



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: September 2004

Date of this certificate: September 2004

TPO-1

Certified Reference Material for Iron Sulphide Concentrate and Tailings

Table 1 Certified Values

Element	Unit	Mean	Within-Lab Standard Deviation	Between-Labs Standard Deviation	95% Confidence Limit
Co	%	0.021	0.002	0.003	± 0.002
Cu	%	0.118	0.004	0.006	± 0.003
Fe	%	34.85	0.19	0.32	± 0.16
Ni	%	0.617	0.009	0.019	± 0.010
S	%	18.03	0.14	0.25	± 0.14
SiO ₂	%	25.52	0.40	0.36	± 0.20

DESCRIPTION

TPO-1 is a mixture of iron sulphide concentrate and tailings from the Clarabell Mill that was originally prepared as a custom certified reference material for Inco Limited, Copper Cliff, Ontario, Canada in 1998. Inco agreed to donate some of the custom certified reference material to Natural Resources Canada.

The raw material was dried at 60°C for 30 hours, crushed, sieved, and blended to obtain a minus 150 micron (100 mesh) product. The yield was 86%. The material comes in glass bottles containing 25-30 g each. This is the only size available. Each bottle was sealed under nitrogen in a laminated aluminum foil-mylar pouch to prevent oxidation.

INTENDED USE

TPO-1 is suitable for the analysis of elements at major, minor and trace levels. Examples of intended use are for quality control in the analysis of samples of a similar type, method development, arbitration and the calibration of equipment.

INSTRUCTIONS FOR THE CORRECT USE OF THE REFERENCE MATERIAL

The assigned values pertain to the date when issued. TPO-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 possible time. After opening the sealed pouch, the bottle should be kept in a desiccator, or preferably, resealed under nitrogen in a new heat-sealed laminated foil pouch to prevent oxidation.

HAZARDOUS SITUATION

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

LEVEL OF HOMOGENEITY

The homogeneity of the stock with respect to its copper, nickel and sulphur was investigated using twenty-two bottles chosen according to the bottling sequence and a stratified random sampling scheme. Two splits were analysed from each bottle. The analyses for copper, nickel and sulphur were performed. The method used for the analysis of copper and nickel was digestion of a 0.5 g-sample with hydrochloric, nitric, hydrofluoric and perchloric acids followed by determination by atomic absorption spectroscopy. The method for the analysis of sulphur was combustion on a 0.5 g sample using a combustion apparatus with infrared detection.

A one-way analysis of variance technique (ANOVA) was used to assess the homogeneity of these elements (1). The ratio of the between-bottles to within-bottle mean squares is compared to the F statistic at the 95% level of probability. No evidence of inhomogeneity was observed for all three elements. Use of a smaller mass will invalidate the use of the certified value and associated parameters.

CERTIFIED VALUES AND THEIR UNCERTAINTIES

Eighteen government, industrial and commercial laboratories participated in the interlaboratory measurement program. Cobalt, copper, iron, nickel and silicon were analyzed by methods of each laboratory's choice. Methods included multi acid digestions, fusion, electroplating, atomic absorption spectroscopy, combustion, titration, gravimetric methods, X-ray fluorescence and inductively coupled plasma - atomic emission spectroscopy. A one-way analysis of variance technique (ANOVA) was used

to estimate the consensus value and other statistical parameters (1). Table 1 contains the means and associated statistical parameters for the six certified elements.

UNCERTIFIED VALUES

Table 2 contains the informational values derived from semi-quantitative analysis.

Table 2 - Informational Values

Element	Unit	Mean
Al	%	3.51
Ca	%	2.17
Cr	%	0.03
K	%	0.56
Mg	%	1.66
Mn	%	0.08
Na	%	0.85
P	%	0.03
Pb	%	0.02
Ti	%	0.35
Zn	%	0.02

TRACEABILITY

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

DATE OF CERTIFICATION

TPO-1 was certified in 1998 and is being added to the CCRMP inventory with the issuance of this certificate.

PERIOD OF VALIDITY

These certified values are valid until December 2009. The stability of the material will be monitored every two years. Purchasers will be notified of any significant changes.

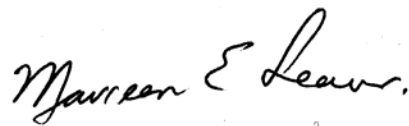
LEGAL NOTICE

CANMET - Mining and Mineral Sciences Laboratories (MMSL) has prepared this reference material and statistically evaluated the analytical data of the interlaboratory certification program to the best of its ability. The purchaser, by receipt hereof, releases and indemnifies CANMET- MMSL from and against all liability and costs arising out of the use of this material and information.

CERTIFYING OFFICERS



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FOR FURTHER INFORMATION

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REFERENCE

1. Brownlee, K.A., Statistical Theory and Methodology in Science and Engineering; John-Wiley and Sons, Inc.; New York; 1960