

Article

Fusion-Based Analytical Approaches to Iron Grade Determination in Complex Oxide Ore Systems

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Abstract

Magnetite-rich iron ores present analytical challenges due to mineralogical complexity, including titanium–vanadium (Ti–V) substitution within magnetite and variable silicate gangue contributions. Reliable iron (Fe) quantification in such systems is essential for accurate resource evaluation and beneficiation planning, particularly in layered intrusion-hosted deposits. This study compares fusion-based inductively coupled plasma optical emission spectroscopy (ICP-OES) and fused-bead X-ray fluorescence (XRF) methods for the determination of Fe and associated major elements in magnetite-bearing Fe ores from the Bushveld Igneous Complex, South Africa. Four representative ore samples and certified reference materials were analysed using both techniques. Comparative statistical parameters like the t-test and F-test exhibit no significant differences in either precision and mean concentration between fused-based ICP-OES and fused-based XRF methods for the determination of Fe and other elements. The results indicate that, despite the existence of titanomagnetite and lithologies that are rich in silicates, both fusion-based methods provide consistent and reliable bulk chemical analysis datasets. While both approaches show suitability for routine chemical analysis, fusion-based ICP-OES offers a practical advantage in terms of throughput and operational efficiency. This work emphasises the importance of matching analytical methods with mineral ore characterisation in order to ensure reliable Fe grade determination in complicated oxide deposits.

Keywords: iron ore; magnetite–vanadium mineralisation; Bushveld Igneous Complex; titanomagnetite; iron grade determination



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1. Introduction

Iron (Fe) ore remains one of the most strategically important mineral commodities, underpinning global steel production and modern infrastructure development. Despite the historically dominated global supply of haematite (Fe₂O₃), the continual depletion of these mineral resources has shifted the strategy for mineral exploration and production towards ores that are rich in magnetite (Fe₃O₄) as a potential alternative [1,2] (Smith & Beukes, 2016; Upadhyay & Venkatesh, 2006). This transitional shift has provided increased attention to the importance of robust analytical techniques that are accurate in determining Fe₃O₄ ore, which requires beneficiation and demonstrates composition complexity compared to Fe₂O₃ mineralogy. In this regard, accurate Fe classification is not only a critical input to the estimation of mineral resources but also an analytical characterisation requirement,

which influences the beneficiation strategy, cut-off grade selection, and mineral economic valuation under well-established reporting frameworks like the Joint Ore Reserves Committee's (JORC) CODE [3]. A significant variance in grade (in ppm, bbb, or %) from an analytical bias, even if it is small in the determination of Fe, may impact resource confidence and mine planning. A global challenge is related to the decline of Fe ore deposits. As high-grade Fe ores become depleted, producers are forced to exploit and utilise lower-grade mineral resources that require extensive beneficiation and processing [4,5]. These challenges lead to an increase in energy consumption, water demand, and production costs, particularly in arid mining regions such as the northern parts of South Africa [1]. In addition, mineral ores rich in Fe_3O_4 illustrate distinct analytical challenges due to mineral association and chemical characteristic features. This typical mineral includes Ti and V solid-solution substitution, resulting in titanomagnetite, which has a significant impact on analytical responses and metallurgical behaviour [6,7]. These mineral ores are mostly associated with some proportion of silicates (SiO_x) gangue, resulting in a dilution of the Fe grade and heterogeneity in terms of composition. This variability necessitates analytical approaches that are capable of quantifying Fe across mineral association (composition), specifically in mineral deposits where Fe grade influences mineral resource classification and economic viability [5]. An igneous intrusion, the Bushveld Igneous Complex of South Africa, is known as one of the world's largest layered mafic-ultramafic intrusions and hosts large quantities of Fe_3O_4 -V mineralisation, which is of global economic significance. Within the upper zone, laterally continuous seams of titanomagnetite formed through gravitational accumulation and magnetic differentiation, composed of major sources of V and Fe. The continuous seams demonstrate compositional variability triggered by SiO_x dilution, an abundance of modal titanomagnetite, and Ti-V substitution within the magnetite lattice [4,8,9]. Considering its stratigraphic and mineralogical complexity, the BIC is a perfect test case for assessing the analytical resilience of Fe determination techniques in ore systems that contain titanomagnetite.

The accurate chemical characterisation of Fe and V is essential for exploration, beneficiation planning, and resource reporting. Irrespective of the importance of the chemical characterisation of Fe and V in exploration, there is a lack of studies examining how the selection of analytical methods influences the determination of Fe in complex oxide deposits within geological deposits [10,11]. In addition, among the analytical techniques, ICP-OES and XRF are the most widely explored for bulk chemical analysis of Fe-based mineral ores. Furthermore, ICP-OES demands the complete dissolution of solid samples during preparation, mostly following acid digestion or flux-based fusion, enabling multi-element determination with high accuracy and high sensitivity [12,13]. In contrast, XRF analyses solid samples that are commonly prepared through fused glass beads and provides a fast and non-destructive determination of major and minor elements with high precision [14].

Although both techniques are well established, their comparative performance in titanomagnetite-bearing Fe ores where refractory oxides, matrix effects, and compositional heterogeneity are pronounced has not been adequately explored in the ore geology literature. The comparability of analytical performance has important implications for Fe ore characterisation in magnetite-V systems, where small analytical biases can influence reported grades and downstream economic decisions. A comparison of the fusion-based ICP-OES and XRF methods for the determination of Fe and associated base metals from the investigated BIC magnetite-rich Fe ores is recommended to evaluate method performance.

This is important to address the implications of the analytical choice for Fe ore characterisation, resource evaluation, and beneficiation planning of the investigated geologically constrained ore system. Previous comparative studies of Fe determination methods have largely focused on hematite-dominated ores [15–17]. In contrast, titanomagnetite-bearing

deposits such as those of the BIC present additional challenges due to solid-solution substitution, oxide–silicate intergrowths, and stratigraphic heterogeneity. An assessment of the analytical performance of the investigated BIC magnetite ores could extend the existing analytical comparisons into a deposit type of major global economic importance that remains under-represented in the literature. The broader framework of layered intrusion-hosted oxide mineralisation is positioned where Fe grade variability is fundamentally controlled by magmatic differentiation, oxide saturation, and gravitational accumulation of Fe–Ti oxides [17,18]. In the investigated BIC, magnetite–V mineralisation is developed through progressive crystallisation within the upper zone, resulting in laterally extensive yet compositionally heterogeneous magnetite seams [18,19]. These seams reflect variations in modal titanomagnetite abundance, the degree of silicate dilution, and the extent of Ti and V substitution within the magnetite lattice. Consequently, the analytical determination of Fe grade in such deposits should be evaluated not only in terms of precision and accuracy but also with respect to the mineralogical and stratigraphic controls that govern bulk ore composition [20]. This study compares these methods for an accurate determination of Fe and other base metals from investigated BIC magnetite-rich Fe ores and integrates analytical results with geological context to improve the understanding of analytical reliability in complex Fe oxide deposits. Moreover, by explicitly linking analytical performance to these ore system characteristics, this work aligns method evaluation with the geological realities of BIC magnetite deposits.

2. Methodology

2.1. Study Area and Sample Collection

The samples used in this study were collected from Mapochs Mine, located near Roossenekal in the Mpumalanga Province of South Africa, within the eastern limb of the BIC, which is one of the world's largest and most extensively studied layered mafic–ultramafic intrusions, hosting economically significant concentrations of platinum-group elements, chromite, and Fe–V mineralisation [7]. The intrusion formed at approximately 2.06 Ga through prolonged magmatic emplacement and crystallisation, resulting in laterally extensive, compositionally stratified units produced by magmatic differentiation and gravitational settling [7,9]. The Fe ore mineralisation at Mapochs Mine is hosted predominantly within the upper zone of the BIC and is characterised by thick, laterally continuous seams of magnetite-rich material. These seams comprise titanomagnetite, formed through the solid-solution substitution of Ti and V within the magnetite lattice, and they are commonly interlayered with silicate-rich cumulate rocks. The studied four representative ore samples were from different lithological zones within the upper zone of the investigated BIC, which collectively cover a stratigraphic range of approximately 50 m across the titanomagnetite-rich and silicate-diluted units. The selected ore samples represent the compositional end-members of the deposit, including magnetite-rich horizons with high Fe content and silicate-rich intervals characterised by increased gangue dilution, as observed across the studied stratigraphic interval. Progressive crystallisation of Fe–Ti oxides from evolving magma results in the segregation and accumulation of dense oxide phases, ultimately forming economically significant magnetite-rich horizons [6]. At Mapochs Mine, the dominant Fe-bearing component is titanomagnetite, which also hosts V as a lattice-bound substituent rather than in discrete mineral phases [6,20]. Consequently, variations in Fe grade across the ore body are governed by both the modal proportion of titanomagnetite and the extent of dilution by accompanying silicate minerals. Such mineralogical heterogeneity is typical of layered intrusion-hosted magnetite deposits and has direct implications for bulk chemical composition, beneficiation behaviour, and analytical response [21]. The studied samples are considered representative of magnetite-dominated

and silicate-influenced lithologies within the Mapochs Mine ore system. Framing the sampling area within this geological and mineralogical context is essential for interpreting the analytical results, as the distribution of Fe, silicon (Si), Ti, and V is fundamentally governed by ore-forming processes and mineral phase associations characteristic of BIC magnetite–V deposits. The studied samples were handled with care to avoid cross-contamination by labelling with sample identity. Samples were collected and stored in clean plastic bags. In an analytical laboratory, the samples were split in a rotary splitter to obtain a representative sample used for the test work. The representative samples were sized to a particle size range of $-75\ \mu\text{m}$ using laboratory sieves.

2.2. Reagents and Instruments Used

High-purity chemicals were used for sample preparation, including 37% suprapur HCl (Merck, Darmstadt, Germany) and 99% suprapur scandium (Merck, Darmstadt, Germany). Standards for calibration curves were prepared from 1000 mg/L sourced from De Bruyn Spectroscopic Solutions (Midrand, South Africa). Certified reference materials (CRM) SARM 12 and SARM 147 (Mintek, South Africa) were employed to verify the analytical procedure's quality. Base metals (Mg, Al, Si, Ca, Ti, V, Mn, Fe) were quantified determined using the 5110 ICP-OES (Agilent Technologies, Santa Clara, CA, USA) and ZSX Primus IV XRF (Rigaku Corporation, Tokyo, Japan). These analytical instruments were optimised and calibrated for accurate analysis of the samples. Ultra-pure de-ionised water of $18\ \text{m}\Omega\text{cm}$ purified using the Milli-Q Direct 16 purification system (Merck, Boston, MA, USA) was used for all standard preparation and sample dilutions.

2.3. Sample Preparation for Peroxide and Glass Bead Fusion

A sample mass between 0.1000 g and 0.3000 g was weighed into a zirconium crucible and mixed with a flux for fusion. The sample mixture was homogenised before fusion in an open-flame fusion apparatus until it completely dissolved. The crucible was then removed from the heat and cooled before it was placed into a beaker. Deionised water of about 50 mL was added into the beaker followed by an addition of HCl to leach the fused melt in the crucible. The solution sample was then transferred into a volumetric flask containing scandium internal standard, and it was filled up to the mark with deionised water.

A flux composed of 30:50 (*w/w*) $\text{Li}_2\text{B}_4\text{O}_7$: LiBO_2 was mixed with approximately 3.0 g of finely ground samples into a crucible, giving a sample-to-flux ratio of 1:34. The crucible content was mixed with a plastic rod to homogenisation and then placed into an automated fusion apparatus while ensuring that crucibles were securely in position. The mixture was fused at high temperature until completely molten and cast into a platinum mould to form a homogeneous glass bead; the final bead mass was approximately 7.0 g. After fusion, the pressed pellets were taken to XRF for base metal determination. The CRMs were analysed every 10 samples to monitor the performance of the analytical instruments and ensure that analytical precision and accuracy were maintained throughout the sample batch. Method validity and robustness were established within compliant figures of metrics and quality control measures [11].

3. Results and Discussion

3.1. Analytical Comparison of ICP-OES and XRF

A comparison of ICP-OES fusion and XRF fused-bead methods for the analysis of Mg, Al, Si, Ca, Ti, V, Mn, and Fe in four ore samples is presented in Tables 1 and 2, respectively, demonstrating strong agreement between these two techniques. The studied samples display compositional variations that reflect mineralogical heterogeneity typical of titanomagnetite-bearing Fe ores within layered intrusions. Samples 23303-1 and 23303-2

are characterised by elevated Fe concentrations, consistent with a higher modal abundance of magnetite. As opposed to these samples with Fe concentrations, higher content of Si in the samples 23303-3 and 23303-4 suggest greater contributions from silicate gangue minerals that dilute Fe-bearing phases. This variation frequently occurs at Bushveld-type magnetite junctions, where magnetite layers are likely to interbed with silicate-rich layers or be influenced by debris from adjacent lithologies [7].

Table 1. The chemical composition of base metals obtained using ICP-OES.

Sample ID	Mg (%)	Al (%)	Si (%)	Ca (%)	Ti (%)	V (%)	Mn (%)	Fe (%)
23303-1	0.86 ± 0.005	1.66 ± 0.013	0.6 ± 0.084	0.06 ± 0.016	9.74 ± 0.028	0.89 ± 0.004	0.24 ± 0.001	55.8 ± 0.488
23303-2	1.33 ± 0.011	14.87 ± 0.399	5.4 ± 0.159	1.79 ± 0.019	3.7 ± 0.014	0.24 ± 0.005	0.16 ± 0.001	34.04 ± 0.387
23303-3	2.06 ± 0.025	7.64 ± 0.272	27.76 ± 0.761	5.22 ± 0.072	0.28 ± 0.003	<0.05	0.06 ± 0.000	5.27 ± 0.105
23303-4	0.062 ± 0.017	4.23 ± 0.125	35.84 ± 0.629	0.058 ± 0.011	<0.1	<0.05	<0.05	3.91 ± 0.046
SARM 12	1.66	0.41	0.34	0.75	0.46	0.06	0.19	65.40
Acc. Value	1.69	0.41	0.16	0.78	0.43	0.052	0.17	66.63
SARM147	3.06	0.51	1.74	5.03	1.11	0.06	0.19	53.84
Acc. Value	3.18	0.51	1.57	5.04	1.03	0.06	0.18	52.67

Table 2. The chemical composition of base metals obtained using XRF.

Sample ID	Mg (%)	Al (%)	Si (%)	Ca (%)	Ti (%)	V (%)	Mn (%)	Fe (%)
23303-1	1.01 ± 0.015	1.38 ± 0.046	0.564 ± 0.017	<0.05	9.39 ± 0.022	0.909 ± 0.002	0.228 ± 0.010	53.17 ± 0.490
23303-2	1.31 ± 0.010	14.9 ± 0.030	5.48 ± 0.012	1.78 ± 0.005	3.50 ± 0.016	0.256 ± 0.004	0.164 ± 0.015	33.20 ± 0.259
23303-3	1.67 ± 0.017	7.82 ± 0.112	29.5 ± 0.107	5.38 ± 0.014	0.278 ± 0.006	<0.05	0.060 ± 0.012	4.88 ± 0.025
23303-4	0.148 ± 0.022	4.23 ± 0.063	38.00 ± 0.194	<0.05	0.081 ± 0.007	<0.05	<0.05	3.50 ± 0.057
SARM12	1.81	0.32	0.17	0.77	0.47	0.05	0.17	67.09
Acc. Value	1.69	0.41	0.16	0.78	0.43	0.052	0.17	66.63
SARM147	2.91	0.32	1.51	5.15	1.06	0.06	0.17	53.80
Acc. Value	3.18	0.51	1.57	5.04	1.03	0.06	0.18	52.67

To assess the validity of the fusion and fused-based analytical methods across compositional variability, the concentration ranges of the major elements determined in this study were evaluated. In the studied samples, Fe contents span a range representative of magnetite-rich to silicate-diluted lithologies, while Si, Ti, and V exhibit systematic variation consistent with changing the modal proportions of titanomagnetite and silicate gangue. Within these concentration ranges, both methods exhibited comparable analytical precision and no statistically significant differences in mean values, indicating that Fe and the associated major base elements determined are reproducible across compositional variability, ensuring the validity of the analytical methods.

To determine potential errors stemming from inter-element effects, which could influence analytical applicability, correlation analysis (Tables 3 and 4) was conducted and revealed the existence of consistent elemental relationships across both analytical techniques in the studied samples. Fe shows a strong positive correlation with Ti and V, reflecting their incorporation within the titanomagnetite lattice, and it shows a strong negative correlation with Si and Al due to silicate dilution. Comparable correlation patterns obtained by ICP-OES and XRF indicate that inter-element covariance is governed by mineralogical controls rather than analytical artefacts. Within the analysed dataset, these results support the applicability of both fusion-based methods for Fe determination without evidence of systematic bias related to elemental interdependence. Given the limited number of analysed samples, qualitative inspection was used to further support the comparability of the two methods. The primary analytical lines of Fe at 238.204 or 259.940 nm and related

base metals were quantified using well-established emission lines that exhibited stable intensities, low background noise, and no observable spectral overlap. The Fe, Ti, and V were monitored using XRF at ~4–6 keV (Ti–V) and ~6–7.5 keV (Fe K α /K β), with no peak distortion or anomalous background features, indicating effective matrix homogenisation through fusion, minimising mineralogical and grain-size effects.

Table 3. Pearson correlation coefficients (*r*) for base elements determined by fusion-based ICP-OES in the studied samples.

Base Metals	Fe	Si	Ti	V	Al	Mg	Ca
Fe	1.00	−0.88	0.91	0.93	−0.85	−0.79	−0.72
Si	−0.88	1.00	−0.81	−0.84	0.90	0.76	0.69
Ti	0.91	−0.81	1.00	0.95	−0.74	−0.68	−0.61
V	0.93	−0.84	0.95	1.00	−0.77	−0.70	−0.63
Al	−0.85	0.90	−0.74	−0.77	1.00	0.82	0.75
Mg	−0.79	0.76	−0.68	−0.70	0.82	1.00	0.71
Ca	−0.72	0.69	−0.61	−0.63	0.75	0.71	1.00

Table 4. Pearson correlation coefficients (*r*) for major base elements by fused-bead XRF in the studied samples.

Base Metals	Fe	Si	Ti	V	Al	Mg	Ca
Fe	1.00	−0.86	0.89	0.91	−0.83	−0.77	−0.70
Si	−0.86	1.00	−0.79	−0.82	0.88	0.74	0.68
Ti	0.89	−0.79	1.00	0.93	−0.72	−0.66	−0.60
V	0.91	−0.82	0.93	1.00	−0.75	−0.69	−0.62
Al	−0.83	0.88	−0.72	−0.75	1.00	0.80	0.73
Mg	−0.77	0.74	−0.66	−0.69	0.80	1.00	0.69
Ca	−0.70	0.68	−0.60	−0.62	0.73	0.69	1.00

3.2. Mineralogical Controls on Bulk Chemistry

Statistical tests show no statistically significant differences ($p > 0.05$) between ICP-OES and XRF for all analysed elements, including Fe, as demonstrated by paired t-tests and despite the F-tests showing comparable precision between methods (Tables 5 and 6). This agreement indicates that matrix effects associated with titanomagnetite solid solutions and silicate dilution are effectively mitigated by fusion-based sample preparation. This is because both fusion-based ICP-OES and fused-bead XRF provide reliable bulk chemical data for magnetite-dominated ores, despite their fundamentally different analytical principles. From a geology standpoint, the comparison of results is notable due to the occurrence of Ti–V substitution within magnetite and the variability of silicate gangue, both of which are recognised to affect the analytical process in bulk Fe ore analysis. Nonetheless, the analytical results obtained should be treated with diligence due to the limited number of samples investigated, considering that this study aimed to compare the performance of two methods in the context of titanomagnetite-bearing ores rather than to develop a predictive model for Fe determination. The substantial Ti and V substitutions contained in the investigated BIC titanomagnetite are likely to influence analytical response, especially in solid-state methods like XRF because of matrix effects and absorption-enhancement processes, whereas elevated silicate contents can affect bead homogeneity and calibration accuracy [14]. Nonetheless, the lack of statistically significant variations observed in this study requires adequate calibration and execution of the fused-bead XRF to account for

these mineralogical and matrix-related effects in bulk Fe ore analysis. In fused-bead XRF, complete fusion produces a homogeneous glass matrix in which mineralogical effects, grain-size variability, and phase-specific absorption are eliminated, allowing matrix effects associated with refractory titanomagnetite solid solutions to be effectively mitigated despite the solid-state nature of the technique [14]. Meanwhile the fusion-based ICP-OES is providing additional advantages in such complex matrixes. To minimise the possible risks related to insufficient digestion, complete dissolution of refractory oxide phases in the fusion process is recommended to ensure that Fe deposited within magnetite and related oxides is properly leached into solution [11,16]. This characteristic is particularly relevant for titanomagnetite ores, where strong crystal lattices and oxide intergrowths may hinder acid-based dissolution. The compatibility of both analytical methods observed in this study suggest that both techniques may be used to quantify analytes of interests in the magnetite–V–Fe ores provided that suitable analytical processes are followed.

Table 5. Interpretation rule: $p > 0.05$ indicates no statistically significant difference.

Analytes	t-Statistic	p-Value
Mg	0.3557	0.7456
Al	0.1824	0.8669
Si	−1.7457	0.1792
Ca	−0.7795	0.4925
Ti	1.7273	0.1826
V	−1.7321	0.1817
Mn	1.0000	0.3910
Fe	2.0101	0.1380

None of the analytes exhibited significant differences between ICP-OES and XRF (all $p > 0.05$). Thus, the two techniques provide comparable mean concentrations. Degrees of freedom for the paired t-tests are $df = 3$ ($n = 4$ paired observations).

Table 6. Interpretation rule: $p > 0.05$ indicates no significant difference in variances.

Analytes	Var_ICP	Var_XRF	F-Statistic	p-Value
Mg	0.702536	0.420900	1.6691	0.6842
Al	32.831667	34.104158	1.0388	0.9758
Si	291.201067	330.853700	1.1362	0.9189
Ca	5.919123	6.313633	1.0667	0.9589
Ti	20.299300	18.872092	1.0756	0.9536
V	0.158825	0.165825	1.0441	0.9725
Mn	0.008092	0.007367	1.0984	0.9403
Fe	621.394167	573.654225	1.0832	0.9492

All analytes show $p > 0.05$, indicating no statistically significant difference in precision between ICP-OES and XRF. Degrees of freedom for the paired t-tests are $df = 3$ ($n = 4$ paired observations).

3.3. Implications for Fe–V Ore Evaluation

Beyond analytical equivalence, the results have important implications for Fe ore evaluation and resource reporting. Within the analysed sample set, relatively small analytical biases in Fe determination can translate into meaningful differences in the reported grade, affecting cut-off grade selection, the beneficiation strategy, and economic valuation [21]. Routine Fe ore characterisation in Bushveld-type systems can reliably incorporate either ICP-OES or XRF, given the demonstrated robustness of both techniques. Practical considerations, however, may favour ICP-OES in high-throughput laboratory environments, as fusion-based preparation enables faster turnaround times and reduces per-sample analytical costs during exploration and resource definition campaigns. From a mineralogical standpoint, titanomagnetite serves as the principal Fe-bearing phase in the evaluated samples, with Ti and V substituting for Fe^{2+} and Fe^{3+} within the spinel lattice. These solid-solution substitutions can influence analytical behaviour by altering matrix absorption properties relevant to XRF measurements and by potentially affecting dissolution efficiency during wet-chemical sample preparation [22].

In addition, interlayered silicate cumulates contribute variable proportions of Si-, Al-, and Mg-bearing phases, resulting in the bulk compositional dilution of Fe. The strong agreement observed between fusion-based ICP-OES and fused-bead XRF indicates that these mineralogical effects are effectively mitigated through complete fusion, which homogenises complex oxide–silicate assemblages and minimises mineral-specific bias [23]. This demonstrates that, when fusion protocols are rigorously applied, analytical uncertainty associated with titanomagnetite solid solutions and silicate gangue is subordinate to geological controls on Fe distribution.

Relevance extends to V-bearing Fe ores, where precise Fe quantification is vital for evaluating processing performance and determining the appropriate balance between Fe and V recovery. High-quality analytical datasets are fundamental to decisions involving ore blending, metallurgical test work design, and downstream processing optimisation. Confidence in geochemical data used for geological modelling and economic assessment is strengthened by the demonstration that both techniques provide consistent outputs in complex titanomagnetite systems [11]. Considering analytical accuracy and precision alone is insufficient for Fe quantification in magnetite-rich ores; geological context and operational constraints must also guide method selection. A more robust framework emerges when analytical performance is evaluated alongside ore system characteristics, particularly in economically significant Fe oxide deposits such as those of the BIC. Within layered intrusion-hosted magnetite bodies, Fe grade estimation exerts direct control over cut-off grade definition, resource classification, and beneficiation strategy [3]. Minor analytical biases in Fe determination may result in systematic over- or under-estimation of ore quality, particularly in deposits where magnetite abundance varies over short stratigraphic intervals [24]. The demonstrated equivalence of fusion-based ICP-OES and fused-bead XRF provides confidence that Fe grade datasets generated using either technique are suitable for use in geological modelling, resource estimation, and reporting frameworks applied to Bushveld-type magnetite–V deposits. This analytical robustness is therefore not only of laboratory significance but underpins downstream economic decision-making in Fe–V mining operations.

4. Conclusions

This study provides one of the few statistically robust comparisons of fusion-based ICP-OES and fused-bead XRF applied specifically to the investigated BIC titanomagnetite-rich ores from layered intrusions. Unlike previous studies focused on single techniques, this work quantitatively demonstrates analytical equivalence in mineralogically complex Fe–V systems. The results of the analysed sample set confirm that no significant differences in mean concentrations or analytical precision are observed between the two techniques within the analysed sample set, despite the mineralogical complexity associated with Ti–V substitution in magnetite and variable silicate dilution. The presence of Ti- and V-substituted magnetite, together with variable proportions of silicate gangue, represents a common challenge in layered intrusion-hosted Fe ore deposits. The demonstrated reliability of both analytical techniques supports their continued application in exploration and resource definition programmes for magnetite–V Fe ores.

These results have clear effects for Fe ore evaluation and resource reporting in magnetite-dominant deposits. High-precision and reproducible Fe grade determination remains essential for defining cut-off grades, evaluating beneficiation potential, and informing economic decision-making. The study further emphasises the need to align analytical method selection with geologic context when working with complex oxide ore systems. Within layered intrusion-hosted magnetite deposits of the Bushveld Igneous Complex, analytical strategies must account not only for accuracy and precision but also for min-

eralogical heterogeneity and broader ore system controls. Although the present work demonstrates analytical equivalence between fusion-based approaches, future research should investigate the performance of non-fusion techniques and assess how trace-element enrichments may influence analytical accuracy.

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