



**CCRMP**  
Canadian Certified Reference Materials Project

CANMET Mining and Mineral Sciences Laboratories  
555 Booth Street, Ottawa, Ontario, Canada K1A 0G1  
Tel.: (613) 995-4738, Fax: (613) 943-0573  
E-mail: ccrmp@nrcan.gc.ca  
www.ccrmp.ca

**PCMRC**  
Projet canadien de matériaux de référence certifiés

Laboratoires des mines et sciences minérales de CANMET  
555, rue Booth, Ottawa (Ontario) Canada K1A 0G1  
Tél. : (613) 995-4738, Téléc. : (613) 943-0573  
Courriel : pcmrc@nrcan.gc.ca  
www.pcmrc.ca

# Certificate of Analysis

First issued: September 2004

Version: January 2010

## TPO-1

### Certified Reference Material for Iron Sulphide Concentrate and Tailings

**Table 1 - Certified Values**

Constituent	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 <sub>2</sub>	%	25.52	0.40	0.36	± 0.20



**Table 2 - Informational Values**

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

**DESCRIPTION**

TPO-1 is a mixture of iron sulphide concentrate and tailings from the Clarabelle 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 25-30g 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 USE**

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 0.5g-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

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.

### 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 for Inco Limited. In 2004 the material was added to the inventory and a certificate was issued. The September 2004 version of the certificate was re-issued in January 2010 with no changes due to the expiration of the former.

### PERIOD OF VALIDITY

These certified values are valid until January 31, 2032. The stability of the material will be monitored every two years. Updates will be published on the CCRMP web site.

### LEGAL NOTICE

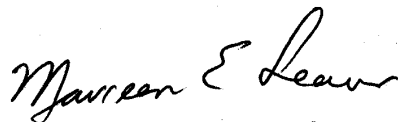
CANMET - Mining and Mineral Sciences Laboratories (MMSL) has prepared this reference material and statistically evaluated the analytical data of the interlaboratory certification program to the best of its ability. The purchaser, by receipt hereof, releases and indemnifies CANMET- MMSL from and against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

### CERTIFYING OFFICERS



---

Joseph Salley



---

Maureen E. Leaver

**FOR FURTHER INFORMATION**

**CCRMP**  
**CANMET - MMSL (NRCan)**  
**555 Booth Street**  
**Ottawa, Ontario, Canada K1A 0G1**  
**Telephone: (613) 995-4738**  
**Facsimile: (613) 943-0573**  
**E-mail: [ccrmp@nrcan.gc.ca](mailto:ccrmp@nrcan.gc.ca)**

**REFERENCE**

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