

CCRMP Canadian Certified Reference Materials Project

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PCMRC Projet canadien de matériaux de référence certifiés

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Certificate of Analysis

First issued: July 1994

Version: May 2007

NBM-1

Certified Reference Material for Acid Base Accounting

Table 1 - NBM-1 certified values

Test	Units	Mean	Between- Labs SD	Within- Lab SD	95% confidence interval of mean
AP-MS	kgCaCO₃/t	8.48	0.95	0.44	± 0.57
AP-S	kgCaCO ₃ /t	8.73	0.81	0.35	± 0.35
Paste pH	pН	8.45	0.11	0.04	± 0.05
S	%	0.28	0.03	0.01	± 0.01

Table 2 - Acronyms for acid base accounting tests

Sobek and modified Sobek methods			
AP	Acid potential		
NP	Neutralization potential		
S	Sobek method		
MS	Modified Sobek method		
m	Moderate fizz rating		
S	Slight fizz rating		

Table 3 - NBM-1 method-specific values

Test	Units	Mean	Between-Labs SD	Within- Lab SD	95% confidence interval of mean
NP-S-m	kgCaCO₃/t	72.1	8.5	2.1	± 10.7
NP-S-s	kgCaCO₃/t	49.6	3.0	1.8	± 1.9



Canada

Canada

Test	Units	Mean	SD
NP-MS-m	kgCaCO3/t	52.3	1.4
NP-MS-s	kgCaCO3/t	46.6	10.1
S(SO ₄)	%	0.02	0.002

Table 4 - NBM-1 informational values

Table 5 - NBM-1 informational values for analytes

Analytes	Units	Mean	SD
AI	%	7.86	0.09
Ва	%	0.117	0.001
С	%	0.79	0.08
C (CO ₃)	%	0.50	0.18
Са	%	2.30	0.02
Fe	%	4.09	0.03
К	%	2.36	0.18
Loss on ignition	%	3.45	0.33
Loss of moisture	%	0.32	0.02
Mg	%	1.39	0.02
Mn	%	0.046	0.001
Na	%	2.70	0.13
Ρ	%	0.10	0.00
Si	%	28.47	0.10
Ti	%	0.335	0.006

SOURCE

The raw material used to prepare NBM-1 was a biotic altered feldspar porphyry non-ore grade pit rock from the Bell Mine in Granisle, British Columbia and was donated by Noranda Minerals Incorporated.

DESCRIPTION

Major species in NBM-1 include sodium-plagioclase (30.7%), orthoclase (27.9%), quartz (21.8%), biotite (6.7%), kaolinite (3.7%), hematite plus magnetite (3.9%), siderite (2.5%), and ankerite (1.5%). Minor species include chalcopyrite (0.3%), and calcite, apatite, bornite, pyrite, and rutile, each with a concentration of 0.2%. Also, it was estimated visually that the weight ratio of hematite to magnetite is about 3:1.

INTENDED USE

NBM-1 is suitable for the analysis of rocks for sulphur and various static prediction tests for acid base accounting by the Sobek and modified Sobek methods as described in reference 1. Examples of intended use are for quality control in the analysis of samples of a similar type, method development, environmental assessment and the calibration of equipment.

INSTRUCTIONS FOR USE

NBM-1 should be used "as is", without drying. The contents of the bottle should be thoroughly mixed before taking samples. The contents of the bottle should be exposed to air for the shortest time possible. Unused material should be stored under an inert gas in a desiccator, or in a new, heat-sealed laminated foil pouch. The values herein pertain to the date when issued. CANMET is not responsible for changes occurring after receipt by the user.

HANDLING PRECAUTIONS

Normal safety precautions for handling fine particulate matter are suggested, such as the use of safety glasses, breathing protection, gloves and a laboratory coat.

METHOD OF PREPARATION

The raw material was crushed, ground, and sieved to separate a minus 74 µm fraction which was blended. The yield was 86%. The product was bottled in one size, 100-gram units. Each bottle was sealed under nitrogen in a laminated aluminum foil-mylar pouch to prevent oxidation.

HOMOGENEITY

The homogeneity of the stock with respect to iron and sulphur was investigated using twenty-two bottles chosen according to a stratified random sampling scheme. Two splits were analysed from each bottle for both elements. Samples of 0.25 g were analysed for sulphur using a combustion analyzer. For iron analysis, samples of 1.25 g were digested with four acids and a titration was performed with potassium dichromate after a stannous chloride reduction. Use of a smaller sub-sample will invalidate the use of the certified values and associated parameters. A one–way analysis of variance technique (ANOVA) was used to assess the homogeneity of these elements ⁽²⁾. The ratio of the between-bottles to within-bottle mean squares was compared to the F statistic at the 95% level of probability. No evidence of inhomogeneity was observed for either element.

CERTIFIED VALUES

Twenty-six industrial, commercial, and government laboratories participated in an interlaboratory measurement program. Gravimetric and combustion methods for sulphur, and various static tests for acid base accounting ⁽¹⁾ involving wet chemistry were performed at the discretion of each laboratory. A one-way analysis of variance technique was used to estimate the consensus value and other statistical parameters ⁽²⁾. The certified value listed for each element or test is the best estimate of the "true" value based on the results of the interlaboratory measurement program. The mean values for AP-MS, AP-S, sulphur and paste pH were certified (see Table 1).

Full details of all phases of the work, including the statistical analyses, the methods and the names of the participating laboratories are contained in CCRMP Report 01-1E, Version 2.

UNCERTIFIED VALUES

Two tests for acid base accounting, NP-S-m and NP-S-s, were given "method-specific" values (see Table 3). "Method-specific" refers to the use of the Sobek and modified Sobek methods for acid base accounting, as described in reference 1. The term "method-specific", is not equivalent to "certified".

The value for NP-MS-m was derived from two sets of data and is therefore an "informational value." The value for NP-MS-s derived from sixteen sets of data was given the rating of an "informational value" due to the standard deviation (see Table 4).

Informational values for fifteen elements, shown in Table 5, were derived from the means of fewer than four sets of results.

TRACEABILITY

The values quoted herein are based on the consensus values derived from the statistical analysis of the data from the interlaboratory measurement program.

CERTIFICATION HISTORY

NBM-1 was originally released in 1994 with values for sulphur, NP-S-s. In 2002 a new certificate was issued with values for several acid base accounting tests as a result of an additional interlaboratory measurement program. This 2007 version of the certificate was issued due to the expiration of its predecessor. Based upon a reassessment of the data, two means, AP-MS and NP-S-m, have minor changes. The statistical parameters for some parameters have been revised also. The principle mineralogy of the source material has been changed to correct a typographical error. Additional information in the text has been included in accordance with ISO Guide 31:2000.

PERIOD OF VALIDITY

These certified values are valid until December 31, 2030. The stability of the material will be monitored every two years for the duration of the inventory. Updates will be made via CCRMP web site.

LEGAL NOTICE

CANMET has prepared this reference material and statistically evaluated the analytical data of the interlaboratory measurement program to the best of its ability. The purchaser, by receipt hereof, releases and indemnifies CANMET from and against all liability and costs arising out of the use of this material and information.

CERTIFYING OFFICER

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FOR FURTHER INFORMATION

The NBM-1 certification report is available free of charge upon request to:

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REFERENCES

- 1. Coastech Research (1991), Acid Rock Drainage Prediction Manual, the Mine Environment Neutral Drainage (MEND) Program (Project 1.16.1b).
- 2. Brownlee, K.A., Statistical Theory and Methodology in Science and Engineering; John-Wiley and Sons, Inc.; New York; 1960.