Reference Material Data Sheet

IAG FeMn-1 Manganese Nodule

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Description of the reference material

FeMn-1, manganese nodule, was prepared as a candidate reference material by C. Kriete of Bundesanstalt für Geowissenschaften und Rohstoffe (BGR), Hannover, Germany. The material was produced from a mix of nodule samples obtained from the Peru Basin in the Pacific Ocean. Testing for homogeneity was carried out by the originating laboratory and the material considered suitable for use as a supplementary test material in the GeoPT proficiency testing programme. Special instructions were issued to analysts in respect of FeMn-1. On account of the material being hygroscopic, the direction was issued that it must be dried for 48 h at 105 °C and then kept in a desiccator prior to analysis. Warning was given of the high cobalt content and the possibility that the material might sinter during LOI determination. Reference values were characterised for use of this material as a reference material through the GeoPT23A round of the International Association of Geoanalysts' GeoPT proficiency testing scheme undertaken in 2008. This scheme operates in accord with a protocol (IAG, 2002, 2018) that is designed to comply with statistical methods described in ISO 13528:2015. The Proficiency Testing Steering Committee for this round comprised Dr P.C. Webb (results coordinator), Prof. M. Thompson (statistician), Prof. P.J. Potts, J.S. Watson and C. Kriete.

Intended use

This reference material is designed for use by laboratories undertaking the geochemical characterisation of the major and trace element mass fractions of rocks (in particular sediments and concretionary materials of a similar matrix type). Appropriate uses include the calibration of a measurement system, the assessment of a measurement procedure, assigning values to other materials, and quality control. Note that the material may be used only for a single purpose in the same measurement process. For example, it must not be used for both calibration and method validation at the same time.

Minimum sample size

On the basis of the homogeneity results and an assessment of the methods used to contribute results to the GeoPT23A round, the minimum sample size recommended for use as a test portion is 0.2 g.

Period of validity

Provided the storage and handling conditions are met, this reference material is not expected to deteriorate with time. On exposure to air, the material may absorb moisture, and instructions for handling must be followed, especially noting that the material is reported to be hydroscopic. Evidence for the stability of typical GeoPT test materials and of the scheme itself have been presented by Thompson et al. (2018) and Webb et al. (2019).

Storage information

Store in a sealed container in a cool dry environment, noting the instructions for handling in the next section.

Instructions for handling

Before any measurements are made, every test portion of the material must be dried at 105 ± 5°C for at least 48 hours and stored in a desiccator, if necessary, prior to use. Avoid contamination and cross-contamination of the test material. Note that the material has a high cobalt content (which may affect neutron activation measurements, for example) and the test material may sinter during loss on ignition measurements.

Assessment of reference values

*The reference values (**Table 1**) were derived as consensus values from a rigorous assessment of measurement results submitted by participating laboratories to the GeoPT23A proficiency testing round, as reported by Webb et al. (2008). Consensus values were determined as a robust mean if the distribution of results was unimodal and, outliers aside, close to symmetrical. If some asymmetry was apparent in a unimodal distribution, the median was usually preferred as the consensus value. Sometimes, especially when a noteworthy skew was apparent in the data distribution and an objective explanation for this outcome was forthcoming, the mode of the results might be used. If the number of valid results contributing to the location was less than 15 or their dispersion was unusually great, no reference value was assigned, although values may be reported as indicative values (**Table 2**). These judgements were made in accordance with the revised GeoPT protocol (IAG, 2018) by the IAG Proficiency Testing Steering Committee.*

Metrological traceability

Traceability was not formally demonstrated for this reference material. However, traceability can be demonstrated through laboratories participating in this round using certified reference materials as calibrators or for data assessment (although this information is not currently recorded by the GeoPT programme) and is implied by the overall agreement of contributed data for individual elements/oxides submitted to the programme from which robust consensus values were derived. A review of all the factors that contribute to demonstrating the traceability of consensus values derived from the GeoPT proficiency testing scheme has been described by Potts et al. (2019).

Reference to characterisation report

Further details of the results and organisation of the round can be found in the report of the GeoPT23A proficiency testing round (http://www.geoanalyst.org/geopt-previous-rounds/), noting that a reassessment of data and the application of statistical procedures were undertaken in accordance with the revised GeoPT protocol (IAG, 2018).

Safety information

Silicate powders containing heavy metals can cause harm especially if inhaled or in contact with the skin. User organisations must undertake a health and safety risk assessment and ensure that the appropriate procedures are followed in the handling and use of this material. Further details are available on the relevant Material Safety Data Sheet, which is available from the supplier on request.

Legal notice – terms and conditions

1. The IAG shall not be liable to the user of this material for loss (whether direct or indirect) of profits, business, anticipated savings or reputation or for any indirect or consequential loss or damage whatsoever even if previously advised thereof and whether arising from negligence, breach of these Terms and Conditions or howsoever occurring.

2. In any event, and notwithstanding anything contained in these Terms and Conditions, IAG's liability in contract, tort (including negligence, defamation or breach of statutory duty) or otherwise arising by reason of or in connection with these Terms and Conditions (including as a result of proficiency testing) shall be limited to the price paid for the material giving rise to such liability.

3. The IAG does not grant any warranties in relation to Geo*PT* products or the supply of analytical services or distribution of the proficiency test, and all other conditions, warranties, stipulations or other statements whatsoever, whether express or implied, by statute, at common law or otherwise howsoever, relating to the Geo*PT* products, analytical services or proficiency tests are hereby excluded. In particular, (but without limitation to the foregoing) no warranties are granted regarding the fitness for purpose, performance, use, quality or merchantability of the Geo*PT* products, whether express or implied, by statute, at common law or otherwise howsoever.

Revisions

Any revisions to this reference material data sheet will made available on the IAGeo Limited website (www.iageo.com).

Acknowledgements

Colleagues at the Bundesanstalt für Geowissenschaften und Rohstoffe, under the direction of Connie Kriete are gratefully acknowledged for assistance with this round of the GeoPT proficiency testing programme.

Approvals

This reference material information sheet was approved on behalf of the International Association of Geoanalysts.

Name *Philip J. Potts* **Date** **21st May 2020**
Peter C. Webb

References

IAG (2002)
Protocol for the operation of the GeoPT proficiency testing scheme.
International Association of Geoanalysts (Keyworth, Nottingham), 12pp.

IAG (2018)
Protocol for the operation of the GeoPT proficiency testing scheme.
International Association of Geoanalysts (Keyworth, Nottingham), 18pp.

ISO 13528:2015
Statistical methods for use in proficiency testing by interlaboratory comparison.
International Organisation for Standardisation (Geneva), 89pp.

Potts P.J., Webb P.C. and Thompson M. (2019)
GeoPT proficiency testing programme as a scheme for the certification of geological reference materials.
Geostandards and Geoanalytical Research, 43, 409-418.

Thompson M., Webb P.C., Potts P.J. and Wilson S. (2018)
The stability of 57 consensus values in a proficiency test material re-issued blind after an interval of 18 years.
Analytical Methods, 10, 1547-1551.

Webb P.C., Thompson M., Potts P.J., Watson J.S. and Kriete C. (2008)
GeoPT23 - an international proficiency test for analytical geochemistry laboratories - report on round 23 / September 2008 (Separation Lake pegmatite, OU-9) and 23A (Manganese nodule, FeMn-1).
International Association of Geoanalysts, 64pp (http://www.geoanalyst.org/wp-content/uploads/2017/10/GeoPT23FullReport.pdf).

Webb P.C., Potts P.J., Thompson M., Wilson S.A. and Gowing C.J.B. (2019)
The long term robustness and stability of consensus values as composition location estimators for a typical geochemical test material in the GeoPT proficiency testing programme.
Geostandards and Geoanalytical Research, 43, 397-408.

Table 1

IAG FeMn-1 Manganese Nodule
Reference values

Certified values for elemental/oxide concentrations and uncertainties on a dried (105 °C) basis

Oxide / element	Reference value g 100g^{-1}	Uncertainty g 100g^{-1}	p		Element	Reference value mg kg^{-1}	Uncertainty mg kg^{-1}	p
MgO	2.82	0.06	52		**Nb**	13.2	0.3	47
CaO	2.50	0.05	55		**Nd**	63.0	1.7	42
	mg kg^{-1}	**mg kg^{-1}**			**Pb**	126	5	57
Ce	110	4	45		**Pr**	14.1	0.5	35
Cs	0.82	0.06	30		**Rb**	12.2	0.6	47
Dy	15.9	0.5	34		**Sm**	14.1	0.3	38
Er	9.81	0.30	33		**Sr**	680	15	62
Eu	3.82	0.25	36		**Tb**	2.52	0.10	35
Gd	15.6	0.6	34		**Th**	6.80	0.25	43
Hf	4.74	0.12	34		**Tm**	1.52	0.12	32
Ho	3.42	0.11	35		**U**	4.49	0.15	39
La	68.1	1.7	45		**Y**	69.4	2.2	54
Li	299	11	28		**Yb**	10.1	0.3	36
Lu	1.59	0.05	34		**Zr**	323	9	53

Reference values *are the GeoPT assigned values assessed from the robust statistical analysis of results submitted to the GeoPT23A round, following assessment of both the consistency of the data distribution and the agreement between methods, where possible (see text).*

Uncertainties *are the robust standard deviation of the mean, median or mode of the assigned value expanded by a coverage factor of two.*

p *is the number of laboratories reporting results for that element/oxide in the GeoPT23A round.*

Table 2

IAG FeMn-1 Manganese Nodule
Indicative values

Indicative values for elemental/oxide concentrations and uncertainties on a dried (105 °C) basis

Oxide / element	Indicative value g 100g^{-1}	Uncertainty g 100g^{-1}	p		Element	Indicative value mg kg^{-1}	Uncertainty mg kg^{-1}	p
SiO_2	10.40	0.2	49		**Cd**	19.8	1.5	34
TiO_2	0.285	0.014	58		**Co**	475	16	57
Al_2O_3	2.92	0.08	53		**Cu**	5890	175	62
Fe_2O_3T	8.67	0.22	58		**Mo**	572	16	51
MnO	44.21	0.63	58		**Ni**	13100	275	57
P_2O_5	0.352	0.012	49		**Sb**	60.8	3.8	33
	mg kg^{-1}	**mg kg^{-1}**			**Sc**	7.58	0.84	36
As	74.5	5.1	39		**Ta**	0.26	0.04	25
Ba	3165	110	60		**Tl**	129	7	30
Be	1.49	0.21	19		**V**	469	27	53
Bi	3.42	0.44	27		**Zn**	1885	60	59

Indicative values *are 'provisional' data from the relevant GeoPT23A report where, data distributions were judged not to be good enough to meet the criteria for designation as reference values. These data are provided for information purposes only and* ***not*** *for the calibration of methods or the assessment of data.*

Uncertainties *are the robust standard deviation of the mean, median or mode expanded by a coverage factor of two.*

p *is the number of laboratories reporting results for that element/oxide in the GeoPT23A round.*

Fe_2O_3T *is the total iron expressed as Fe_2O_3.*