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AMIS0304

Certified Reference Material

Rare Earth Elements
Glenover Carbonatite Complex, South Africa

Certificate of Analysis

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

Certified Concentrations

Ce M/ICP	8090	±	692	ppm
La M/ICP	3610	±	311	ppm
Nd M/ICP	3875	±	442	ppm
Pr M/ICP	1007	±	89	ppm
U M/ICP	24	±	1.7	ppm
Y M/ICP	410	±	39	ppm
Al M/ICP	8070	±	676	ppm
Ca M/ICP	20.11	±	1.34	%
Fe M/ICP	14.73	±	0.93	%
Mg M/ICP	1.67	±	0.18	%
P M/ICP	7.88	±	0.35	%
P XRF	7.99	±	0.32	%
Si M/ICP	5.81	±	0.34	%
Specific gravity	3.37	±	0.10	

Provisional Concentrations

Sm M/ICP	575	±	70	ppm
Th M/ICP	437	±	67	ppm
Th XRF	450	±	71	ppm

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, 9 and 12.
2. There is additional certified major element data presented on p2 and uncertified trace element data presented as an appendix.
3. CREO = (Nd+Eu+Tb+Dy+Y)₂O₃ = 0.54% (see Appendix 2)

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

Certified Concentrations

Al ₂ O ₃	1.52	±	0.08	%
CaO	28.50	±	0.72	%
Fe ₂ O ₃	20.93	±	1.00	%
K ₂ O	0.28	±	0.02	%
MgO	2.87	±	0.12	%
MnO	0.46	±	0.03	%
P ₂ O ₅	18.35	±	0.74	%
SiO ₂	12.31	±	0.40	%
TiO ₂	1.80	±	0.08	%
LOI	7.45	±	0.66	%

Indicated Means

Cr ₂ O ₃	0.01	%
Na ₂ O	0.09	%

1. **Intended Use:** AMIS0304 can be used to check analysis of samples of rare earth element bearing rocks with a similar grade and matrix.

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

2. **Origin of Material:** AMIS0304 is a commissioned CRM made up of material supplied by Fer-Min-Ore (Pty) Ltd from the Glenover Phosphate Mine on the Glenover Carbonatite Complex. This deposit is located 88km north of Thabazimbi, Limpopo Province, South Africa. The Glenover complex is an irregular pipe like carbonatite body approximately at the centre of a large biotite pyroxenite plug with cone sheets and irregular intrusions of carbonatite emanating from the pipe. Rare Earth Elements are associated with this Carbonatite.

An apatite bearing hematite breccia body, which has been exploited for its phosphate content, is situated at the centre of the biotite pyroxenite, immediately north of the central carbonatite pipe.

3. **Mineral and Chemical Composition:** The central body of the carbonatite is composed of beforite, an ankerite rich rock with crystallographic and physical characters resembling dolomite and siderite. The pyroxenite was an earlier event and comprises mainly decomposed biotite-phlogopite. The pyroxenite has been intruded by carbonatite dykes, sills and cone sheets and, where cut by the carbonatite, it has undergone carbonatisation and is veined with beforite and sovite.

4. **Appearance:** The material is a very fine powder. It is colored a Moderate Red (Corstor 5R 4/8).

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 ore is crushed, then dry-milled and air classified to 100% <54 μ . This fine powder is mixed in a blender for 14 hours and then split down into numbered 1 kg tubs. These lots are sampled for quality control and for round robin analysis. Quality control will typically comprise sampling 30 tubs selected from the whole stream. Round robin samples are selected the same way, so that one laboratory will receive samples from the beginning, end, and from throughout the batch.

7. **Methods of Analysis requested:**

1. Multi-acid digests, including HF, ICP- OES or ICP-MS. Multi element scan.
2. Fusion, ICP- OES or ICP-MS. Multi element scan to include REE's, Nb, Y, Al, Mg, Si, P, Fe, Ca, U and Th.
3. XRF. Multi element scan to include REE's, Nb, Y, Al, Mg, Si, P, Fe, Ca, U and Th.
4. XRF fusion. Majors (Al₂O₃, CaO, Cr₂O₃, Fe₂O₃, K₂O, MgO, MnO, Na₂O, P₂O₅, SiO₂, TiO₂. LOI.)
5. SG (gas pycnometer

8. **Information requested:**

1. State and provide brief description of analytical techniques used.
2. State aliquots used for all determinations.
3. Results for individual analyses to be reported (not averages)
4. All results for Rare Earth Elements to be reported in ppm (not as oxides).
5. All results for multi-element scans to be reported in ppm.
6. All results for major elements to be reported in %, as oxides.
7. Report all QC data, to include replicates, blanks and certified reference materials used.

9. **Method of Certification:** Twenty one laboratories were each given eight packages, comprising eight samples scientifically selected from throughout the batch. Sixteen laboratories reported results in time for certification of the economic elements. Fourteen of these laboratories reported results for the major elements.

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"

Assay data (cont) - Major Oxides and Specific Gravity

Lab Code	Al ₂ O ₃ XRF %	CaO XRF %	Cr ₂ O ₃ XRF %	Fe ₂ O ₃ XRF %	K ₂ O XRF %	MgO XRF %	MnO XRF %	Na ₂ O XRF %	P ₂ O ₅ XRF %	SiO ₂ XRF %	TiO ₂ XRF %	LOI %	SG pyc %
U	1.52	28.73		20.36	0.28	2.77	0.44	0.07	18.67	12.67	1.83	7.88	3.30
U	1.51	28.82		20.42	0.28	2.81	0.44		18.79	12.67	1.82	7.95	3.29
U	1.52	28.45		20.14	0.29	2.76	0.43	0.05	18.67	12.51	1.81	7.90	3.32
U	1.51	28.43		20.18	0.28	2.76	0.43	0.06	18.52	12.46	1.80	7.82	3.29
U	1.54	28.69		20.29	0.29	2.79	0.43	0.09	18.63	12.67	1.83	8.04	3.33
U	1.46	28.51		20.13	0.28	2.77	0.43	0.06	18.68	12.62	1.81	7.94	3.30
U	1.53	28.57		20.14	0.28	2.78	0.43	0.06	18.65	12.52	1.80	7.86	3.31
U	1.54	28.87		20.42	0.29	2.79	0.44	0.07	18.74	12.64	1.83	7.83	3.30

12. Measurement of Uncertainty: The samples used in the certification process were 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
Ce	M/ICP	ppm	346.3	242.7	209.1	76.57
La	M/ICP	ppm	155.3	111.6	89.50	35.01
Nd	M/ICP	ppm	220.9	151.2	114.5	43.47
Pr	M/ICP	ppm	44.31	29.63	22.16	8.529
Sm	M/ICP	ppm	35.05	25.38	14.96	7.195
Th	M/ICP	ppm	33.50	25.57	14.63	7.530
Th	XRF	ppm	35.50	36.13	15.03	13.81
U	M/ICP	ppm	0.832	0.458	0.635	0.155
Y	M/ICP	ppm	19.66	16.40	7.708	5.261
Al	M/ICP	ppm	337.9	247.5	184.4	77.25
Ca	M/ICP	ppm	6716	5620	2943	1808
Fe	M/ICP	ppm	4649	3659	2071	1125
Mg	M/ICP	ppm	877.2	714.9	303.6	218.1
P	M/ICP	ppm	1761	1127	1391	436
P	XRF	ppm	1590	1435	347.0	455.5
Si	M/ICP	ppm	1716	1225	1091	406.5
Al ₂ O ₃	XRF	%	0.037	0.030	0.017	0.010
CaO	XRF	%	0.356	0.281	0.139	0.086
Cr ₂ O ₃	XRF	%	0.002	0.001	0.001	0.001
Fe ₂ O ₃	XRF	%	0.501	0.408	0.142	0.119
K ₂ O	XRF	%	0.009	0.007	0.006	0.002
LOI		%	0.327	0.285	0.064	0.086
MgO	XRF	%	0.059	0.045	0.024	0.013
MnO	XRF	%	0.013	0.012	0.005	0.004
Na ₂ O	XRF	%	0.020	0.017	0.010	0.006
P ₂ O ₅	XRF	%	0.367	0.331	0.079	0.105
SiO ₂	XRF	%	0.202	0.159	0.090	0.049
TiO ₂	XRF	%	0.043	0.037	0.013	0.012
SG	pyc	%	0.046	0.044	0.025	0.017

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 Stc 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 each certificate fulfill the AMIS statistical criteria regarding agreement for certification and have been independently validated by Dr Barry Smee, BSc, PhD, P.Geo, (B.C.).

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, who have maintained measurement traceability during the analytical process.

15. Certification: AMIS0304 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 for the ICP. 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 50g to 250g) of material. The Laboratory Packs are sealed bottles delivered in sealed foil pouches. The Explorer Packs contain material in standard geochem envelopes, 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 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.

10 May 2012 (*Appendix 2 Added: 05 November 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 1– uncertified trace element statistics

Analyte	Method	Unit	Mean	2SD	RSD%	n
Ag	M/ICP	ppm	2.2	0.37	8.6	14
As	M/ICP	ppm	54.3	28.3	26.0	41
Au	M/ICP	ppm	105	5.8	2.8	8
Ba	M/ICP	ppm	2525	298	5.9	48
Be	M/ICP	ppm	35.8	9.9	13.8	48
Bi	M/ICP	ppm	3.1	2.9	47.4	38
Cd	M/ICP	ppm	0.52	0.09	8.5	40
Co	M/ICP	ppm	98.7	8.6	4.4	54
Cr	M/ICP	ppm	86.2	40.0	23.2	46
Cs	M/ICP	ppm	0.43	0.11	13.4	38
Cu	M/ICP	ppm	254	80.6	15.9	48
Ga	M/ICP	ppm	35.1	64.7	92.3	31
Ge	M/ICP	ppm	11.1	5.3	23.8	16
Hf	M/ICP	ppm	16.9	22.6	66.9	64
In	M/ICP	ppm	1.04	0.14	6.9	38
K	M/ICP	%	0.27	0.05	9.6	55
Li	M/ICP	ppm	19.5	11.9	30.4	45
Mn	M/ICP	ppm	3347	405	6.1	52
Mo	M/ICP	ppm	7.2	2.0	13.7	47
Na	M/ICP	%	0.08	0.01	7.0	32
Ni	M/ICP	ppm	53.5	17.4	16.2	45
Pb	M/ICP	ppm	81.9	65.0	39.7	31
Rb	M/ICP	ppm	10.9	1.6	7.5	54
Re	M/ICP	ppm	0.01	0.00	16.5	8
S	M/ICP	%	0.05	0.04	42.2	32
Sb	M/ICP	ppm	203	712	175	32
Se	M/ICP	ppm	19.3	1.1	2.9	8
Sn	M/ICP	ppm	16.2	15.6	48.2	63
Sr	M/ICP	ppm	3515	324	4.6	46
Ta	M/ICP	ppm	5.4	10.7	98.2	64
Te	M/ICP	ppm	0.69	0.12	8.9	22
Ti	M/ICP	%	0.56	0.83	73.6	24
Tl	M/ICP	ppm	304	1797	295	34
V	M/ICP	ppm	332	67.9	10.2	40
W	M/ICP	ppm	3.7	4.3	57.6	40
Zn	M/ICP	ppm	79.4	10.9	6.9	46
Zr	M/ICP	ppm	1002	396	19.8	45

Appendix 2. – Rare Earth Oxide content

AMIS0304 Rare Earth Element content by different reporting conventions (Total, Critical, Light, Medium, Heavy).

TREE	1.8	%	(La+Ce+Pr+Nd+Sm+Eu+Tb+Dy+Yb+Y)
CREE	0.463	%	(Nd+Eu+Tb+Dy+Y)
LREE	1.658	%	La+Ce+Pr+Nd
MREE	739	ppm	(Sm+Eu)
HREE	763	ppm	(Tb+Dy+Yb+Y)

AMIS0304 Rare Earth Oxide content by different reporting conventions.

TREO	2.149	%	(La+Ce+Pr+Nd+Sm+Eu+Tb+Dy+Yb+Y)2O3
CREO	0.54	%	(Nd+Eu+Tb+Dy+Y)2O3
LREO	1.99	%	(La+Ce+Pr+Nd)2O3
MREO	0.086	%	(Sm+Eu)2O3
HREO	0.076		(Tb+Dy+Yb+Y)2O3

Ref: *Rare-Earth Terminology - A Quick Refresher On The Basics*, by Gareth Hatch, December 11, 2012; <http://www.techmetalsresearch.com/2012/12/rare-earth-terminology-a-quick-refresher-on-the-basics/>