

CERTIFICATE OF ANALYSIS

81X PA12.5 (batch D)

Certified Reference Material Information

Type: LEAD / ANTIMONY BINARY (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	Sb
Value ¹	12.72
Uncertainty ²	0.13

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 30th November 2001

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

Discs were checked for uniformity using an optical emission spectrometer. One additional disc was checked for vertical uniformity using the same method.

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

The vertical uniformity check showed that this material is of satisfactory homogeneity for the first 10 mm of depth from the working face.

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 - 2000, 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	12.55	ICP
2	12.6	ICP
3	12.66	ICP
4	12.68	AAS
5	12.68	AAS
6	12.78	VOL
7	12.82	VOL
8	12.85	AAS
9	12.89	AAS
Mean	12.72	
Std Dev	0.12	
C_(95%)	0.09	

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.

See page 4 for details of the analytical methods used.

This material also contains approximately 0.035% Bi, 0.01% each of As and Sn, and 0.001% each of Cu and Ag.

Uncertainty Calculation

Using the individual data from the material homogeneity checks, an overall standard deviation value was derived. This value was combined with the 95% half-width confidence interval (C_(95%)) obtained from the wet analysis programme above, using the square-root of the summed squares, to derive the final uncertainty value.

Participating Laboratories

Rotech Laboratories Ltd	Wednesbury, England	UKAS accreditation 0366
London & Scandinavian Met Co Ltd	Rotherham, England	UKAS accreditation 1091
Metals Technology Testing	Sheffield, England	UKAS accreditation 0963
University Metals Advisory Centre	Sheffield, England	UKAS accreditation 0411
Universal Scientific Laboratory Pty Ltd	Milperra, NSW, Australia	NATA accreditation 492
Laboratory Testing Inc	Hatfield, PA, USA	A2LA accreditation 0117
Spectro Analytical Instruments Ltd	Kempton Park, South Africa	
University Dept of Science and Eng	Shanghai, China	

Note: to achieve National Accreditation (eg UKAS, A2LA, NATA), test houses must demonstrate conformity to the general requirements of EN ISO/IEC 17025 and ISO9002.

Analytical Methods Used

AAS - Flame Atomic Absorption:

ICP - Inductively-Coupled plasma - OES detection

Vol - Volumetric, using potassium bromate.

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.

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 ASTM E1724, ASTM E1831 and the ISO Guide to the Expression of Uncertainty in Measurement (GUM).

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, 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 2021, although we reserve the right to make changes as issue revisions, in the intervening period.

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