

## Certificate of Analysis

### Certified Reference Material: LBMA AgRM4 and AgRM5

#### Trace Elements in High Purity Silver

#### General Information

The London Bullion Market Association (LBMA) promotes quality and good practice in the area of gold and silver refining and trade. The production and sale of the Reference Materials referred to herein represent part of this effort. This set of reference materials is meant to replace AgRM1 and 2. The new reference materials, AgRM4 and AgRM5, were manufactured by Tanaka Kikinzoku Kogyo K.K., Japan, under the guidance of the Steering Committee. The chosen composition reflects the needs expressed by LBMA accredited refiners.

The following table lists the elements for which certified values have been established with expanded uncertainty ( $U_{CRM} = k u_c$ , where  $u_c$  is the combined standard uncertainty calculated according to the ISO Guide [1] and  $k=2$  is the coverage factor).

Element Concentrations, mg/kg

	AgRM4	AgRM5
Al		8.5 ± 0.7
As	20.1 ± 1.4	40.1 ± 2.5
Au	10.0 ± 1.0	28.4 ± 1.3
Bi	28.4 ± 2.5	9.8 ± 0.9
Ca		2.5 ± 1.8
Cd	10.0 ± 0.5	29.2 ± 1.3
Co	9.5 ± 0.5	
Cr		8.0 ± 0.6
Cu	11.5 ± 0.6	32.4 ± 1.3
Fe	11.4 ± 0.8	28.6 ± 3.4
In	10.8 ± 0.5	
Mg		9.9 ± 1.0
Mn	11.8 ± 0.3	1.5 ± 0.2
Ni	10.3 ± 0.7	23.5 ± 1.4
Pb	29.2 ± 2.1	10.2 ± 0.8
Pd	11.1 ± 0.4	31.2 ± 1.0
Pt	9.6 ± 0.8	28.4 ± 1.2
Sb	19.1 ± 1.2	
Se	30.5 ± 2.8	10.4 ± 0.7
Si	10.1 ± 0.9	28.7 ± 2.4
Sn		10.4 ± 1.0
Te	28.6 ± 3.7	9.7 ± 3.3
Zn	10.0 ± 1.3	28.9 ± 2.7

#### Manufacture of the Reference Materials

These reference materials were produced by melting high-purity silver with master alloys in order to include trace impurities of a number of elements as shown in the table. The target level of each element was agreed upon within the Steering Committee. After casting in a vertical graphite mould designed for rapid cooling, the ingot was rolled to a thickness of 7 - 8 mm. The surfaces at the top and bottom faces were removed using a milling machine. The ingot was cut into individual pieces and machined to produce blocks 21 x 21 mm and 6 mm thickness with a weight of approximately 28 g.

## Homogeneity

Samples were cut from the rolled ingot according to a grid pattern. Fifteen pieces were selected systematically from the grid pattern which encompassed 3 samples from each of 5 evenly spaced rows of cut pieces. The samples were chosen to cover the edges and the middle of the rolled ingot. Samples were analysed at the top, bottom, and at three intermediate

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depths for each of the elements in a random order. Concentration data was obtained from the producer by using spark optical emission spectrometry. Results from these tests were evaluated using ANOVA and found to be satisfactory.

## Quantitative analysis of trace elements

Shavings from the milling of the disks were acid washed in 50% HCl, rinsed several times with distilled deionised water, and then dried in a clean hood. Portions of the shavings (25 g) of each reference material were distributed to 8 laboratories for analysis. Each participant laboratory was requested to perform their trace elements determination on at least 5 sub samples. All the laboratories determined the trace element concentrations by inductively coupled plasma optical emission spectrometry of the solutions prepared from the shavings.

**Instructions for the storage, handling and correct use of these reference materials** Keep the materials in a box to avoid exposure to industrial environment. Metallic dusts or vapour may deposit on the surface. In case of doubt, clean with ethanol, then high-purity water. If not sufficient, it is recommended that possible surface contamination be removed by placing the sample in hot 18 % HCl for approx. 10 minutes, followed by rinsing with high purity water. Once impacts of a spark spectrometer cover the surface, remove about 50 micrometers by milling, or by polishing.

**Hazardous information:** There are no hazards associated with this material.

## Intended use

These reference materials are intended to be used for the validation of analytical methods as well as for calibration of analytical instruments.

## Traceability

The results in this certificate are traceable to the SI through gravimetrically prepared standards of established purity and international measurement inter-comparisons.

**Date of certification:** 08.10.2019

**Expiration date of the certificate: 31.12.2029.** These reference materials and their certified property values are expected to remain unchanged for more than 50 years, but new analytical techniques or instruments with better characteristics of accuracy and precision are likely to appear as laboratory equipment is renewed. Accordingly, analyses may be performed again by some of the laboratories.

## Acknowledgements

The following laboratories participated in the analysis of the reference material:

Agosi, Germany	Great Wall Gold and Silver Refinery, China,
Krastsvetmet, Russia	Metalor Technologies, Switzerland
Rand Refinery PTY Ltd, South Africa	Royal Canadian Mint, Canada
Tanaka Kikinzoku Kogyo K.K., Japan	Umicore Precious Metals Refining, Belgium

## For the LBMA:

Neil Harby, Chief Technical Officer and Dr. Stewart Murray, Good Delivery Consultant

## Steering Committee members:

Chen Jie, Great Wall Gold and Silver Refinery, China,	Dr. Michael Hinds (Chair), Royal Canadian Mint, Canada
Dr. Jonathan Jodry, Metalor Technologies, Switzerland	Stefan Zorn, Agosi, Germany
Madeleine Theron, Rand Refinery PTY Ltd, South Africa	Hiroshi Sawai, Hitoshi Kosai, Tanaka Kikinzoku Kogyo K.K., Japan

## **References**

[1] Guide to Expression of Uncertainty in Measurement, ISBN 92-67-10188-9, 1<sup>st</sup> ed. ISO, Geneva, Switzerland (1993).

## **Disclaimer**

The LBMA, the Steering Committee, the manufacturer and the laboratories involved in the chemical analysis of the Reference Materials have used their best endeavours to ensure that the reference materials are homogeneous in respect of the contained elements and that their concentrations are accurately determined.

However, all assayers will recognize that there can be no absolute guarantees in relation to these parameters. For example, it cannot be ruled out totally that the reference Material may contain extraneous inclusions (though such foreign bodies would be readily detected by the using laboratory). In addition, minor deviations from complete homogeneity which are not detected by the homogeneity testing are conceivable.

London, October, 2019