

CERTIFICATE OF ANALYSIS

24X 07001 (batch B)

Certified Reference Material Information

Type: NICKEL WASSPALLOY (WROUGHT)
Form and Size: Disc ~32mm diameter
Manufactured by: ATI Allvac, SC, USA
Certified and Supplied by: MBH Analytical Ltd

Assigned Values

Percentage element by weight

Element	C	Si	S	P	Mn	Cu	Cr	Fe
Value ¹	0.0357	0.064	(0.0007)	0.0033	0.0311	0.0115	19.52	0.997
Uncertainty ²	0.0012	0.005	-	0.0004	0.0011	0.0008	0.07	0.007

Element	Mo	Co	Ti	Al	Nb	B	Zr	Ni
Value ¹	4.29	13.31	3.01	1.384	0.0314	0.0060	0.0563	57.2
Uncertainty ²	0.04	0.07	0.02	0.009	0.0014	0.0003	0.0016	0.2

Note: values given in parentheses are not certified - they are provided for information only.

Definitions

- ¹ The above values are the present best estimates of the true content for each element. Each value is a panel consensus, based on the averaged results of an interlaboratory testing programme, detailed on page 3.
- ² The uncertainty values are generated from the 95% confidence interval derived from the wet analysis results, in combination with a statistical assessment of the homogeneity data, as described on page 2.

Certified by:

MBH ANALYTICAL LIMITED _____

on 2nd July 2012

C Eveleigh



Method of Preparation

This reference material was produced from commercial barstock to UNS N07001, VIM/VAR melted and hot finished. The discs in this batch are the product of one length of bar from a single heat.

Sampling

Samples for chemical analysis were taken from various positions within the bar. At least 5% of all discs were selected for non-destructive homogeneity testing.

Homogeneity

The discs were checked for sample and batch uniformity using an optical emission spectrometer.

Using the meaned data from each surface, standard deviation values were derived for each element as an indicator of any non-homogeneity (as determined for the specific sample size taken by the spectrometer).

Chemical Analysis

Analysis was carried out on millings taken from samples representative of the product. It was performed by a panel of laboratories mostly operating within the terms of EN ISO/IEC 17025 - 2005, using documented standard reference methods and validated by appropriate reference materials.

The individual values listed overpage are the average of each analyst's results.

Estimation of Uncertainties

Each element certified has been analysed by several laboratories, and 95% half-width confidence intervals ($C_{(95\%)}$) for the resultant mean values have been derived by the method shown on page 3.

As a separate exercise, the degree of non-homogeneity of the batch for each element has been quantified by a programme of non-destructive application testing, discussed above.

The final certified uncertainty for each element has been derived by combining these two factors, using the square-root of the summed squares.

Traceability

Much of the analytical work performed to assess this material has been carried out by laboratories with proven competence, as indicated by their accreditation to ISO 17025. It is an implicit requirement for this accreditation that analytical work should be performed with due traceability, via an unbroken chain of comparisons, each with stated uncertainty, to primary standards such as the mole, or to nationally- or internationally-recognised reference materials. In addition, some of the results derived as part of this testing programme have traceability to NIST standards, as part of the analytical calibration or process control.

Usage

Intended use: With optical emission and X-ray fluorescence spectrometers.

Recommended method of use: Nickel-base alloys are generally prepared by finishing, milling, turning or polishing. However, users are recommended to follow the calibration and sample preparation procedures specified by the relevant instrument manufacturer.

Preparation should be the same for reference materials and the samples for test.

For OES, a minimum of five consistent replicate analyses is recommended to provide the necessary sample size. Users are advised to check against possible bias between reference materials and production samples due to differences in metallurgical history, and be aware of possible inter-element effects.

Analytical Data

Percentage element by weight

Sample	C	Si	S	P	Mn	Cu	Cr	Fe
1	0.0340	0.0559	0.0002	0.0026	0.0280	0.0100	19.41	0.980
2	0.0342	0.0564	0.0003	0.0029	0.0284	0.0100	19.43	0.981
3	0.0347	0.0605	0.0004	0.0033	0.0290	0.0100	19.43	0.985
4	0.0348	0.0610	0.0006	0.0033	0.0292	0.0107	19.48	0.987
5	0.0348	0.0643	0.0006	0.0033	0.0297	0.0109	19.48	0.991
6	0.0350	0.0681	0.0006	0.0034	0.0310	0.0110	19.51	0.997
7	0.0350	0.0682	0.0007	0.0037	0.0313	0.0112	19.56	0.997
8	0.0353	0.0710	0.0010	0.0041	0.0314	0.0119	19.57	0.998
9	0.0361	0.0724	0.0010		0.0320	0.0122	19.57	0.999
10	0.0370		0.0010		0.0321	0.0125	19.58	1.000
11	0.0370		0.0012		0.0321	0.0126	19.58	1.004
12	0.0371		0.0012		0.0325	0.0131	19.59	1.006
13					0.0329	0.0138		1.009
14					0.0351			1.023
Mean	0.0357	0.064	0.0007	0.0033	0.0311	0.0115	19.52	0.997
Std Dev	0.0011	0.006	0.0003	0.0005	0.0020	0.0013	0.07	0.012
C (95%)	0.0007	0.005	0.0002	0.0004	0.0011	0.0008	0.04	0.007

Sample	Mo	Co	Ti	Al	Nb	B	Zr	Ni
1	4.230	13.19	2.971	1.367	0.0290	0.0054	0.0525	56.97
2	4.240	13.19	2.985	1.367	0.0298	0.0055	0.0530	57.05
3	4.243	13.22	2.986	1.372	0.0307	0.0059	0.0533	57.08
4	4.280	13.23	2.989	1.372	0.0310	0.0060	0.0537	57.12
5	4.290	13.26	2.994	1.375	0.0314	0.0060	0.0543	57.21
6	4.295	13.30	2.995	1.377	0.0315	0.0060	0.0547	57.35
7	4.300	13.32	3.003	1.377	0.0319	0.0061	0.0558	57.46
8	4.304	13.33	3.013	1.378	0.0325	0.0061	0.0559	57.57
9	4.312	13.40	3.016	1.389	0.0327	0.0063	0.0560	
10	4.325	13.44	3.025	1.391	0.0336	0.0064	0.0565	
11	4.325	13.49	3.030	1.399		0.0068	0.0586	
12	4.331		3.039	1.409			0.0589	
13	4.336		3.050	1.415			0.0600	
14	4.345		3.051				0.0601	
15			3.055				0.0611	
Mean	4.294	13.31	3.013	1.384	0.0314	0.0060	0.0563	57.23
Std Dev	0.038	0.10	0.027	0.016	0.0014	0.0004	0.0028	0.21
C (95%)	0.022	0.07	0.015	0.009	0.0010	0.0003	0.0016	0.18

Note: $C_{(95\%)}$ is the 95% half-width confidence interval derived from the equation:

$$C_{(95\%)} = (t \times SD) / \sqrt{n}$$

where n is the number of available values, t is the Student's t value for n-1 degrees of freedom, and SD is the standard deviation of the test results.

Participating Laboratories

Exova Materials Testing	Middlesbrough, England	UKAS accreditation 0239
IncoTest Ltd	Hereford, England	UKAS accreditation 0281
Metals Technology (Testing) Ltd	Sheffield, England	UKAS accreditation 0963
Sheffield Assay Office	Sheffield, England	UKAS accreditation 0012
Laboratory Testing, Inc	Hatfield, PA, USA	A2LA accreditation 0117
Genitest, Inc	Montreal, Canada	PRI accreditation 123077
Institute of Iron and Steel Technology	Shanghai, China	CNAL accreditation 0783
Raghavendra Spectrometallurgical Lab.	Bangalore, India	NABL accreditation 0371
Sargam Laboratory Pvt Ltd	Chennai, India	NABL accreditation 0025
Shriram Institute for Industrial Research	Delhi, India	NABL accreditation 0045
Instytut Metalurgii Zelaza	Gliwice, Poland	PCA accreditation AB554
London & Scandinavian Met Co Ltd	Rotherham, England	
Coleshill Laboratories Ltd	Birmingham, England	
De Bruyn Spectroscopic Solutions Ltd	Johannesburg, South Africa	
LECO Corporation	St Joseph, MI, USA	

Note: to achieve the above-noted accreditation (UKAS, A2LA, etc), test houses must demonstrate conformity to the general requirements of EN ISO/IEC 17025.

Analytical Methods Used

ELEMENT	RESULT No. & METHOD				
	ICP-AES	FAAS	GD-AES	OTHER	
Carbon	-	-	-	all	combustion (IR or volumetric detection)
Silicon	5, 7, 9	-	-	4	photometric (molybdenum blue)
Sulfur	4, 12	-	-	1-3, 6, 8	gravimetric (perchloric acid)
Phosphorus	1-5	-	8	1-3, 5-11	combustion (IR or volumetric detection)
Manganese	1-5, 8-10, 12, 13	7, 11, 14	6	6	ICP-MS
Copper	2-4, 7-10, 13	5, 11, 12	6	7	photometric (molybdenum blue)
Chromium	1, 5, 7-10, 12	-	6	1	photometric (BCO)
Iron	1, 2, 4, 7-11, 13, 14	3, 5, 10	6	2-4, 11	volumetric (ferrous ammonium sulfate)
Molybdenum	2, 4, 5, 8-10, 12, 13	7	3	12	volumetric (dichromate)
Cobalt	2-4, 7, 9	6	10	6, 11	photometric (thiocyanate)
Titanium	1, 2, 4-8, 10-12, 15	13, 14	9	1, 14	gravimetric (α -benzoin oxime)
Aluminium	1-4, 6, 9, 10, 12, 13	5, 11	8	5	gravimetric
Niobium	3-5, 7-10	6	1	8	volumetric (ferricyanide)
Boron	1-3, 5-9	-	10	3	photometric (nitroso-R)
Zirconium	1-6, 8-12, 14, 15	-	13	7	photometric (DAP)
Nickel	2, 4-6	-	-	2	photometric (chrome azurol S)
				4	photometric (chlorosulfophenol)
				7	ICP-MS
				3	ICP-MS
				1, 7, 8	volumetric (EDTA/DMGO)
					gravimetric (dimethyl glyoxime)

Notes

This Certified Reference Material has been produced and certified in accordance with the requirements of ISO Guide 34-2009, ISO Guide 31-2000 and ISO Guide 35-2006, taking into account the requirements of the ISO Guide to the Expression of Uncertainty in Measurement (GUM).

This certification is applicable to the whole of the disc. However, in accordance with normal practice for emission spectrometry, it is appropriate to avoid usage of the central portion of the disc, ~6 mm diameter.

This material will remain stable indefinitely, provided adequate precautions are taken to protect it from cross-contamination, extremes of temperature and atmospheric moisture. All production records will be retained for a period of 20 years from the date of this certificate. This certification will therefore expire in July 2032, although we reserve the right to make changes as issue revisions, in the intervening period.

The testing, analysis and certification of this product were supervised by C Eveleigh, PhD, Technical Director, MBH Analytical Ltd.

The material to which this certificate of analysis refers is supplied subject to our general conditions of sale.