

CERTIFICATE OF ANALYSIS

13X NSC6 (batch A)

Certified Reference Material Information

Type: NITRONIC 40-type (HIPped POWDER)
Form and Size: Disc 40mm Diameter x 13mm Thickness
Produced by: Crucible Materials Corp.
Certified and supplied by: MBH Analytical Ltd.

Assigned Values

Percentage element by weight

Element	C	Si	S	P	Mn	Ni
Value ¹	0.0266	0.523	0.0055	0.0049	8.85	6.52
Uncertainty ²	0.0015	0.011	0.0004	0.0006	0.03	0.03

Element	Cr	Mo	Al	Cu	V	N
Value ¹	20.47	(0.002)	(0.009)	0.0064	0.0052	0.235
Uncertainty ²	0.10	-	-	0.0007	0.0014	0.009

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 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 21st 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	Ni
1	0.0232	0.496	0.0042	0.0034	8.78	6.451
2	0.0248	0.496	0.0048	0.0035	8.791	6.469
3	0.0250	0.509	0.005	0.0045	8.80	6.471
4	0.026	0.510	0.0053	0.0045	8.80	6.48
5	0.0261	0.515	0.0054	0.0047	8.838	6.482
6	0.0262	0.524	0.0055	0.0050	8.852	6.516
7	0.0264	0.530	0.0058	0.0050	8.862	6.517
8	0.0266	0.535	0.0059	0.0053	8.87	6.54
9	0.0282	0.535	0.0059	0.006	8.870	6.550
10	0.0290	0.535	0.0061	0.0060	8.884	6.551
11	0.0312	0.543	0.0061	0.0063	8.885	6.57
12		0.546	0.0064		8.90	6.584
13					8.929	6.584
Mean	0.0266	0.523	0.0055	0.0049	8.851	6.520
Std Dev	0.0022	0.017	0.0006	0.0010	0.046	0.046
C (95%)	0.0015	0.011	0.0004	0.0006	0.028	0.028

Sample	Cr	Mo	Al	Cu	V	N
1	20.23	0.0010	0.0059	0.0049	0.0026	0.218
2	20.31	0.0013	0.0067	0.0049	0.0027	0.222
3	20.37	0.0013	0.0070	0.0053	0.0032	0.225
4	20.40	0.0020	0.0085	0.0056	0.0037	0.230
5	20.41	0.0026	0.0089	0.0056	0.0043	0.241
6	20.44	0.0035	0.0089	0.0064	0.0044	0.242
7	20.56	0.0040	0.0135	0.0066	0.0050	0.242
8	20.57		0.0147	0.0068	0.0051	0.247
9	20.58			0.007	0.0061	0.249
10	20.65			0.0071	0.0073	
11	20.66			0.0072	0.009	
12				0.0079	0.0090	
13				0.0083		
Mean	20.47	(0.0022)	0.009	0.0064	0.0052	0.235
Std Dev	0.14	-	0.003	0.0011	0.0022	0.011
C (95%)	0.10	-	0.003	0.0007	0.0014	0.009

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
Bodycote Materials Testing
IncoTest Ltd
Metals Technology (Testing) Ltd
Universal Scientific Laboratory
Genitest, Inc
Laboratory TUV-Nord Czech
Institute of Iron and Steel Technology
Luo Yang Copper Co
Sargam Metals Pvt Ltd
TCR Engineering Services Ltd
De Bruyn Spectroscopic Solutions Ltd
Rigaku America Applications Laboratory
Crucible Materials Corp

Sheffield, England
Middlesbrough, England
Hereford, England
Sheffield, England
Milperra, NSW, Australia
Montreal, Canada
Brno, Czech Republic
Shanghai, China
Luo Yang, He Nan, China
Chennai, India
Mumbai, India
Johannesburg, South Africa
Woodlands, TX, USA
Pittsburg, PA, USA

UKAS accreditation 1385
UKAS accreditation 0239
UKAS accreditation 0281
UKAS accreditation 0963
NATA accreditation 0492
PRI accreditation 123077
CAI accreditation 1060
CNAL accreditation 0783
CNAL accreditation 0173
NABL accreditation 0025
NABL accreditation 0367

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	2, 3, 5, 7, 9	3	-	1, 4, 6, 10 8, 11, 12	gravimetric (perchloric acid) photometric (molybdenum blue)
Sulfur	-	-	-	all	combustion (infra-red detection)
Phosphorus	1-3, 6, 8, 10	5	-	7, 9, 11 4	photometric (molybdenum blue) ICP-MS
Manganese	3, 8-11, 13	1	2	6, 7 4, 5, 12	photometric (periodate) volumetric (arsenite)
Nickel	1, 6, 7, 11-13	4, 9	10	7 2, 3 5, 8	photometric (dimethyl glyoxime) volumetric (dimethyl glyoxime) gravimetric (dimethyl glyoxime)
Chromium	1, 4, 7-9, 11	5	-	2, 3, 6, 10	volumetric (ferrous ammonium sulfate)
Molybdenum	1, 2, 5, 7	-	3, 4	6	ICP-MS
Aluminium	1, 2, 5-7	-	3, 4, 8		
Copper	1, 2, 6, 8, 10, 13	3, 5, 7	9, 11, 12	4	photometric (BCO)
Vanadium	1, 4, 6, 8, 10-12	5	2, 7, 9	3	ICP-MS
Nitrogen	-	-	-	1, 5-9 2, 4 3	inert gas fusion (thermal conductivity) photometric (Nessler reagent) volumetric (hydrochloric acid)

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.