

CERTIFICATE OF ANALYSIS

82X PAg2.5R (batch D)

Certified Reference Material Information

| | |
|----------------------------|-------------------------------------|
| Type: | LEAD/SILVER ALLOY (CAST) |
| Form and Size: | Disc 40mm Diameter x 15mm Thickness |
| Produced by: | MBH Analytical Limited |
| Certified and supplied by: | MBH Analytical Limited |

Certified Analysis

Percentage element by weight

| Element | Sn | Sb | Bi | Cu | As | Ag | Zn |
|--------------------------|-------|-------|------|------|-------|------|----------|
| Value ¹ | 0.082 | 0.246 | 0.13 | 0.26 | 0.009 | 2.21 | (0.0024) |
| Uncertainty ² | 0.006 | 0.009 | 0.01 | 0.02 | 0.003 | 0.03 | 0.001 |

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

Definitions

- ¹ The certified values are derived from the results of an interlaboratory testing programme, detailed on page 3.
- ² The uncertainty values are generated from the 95% confidence interval derived from the analysis results (page 3). When appropriate, these values have been modified to account for additional information from the material homogeneity checks.

Certified by:

MBH ANALYTICAL LIMITED _____ on 19th December 2000

Method of Preparation

This reference material was produced from commercially-available lead and high-purity silver. The other minor and trace elements were added as single elements or as binary alloys. 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

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

Samples representative of the batch were checked for uniformity using an optical emission spectrometer.

Multiple measurements were taken from each surface under test.

The mean value of the material was then calculated from the multiple measurement averages.

For each of the surfaces checked, the differences between the averaged result and the overall mean value were evaluated to ensure that the homogeneity of the material satisfied the acceptance criteria defined in ISO guide 30 - 1992, and fell within 95% probability limits.

Chemical Analysis

Analysis was carried out on millings taken from samples representative of the product. It was performed by participating laboratories, normally following the requirements of ISO guide 25 - 1990, using documented standard methods of analysis.

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

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 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 | Sn | Sb | Bi | Cu | As | Ag | Zn |
|--------------------------|--------------|--------------|--------------|--------------|--------------|--------------|---------------|
| 1 | 0.075 | 0.238 | 0.106 | 0.23 | 0.006 | 2.17 | 0.0009 |
| 2 | 0.076 | 0.240 | 0.12 | 0.237 | 0.0082 | 2.18 | 0.0011 |
| 3 | 0.083 | 0.24 | 0.120 | 0.25 | 0.009 | 2.20 | 0.002 |
| 4 | 0.085 | 0.24 | 0.12 | 0.27 | 0.010 | 2.21 | 0.002 |
| 5 | 0.085 | 0.24 | 0.13 | 0.27 | 0.010 | 2.22 | 0.0028 |
| 6 | 0.089 | 0.248 | 0.135 | 0.27 | 0.012 | 2.22 | 0.003 |
| 7 | | 0.26 | 0.14 | 0.291 | | 2.250 | 0.0035 |
| 8 | | 0.26 | 0.14 | | | 2.26 | 0.004 |
| Mean | 0.082 | 0.246 | 0.126 | 0.260 | 0.009 | 2.214 | 0.0024 |
| Std Dev | 0.006 | 0.009 | 0.012 | 0.022 | 0.002 | 0.031 | 0.0011 |
| C_(95%) | 0.006 | 0.008 | 0.010 | 0.020 | 0.002 | 0.026 | 0.0009 |

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

| | | |
|---|--------------------------|---------------------|
| Bodycote Materials Testing Ltd | Middlesbrough, England | NAMAS approval 0239 |
| Metals Technology Testing | Sheffield, England | NAMAS approval 0963 |
| Rotech Laboratories Ltd | Wednesbury, England | NAMAS approval 0366 |
| University Metals Advisory Centre | Sheffield, England | NAMAS approval 0411 |
| Birmingham Assay Office | Birmingham, England | NAMAS approval 0667 |
| Universal Scientific Laboratory Pty Ltd | Milperra, NSW, Australia | NATA approval 492 |
| J B Elds Ltd | Stoke, England | |
| Shiva Analyticals Ltd | Bangalore, India | |

Analytical Methods Used

| | | | | |
|-----------|------|-----|-------------|--------------------------|
| Tin: | FAAS | ICP | | |
| Antimony: | FAAS | ICP | | |
| Bismuth: | FAAS | ICP | | |
| Copper: | FAAS | ICP | | |
| Arsenic: | FAAS | ICP | GF-AAS | |
| Silver: | FAAS | ICP | cupellation | volumetric (thiocyanate) |
| Zinc: | FAAS | ICP | | |

Notes

This product was originally certified in January 2000. The certificate has been re-formatted for clarity. It has also been changed to include additional results, and to show estimates of uncertainty.

This Certified Reference Material was originally produced in accordance with the general principles of ISO Guide 34 - 1996. This revised certificate conforms with the requirements of ISO Guide 31 - 2000.

To achieve NAMAS (UK National Measurement Accreditation Scheme) approval, test houses were required to demonstrate conformity to the general requirements of BS EN 45001, ISO Guide 25 and ISO9002.

The combination of alloying elements used in a complex cast material of this type may produce a structure which exhibits micro-porosity on the rear (engraved) surface of the disc. In addition, the unidirectional solidification effects associated with semi-chill casting may lead 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 10mm. Material to the rear of the disc, to a depth of 5mm, is not certified.

This material is liable to superficial corrosion; however, it will otherwise remain stable provided adequate precautions are taken to protect it from cross-contamination, extremes of temperature and atmospheric moisture.

The material to which this certificate of analysis refers is supplied subject to our general conditions of sale.