

# CERTIFICATE OF ANALYSIS

**24X 07001 (batch C)**

## 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	Mo
Value <sup>1</sup>	0.0360	0.042	0.0005	0.0023	0.024	0.0118	19.62	1.023	4.31
Uncertainty <sup>2</sup>	0.0015	0.004	0.0001	0.0004	0.002	0.0013	0.06	0.017	0.03

Element	Co	Ti	Al	Nb	V	W	Zr	B	Ni
Value <sup>1</sup>	13.20	3.14	1.476	0.050	0.041	0.041	0.060	0.0062	56.92
Uncertainty <sup>2</sup>	0.04	0.02	0.019	0.002	0.002	0.003	0.002	0.0005	0.15

## Definitions

- <sup>1</sup> 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.
- <sup>2</sup> 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 27<sup>th</sup> December 2017

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. Approximately 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, 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.

Of the individual results herein, some have traceability (to the mole) via primary analytical methods. Some are traceable to substances of known stoichiometry. Most have traceability via commercial solutions. Furthermore, some results have additional 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	Mo
1	0.0321	0.0364	0.00040	0.0017	0.0193	0.0087	19.53	0.987	4.227
2	0.0329	0.0375	0.00040	0.0017	0.0198	0.0093	19.54	0.998	4.259
3	0.0340	0.0392	0.00043	0.0019	0.0203	0.0104	19.56	1.007	4.260
4	0.0348	0.0406	0.00045	0.0019	0.0218	0.0108	19.57	1.007	4.273
5	0.0364	0.0441	0.00050	0.0025	0.0226	0.0110	19.59	1.008	4.290
6	0.0367	0.0442	0.00060	0.0027	0.0227	0.0126	19.61	1.021	4.293
7	0.0368	0.0446	0.00065	0.0028	0.0255	0.0128	19.63	1.022	4.298
8	0.0375	0.0495	0.00066	0.0030	0.0262	0.0128	19.64	1.037	4.340
9	0.0376		0.00080		0.0267	0.0134	19.66	1.044	4.346
10	0.0381				0.0267	0.0142	19.70	1.059	4.350
11	0.0386				0.0271	0.0143	19.70	1.062	4.354
12					0.0312		19.71		4.362
13									4.381
<b>Mean</b>	<b>0.0360</b>	<b>0.0420</b>	<b>0.00054</b>	<b>0.0023</b>	<b>0.0242</b>	<b>0.0118</b>	<b>19.62</b>	<b>1.023</b>	<b>4.310</b>
<b>Std Dev</b>	0.0022	0.0044	0.00014	0.0005	0.0036	0.0019	0.06	0.025	0.048
<b>C (95%)</b>	0.0015	0.0037	0.00011	0.0004	0.0023	0.0013	0.04	0.017	0.029

Sample	Co	Ti	Al	Nb	V	W	Zr	B	Ni
1	13.11	3.100	1.427	0.0462	0.0372	0.0367	0.0570	0.0051	56.69
2	13.14	3.101	1.439	0.0465	0.0373	0.0382	0.0571	0.0053	56.82
3	13.16	3.105	1.442	0.0482	0.0381	0.0391	0.0581	0.0053	56.83
4	13.16	3.120	1.446	0.0492	0.0393	0.0396	0.0595	0.0062	56.87
5	13.17	3.122	1.464	0.0494	0.0400	0.0397	0.0600	0.0062	56.93
6	13.19	3.137	1.468	0.0503	0.0401	0.0414	0.0603	0.0063	57.00
7	13.21	3.145	1.480	0.0522	0.0405	0.0415	0.0609	0.0063	57.07
8	13.22	3.146	1.489	0.0533	0.0410	0.0433	0.0625	0.0068	57.12
9	13.24	3.161	1.489	0.0536	0.0412	0.0453	0.0639	0.0070	
10	13.25	3.165	1.491	0.0543	0.0428	0.0476	0.0649	0.0073	
11	13.28	3.180	1.508		0.0428				
12	13.28	3.198	1.510		0.0448				
13			1.533		0.0460				
<b>Mean</b>	<b>13.20</b>	<b>3.140</b>	<b>1.476</b>	<b>0.0503</b>	<b>0.0409</b>	<b>0.0412</b>	<b>0.0604</b>	<b>0.0062</b>	<b>56.92</b>
<b>Std Dev</b>	0.06	0.032	0.032	0.0029	0.0027	0.0033	0.0027	0.0007	0.14
<b>C (95%)</b>	0.04	0.020	0.019	0.0021	0.0016	0.0024	0.0019	0.0005	0.12

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 Ltd	Middlesbrough, England	UKAS accreditation 0239
Sheffield Analytical Services	Sheffield, England	UKAS accreditation 0012
Metals Technology (Testing) Ltd	Sheffield, England	UKAS accreditation 0963
Laboratory Testing, Inc	Hatfield, PA, USA	A2LA accreditation 0117
Genitest, Inc	Montreal, Canada	PJ accreditation L17-153
Shanghai Jinyi Test Tech Co	Shanghai, China	CNAS accreditation L0041
Shandong Metallurgical & Science Research	Jinan, Shandong, China	CNAS accreditation 1461
Raghavendra SpectroMet Laboratory	Bangalore, India	NABL accreditation 0371
TCR Engineering Services Ltd	Mumbai, India	NABL accreditation 0367
Instytut Metalurgii Zelaza	Gliwice, Poland	PCA accreditation AB554
Tec-Eurolab	Campogalliano, Italy	ACCREDIA accreditation 52
TUV Nord Czech	Brno, Czech Republic	CAI accreditation L1060
Mineral & Metallurgical Laboratories	Bangalore, India	
AMG Superalloys UK Ltd	Rotherham, England	
Analyticka Laborator Lithea sro	Brno, Czech Republic	

Note: to achieve the above accreditation (UKAS, 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	OTHER
Carbon	-	-	all combustion (infra-red detection)
Silicon	2-4, 6	-	1, 5, 8 gravimetric (perchloric acid)
			7 photometric (molybdenum blue)
Sulfur	4	-	1-3, 5-9 combustion (infra-red detection)
Phosphorus	1, 2, 4, 6, 7	-	3, 5 photometric (molybdenum blue)
			8 volumetric (alkalimetric)
Manganese	1-3, 6, 8-12	-	4, 5, 7 photometric (periodate)
Copper	1-3, 5, 8-11	7	4, 6 photometric (BCO)
Chromium	2, 5-8, 11, 12	-	1, 3, 4, 9, 10 volumetric (ferrous ammonium sulfate)
Iron	1-3, 5, 6, 8-11	-	4 photometric (sulfosalicylic acid)
			7 volumetric (dichromate)
Molybdenum	1-3, 5, 6, 8, 9, 11, 12	10	4, 13 photometric (thiocyanate)
			7 gravimetric
Cobalt	1, 3, 4, 6, 7, 9-12	-	2, 5 photometric (5-chloro-PADAB)
			8 gravimetric
Titanium	1, 2, 6, 8-12	4	3, 5, 7 photometric (diantipyryl methane)
Aluminium	1-4, 6, 9-12	7	5, 8 photometric (chrome azurol S)
			13 gravimetric
Niobium	1, 3-6, 9, 10	-	2, 7, 8 photometric (chlorosulfophenol)
Vanadium	1-6, 8-13	7	
Tungsten	1, 3-6, 8-10	-	2, 7 photometric (thiocyanate)
Zirconium	1-10	-	
Boron	1-10	-	
Nickel	3-6	-	1, 2, 7, 8 gravimetric (dimethyl glyoxime)

## Notes

This Certified Reference Material has been produced and certified in accordance with the requirements of ISO Guide 34, ISO Guide 31 and ISO Guide 35, 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 December 2037, 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.