	48.23			0.42	0.90	0.66	1.80	7.80	1.05					35.36	35.70	36.75	
SnO2 NTNU	0.01	0.00	0.00	0.00	0.01	0.00			0.00	0.00	0.00	0.00	0.00	0.00					0.01	0.00	0.00	
ZnO NTNU	0.04	0.04	0.04	0.01	0.01	0.01			0.00	0.01	0.00	0.00	0.01	0.01					0.00	0.00	0.01	
Al2O3 NTNU	0.02	0.01	0.01	0.02	0.02	0.01			0.07	0.13	0.09	0.06	0.10	0.03					1.51	1.31	1.04	
V2O5[#] NTNU	0.29	0.35	0.37	0.35	0.49	0.45			1.43	1.66	1.65	2.06	1.91	2.00					0.59	0.81	0.67	
Cr2O3 NTNU	0.01	0.01	0.01	0.01	0.02	0.02			0.04	0.04	0.04	0.09	0.08	0.10					0.02	0.01	0.01	
FeO NTNU	48.89	48.28	48.93	49.02	49.32	49.23			95.73	94.40	95.94	93.82	87.56	95.02					2.15	1.69	2.16	
MnO NTNU	2.00	1.97	1.97	1.49	1.34	1.38			0.02	0.04	0.03	0.06	0.25	0.03					0.04	0.04	0.07	
MgO NTNU	0.15	0.15	0.14	0.01	0.02	0.03			0.01	0.04	0.01	0.01	0.02	0.01					0.22	0.16	0.01	
CaO NTNU	0.08	0.08	0.13	0.01	0.01	0.04			0.11	0.12	0.09	0.02	0.01	0.05					26.55	27.11	27.05	
TOTAL NTNU	100.17	99.66	100.11	99.93	100.24	99.49			97.99	97.58	98.62	98.05	97.87	98.42					97.62	98.00	98.59	

***** *Background correction in all samples from NTNU is off-peak, but the name in the table correspond to the spots measured at GTK.***

[#] ***V measured on Kβ-line.***

Table 1. EPMA results Continues.

EPMA average results from AGH with 20kV accelerating voltage and 20nA beam current. V measured on Kβ-line.

beam size (if other than 1 my)																	*20my*	*20my*				
mineral	*ilmenite*								*magnetite*										*titanite*			
*background correction**	*MAN*						*off-peak*		*MAN*						*off-peak*		*off-peak*		*MAN*			*off-peak*
No. of analyses	*7*	*6*	*6*	*6*	*6*	*6*	*3*	*3*	*5*	*5*	*5*	*5*	*5*	*5*	*3*	*3*	*2*	*2*	*6*	*4*	*4*	
	175 site1	*175 site2*	*175 site3*	*176 site4*	*176 site5*	*176 site6*	*175 site1b*	*176 site4b*	*175 site1*	*175 site2*	*175 site3*	*176 site4*	*176 site5*	*176 site6*	*175 site1b*	*176 site4b*	*175 site1c*	*176 site4c*	*175 site1*	*175 site2*	*175 site3*	*175 site1b*
Nb2O5 AGH VKb	0.01	0.01	0.01	0.00	0.01	0.01	0.01	0.01	0.00	0.00	0.01	0.01	0.00	0.00	0.00	0.00	0.01	0.01	0.01	0.01	0.00	
Ta2O5 AGH VKb	0.00	0.00	0.00	0.00	0.01	0.01	0.00	0.01	0.01	0.02	0.01	0.00	0.01	0.00	0.02	0.00	0.00	0.01	0.00	0.01	0.00	
WO3 AGH VKb	0.00	0.02	0.00	0.01	0.01	0.00	0.01	0.02	0.01	0.01	0.02	0.02	0.02	0.02	0.01	0.02	0.02	0.02	0.00	0.00	0.00	
SiO2 AGH VKb	0.02	0.02	0.01	0.15	0.01	0.01	0.00	0.13	0.19	0.02	0.03	0.02	0.01	0.02	0.06	0.01	2.38	0.10	30.46	30.40	30.77	
TiO2 AGH VKb	51.55	51.86	51.40	51.44	51.51	51.88	51.35	51.86	0.58	0.67	0.82	2.39	4.16	2.74	0.51	2.04	3.33	11.06	37.55	37.97	37.92	
SnO2 AGH VKb	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	
ZnO AGH VKb	0.04	0.05	0.05	0.00	0.00	0.00	0.02	0.01	0.03	0.00	0.01	0.01	0.01	0.00	0.00	0.00	0.00	0.00	0.00	0.01	0.02	
Al2O3 AGH VKb	0.02	0.04	0.01	0.14	0.02	0.03	0.02	0.07	0.07	0.05	0.05	0.04	0.03	0.03	0.06	0.02	0.36	0.10	1.28	0.98	1.07	
V2O5[#] AGH VKb	0.30	0.35	0.38	0.41	0.50	0.50	0.35	0.37	1.47	1.72	1.72	2.13	2.14	2.10	1.50	2.14	1.42	1.86	0.71	0.82	0.73	
Cr2O3 AGH VKb	0.02	0.01	0.01	0.02	0.02	0.02	0.01	0.01	0.05	0.04	0.05	0.07	0.08	0.07	0.03	0.06	0.02	0.05	0.02	0.02	0.02	
FeO AGH VKb	46.18	45.95	46.35	46.05	47.05	47.03	46.66	46.74	90.40	90.53	91.00	89.06	87.60	89.14	90.64	89.89	84.37	81.51	1.64	1.21	1.51	
MnO AGH VKb	1.92	1.89	1.87	1.40	1.26	1.33	1.90	1.44	0.02	0.03	0.02	0.06	0.11	0.08	0.02	0.06	0.07	0.30	0.04	0.03	0.04	
MgO AGH VKb	0.00	0.03	0.00	0.02	0.00	0.00	0.02	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.14	0.00	0.13	0.00	0.00	
CaO AGH VKb	0.11	0.11	0.11	0.00	0.01	0.02	0.02	0.01	0.20	0.06	0.10	0.03	0.00	0.04	0.12	0.01	1.75	0.08	27.30	27.83	27.78	
TOTAL AGH VKb	100.18	100.33	100.20	99.66	100.40	100.85	100.37	100.67	93.04	93.15	93.83	93.83	94.17	94.24	92.95	94.26	93.86	95.08	99.12	99.27	99.87	

** Background correction in all samples from AGH is off-peak, but the name in the table correspond to the spots measured at GTK.*

[#] *V measured on Kβ-line.*

EPMA average results from AGH with 20kV accelerating voltage and 20nA beam current. V measured on Kα-line.

beam size (if other than 1 my)																	*20my*	*20my*				
mineral	*ilmenite*								*magnetite*										*titanite*			
*background correction**	*MAN*						*off-peak*		*MAN*						*off-peak*		*off-peak*		*MAN*			*off-peak*
No. of analyses	*7*	*6*	*6*	*6*	*6*	*6*	*3*	*3*	*5*	*5*	*5*	*5*	*5*	*5*	*3*	*3*	*2*	*2*	*3*	*4*	*4*	*2*
	175 site1	*175 site2*	*175 site3*	*176 site4*	*176 site5*	*176 site6*	*175 site1b*	*176 site4b*	*175 site1*	*175 site2*	*175 site3*	*176 site4*	*176 site5*	*176 site6*	*175 site1b*	*176 site4b*	*175 site1c*	*176 site4c*	*175 site1*	*175 site2*	*175 site3*	*175 site1b*
Nb2O5 AGH Vka	0.01	0.01	0.00	0.00	0.01	0.01	0.01	0.01	0.00	0.00	0.00	0.01	0.00	0.00	0.00	0.01	0.01	0.00	0.00	0.00	0.01	
Ta2O5 AGH Vka	0.01	0.01	0.00	0.02	0.01	0.00	0.00	0.01	0.00	0.01	0.00	0.01	0.00	0.01	0.00	0.01	0.00	0.01	0.01	0.02	0.00	
WO3 AGH Vka	0.01	0.01	0.00	0.01	0.00	0.02	0.01	0.01	0.02	0.02	0.01	0.02	0.01	0.01	0.01	0.03	0.02	0.02	0.00	0.00	0.00	
SiO2 AGH Vka	0.02	0.02	0.00	0.01	0.01	0.01	0.01	0.08	0.12	0.02	0.16	0.01	0.04	0.01	0.06	0.01	2.34	0.04	30.46	30.52	30.66	
TiO2 AGH Vka	51.57	51.73	51.44	51.78	51.52	51.87	51.26	51.91	0.50	0.72	1.16	1.74	5.38	2.45	0.54	1.46	3.28	10.45	37.53	37.83	38.13	
SnO2 AGH Vka	0.00	0.00	0.00	0.00	0.00	0.00	0.01	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	
ZnO AGH Vka	0.05	0.03	0.03	0.01	0.00	0.01	0.03	0.01	0.01	0.01	0.01	0.01	0.01	0.02	0.00	0.00	0.00	0.00	0.01	0.01	0.00	
Al2O3 AGH Vka	0.03	0.02	0.01	0.03	0.02	0.03	0.05	0.05	0.14	0.04	0.04	0.01	0.06	0.04	0.02	0.01	0.31	0.05	1.38	1.11	1.06	
V2O5[#] AGH Vka	0.23	0.32	0.33	0.36	0.49	0.45	0.27	0.32	1.45	1.75	1.77	2.17	2.10	2.17	1.43	2.19	1.41	1.88	0.66	0.84	0.73	
Cr2O3 AGH Vka	0.01	0.02	0.02	0.03	0.02	0.03	0.00	0.01	0.05	0.03	0.04	0.08	0.08	0.07	0.05	0.08	0.02	0.07	0.02	0.02	0.01	
FeO AGH Vka	46.16	46.27	46.50	46.56	47.17	47.15	46.71	46.54	90.86	90.60	90.19	90.36	86.07	89.36	91.22	90.27	84.43	81.95	1.95	1.33	1.82	
MnO AGH Vka	1.92	1.90	1.89	1.38	1.28	1.30	1.81	1.55	0.03	0.02	0.03	0.04	0.15	0.07	0.03	0.05	0.07	0.31	0.04	0.02	0.06	
MgO AGH Vka	0.02	0.01	0.01	0.00	0.00	0.00	0.02	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.13	0.00	0.23	0.02	0.01	
CaO AGH Vka	0.14	0.09	0.11	0.01	0.02	0.03	0.04	0.05	0.15	0.07	0.36	0.01	0.01	0.04	0.14	0.01	1.71	0.00	27.12	27.85	27.48	
TOTAL AGH Vka	100.19	100.44	100.34	100.19	100.56	100.91	100.23	100.54	93.32	93.29	93.76	94.46	93.90	94.24	93.49	94.13	93.71	94.79	99.42	99.58	99.98	

** Background correction in all samples from AGH is off-peak, but the name in the table correspond to the spots measured at GTK.*

[#] *V measured on Kα-line.*