

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@rncan.gc.ca www.pcmrc.ca

# **Certificate of Analysis** Version: January 2010

First issued: September 2004

CH-4

## Gold Ore Certified Reference Material

Element	Unit	Mean	Within-Lab Standard Deviation	Between- Labs Standard Deviation	Expanded Uncertainty at 95% Confidence
Ag	µg/g	2.1	0.2	0.2	0.1
Au	µg/g	0.88	0.04	0.04	0.05
Cd	µg/g	1.14	0.08	0.15	0.10
Со	µg/g	26	2	1	1
Cu	%	0.20	0.01	0.01	0.00
Fe	%	5.42	0.07	0.18	0.07
K	%	1.81	0.05	0.12	0.05
Mg	%	1.43	0.01	0.03	0.02
S	%	0.63	0.02	0.06	0.04
Se	µg/g	2.1	0.2	0.4	0.2
Ti	%	0.31	0.01	0.01	0.01
Zn	%	0.020	0.001	0.002	0.00

### Table 1 - Certified Values

Element	Unit	Mean	Within Lab- Standard Deviation	Between- Labs Standard Deviation	95 % Confidence Limit
AI	%	7.73	0.08	0.45	0.22
As	µg/g	8.8	0.5	1.7	0.6
С	%	0.12	0.01	0.01	0.01
Ca	%	1.96	0.04	0.13	0.03
Cr	µg/g	114	4	11	7.12
Mn	%	0.043	0.002	0.005	0.00
Na	%	3.26	0.06	0.55	0.06
Ni	µg/g	51	1	4	2.59
Р	%	0.061	0.005	0.006	0.01
Sb	µg/g	0.77	0.18	0.58	0.40
SiO <sub>2</sub>	%	63.10	0.31	1.04	0.47

Table 2 - Provisional Values

Table 3 - Informational Values

			Standard				Standard
Element	Unit	Mean	Deviation	Element	Unit	Mean	Deviation
Ba	µg/g	425	60	Nd	µg/g	16	1
Bi	µg/g	0.6	0.2	Pb	μg/g	14	7
Ce	µg/g	35	5	Pr	µg/g	4	0.3
Cs	µg/g	2	0.3	Rb	µg/g	79	4
Dy	µg/g	2	0.2	Sc	µg/g	13	1
Er	µg/g	1.2	0.1	Sm	µg/g	3	0.2
Eu	µg/g	0.7	0.07	Sr	µg/g	209	20
Ga	µg/g	18	1	Та	µg/g	0.3	0.08
Gd	µg/g	3	0.2	Tb	µg/g	0.4	0.05
Hf	µg/g	3	0.2	Th	µg/g	2	0.2
Hg	µg/g	0.03	0.007	TI	µg/g	0.4	0.09
Но	µg/g	0.4	0.03	Tm	µg/g	0.2	0.01
La	µg/g	16	3	U	µg/g	0.7	0.3
Li	µg/g	12	2	V	µg/g	87	6
LOI	%	0.9	0.2	W	µg/g	3	1
Lu	µg/g	0.2	0.02	Y	µg/g	11	3
Мо	µg/g	3	1	Yb	µg/g	1	0.07
Nb	µg/g	4	0.5	Zr	µg/g	117	7

LOI = loss on ignition

#### DESCRIPTION

The source material for CH-4 was donated by Corporation Minière Inmet, Division Troilus, Chibougamau, Quebec in 2000. After crushing, milling, sieving and blending, the yield was 37%. The material is a fine powder with a mesh size of less than 45 µm (325 mesh) which comes in a glass bottle containing 200 g. This is the only size available. The host rock of the raw material is meta-anorthosite. The mineralogy includes pyrrhotite, pyrite and chalcopyrite, and small amounts of sphalerite, galena and molybdenite.

#### INTENDED USE

CH-4 is suitable for use in applications involving the analysis of various elements in gold ores with a siliceous matrix. 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

CH-4 should be used "as is", without pre-treatment. The contents of the bottle should be thoroughly mixed before taking samples. The material can be stored at room temperature and pressure with no special precautions.

#### 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

Twenty-two bottles of CH-4 were chosen according to a random stratified sampling scheme. Samples were analyzed from two splits taken from each selected bottle. Gold and silver in 30-g samples were determined using fire assay fusion with lead collection followed by atomic absorption spectroscopy (FAA). Samples of 0.25 g were digested in hydrochloric, nitric, perchloric and hydrofluoric acids and the resulting solution was analyzed for copper using FAA. Samples of 1 g were analyzed for carbon and sulphur using a combustion furnace.

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 these five elements. Further details are available in the certification report. Use of a sample mass smaller than indicated for each element will invalidate the statistical parameters contained herein.

#### **CERTIFIED VALUES**

Thirty-three industrial, commercial, and government laboratories participated in an interlaboratory measurement program. Gold was analyzed by a variety of fire assay methods and instrumental neutron activation. Silver was analyzed by a variety of acid digestions, fire assay and instrumental neutron activation. Other elements were analyzed by various acid digestions, fusion, x-ray fluorescence, inductively coupled plasma – optical emission spectroscopy, inductively coupled plasma – mass spectroscopy and instrumental neutron activation.

A one-way analysis of variance technique was used to evaluate the data with regards to the criteria for certification and to estimate the consensus value and other statistical parameters (1). The two criteria for certification involve the agreement of within- and between-laboratories standard deviations and the number of sets with acceptable agreement. Table 1 contains the means and associated statistical parameters for the certified elements. The expanded uncertainty at 95% confidence was calculated for the certified values from the variance derived from the interlaboratory program and the homogeneity assessment, if performed, as in the case of gold, silver, copper, and sulphur. Full details of all phases of the work, including statistical analysis, the methods and the names of the participants are contained in CCRMP Report 04-2E.

#### **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 calculated from the mean of two or more sets of results which were considered to be in good agreement.

#### 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

The first issue of CH-4 was September 2004. The May 2005 certificate contains revised certified or provisional values for aluminum, arsenic, iron, magnesium, potassium, silica, sodium and sulphur. Also, the expanded uncertainty at 95% confidence instead of the 95% confidence interval is used. The May 2005 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 December 31, 2032. The stability of the material will be monitored every five 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**

Joreph Salley

Joseph Salley

Maureen E Leaver.

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

#### Reference

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