

CERTIFICATE OF ANALYSIS

81X PA3.5 (batch E)

Certified Reference Material Information

Type: LEAD / ANTIMONY BINARY (CAST)

Form and Size: Disc ~40mm diameter

Produced by: MBH Analytical Ltd

Certified and supplied by: MBH Analytical Ltd

Assigned Values

Percentage element by weight

Element	Sb
Value ¹	3.49
Uncertainty ²	0.05

Definitions

- ¹ The certified value for antimony is the present best estimate of the true content of the sample. It is a panel consensus, based on the averaged result of an interlaboratory testing programme, detailed on page 3.
- ² The uncertainty value is 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 3.

Certified by:

MBH ANALYTICAL LIMITED _____

on 5th November 2011

C Eveleigh

Method of Preparation

This reference material was produced from commercially-available lead and antimony. Individual discs were prepared by sequential transfer of aliquots from the melt holding pot, and each was cast into a steel mould. 2mm has been removed from the cast face to minimise any surface effects.

Sampling

At least 10% of discs, taken from throughout the casting process, were used to monitor homogeneity within the cast. Chemical analyses were carried out on turnings taken from the working faces of several discs.

Homogeneity

The discs were checked for sample and batch uniformity using an optical emission spectrometer.

Using the combined data for each surface, standard deviation values were derived for antimony as an indicator of any non-homogeneity (as determined for the specific sample size taken by the spectrometer).

Estimation of Uncertainties

The antimony in this sample has been analysed by several laboratories, and the 95% half-width confidence interval (C(95%)) for the resultant mean value has been derived by the method shown above.

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.

Usage

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

Recommended method of use: Lead alloys are generally prepared by machining on a lathe. However, users are recommended to follow the calibration and sample preparation methods specified by the relevant instrument manufacturer.

Preparation should be the same for reference materials and the samples for test.

A minimum of three 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.

Chemical Analysis

Analysis was carried out on millings taken from samples representative of the product. It was performed by participating laboratories mostly operating within the terms of EN ISO/IEC 17025 - 2005, using documented standard methods of analysis.

The individual values listed below are the average of each analyst's results, for each method used. However, results from the same analyst but derived by different methods are stated separately.

Analytical Data

Percentage element by weight

Sample	Sb	Method
1	3.386	AAS
2	3.387	VOL
3	3.419	VOL
4	3.445	ICP
5	3.478	VOL
6	3.521	ICP
7	3.530	AAS
8	3.538	ICP
9	3.545	VOL
10	3.590	AAS
11	3.596	AAS
Mean	3.494	
Std Dev	0.076	
C_(95%)	0.051	

Notes: $C_{(95\%)}$ 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.

Analytical Methods Used

AAS - Flame Atomic Absorption

ICP - Inductively-Coupled plasma - AES detection

VOL - Volumetric, using potassium bromate.

Participating Laboratories

Universal Scientific Laboratory Pty	Milperra, NSW, Australia	NATA accreditation 0492
Laboratory Testing, Inc	Hatfield, PA, USA	A2LA accreditation 0117
Institute of Iron & Steel Technology	Shanghai, China	CNAL accreditation 0783
Luo Yang Copper Co	Luo Yang, He Nan, China	CNAL accreditation 0173
Sargam Metals Pvt Ltd	Chennai, India	NABL accreditation T025
TCR Engineering Servs Pvt Ltd	Mumbai, India	NABL accreditation T367
Raghavendra SpectroMet Laboratory	Bangalore, India	NABL accreditation T371
Institute of Non-Ferrous Metals	Gliwice, Poland	PCA accreditation AB274
AIM Metals and Alloys LP	Montreal, Canada	
Laboratory Inppamet	Calama, Chile	
Envirowales Ltd	Ebbw Vale, Wales	

Note: to achieve the above accreditation (eg NATA, A2LA, etc), test houses are required to demonstrate conformity to the general requirements of EN ISO/IEC 17025.

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.

Notes

This Certified Reference Material has been produced and certified in accordance with the requirements of ISO Guide 34-2009, ISO Guide 31-2000 and ISO Guide 35-2006, taking into account the requirements of the ISO Guide to the Expression of Uncertainty in Measurement (GUM).

The unidirectional solidification effects associated with semi-chill casting have led to the formation of inhomogeneous segregates in the rear portion of the disc. The above certification is therefore only applicable from the front face of the disc to a depth of 12mm. The rear portion of the disc, to a depth of ~3mm, is not certified.

This material is liable to superficial corrosion, and there is some possibility of microstructural changes due to recrystallisation; however, it will otherwise 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 2031, although we reserve the right to make changes as issue revisions, in the intervening period.

This sample is also available in the form of chippings.

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.