

# Certificate of Analysis IARM 21D

PH13-8Mo / UNS S13800

Certified Reference Material

#### Certified Values listed in wt.% with associated uncertainties

Al	$1.03 \pm 0.02$	C	$0.032 \pm 0.003$	Co	$0.078 \pm 0.006$	Cr	$12.69 \pm 0.07$
Cu	$0.017 \pm 0.002$	Mn	$0.052 \pm 0.007$	Мо	$2.23 \pm 0.02$	N	$0.0037 \pm 0.0003$
Nb	$0.005 \pm 0.002$	Ni	$8.29 \pm 0.06$	Р	$0.008 \pm 0.001$	S	$0.0014 \pm 0.0008$
Si	$0.039 \pm 0.004$	Ti	<b>0.016</b> ± 0.001	V	$0.017 \pm 0.001$	W	$0.012 \pm 0.003$

## **Indicative Values listed in ppm**

As (30)	B (4)	Ca (4)	Mg (<5)	O (6)	Pb (<10)	Sb (<10)
Se (<10)	Sn (10)	Ta (<30)	Zn (<20)	Zr (20)		

#### **Description and Intended Use**

This CRM may come in the form of a solid disc or chips. The intended use of this CRM may include, but is not limited to, the calibration of instruments and the validation of analytical methods.

# **Interpretation of Data**

- 1. Certified values listed reflect analysis results submitted by qualified analytical laboratories using a combination of methods and instrumentation that emulate actual methods and instrumental techniques currently utilized in the analytical community, and are reported as wt% unless otherwise noted.
- 2. This material was tested using both the solid disks and chips prepared from individual sections of bar. The certified values are considered representative of the overall average composition of the material.
- 3. Any data reported and enclosed by a parentheses () is a "best estimate" and is not certified. This data could not be quantified sufficiently for certification. It was, however, reported by enough laboratories to be considered as potentially present in the matrix of the material being examined.
- 4. "Provisional Certificate of Analysis" reports values that support a fully certified reference material; it also indicates that values may be in a continued process of statistical evaluation and are subject to change.
- 5. Chips are not certified for Oxygen analysis.



The following data and accompanying statements represent all pertinent information reported in the ILAP as it applies to the chemical characterization of this material.

	Al	As	В	С	Са	Со	Cr	Cu	Mg	Mn	Мо	N	Nb	Ni	0	Р
1	0.9857	0.0013	0.0003	0.028	0.0003	0.058	12.531	0.0095	0.0005	0.029	2.187	0.00332	0.0009	8.107	0.0003	0.0058
2	0.9933	0.0027	0.00033	0.0284	0.0004	0.074	12.587	0.014	<0.0001	0.04	2.20	0.0035	0.0038	8.20	0.00039	0.0064
3	0.995	0.004	0.0004	0.029	<0.0001	0.074	12.60	0.016		0.0448	2.2097	0.0036	0.004	8.208	0.001	0.007
4	1.015		0.0005	0.031		0.0764	12.6016	0.016		0.0451	2.212	0.0036	0.005	8.2233	<0.005	0.007
5	1.018		0.0006	0.031		0.0771	12.6189	0.0161		0.0461	2.2144	0.004	0.0056	8.226		0.007
6	1.02		<0.0005	0.03117		0.08	12.6447	0.0162		0.047	2.22	0.004	0.007	8.2436		0.007
7	1.0279		<0.0005	0.0317		0.08	12.647	0.0173		0.0475	2.22	<0.005	0.0072	8.2961		0.007
8	1.031			0.032		0.081	12.66	0.018		0.052	2.222		0.01	8.30		0.0072
9	1.05			0.0322		0.081	12.673	0.018		0.0535	2.2246		<0005	8.31		0.0074
10	1.057			0.038		0.0869	12.69	0.02		0.056	2.24			8.32		0.008
11	1.0787			0.0417		0.0923	12.7056	0.02		0.056	2.241			8.344		0.0082
12	1.0788						12.884	0.02		0.0643	2.245			8.36193		0.0088
13	1.0879						12.91			0.068	2.2825			8.4405		0.0105
14							12.9334			0.078	2.2834			8.49		0.0157
15																
Mean	1.03	0.003	0.0004	0.032	0.0004	0.078	12.69	0.017		0.052	2.23	0.0037	0.005	8.29	0.0006	0.008
STDV.	0.03	0.001	0.0001	0.004	0.00007	0.009	0.1	0.003		0.01	0.03	0.0003	0.003	0.1	0.0004	0.002
Certified	1.03	(0.003)	(0.0004)	0.032	(0.0004)	0.078	12.69	0.017	(<0.0005)	0.052	2.23	0.0037	0.005	8.29	(0.0006)	0.008
95% C.I.	0.02			0.003		0.006	0.07	0.002		0.007	0.02	0.0003	0.002	0.06		0.001
Methods	X,O,I	X,O,I	O,I	O,I,C	0	X,O,I	X,W,O,I	X,O,I	O,I	X,O,I	X,O,I	O,F	X,O,I	X,O,I	I,F	X,O,I
	Pb	S	Sb	Se	Si	Sn	Ta	Ti	V	W	Zn	Zr	T	T	T	
1 1	0.00035	0.0007	0.0003	<0.001	0.031	0.0003	0.0034	0.0146	0.0103	0.0072	0.0016	0.0008				
2	0.001	0.0008	<0.001	<0.001	0.0319	0.0003	<0.001	0.015	0.016	0.0103	<0.001	0.0018				
3	<0.001	0.00085			0.0326	0.0009	<0.001	0.015	0.016	0.011		0.003				
4		0.00091			0.0349	0.0009		0.015	0.0162	0.012		0.0034				
5		0.001			0.035	0.0027		0.015	0.0164	0.0129						
6		0.001			0.0353	<0.005		0.0159	0.0164	0.014						
7		0.00103			0.038			0.0163	0.0164	0.0163						
8		0.0023			0.038			0.0168	0.0176							
9		0.004			0.04			0.018	0.0177							
10					0.043			0.02	0.018							
11					0.0483				0.018							

Legend: W = Classical, C = Combustion, F = Fusion, A = AA or GFAA, I = ICP or DCP, IM=ICP-MS, D = DC Arc, O = AES, X = XRF, G = GDAES or GDMS, H = Hollow Cathode AES

0.016

0.002

0.016

0.001

X,O,I

(<0.003)

X,O,I

0.019

0.017

0.002

0.017

0.001

X,O,I

0.012

0.003

0.012

0.003

X,O,I

0.002

0.001

(0.002)

X,O,I

(<0.002)

X,I

0.05

0.0541

0.039

0.007

0.039

0.004

X,O,I

0.001

0.001

(0.001)

X,O,I



12 13 14

15

Mean STDV.

**Certified** 95% C.I.

Methods

0.0014

0.001

0.0014 0.0008 X,O,I,C

(<0.001)

X,O,I

(<0.001)

X,I

0.001

0.0005

(<0.001)

O,I

# **Participating Laboratories**

Anderson Laboratories, Inc.
Chicago Spectro Service Laboratory
Kennametal Stellite, Inc.
Laboratory Testing, Inc.
TimkenSteel Corporation

Greendale, WI Chicago, IL Belleville, ON Hatfield, PA Canton, OH Carpenter Technology - Athens Operations Exova - Burlington Laboratorio Prove Materiali S. Marco srl Latrobe Specialty Metals, A Carpenter Co. VHG Labs Tanner, AL Burlington, ON Schio, Italy Latrobe, PA Manchester, NH

### **Traceability**

Members of the "Inter-Laboratory Analysis Program" (ILAP) validate test methods and instrument performance utilizing SRMs, CRMs, and RMs produced by recognized Certifying Bodies. The specific SRMs, CRMs, and RMs applicable to the material covered by this certificate are:

ALPHA AR 667	BAS 406/1	BS 13B	ALPHA AR871	IARM 152B	IARM8A	JSS 653-11	NIST 1152	NIST 1261A	NIST 1767
ALPHA AR1652	BAS 407/2	BS 184	BAS 404/1	IARM 154A	JSS 172-4	JSS 654-11	NIST 1155	NIST 1262	NIST 2166
ALPHA AR654	BAS 408/1	BS 184A	BAS 465/1	IARM 16C	JSS 173-4	JSS 655-11	NIST 1155A	NIST 1262B	NIST 339
ALPHA AR871	BAS 409/1	BS 81N	BS 184	IARM 21A	JSS 174-4	JSS ST01	NIST 1160	NIST 1263	NIST 361
ALPHA AR874	BAS 410/2	BS 84J	CZECH 188A	IARM 21B	JSS 175-4	JSS ST01-5	NIST 1161	NIST 1264	<b>NIST C1151</b>
ASTM 1021	BAS 421	BS CA316-4	IARM 21B	IARM 21C	JSS 190-1	JSS ST02-5	NIST 1162	NIST 1754	NIST C1152
ASTM 9921	BAS 422	CZECH 181A	IARM21A	IARM 2C	JSS 191-1	JSS ST03-5	NIST 1163	NIST 1760	NIST C1153
ASTM 9922	BAS 465/1	CZECH 186A	JSS 193-1	IARM 302B	JSS 192-1	JSS ST04-5	NIST 1164	NIST 1761	NIST C1154
BAS 401/1	BAS 466/1	CZECH 187A	JSS ST01	IARM 327A	JSS 193-1	JSS ST05-5	NIST 1171	NIST 1762	NIST C1173
BAS 401/2	BAS 467/1	CZECH 187B	MAI 13-8MO	IARM 4C	JSS 194-1	LECO 501-503	NIST 1172	NIST 1763	NIST C1287
BAS 402/1	BAS 65	CZECH 188A	NIST 1163	IARM 5G	JSS 195-1	LECO 501-645	NIST 1185	NIST 1764	NIST C1288
BAS 403/1	BCS467-1	CZECH 189A	NIST 1262	IARM 6A	JSS 650-11	LECO 502-459	NIST 1230	NIST 1764A	NIST C1289
BAS 404/1	BCS474	ECRM0971	NIST 1764	IARM 9A	JSS 651-11	MBH 12X353	NIST 1260	NIST 1765	NIST C2400
BAS 405/1	BCS475	IARM 152A	NIST C1152	IARM21A	JSS 652-11	NBS 1155	NIST 1261	NIST 1766	NIST C2401

## **Homogeneity and Uncertainty**

"Uncertainty" values, as reported adjacent to certified concentration values, are based on a 95% Confidence Interval. These estimated uncertainties include the combined effects of method imprecision, material inhomogeneity, and any bias between methods. Homogeneity data from experimental XRF results are reflected in both the overall statistics and certified data. Homogeneity samples are selected by a systematic sampling procedure. The number of samples may be determined by equation 1, where N<sub>prod</sub> is the number of units produced and N<sub>min</sub> is the number of samples used for homogeneity testing. These samples are arranged in a simple randomized design such that each sample is analyzed multiple times by XRF. Homogeneity is also determined within sample using an applied version of ASTM E826. A single factor ANOVA is used to calculated uncertainty due to inhomogeneity (U<sub>hom</sub>). Uncertainty of the material is calculated by equation 2, where H=U<sub>hom</sub>, S= Standard deviation, t= t-value at 95% CI, and n= number of observations.

1. 
$$N_{min} = \max(10, \sqrt[3]{N_{prod}})$$
2.  $U_{CRM} = \frac{\sqrt{H^2 + S^2}}{\sqrt{n}} * t$ 

The International Standards Organization (ISO) definitions, expressed in ISO Guide 30–1992 list the following:

<u>Certifying Body:</u> Any technically competent body (organization or firm, public or private) that issues a reference material certificate with the information detailed in ISO Guide 31. The only generally accepted certifying body in the United States for primary standards or Standard Reference Materials (SRM) is the U. S. Department of Commerce, National Institute of Standards & Technology (NIST), Gaithersburg, MD. All other certifying bodies in the United States produce Reference Materials (RM) or Certified Reference Materials (CRM).

Reference Material (RM): Material or substance, with one or more property values that are sufficiently homogeneous and well established, to be used for the calibration of an apparatus, the assessment of a measurement method, or for assigning values to materials.

<u>Certified Reference Material (CRM):</u> Reference material, accompanied by a certificate, with one or more property values certified by a procedure, which establishes its traceability to an accurate realization of the unit in which the property values are expressed, and for which each certified value is accompanied by an uncertainty at a stated level of confidence.

<u>Inter-Laboratory Analysis Program (ILAP):</u> ASTM Standard E691-87 applies to inter-laboratory studies to "Determine the Precision of a Single Test Method", but also outlines a well thought out and logical plan for conducting an inter laboratory program involving multiple analytical techniques. Therefore, the guidelines established in ASTM E691-87 were applied to all aspects of this inter laboratory program, including the protocols for planning, handling, analysis and treatment of resulting data.

Methods of Analysis: The "Inter Laboratory Analysis Program" analyzes a wide variety of materials, and as a result, no single analytical method would provide optimum analytical results. Therefore, a combination of ASTM Standard Methods for classical wet chemistry, ICP, AA, Optical Emission, X-Ray spectrometric, and other accepted methods were used to produce analytical data. Carbon, Sulfur, Nitrogen, and Oxygen results were supplied from combustion and OE instrument procedures.

**Expiration of Certification:** The certification of this IARM is valid indefinitely, within the uncertainty specified, provided the IARM is handled and stored in accordance with the instructions stated on this certificate. The certification is nullified if the IARM is damaged, contaminated, otherwise modified, or used in a manner for which it was not intended.

Instructions for Use: The test surface is on the side opposite to the labeled surface, which includes the IARM number. The entire thickness of the unit is certified. However, the user is cautioned not to measure disks less than 2 mm thick when using X-ray fluorescence spectrometry. Each packaged disk has been prepared by finishing the test surface using a lathe. The user must determine the correct surface preparation procedure for each analytical technique. The user is cautioned to use care when either resurfacing the disk or performing additional polishing, as these processes may contaminate the surface. The minimum sample size for chips should be individually evaluated based on the analytical technique used; this would typically be greater than 0.1 grams. The material should be stored in a cool, dry location when not in use. Chips are not to be used for Oxygen analysis.

<u>Selection of Materials:</u> A "batch" or "series" is defined as a continuous length of bar produced from a single heat. The majority of IARM materials are in wrought condition; other methods of manufacture are utilized if necessary. ILAP samples are removed from equal sections from the total length of the bar. A portion of each section is converted to chips and a thin (pin) disk for analysis by classical wet chemistry, ICP, AA, and combustion procedures, and the balance remains as a thick disk for OES and X-Ray analysis.

David Coler, General Manager

**Analytical Reference Materials International** 

