ANALYTICAL REFERENCE MATERIALS INTERNATIONAL

High Purity Alloying Nickel, Grade: **HPNi-MI**

Mid-Level Impurities Added

Part Number (Q.A. No.): **IARM-189A**

CERTIFICATE OF A *CERTIFIED REFERENCE MATERIAL*

	# Labs	# Labs Used	Range µg/g	Average Value µg/g	Std Dev	Homo Std Dev	Certified Value µg/g	Confidence Interval
Ag	14	11	2 - 3	2.39	0.33	0.22	2.4	0.2
Al	9	6	34 - 50	44	5	-	44	6
As	17	12	0.3 - 1.6	0.69	0.39	0.19	0.7	0.3
B	5	4	(<2 - 3)	(<5)	-	-	(<5)	-
Be	3	3	<0.1 - <1	(<1)	-	-	(<1)	-
Bi	15	15	1.8 - 3.3	2.62	0.46	0.23	2.6	0.3
C	10	9	18 - 31	22.7	4.4	-	23	4
Ca	5	5	1 - 10	(<10)	(4)	-	<10	(5)
Cd	8	6	0.3 - 1.0	0.75	0.27	-	0.8	0.3
Co	13	9	1.0 - 5.0	3.1	1.2	0.53	3.1	0.9
Cr	9	5	(1 - 22)	(10)	(9)	-	(10)	(9)
Cu	14	14	7.0 - 12.7	9.0	1.5	0.82	9.0	0.9
Fe	15	10	35 - 40	37.9	1.5	1.2	38	1
Ga	9	8	0.09 - 0.5	<0.5(0.2)	0.2	0.07	<0.5	0.2
Mg	6	5	6 - 10	(7.7)	(1.5)	-	(8)	(2)
Mn	9	6	1.6 - 2.0	1.9	0.15	-	1.9	0.2
Mo	4	2	<1	(<1)	-	-	(<1)	-
N	6	5	0.3 - 2.0	1.1	0.6	-	(1)	(1)
Ni	-	-	-	BAL	-	-	BAL	-
O	6	5	13 - 24	(18)	(4)	-	(18)	(5)
P	14	12	2 - 5.3	3.72	0.93	0.58	3.7	0.6
Pb	16	15	2.1 - 3.8	2.86	0.42	0.18	2.9	0.3
S	10	8	0.8 - 4.0	1.8	1.3	-	1.8	1.1
Sb	18	16	1.6 - 6.2	3.9	1.1	-	3.9	0.6
Se	14	14	1.0 - 3.3	2.06	0.80	0.53	2.1	0.5
Si	11	6	15 - 24	18.8	3.1	2.6	19	3
Sn	14	13	1.5 - 3.0	2.2	0.4	0.23	2.2	0.3
Te	15	13	0.8 - 2.3	1.70	0.48	0.38	1.7	0.3
Ti	7	6	2.3 - 4.0	(3.3)	(0.6)	-	(3.3)	(0.7)
Tl	6	6	0.6 - 3.5	2.30	0.54	-	2.3	0.6
V	5	3	<0.1 - 0.2	(<0.5)	-	-	(<0.5)	-
Zn	15	13	2.0 - 4.0	2.80	0.76	0.64	2.8	0.5

Notes: All values are expressed in micrograms per gram (µg/g). Confidence Interval is at the 95% level.

"Homo Std Dev" is the standard deviation from the analysis of 10 randomly selected samples by the glow discharge/mass spectrometry method. This value gives an indication of the homogeneity of the material.

Values in parentheses ( ) indicate that insufficient results were available for certification. These values are given for information only and therefore caution must be exercised in using the data given.

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The Certification of Nickel and Cobalt Reference Materials

In 1992 Inco Limited (Ontario Division) initiated a program with Analytical Reference Materials Inc. to prepare six samples of nickel and one of cobalt with a view to certification as standard reference materials. Fourteen trace elements were of particular interest. Currently there are no certified reference materials for nickel and cobalt covering a range of trace elements.

Three major categories of sample were selected:

1. Four samples. One high purity nickel produced by melting of nickel pellet produced by the carbonyl process, and three samples with low (0.5-5µg/g), medium (5-10µg/g), and high (10-30µg/g) levels of trace elements. The three spiked samples were prepared from carbonyl nickel pellet.

2. Two samples. One prepared by melting nickel produced by the electrolytic process and one containing trace elements in the 1-5µg/g range, except for sulfur at 165µg/g range.

3. One sample of electrolytic cobalt with trace elements in the 0.5-5-µg/g range.

<u>Preparation by Melting</u>

About 320 lbs of nickel (or cobalt) were melted under vacuum in a magnesia crucible. Trace elements were added by wrapping weighed amounts in foil and plunging into the melt under a low pressure of argon. Each heat was cast into a graphite molds to produce two inch diameter circular billets. Each billet was ground by hand to remove surface oxides and hot rolled in three stages into rods of about 1.3 inches diameter. Each heat produced five or six master lengths about 6 ft long. These were divided into sub samples about 15 inches long. All samples were identified such that each sample could be reconstructed into its original bar form. Samples for analysis were prepared by milling using a tungsten carbide tool or by cutting into thin disks and polishing.

<u>Homogeneity Testing</u>

The Process Technology laboratory of Inco Limited did initial testing for homogeneity. Following positive results, 133 samples were strategically removed from the total batch and 70 were selected randomly for homogeneity study using a glow discharge mass spectrometer. The work was contracted to Shiva Technologies.

Each sample was etched with nitric acid, rinsed with water and ethanol. After a 30-minute pre-sputter, five consecutive readings were taken for the elements of highest interest. The average of the five readings was used for the homogeneity evaluation. NIST nickel-based alloy 1244 was used as a control sample.

<u>Inter-laboratory Testing and Data Evaluation</u>

Seventeen laboratories participated in the analysis of the material. Each laboratory reported one result for each element determined; therefore no evaluation could be made of the repeatability of the analysis within an individual laboratory. However, this is normally much better than between laboratories that it is not considered a contributing factor. Not all laboratories reported results for all elements of interest; therefore there was insufficient data for certification for some elements.

The methods used were electrothermal atomic absorption (some using ISO 5723), flame atomic absorption for higher levels (ISO 6351), ICP (inductively coupled plasma spectroscopy), glow discharge mass spectrometry, OES (optical emission spectroscopy), and combustion technology for carbon, sulfur, nitrogen, and oxygen, and molecular absorption spectrometry for phosphorus.

Data were analyzed for outliers and stragglers using the Grubb test (ASTM EI78). Laboratories reporting results identified as such were invited to reanalyze for the specific elements. Only a few laboratories reported recheck results. For the final critique of the data outlying results were rejected but some stragglers were retained. A few results for some elements were rejected on the basis of chemical intuition or the method used. This technique was used in a few cases where the data were not normally distributed and statistical methods failed. Some results reported to very low levels were rejected or the basis of incompatibility with the detection limits reported by other laboratories.

Although at first sight the confidence intervals appear to be very large, one must realize the difficulties in obtaining a sufficient number of reliable results at low levels of concentration. Unfortunately, some laboratories reported results to only one significant figure, which appeared to have been rounded. This contributed to higher values for the confidence interval. Thus it was not possible to assign a certified value to a higher number of significant figures or decimal places. However, the confidence interval has been extended to an extra decimal place for some elements where such an action seems reasonable.

The labs that participated in the certification process were as follows:

AB Sandvik Steel R & D Centre, Sandviken, Sweden
Falconbridge Metallverk als, Kristiansand, Norway
Haynes International, Inc., Kokomo, IN
Inco Alloys International, Huntington, WV
Inco Limited, Copper Cliff, Canada
Inco Limited, Port Colborne, Canada
Inco Limited, Manitoba, Canada
Inco Limited, Mississauga, Canada
Lakefield Research, Ontario, Canada
Leco Corporation, St. Joseph, MI
Outokumpu Harjvalta Metals Oy, Harjvalta, Finland
Sherritt Technologies, Fort Saskatchewan, AB
Shiva Technologies, Inc., Cicero, NY
Sumitomo Metal Mining Co., Ltd., Ehime, Japan
Teledyne Allvac, Monroe, NC
Wiggin Alloys Limited, Hereford, England

R Dan Brown, President

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