

# CERTIFICATE OF ANALYSIS

**85X 0494Pb1 (batch A)**

## Certified Reference Material Information

Type:	LEAD (CAST)
Form and Size:	Disc 40mm Diameter x 15mm Thickness
Manufactured by:	MBH Analytical Limited
Certified and Supplied by:	MBH Analytical Limited

## Certified Analysis

### Percentage element by weight

Element	Sb	As	Sn	Cu	Se	Bi	Pb
Value <sup>1</sup>	0.95	0.049	0.051	0.012	0.004	0.0017	(bal)
Uncertainty <sup>2</sup>	0.02	0.002	0.005	0.001	0.001	0.0003	-

Values in brackets are not certified - they are provided for information only

## Definitions

- <sup>1</sup> 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.
- <sup>2</sup> 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 31st January 2002

## **Method of Preparation**

This reference material was produced from commercial-purity lead, with additions of pure elements or binary alloys. Individual discs were prepared by sequential transfer of aliquots from the melt holding pot, and each was cast into a steel mould.

## **Sampling**

Samples for chemical analysis were taken from throughout the casting process. In addition several discs, chosen at random from the complete batch, were checked for homogeneity.

## **Homogeneity**

Discs were checked for uniformity using an optical emission spectrometer. One additional disc was checked for vertical uniformity using the same method. Multiple measurements were taken from each surface under test, and averaged. The mean value of the material was then calculated from these averages.

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.

For all elements, 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 participating laboratories mostly operating within the terms of ISO Guide 25 - 1990 or EN ISO/IEC 17025 - 2000, using documented standard methods of analysis.

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

## **Usage**

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

Recommended method of use: Lead and its alloys are generally prepared by machining on a lathe. 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.

Ideally, samples should be prepared immediately prior to use, in order to minimise the effects of oxidation.

## Analytical Data

Sample	<u>Percentage element by weight</u>					
	Sb	As	Sn	Cu	Se	Bi
1	0.91	0.045	0.04	0.010	0.003	0.0012
2	0.92	0.047	0.049	0.012	0.003	0.0012
3	0.93	0.049	0.050	0.012	0.0038	0.0015
4	0.942	0.050	0.051	0.012	0.0040	0.0015
5	0.943	0.050	0.052	0.012	0.004	0.002
6	0.95	0.050	0.052	0.0121	<0.005	0.002
7	0.956	0.053	0.055	0.013	<0.01	0.002
8	0.98		0.06	0.013		0.002
9	0.98			0.014		
<b>Mean</b>	<b>0.946</b>	<b>0.049</b>	<b>0.051</b>	<b>0.0122</b>	<b>0.0036</b>	<b>0.0017</b>
<b>Std Dev</b>	0.024	0.003	0.006	0.0011	0.0005	0.0004
<b>C<sub>(95%)</sub></b>	0.019	0.002	0.005	0.0008	0.0006	0.0003

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 accreditation 0239
Metals Technology (testing) Ltd	Sheffield, England	NAMAS accreditation 0963
Rotech Laboratories Ltd	Wednesbury, England	NAMAS accreditation 0366
Sheffield Assay Office	Sheffield, England	NAMAS accreditation 0012
University Metals Advisory Centre	Sheffield, England	NAMAS accreditation 0041
Birmingham Assay Office	Birmingham, England	NAMAS accreditation 0667
J B Elds Ltd	Stoke, England	NAMAS accreditation 1173
Universal Scientific Laboratory Pty Ltd	Milperra, NSW, Australia	NATA accreditation 0492
British Lead Mills Ltd	Welwyn, England	
Analytichem Ltd	Solihull, England	
UK Battery Manufacturer	Manchester, England	

Note: to achieve UK National Accreditation (NAMAS), test houses were required to demonstrate conformity to the general requirements of ISO Guide 25 - 1990. For Australian National Accreditation (NATA), test houses must demonstrate conformity to the general requirements EN ISO/IEC 17025.

## Analytical Methods Used

ELEMENT	RESULT No. & METHOD		
	ICP-AES	FAAS	OTHER
Antimony:	1, 3, 4, 9	2, 6, 7, 8	5 OES
Arsenic:	3, 4, 7	1, 5, 6	2 OES
Tin:	1, 4, 5	2, 6, 7, 8	3 OES
Copper:	4, 5, 7, 8	1, 2, 6, 9	3 OES
Selenium:	1, 2, 3	5, 6, 7	4 OES
Bismuth:	1, 2, 5, 6	3, 4, 7, 8	

## 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 was originally produced and certified in October 1999. The certification has been revised to incorporate additional analytical results, and to indicate formal assessments of uncertainty for each certified value. This revised certification is 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 combination of alloying elements used in a complex cast material of this type, coupled with 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. 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 original date of analysis. This certification will therefore expire in October 2019, 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.