



African Mineral Standards

MATRIX REFERENCE MATERIALS

Tel: +27 (0) 11 923 0800, Fax: +27 (0) 11 392 4715, web: www.amis.co.za
11 Gewel Street (off Hulley Road), D1 Isando Business Park, Kempton Park, 1609
P.O. Box 856, Isando, 1600, Gauteng, South Africa, a division of the Set Point Group

AMIS0368

**Vanadium bearing titaniferous magnetite,
Rhovan Vanadium Mine
South Africa**

Certified Reference Material

Certificate of Analysis

**Recommended Concentrations and Limits^{1, 2}
(at two Standard Deviations)**

Certified Concentrations

Fe M/ICP per	53.01	±	1.74	%
Ti M/ICP per	8.26	±	0.40	%
V M/ICP per	0.84	±	0.04	%
Specific Gravity	4.47	±	0.20	

1. *Manufacturers recommended limits for use of the material as control samples, based on two standard deviations, calculated using "Between Laboratory" statistics for treatment of the data for trivial, non-trivial and technically invalid results. See sections 1, 10 and 13.*
2. *There is additional certified major element data presented on p2 and uncertified trace element data presented as an appendix.*

Major Element Recommended Concentrations and Limits (at two Standard Deviations)

Certified Concentrations

Al ₂ O ₃	3.64	±	0.06	%
Cr ₂ O ₃	0.24	±	0.02	%
Fe ₂ O ₃	75.51	±	0.86	%
MgO	0.68	±	0.02	%
MnO	0.21	±	0.002	%
SiO ₂	2.65	±	0.06	%
TiO ₂	13.91	±	0.18	%
LOI	1.14	±	0.12	%
V ₂ O ₅	1.52	±	0.04	%

Provisional Concentrations

CaO	0.05	±	0.01	%
K ₂ O	0.05	±	0.01	%

Indicated Mean

Na ₂ O	0.03	%
-------------------	------	---

1. Intended Use: AMIS0368 is a certified reference material which may be used to demonstrate the validity of measurement results of a single analysis of vanadium bearing titaniferous magnetite ores hosted by mafic-ultramafic rocks.

It is a matrix matched Certified Reference Material, fit for use as control samples in routine assay laboratory quality control when inserted within runs of samples and measured in parallel to the unknown. Its purpose is to monitor inter-laboratory or instrument bias and within lab precision. It can be used, indirectly, to establish the traceability of results to an SI system of units.

The recommended concentrations and limits for this material are property values based on a measurement campaign (round robin) and reflect consensus results from the laboratories that participated in the round robin.

Slight variations in analytical procedures between laboratories will reflect as slight biases to the recommended concentrations (see 19). Good laboratories will report results within the two standard deviation levels with a failure rate of <10 %.

The material can also be used for method development and for the calibration of equipment.

2. Origin of Material: The material for this CRM was provided by Xstrata plc. from the Rhovan Vanadium Mine, situated approximately 30km to the north-west of Brits in the North-West Province of South Africa. The deposit being mined comprises thick layers of magnetite occurring close to the base of the Upper Zone of the 2060Ma year old Bushveld Complex.

3. Mineral and Chemical Composition: The mineral deposit comprises a vanadiferous, titaniferous magnetite gabbro with the vanadium occurring in the magnetite.

4. **Appearance:** The material is a very fine powder. It is colored Weak Red (Corstor 5R 4/4).
5. **Handling instructions:** The material is packaged in Laboratory Packs and Explorer Packs that must be shaken or otherwise agitated before use. Normal safety precautions for handling fine particulate matter are suggested, such as the use of safety glasses, breathing protection, gloves and a laboratory coat
6. **Method of Preparation:** The material was crushed, dry-milled and air-classified to <54um. Wet sieve particle size analysis of random samples confirmed the material was 98.5% <54um. It was then blended in a bi-conical mixer, systematically divided and then sealed into 1kg Laboratory Packs. Explorer Packs are subdivided from the Laboratory packs as required. Samples were randomly selected for homogeneity testing and third party analysis. Statistical analysis of both homogeneity and the consensus test results were carried out by independent statisticians.
7. **Methods of Analysis requested:**
1. Multi element scan to include Fe, Ti and V. Fusion, ICP-OES or ICP-MS.
 2. Majors (Al₂O₃, CaO, Cr₂O₃, Fe₂O₃, K₂O, MgO, MnO, Na₂O, SiO₂, TiO₂, V₂O₅. LOI.) XRF fusion.
 3. SG, gas pycnometer.
8. **Information requested:**
1. State aliquots used for all determinations.
 2. All results for major elements to be reported as oxides in percentages.
 3. All results for multi-element scans to be reported in ppm.
 4. Report all QC data, to include replicates, blanks and certified reference materials used.
 5. State and provide brief description of analytical techniques used.
9. **Method of Certification:** Twenty four laboratories were each given eight randomly selected packages of sample. Twenty one of the laboratories submitted results in time for certification.

Final limits were calculated after first determining if all data was compatible within a spread normally expected for similar analytical methods done by reputable laboratories. Data from any one laboratory was then removed from further calculations when the mean of all analyses from that laboratory failed a "t test" of the global means of the other laboratories. The means and standard deviations were then re-calculated using all remaining data. Any analysis that fell outside of the new two standard deviations was removed from the ensuing data base. The mean and standard deviations were again calculated using the remaining data.

The "between-laboratory" standard deviation is used in the calculation to eliminate technically and statistically invalid data. Upper and lower limits are based on the standard deviation of the remaining data, which reflect individual analyses and can be used to monitor accuracy in routine laboratory quality control. This is different to limits based on standard deviations derived from grouped set of analyses (see 12), which provide important measures for precision and trueness, but which are less useful for routine QC.

Standards with an RSD of near or less than 5 % are termed "Certified", RSD's of between near 5 % and 15 % are termed "Provisional", and RSD's over 15 % are termed "Informational".

10. **Participating Laboratories:** The 21 out of 24 laboratories that provided results timeously were (not in same order as in the table of assays):
- 1 ACME Analytical Laboratories Ltd CA
 - 2 Activation Laboratories Pty Ltd (ActLabs) CA
 - 3 ALS Ammtec (Australia)
 - 4 ALS Chemex Laboratory Group Brisbane Australia
 - 5 ALS Chemex Laboratory Group Johannesburg SA
 - 6 ALS Chemex Laboratory Group Perth WA

- 7 ALS Chemex Laboratory Group Vancouver CA
- 8 ALS OMAC (Ireland)
- 9 Anglo Research (Crown Campus)
- 10 BV Amdel (Australia)
- 11 Genalysis Laboratory Services (South Africa) Pty
- 12 Genalysis Laboratory Services (W Australia P)
- 13 Intertek Utama Services (Indonesia)
- 14 Set Point Laboratories (Isando) SA
- 15 SGS Australia Pty Ltd (Newburn) WA
- 16 SGS Geosol Laboratories Ltda (Brazil)
- 17 SGS Mineral Services Lakefield (Canada)
- 18 SGS South Africa (Pty) Ltd - Booyens JHB
- 19 SGS Townsville (Australia)
- 20 SGS Vancouver (Canada)
- 21 Ultra Trace (Pty) Ltd WA

11. **Assay Data:** Data as received from the laboratories for the important certified elements listed on p1 are set out below.

Lab Code	Fe M/ICP %	Ti M/ICP %	V M/ICP %	Al2O3 XRF %	CaO XRF %	Cr2O3 XRF %	Fe2O3 XRF %	K2O XRF %	MgO XRF %	MnO XRF %	Na2O XRF %	SiO2 XRF %	TiO2 XRF %	LOI %	V2O5 XRF %	S COMB/LECO %	SG pyc
A	53.14	8.34	0.86	3.64	0.06	0.25	75.54	0.05	0.67	0.21	0.03	2.60	13.91	1.44			4.15
A	53.25	8.31	0.86	3.66	0.06	0.25	76.20	0.05	0.69	0.20	0.03	2.60	14.01	1.46			4.42
A	53.34	8.22	0.86	3.63	0.06	0.25	75.40	0.05	0.68	0.20	0.04	2.60	13.89	1.50			4.32
A	52.88	8.32	0.86	3.62	0.05	0.25	75.51	0.04	0.67	0.20	0.02	2.60	13.91	1.47			4.46
A	52.72	8.15	0.85	3.63	0.05	0.24	75.25	0.04	0.67	0.20	0.02	2.60	13.88	1.47			4.30
A	53.88	8.25	0.86	3.63	0.06	0.25	75.50	0.05	0.67	0.20	0.03	2.60	13.85	1.45			4.43
A	53.50	8.34	0.87	3.63	0.06	0.25	75.65	0.04	0.68	0.21	0.03	2.60	13.94	1.40			4.42
A	54.06	8.48	0.87	3.62	0.06	0.25	75.61	0.04	0.67	0.20	0.02	2.60	13.91	1.45			4.37
C	53.60	8.26	0.87	3.60	0.06	0.23	75.50	0.04	0.70	0.21	0.02	2.67	13.70	1.17	1.50		4.47
C	53.40	8.22	0.86	3.62	0.06	0.23	75.80	0.04	0.71	0.21	0.02	2.63	13.80	1.16	1.50		5.04
C	53.20	8.22	0.86	3.62	0.07	0.24	76.10	0.04	0.73	0.21	0.02	2.79	13.80	1.16	1.50		4.47
C	55.30	8.60	0.86	3.62	0.06	0.23	75.80	0.04	0.70	0.21	0.03	2.65	13.80	1.12	1.50		4.73
C	53.00	8.16	0.86	3.62	0.06	0.24	76.10	0.04	0.71	0.21	0.02	2.67	13.80	1.13	1.50		4.44
C	53.70	8.28	0.86	3.63	0.06	0.24	75.90	0.04	0.71	0.21	0.03	2.64	13.80	1.22	1.51		4.48
C	51.50	8.00	0.86	3.63	0.06	0.24	76.10	0.04	0.71	0.21	0.03	2.66	13.80	1.21	1.50		4.48
C	53.60	8.26	0.87	3.64	0.06	0.24	76.00	0.04	0.71	0.21	0.02	2.67	13.80	1.23	1.50		4.49
D	52.60	8.37	0.82														4.42
D	53.00	8.35	0.83														4.34
D	53.70	8.33	0.82														4.40
D	53.20	8.36	0.83														4.42
D	53.30	8.23	0.84														4.43
D	53.20	8.44	0.83														4.31
D	53.30	8.30	0.83														4.35
D	53.60	8.71	0.84														4.35
E	47.00	8.14	0.74	3.59	0.05	0.24	75.49	0.04	0.66	0.21	0.02	2.65	13.95	1.29	1.55		4.29
E	48.10	8.32	0.73	3.61	0.05	0.24	75.52	0.04	0.66	0.21	0.01	2.64	13.95	1.26	1.55		4.31
E	49.10	8.50	0.75	3.59	0.05	0.25	75.51	0.04	0.66	0.21	0.02	2.62	13.95	1.28	1.55		4.29
E		8.84	0.80	3.61	0.05	0.25	75.52	0.04	0.65	0.21	0.02	2.64	13.95	1.27	1.55		4.33
E	50.00	8.78	0.78	3.60	0.05	0.25	75.52	0.04	0.66	0.21	0.02	2.65	13.95	1.24	1.55		4.31
E	49.40	8.56	0.78	3.62	0.05	0.24	75.50	0.04	0.65	0.21	0.01	2.64	14.00	1.24	1.55		4.33
E	48.70	8.46	0.74	3.60	0.05	0.24	75.57	0.05	0.70	0.21	0.02	2.67	13.90	1.22	1.54		4.31
E	48.60	8.40	0.77	3.59	0.05	0.24	75.59	0.04	0.69	0.21	0.02	2.66	13.90	1.23	1.55		4.37
F	54.10		0.83														4.63
F	55.90		0.84														4.67
F	54.90		0.85														4.65
F	58.40		0.87														4.62
F	54.50		0.84														4.60
F	56.50		0.85														4.59
F	54.70		0.84														4.62
F	57.70		0.85														4.62
G	49.60	8.31	0.75	3.84	0.05	0.25	75.34	0.04	0.68	0.21	0.03	2.65	13.95	1.18	1.53		4.37
G		8.41	0.76	3.85	0.05	0.25	75.33	0.04	0.68	0.21	0.03	2.67	13.95	1.17	1.53		4.32
G	50.00	8.35	0.76	3.84	0.05	0.25	75.31	0.05	0.68	0.21	0.03	2.64	13.95	1.18	1.53		4.31
G		8.56	0.77	3.85	0.05	0.25	75.33	0.04	0.68	0.21	0.03	2.65	13.95	1.17	1.53		4.19
G		8.41	0.75	3.85	0.05	0.25	75.33	0.05	0.68	0.21	0.03	2.65	13.90	1.18	1.53		4.28
G	49.50	8.29	0.74	3.85	0.05	0.25	75.35	0.04	0.69	0.21	0.03	2.64	13.90	1.17	1.53		4.29
G	49.10	8.22	0.74	3.85	0.05	0.25	75.33	0.04	0.68	0.21	0.04	2.66	13.95	1.16	1.54		4.25
G	49.10	8.17	0.74	3.85	0.05	0.25	75.31	0.04	0.68	0.21	0.03	2.65	13.95	1.18	1.53		4.21
H	51.15		0.84	3.63	0.03	0.24	74.34	0.06		0.22		2.68	13.98	1.54	1.51		4.45
H	50.82		0.84	3.66	0.04	0.23	74.76	0.06		0.22		2.63	13.98	1.54	1.52		4.44
H	51.01		0.84	3.78	0.03	0.23	74.43	0.05		0.21		2.58	13.90	1.54	1.51		4.46
H	50.09		0.85	3.62	0.04	0.23	74.64	0.05		0.22		2.61	13.96	1.53	1.52		4.46
H	52.69		0.86	3.64	0.04	0.23	74.84	0.06		0.22		2.65	14.02	1.52	1.51		4.44
H	53.20		0.85	3.64	0.04	0.23	74.75	0.06		0.22		2.65	13.98	1.53	1.51		4.45
H	52.11		0.86	3.67	0.05	0.23	74.82	0.05		0.22		2.68	14.00	1.53	1.51		4.45
H	52.68		0.85	3.66	0.05	0.23	74.75	0.05		0.22		2.68	13.99	1.52	1.51		4.44
I	50.50	7.75		3.62	0.06	0.23		0.05	0.67		0.03	2.62	13.80	1.14		0.02	4.58
I	52.80	8.14		3.61	0.06	0.23		0.05	0.66		0.03	2.63	13.80	1.15		0.03	4.55
I	52.20	8.03		3.62	0.06	0.23		0.05	0.67		0.03	2.64	13.75	1.13		0.03	4.43
I	52.10	8.02		3.59	0.06	0.23		0.05	0.67		0.03	2.61	13.75	1.16		0.03	4.44
I	51.10	7.85		3.60	0.06	0.23		0.05	0.67		0.03	2.62	13.75	1.16		0.03	4.61
I	52.40	8.07		3.61	0.05	0.23		0.05	0.67		0.02	2.63	13.80	1.15		0.03	4.41
I	52.00	8.03		3.60	0.06	0.23		0.05	0.67		0.03	2.62	13.80	1.14		0.03	4.53
I	52.70	8.12		3.61	0.06	0.23		0.05	0.67		0.03	2.62	13.80	1.16		0.03	4.28

12. Measurement of Uncertainty : (ref Dr Hugh Bartlett, Hugh Bartlett Consulting CC.)

The samples used in this certification process have been selected in such a way as to represent the entire batch of material and were taken from the final packaged units; therefore all possible sources of uncertainty (sample uncertainty and measurement uncertainty) are included in the final combined standard uncertainty determination.

The uncertainty measurement takes into consideration the between lab and the within lab variances and is calculated from the square roots of the variances of these components using the formula:

$$\text{Combined standard uncertainty} = \sqrt{(\text{between lab.var/no of labs}) + (\text{mean square within lab.var /no of assays})}$$

These uncertainty measurements may be used, by laboratories, as a component for calculating the total uncertainty for method validation according to the relevant ISO guidelines.

Analyte	Method	unit	S ¹	σ_L ²	SW ³	CSU ⁴
Fe	M/ICP	%	0.874	0.477	0.634	0.147
Ti	M/ICP	%	0.196	0.115	0.136	0.036
V	M/ICP	%	0.015	0.011	0.008	0.003
Al ₂ O ₃	XRF	%	0.026	0.015	0.015	0.004
CaO	XRF	%	0.005	0.003	0.003	0.001
Cr ₂ O ₃	XRF	%	0.009	0.006	0.003	0.001
Fe ₂ O ₃	XRF	%	0.433	0.290	0.206	0.080
K ₂ O	XRF	%	0.004	0.002	0.002	0.001
MgO	XRF	%	0.014	0.008	0.008	0.002
MnO	XRF	%	0.001	0.001	0.001	0.0002
Na ₂ O	XRF	%	0.007	0.005	0.005	0.002
SiO ₂	XRF	%	0.028	0.016	0.016	0.004
TiO ₂	XRF	%	0.093	0.061	0.036	0.000
V ₂ O ₅	XRF	%	0.016	0.013	0.006	0.004
LOI		%	0.064	0.045	0.027	0.012
SG	pyc		0.098	0.060	0.045	0.015

1. S - Std Dev for use on control charts.
2. σ_L - Betw Lab Std Dev, for use to calculate a measure of accuracy.
3. SW - Within Lab Std Dev, for use to calculate a measure of precision.
4. CSU - Combined Standard Uncertainty, a component for use to calculate the total uncertainty in method validation.

13. Certified values: The Certified, Provisional and Indicated values listed on p1 of this certificate fulfill the AMIS statistical criteria regarding agreement for certification and have been independently validated by Dr Barry Smee.

14. Metrological Traceability: The values quoted herein are based on the consensus values derived from statistical analysis of the data from an inter laboratory measurement program. Traceability to SI units is via the standards used by the individual laboratories the majority of which are accredited and who have maintained measurement traceability during the analytical process.

15. Certification: AMIS0368 is a new material.

16. Period of validity: The certified values are valid for this product, while still sealed in its original packaging, until notification to the contrary. The stability of the material will be subject to continuous testing for the duration of the inventory. Should product stability become an issue, all customers will be notified and notification to that effect will be placed on the www.amis.co.za website.

17. Minimum sample size: The majority of laboratories reporting used a 0.5g sample size.. This is the recommended minimum sample size for the use of this material.

18. Availability: This product is available in Laboratory Packs containing 1kg of material and Explorer Packs containing custom weights (from 50 to 250g) of material. Laboratory Packs are sealed bottles delivered in sealed foil pouches. Explorer Packs contain material in standard geochem envelopes, nitrogen flushed and vacuum sealed in foil pouches.

19. Recommended use: The data used to characterize this CRM has been scrutinized using outlier treatment techniques. This, together with the number of participating laboratories, should overcome any "inter-laboratory issues" and should lead to a very accurate measure for the given methods; notwithstanding the underlying assumption that what the good inter-laboratory labs reported was accurate. However an amount of bad data might have had an effect, resulting in limits which in some situations might be too broad for the effective monitoring of a single analytical method, laboratory or production process. Users should therefore set their own limits based on their own data quality objectives and control measurements, after determining the performance characteristics of their own particular method, using a minimum of 20 analyses using this CRM. User set limits should normally be within the limits recommended on p1 and 2 of this certificate.

20. Legal Notice: This certificate and the reference material described in it have been prepared with due care and attention. However AMIS, Set Point Technology (Pty) Ltd, Mike McWha, Dr Barry Smee and Smee and Associates Ltd; accept no liability for any decisions or actions taken following the use of the reference material.

25 March 2013

Certifying Officers:



African Mineral Standards: _____

Mike McWha
BSc (Hons), FGSSA, MAusIMM, Pr.Sci.Nat



Geochemist: _____

Barry W. Smee
BSc, PhD, P.Geo, (B.C.)

Appendix – uncertified trace element statistics

Analyte	Method	Unit	Mean	2SD	RSD%	n
Ag	M/ICP	ppm	0.21	0.19	45.6	30
Al	M/ICP	%	1.9	0.21	5.7	111
As	M/ICP	ppm	5.5	10.9	100	41
Ba	M/ICP	ppm	18.2	13.4	36.7	85
Be	M/ICP	ppm	0.13	0.26	97.4	23
Bi	M/ICP	ppm	1.6	13.8	420	42
Ca	M/ICP	%	0.04	0.01	14.8	98
Cd	M/ICP	ppm	5.2	12.2	117	39
Ce	M/ICP	ppm	5.0	1.9	18.7	40
Co	M/ICP	ppm	157	47.1	15.0	109
Cr	M/ICP	ppm	1416	331	11.7	102
Cs	M/ICP	ppm	0.12	0.09	38.7	32
Cu	M/ICP	ppm	220	43.8	10.0	96
Dy	M/ICP	ppm	0.16	0.09	28.6	16
Er	M/ICP	ppm	0.09	0.10	57.8	16
Eu	M/ICP	ppm	0.06	0.04	31.6	15
Ga	M/ICP	ppm	44.6	7.4	8.3	62
Gd	M/ICP	ppm	0.13	0.09	37.0	8
Ge	M/ICP	ppm	0.88	0.72	41.0	32
Hf	M/ICP	ppm	1.00	0.21	10.3	52
Ho	M/ICP	ppm	0.02	0.02	61.7	8
In	M/ICP	ppm	0.11	0.02	9.5	38
K	M/ICP	%	0.04	0.01	13.8	103
La	M/ICP	ppm	1.5	1.5	52.3	53
Li	M/ICP	ppm	1.4	0.78	28.6	68
Lu	M/ICP	ppm	0.31	0.93	150	23
Mg	M/ICP	%	0.38	0.09	11.4	111
Mn	M/ICP	ppm	1524	231	7.6	106
Mo	M/ICP	ppm	1.1	0.91	42.5	54
Na	M/ICP	%	0.02	0.01	29.7	80
Nb	M/ICP	ppm	3.4	2.2	33.0	46
Nd	M/ICP	ppm	0.88	0.38	21.5	23
Ni	M/ICP	ppm	392	85.8	10.9	90
P	M/ICP	ppm	119	171	71.9	38
Pb	M/ICP	ppm	14.7	35.3	120	63
Pr	M/ICP	ppm	2.0	3.8	92.7	32
Rb	M/ICP	ppm	3.0	3.6	60.5	51
Re	M/ICP	ppm	0.01	0.01	50.2	23
S	M/ICP	%	0.02	0.01	18.5	53
S	Comb/LECO	%	0.02	0.01	11.5	32
Sb	M/ICP	ppm	9.6	19.0	98.9	66
Sc	M/ICP	ppm	24.6	5.6	11.4	73
Se	M/ICP	ppm	2.9	5.0	85.3	32
Si	M/ICP	%	1.3	0.05	1.9	24
Sm	M/ICP	ppm	0.16	0.08	26.2	16
Sn	M/ICP	ppm	2.5	7.7	156	38
Sr	M/ICP	ppm	4.1	4.0	49.0	61
Ta	M/ICP	ppm	0.29	0.19	32.7	33
Te	M/ICP	ppm	3.3	14.4	218	28
Th	M/ICP	ppm	0.62	0.53	42.6	56
Tl	M/ICP	ppm	2.0	8.2	200	20
Tm	M/ICP	ppm	0.06	0.01	8.1	8
U	M/ICP	ppm	4.6	0.72	7.9	54
W	M/ICP	ppm	33.9	123	182	34
Y	M/ICP	ppm	0.78	0.52	33.4	63
Yb	M/ICP	ppm	0.11	0.05	20.6	8
Zn	M/ICP	ppm	307	59.2	9.6	95
Zr	M/ICP	ppm	27.4	5.8	10.6	64