

CERTIFICATE OF ANALYSIS

14 X HS1 (batch C)

Certified Reference Material Information

Type: HIGH SPEED STEEL (WROUGHT)
Form and Size: Disc 40mm Diameter x 15mm Thickness
Supplied by: MBH Analytical Limited
Produced by: Stock Bar

Certified Analysis

Percentage element by weight

Element	C	Si	S	P	Mn	Ni	Cr
Value ¹	0.718	0.22	0.020	0.018	0.29	0.27	4.00
Uncertainty ²	0.008	0.04	0.002	0.003	0.02	0.02	0.06

Element	Mo	Cu	Co	W	V	Sn	N
Value ¹	0.37	0.069	0.25	17.0	1.05	(0.035)	0.023
Uncertainty ²	0.01	0.003	0.02	0.3	0.03	-	0.001

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

Definitions

- ¹ The certified 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 16th May 2003

C Eveleigh



Method of Preparation

This reference material was produced from a single bar of 'stock' wrought material.

Sampling

Samples for chemical analysis were taken from various positions within the bar. Approximately 10% 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.

For each of the surfaces checked, the differences between the averaged result and the overall mean value were evaluated to ensure that the overall homogeneity of the material comprising the batch satisfied the definition given in ISO guide 30 - 1992.

Using the individual data from each check, standard deviation values were derived for each element. These values were combined with the 95% half-width confidence intervals ($C_{(95\%)}$) obtained from the wet analysis programme, using the square-root of the summed squares, to derive the final uncertainty values.

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 ISO Guide 25: 1990, using documented standard reference methods and validated by appropriate reference materials.

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

Traceability

Most of the analytical work performed to assess this material has been carried out by laboratories with proven competence, as indicated by their accreditation to a national authority. It is part of the 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 primary reference materials.

Usage

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

Recommended method of use: Steels 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.

A minimum of four consistent replicate analyses is recommended to optimise precision and accuracy. 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	Cr
1	0.71	0.16	0.016	0.013	0.27	0.23	3.88
2	0.71	0.22	0.018	0.016	0.28	0.25	3.95
3	0.72	0.23	0.019	0.017	0.29	0.27	3.97
4	0.72	0.24	0.019	0.019	0.29	0.28	4.04
5	0.72	0.24	0.020	0.02	0.297	0.28	4.04
6	0.73		0.022	0.022	0.30	0.29	4.05
7			0.024		0.32	0.295	4.06
Mean	0.718	0.218	0.020	0.018	0.292	0.271	4.00
Std Dev	0.008	0.033	0.003	0.003	0.016	0.023	0.07
C_(95%)	0.008	0.042	0.002	0.003	0.015	0.021	0.06

Sample	Mo	Cu	Co	W	V	Sn	N
1	0.36	0.068	0.22	16.68	1.00	0.035	0.022
2	0.36	0.068	0.24	16.8	1.03	<0.05	0.022
3	0.37	0.07	0.246	17.04	1.04		0.023
4	0.37	0.07	0.25	17.20	1.05		0.0232
5	0.37	0.07	0.25	17.21	1.065		0.024
6		0.07	0.26		1.08		
7			0.30		1.08		
Mean	0.366	0.069	0.252	17.00	1.05	(0.035)	0.023
Std Dev	0.005	0.001	0.024	0.24	0.03	-	0.001
C_(95%)	0.007	0.001	0.023	0.30	0.03	-	0.001

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

Willan Metals Ltd	Rotherham, England	NAMAS accreditation 0014
IncoTest Ltd	Hereford, England	NAMAS accreditation 0281
University Metals Advisory Centre	Sheffield, England	NAMAS accreditation 0411
Allvac SMP Ltd	Sheffield, England	NAMAS accreditation 1385
Ductile Steel Processors Ltd	Willenhall, England	NAMAS accreditation 0561
RoTech Laboratories	Wednesbury, England	NAMAS accreditation 0366
London & Scandinavian Met Co Ltd	Rotherham, England	NAMAS accreditation 1091
Metals Technology Testing Ltd	Sheffield, England	NAMAS accreditation 0963
Shiva Analyticals Ltd	Bangalore, India	

Note: to achieve UK National Accreditation (NAMAS), test houses were required to demonstrate conformity to the general requirements of ISO Guide 25: 1990 and ISO9002.

Analytical Methods Used

ELEMENT	RESULT No. & METHOD				
	ICP-AES	FAAS	XRF	OTHER	
Carbon	-	-	-	all	combustion (infra-red detection)
Silicon	4	5	1, 3	2	gravimetric (perchloric acid)
Sulfur	-	-	-	all	combustion (infra-red detection)
Phosphorus	2, 5	-	1, 3	4, 6	photometric (molybdenum blue)
Manganese	2, 6	4, 5	3, 7	1	photometric (periodate)
Nickel	6	1, 2, 3, 7	2, 5		
Chromium	3	2, 6	1, 4	5, 7	volumetric (ferrous ammonium sulfate)
Molybdenum	2, 5	3, 4	1		
Copper	5, 6	1, 2, 4	3		
Cobalt	5, 6	1, 3	4, 7	2	photometric (Nitroso-R)
Tungsten	2	-	3	1, 4, 5	photometric (thiocyanate)
Vanadium	3, 6	2, 4, 5	1, 7		
Tin	-	1	2		
Nitrogen	-	-	-	all	inert gas fusion (thermal conductivity)

Notes

This Certified Reference Material has been 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 ASTM E1724, ASTM E1831 and the ISO Guide to the Expression of Uncertainty in Measurement (GUM).

The above 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 May 2023, although we reserve the right to make changes as issue revisions, in the intervening period.

The manufacture, 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.