

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

Version: January 2009

TLS-1

Certified Reference Material for Unoxidized Tailings

Element	Unit	Mean	Within-lab Standard Deviation	Between-labs Standard Deviation	95% Confidence Limit
Cu	%	0.078	0.002	0.005	± 0.002
Fe	%	10.51	0.23	0.34	± 0.15
Ni	%	0.151	0.004	0.007	± 0.003
S	%	1.81	0.03	0.04	± 0.03

Table 1 - Certified Values

Table 2 - Provisional Values

Constituent	Unit	Mean	Within-Lab Standard Deviation	Between-labs Standard Deviation	95% Confidence Limit
Со	%	0.008	0.002	0.002	± 0.002
SiO ₂	%	51.87	0.47	1.48	± 0.69



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Element	Unit	Mean
AI	%	6.92
Са	%	4.73
Cr	%	0.030
K	%	1.025
Mg	%	3.45
Mn	%	0.120
Na	%	1.690
Р	%	0.087
Pb	%	0.023
Ti	%	0.553
Zn	%	0.013

Table 3 - Informational Values

DESCRIPTION

TLS-1 is a tailings material from the Clarabell Mill originally prepared as a custom certified reference material for Inco Limited, Copper Cliff, Ontario, Canada in 1998. The material is the waste product from the production of a copper-nickel sulphide concentrate in the form of a fine powder. Inco Limited agreed to donate some of the custom certified reference material to CANMET, Natural Resources Canada.

The raw material was dried at 93° C for five hours and allowed to return to ambient temperatures overnight. It was then crushed, sieved and blended to obtain a minus and plus 150 µm (100 mesh) fraction. The plus material was ground, sieved and blended to obtain second minus 150 µm fraction. These two minus fractions were then combined. The yield was 80%. The material comes in glass bottles containing 100 g each. This is the only size available.

INTENDED USE

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

INSTRUCTIONS FOR USE

The assigned values pertain to the date when issued. TLS-1 should be used "as is", without drying. The contents of the bottle should be thoroughly mixed before taking samples.

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 final product was investigated using twenty-two bottles of TLS-1 chosen according to a stratified random sampling scheme. Two samples were analyzed from each bottle in a randomized order. Samples of 0.5 g were digested in nitric, hydrochloric, perchloric and hydrofluoric acids and the determination of copper and nickel was performed using atomic absorption spectroscopy.

Samples of 0.5 g were analyzed for sulphur by combustion using a LECO apparatus with infrared detection.

A one-way analysis of variance technique (ANOVA) was used to assess the homogeneity of these elements¹. 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 copper, nickel and sulphur. Use of a smaller mass will invalidate the use of the certified values and associated statistical parameters.

CERTIFIED VALUES

Eighteen industrial, commercial, and government laboratories participated in an interlaboratory measurement program. Various elements were analyzed by methods selected by the individual laboratories. These methods were to be selected so as to yield highly accurate and precise values. A one-way analysis of variance technique was used to estimate the consensus value and other statistical parameters¹. 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 parameters¹. Table 1 contains the means and associated statistical parameters for the six certified elements.

UNCERTIFIED VALUES

Table 2 contains the provisional elements which did not meet either one or both of the two criteria for certification. Table 3 contains the informational values derived from semiquantitative analysis.

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

TLS-1 was prepared in 1998 for Inco Limited. In 2004 Inco donated some of the material to Natural Resources Canada to sell.

PERIOD OF VALIDITY

These certified values are valid until December 31, 2030. Updates will be published on the CCRMP web site.

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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REFERENCE

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