

# CERTIFICATE OF ANALYSIS

**28X 6256 (batch A)**

## Certified Reference Material Information

Type: NICKEL INCONEL 625-TYPE (HIPped)  
Form and Size: Disc 40mm Diameter x 13mm Thickness  
Manufactured by: Crucible Research, Pittsburg, PA  
Certified and Supplied by: MBH Analytical Ltd

## Assigned Values

### Percentage element by weight

Element	C	Si	S	P	Mn	Cr	Nb
Value <sup>1</sup>	0.0173	0.041	(0.0016)	0.0033	(0.0004)	21.29	3.75
Uncertainty <sup>2</sup>	0.0011	0.004	-	0.0005	-	0.07	0.03

Element	Fe	Cu	Mo	Ti	Al	Ni	N
Value <sup>1</sup>	(0.034)	0.018	8.81	0.266	0.301	65.4	0.007
Uncertainty <sup>2</sup>	-	0.002	0.03	0.008	0.006	0.2	0.001

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

## 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 equivalent to the 95% confidence interval derived from the wet analysis results, after assessment of the homogeneity data, as described on page 2.

## Certified by:

MBH ANALYTICAL LIMITED \_\_\_\_\_

on 21<sup>st</sup> November 2008

C Eveleigh



## **Method of Preparation**

This reference material was produced by atomisation of molten alloy using pressurized argon. The resultant powder was sieved, and then loaded into a steel container which was vacuum outgassed prior to sealing. The sealed container was HIPped, to achieve 100% consolidation.

## **Sampling**

Samples for chemical analysis, and discs for homogeneity checks, were taken from several positions within the batch. At least 10% of all discs were incorporated into the schedule for homogeneity checking.

## **Homogeneity**

The discs were checked for sample and batch uniformity using an optical emission spectrometer. No significant inhomogeneity was found, and spectrometer performance was similar at all positions within the sample.

## **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 - 2000, 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.

Since the complete batch of material has been found to be homogeneous (for the main application envisaged), these 95% confidence intervals have been used directly as an indicator of uncertainty.

## **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: These alloys are generally prepared by finishing, milling, turning or polishing, but note that liquid coolants or solvents should not be used as part of this process. Otherwise, users are recommended to follow the calibration and sample preparation procedures specified by the relevant instrument manufacturer.

A minimum of three 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	Cr	Nb
1	0.014	0.035	0.0010	0.0022	0.0001	21.07	3.698
2	0.0150	0.0353	0.0010	0.0028	0.0002	21.16	3.703
3	0.0159	0.0365	0.0011	0.0029	0.0003	21.22	3.71
4	0.0174	0.0396	0.0011	0.003	0.0007	21.23	3.713
5	0.0174	0.0409	0.0013	0.0035	0.0008	21.25	3.728
6	0.0178	0.044	0.0020	0.0035		21.25	3.730
7	0.0179	0.0448	0.0021	0.0037		21.29	3.75
8	0.018	0.048	0.0024	0.0038		21.32	3.77
9	0.0184		0.0028	0.0042		21.33	3.780
10	0.0188					21.35	3.788
11	0.0197					21.40	3.806
12						21.46	3.808
13						21.47	3.812
<b>Mean</b>	<b>0.0173</b>	<b>0.041</b>	<b>0.0016</b>	<b>0.0033</b>	<b>(0.0004)</b>	<b>21.29</b>	<b>3.753</b>
<b>Std Dev</b>	0.0017	0.005	0.0007	0.0006	-	0.11	0.042
<b>C<sub>(95%)</sub></b>	0.0011	0.004	0.0005	0.0005	-	0.07	0.025

Sample	Fe	Cu	Mo	Ti	Al	Ni	N
1	0.0230	0.0158	8.75	0.253	0.283	65.13	0.0060
2	0.0245	0.0164	8.753	0.255	0.290	65.21	0.0063
3	0.026	0.0174	8.762	0.258	0.296	65.24	0.0063
4	0.0290	0.0192	8.801	0.261	0.297	65.33	0.0069
5	0.030	0.0198	8.815	0.261	0.299	65.35	0.0071
6	0.0390	0.020	8.832	0.263	0.301	65.51	0.0086
7	0.0430	0.0203	8.835	0.265	0.304	65.60	
8	0.0442		8.84	0.266	0.305	65.61	
9	0.0482		8.850	0.274	0.308	65.73	
10			8.850	0.283	0.310		
11			8.851	0.290	0.316		
<b>Mean</b>	<b>0.034</b>	<b>0.0184</b>	<b>8.813</b>	<b>0.266</b>	<b>0.301</b>	<b>65.41</b>	<b>0.0069</b>
<b>Std Dev</b>	0.010	0.0018	0.040	0.012	0.009	0.21	0.0009
<b>C<sub>(95%)</sub></b>	0.007	0.0017	0.031	0.008	0.006	0.16	0.0010

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

ATI AllVac Ltd	Sheffield, England	UKAS accreditation 1385
Bodycote Materials Testing	Middlesbrough, England	UKAS accreditation 0239
IncoTest Ltd	Hereford, England	UKAS accreditation 0281
Sheffield Assay Office	Sheffield, England	UKAS accreditation 0012
Metals Technology (Testing) Ltd	Sheffield, England	UKAS accreditation 0963
Universal Scientific Laboratory	Milperra, NSW, Australia	NATA accreditation 0492
Genitest, Inc	Montreal, Canada	PRI accreditation 123077
Laboratory TUV-Nord Czech	Brno, Czech Republic	CAI accreditation 1060
Institute of Iron and Steel Technology	Shanghai, China	CNAL accreditation 0783
Sargam Metals Pvt Ltd	Chennai, India	NABL accreditation 0025
TCR Engineering Services Ltd	Mumbai, India	NABL accreditation 0367
De Bruyn Spectroscopic Solutions Ltd	Johannesburg, South Africa	
Rigaku America Applications Laboratory	Woodlands, TX, USA	
Crucible Materials Corp	Pittsburg, PA, USA	

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

## Analytical Methods Used

ELEMENT	RESULT No. & METHOD				
	ICP-AES	XRF	FAAS		OTHER
Carbon	-	-	-	all	combustion (infra-red detection)
Silicon	1, 4, 5, 7	-	-	6, 8	photometric (molybdenum blue)
Sulfur	6, 8	-	-	2, 3	gravimetric (perchloric acid)
Phosphorus	4, 8	3, 6	-	1-5, 7, 9	combustion (infra-red detection)
				2	ICP-MS
				1, 5, 9	photometric (molybdenum blue)
				7	volumetric (alkalimetric)
Manganese	4, 5	-	1, 3	2	ICP-MS
Chromium	1, 2, 4-6, 8, 9	12	-	3, 7, 10, 11, 13	volumetric (FAS)
Niobium	3, 4, 6, 8-13	5	7	1	gravimetric
				2	photometric (chlorosulfophenol S)
Iron	1, 4, 5, 7	2, 6	3, 9	8	photometric (sulfsalicylic acid)
Copper	3, 4, 6, 7	-	1, 5	2	photometric (BCO)
Molybdenum	3, 5, 8-11	2, 7	1	4	photometric (thiocyanate)
				6	gravimetric ( $\alpha$ -benzoin oxime)
Titanium	5-9, 11	1, 3	2, 10	4	photometric (DAP)
Aluminium	3, 5, 6, 8-11	4, 7	1, 2		
Nickel	1, 2, 7, 9	4	-	6	volumetric (EDTA/DMGO)
				3, 5, 8	gravimetric (dimethyl glyoxime)
Nitrogen	-	-	-	1-5	inert gas fusion (thermal conductivity)
				6	photometric (Nessler's reagent)

## Notes

This Certified Reference Material has been produced and certified in accordance with the requirements of ISO Guide 34-2000, ISO Guide 31-2000 and ISO Guide 35-1989, 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.

This material will remain stable 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 November 2028, although we reserve the right to make changes as issue revisions, in the intervening period.

The sampling, 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.