



## **Tender Document for LBMA Reference Material AgRM6 and AgRM7**

### Introduction

The LBMA has previously sponsored the production and sale of AgRM4 and AgRM5, silver reference material (RM) at the 999.9‰ Ag purity level. These RMs have almost sold out and a replacement is required.

The next phase of this project is now commencing. It will again be led by Dr Mike Hinds, a consultant for the LBMA, who is working with the LBMA Referees Group to complete this project.

These RMs will be suitable for both Spark-Optical Emission Spectrometry and ICP-OES analyses.

### Composition: Table 1

Element Target Concentrations for Silver Reference Materials AgRM6 and AgRM7,  
Concentrations in µg/g

	<b>AgRM6</b>	<b>AgRM7</b>
Al	20	40
As	10	30
Au	15	100
Bi	20	50
Ca	10	35
Cd	15	40
Co	25	35
Cr	25	40
Cu	20	75
Fe	5	35
In	20	40
Mg	20	30
Mn	20	35
Ni	15	35
Pb	15	40
Pd	15	40
Pt	15	50
Sb	10	30
Se	10	40
Si	15	35
Sn	35	20
Te	15	35
Zn	15	35

**Concentrations to be within ± 10% of Target Values**

## Form

Ideally LBMA would like the following dimension: Disks of 30 mm diameter x 10 mm thickness.

Mass  $\approx$  74 g.

If it challenging to manufacture each RM with these dimensions with your equipment (i.e., casting mass, or other issue), please indicate what dimensions are possible, provided the thickness is not less than 6 mm. The disk shape is preferred however a square top face shape will be considered.

## Quantity

81 units of each RM (excluding those judged defective or used for homogeneity testing).

## Manufacturing and Analytical Testing

See the Request for Quotation and Questionnaire (attached).

## Quotation

Good Delivery Refiners interested in being contracted to produce AgRM6 and AgRM7 should submit their quotation and replies to the Request for Quotation and Questionnaire to the LBMA Chief Technical Officer, by email at [gdl@lbma.org.uk](mailto:gdl@lbma.org.uk) by Friday 28 November 2025

29 September 2025

## Request for Quotation and Questionnaire

Objective: The objective of this project is to produce a silver reference material (RM) with set amounts of impurity elements homogeneously distributed within the material. The final product will be approximately 96 individual sub-samples cut from the homogenized cast block. The material will be tested for element homogeneity (15 pieces will be used). Shavings from each of the 15 selected pieces will be sent out to approximately 10 laboratories for analysis. The sub-samples will have a mass of  $\approx 74$  g and have the following approximate block dimensions: 30 mm diameter x thickness 10 mm.

The responses to the questions in this document will enable the Referees' Working Group and the LBMA to select the manufacturer for this project. The general evaluation weighting for each section is given below.

### Experience

20%

- 1) What experience does your group have in manufacturing the type of reference material the LBMA Reference Material Project requires (see attached Table 1). Please list recent accomplishments with dates. What form was the product (disk, rod, bar, granules)?

Number and qualifications of staff who will be working on this project? How many years of experience do these people have?

Indicate the elements that you have experience in adding to silver.

### Equipment

20%

- 2) We require approximately 3.5kg of each silver RM to be cast. It is recognized that the final amount of reference material produced will be reduced because of losses in the casting, milling, and cutting stages. Do you have the equipment to do this?
- 3) Please specify casting equipment (include size, shape and material of the mould), rolling device, milling device, polishing device (paper type used), annealing equipment, and cutting equipment (specify cutter type and thickness) that you have available to use. The inclusion of pictures of

the equipment would also be very helpful.

- 4) What is the maximum amount that you can safely cast and handle in a single piece?

Time for Manufacturing and Logistics 25%

- 5) How long will it take to produce the reference material and complete the element homogeneity testing (steps “a” to “h” in Question 7)? From receipt of order to final product and given your company’s workload how many weeks would this project take?
- 6) Describe the procedures involved in shipping silver reference materials from your facility to the LBMA or another laboratory outside your country. How often has this occurred over the past two years?

Costing for Proposed Manufacturing/Testing Methodology 25%

- 7) Give a cost estimate (in \$US) for the work and shipping (material and labour excluding the cost of precious metal) for the production of the two Ag reference materials. Please provide the costing with a breakdown if possible. The project would require the following steps in the prescribed order (see table below):

<b>Manufacturing Steps</b>	<b>Cost (\$US)</b>
<p>Initial Casting</p> <ul style="list-style-type: none"> <li>a. Melting the metal matrix with the addition of elements at the concentration levels by induction melting and cold casting (preferably in vacuum). See attached tables for elements and concentrations.</li> <li>b. Determine whether elements are near target concentrations by sampling the cast block at two opposite ends determining element concentrations by ICP-AES, ICP-MS, or other equivalent method. The samples must also be cleaned to avoid surface contamination.</li> </ul>	

<p><b>Bar Preparation</b></p> <ul style="list-style-type: none"> <li>c. Anneal the cast bars at 500°C, under argon or nitrogen for a minimum of two hours. This is to homogenize the elements in the metal matrix and to remove thermal stress from casting.</li> <li>d. Mill the cast block for the following reasons: to remove uneven surfaces, to remove the outer layer of metal, and to make the large faces parallel for rolling.</li> <li>e. Roll the milled block to 6mm thickness.</li> <li>f. Re-annealing at 400°C, under argon or nitrogen for a minimum of two hours to minimize grain size.</li> </ul>	
<p><b>Standard Preparation</b></p> <ul style="list-style-type: none"> <li>g. Cut into sub-sample pieces (<math>\approx 74</math> g) and label. Dimensions to be decided by Referees Group. An identifying label “LBMA AgRM6” or “LBMA AgRM7” must be engraved onto the side of each piece.</li> </ul>	
<p><b>Homogeneity Test</b></p> <ul style="list-style-type: none"> <li>h. Conduct the homogeneity test for element homogeneity by spark AES or spark ablation ICP-AES as described in Appendix 1. The data from the homogeneity analysis must be sent via the LBMA to the Referees Group, which will decide if the material can be considered as sufficiently homogeneous.</li> </ul>	
<p><b>Preparation and Distribution of Shavings for Analysis</b></p>	

<p>i. If the materials are determined to be homogeneous then shavings must be taken from different pieces must be combined for analysis. The shavings must be cleaned in 50% hydrochloric acid for 60 minutes, washed several times, and then dried (under clean hood conditions).</p> <p>j. Divide, weigh, package, and send shavings in 11 separate containers each containing 12g to the LBMA for subsequent distribution to 10 assayers worldwide (to be nominated by the Steering Committee). These should be sent to the LBMA which will purchase them at the current gold price.</p> <p>k. The individual pieces must be weighed, labelled, packaged, and shipped to the LBMA. The price will be calculated as the total of the ruling silver price x the fine silver weight of the RMs and the quoted manufacturing cost (including the cost of shipping the RMs to the LBMA's designated vault in London.</p>	
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8) Do you have an alternative approach to making the reference materials that differs from the one outlined above? The product must have very small grain sizes that can be used with Spark - Atomic Emission Spectrometry. Please describe the different steps and provide a quotation for that approach.

Communication

10%

9) The manufacturer must communicate effectively with the Project Lead or delegate throughout the process. What means would be most effective for you to communicate? We would expect bi-monthly updates on the manufacturing process at a minimum.

10) Please include any other relevant factors that this document does not address.

**From experience from previous reference materials, we offer the following points for consideration when manufacturing the reference material:**

- To achieve the specified concentration levels and reduce the risk of element volatilization, vacuum melting should be used.
  - Vacuum melting systems have physical constraints due to limited chamber size, which restricts the allowable shape and size of the casting molds.
  - Given the mold height limits, using cylindrical or rectangular molds results in castings with a thick cross-section. Recommend water cooled copper molds if possible.
  - Such thick castings are prone to segregation due to the uneven cooling between the inner and outer regions—even when cooled promptly.
- Continuous casting is acceptable provided the desired elements are retained at the target concentrations and homogeneously distributed.
- Rolling out such castings to the desired thickness introduces risks of cracking and internal defects.
- To form the final product, outer surface machining and cutoff processing are required, causing significant material loss.
- If a batch is rejected, the machining scrap cannot be reused as feedstock nor as evaluation samples for subsequent lots.
- Additional length is required for work holding during machining, further reducing yield.

## Appendix 1. Silver Homogeneity Testing Procedure

A test for homogeneity is performed to ensure that all pieces in each sample set have the same element concentrations within the limits of the statistical test. The test will be done on final cut pieces that have been numbered and left in the same position from which they were cut. There will be 15 pieces taken for testing from different rows of the rolled strip as shown in Figure 1a. The test samples can be either rectangular or disk shaped. It is important to maintain the orientation of the top and bottom surfaces regardless of the sample shape. Figure 1a displays the samples as rectangle shapes for convenience.

- a) Prior to punching individual disks from the final rolled (and annealed) material, number and label the sample pieces as in Figure 1a, taking care to mark the top and bottom surfaces of the strip to be able to identify these during analysis by Spark Optical Emission Spectrometry.
- b) Cut the samples and place in their original order, ensuring the top side is facing up.
- c) Take 15 pieces from the total number of cut pieces in a systematic manner as shown in Figure 1a. This is necessary for the Analysis of Variance Test (ANOVA) using a randomized pattern of testing based on a block design for determining homogeneity.
- d) Arrange the selected pieces as shown in Figure 2a. Prepare the top and bottom surfaces. Then label each surface with a marking pen (see Figure 3a). Once the top and bottom surfaces of each piece have been analysed twice by Spark OES, remove about half of the silver from the top surface to determine the element concentrations at the middle depth of the disk.
- e) Overview of homogeneity testing:
  - Method: Spark OES (in the middle of each face).
  - 45 individual test sites x 2 replicates = 90 analyses.
    - 15 – disk test sites on top side.
    - 15 – disk test sites on bottom side.
    - 15 – disk test sites at half depth (middle).
  - Analyse each piece in the prescribed random order (as shown in Table 1a).
    - For Top and Bottom of pieces (rolled surface faces).
      - Analyse using Sequence 1 order.
      - Analyse using Sequence 2 order.
    - Remove half the Ag from the top of each piece (half depth).
      - Analyse using Sequence 3 order.
  - Analyse a quality control sample at beginning and every 30 analyses to verify instrument precision.
    - One or more quality control samples may be analysed to cover the elements and concentration ranges.
    - The main concern is having very reproducible results so please analyse the samples in a short period of time to avoid instrumental drift.

- f) If the analyst observes a “bad” burn (poor seal or other obvious problem) then the analysis at that site may be repeated.
- g) Reporting results:
- Place the data in the Excel workbook: *Ag Homogeneity Template.xlsx*.
  - The spreadsheet has the same random order code as shown in Table 1a. Copy the data into the spreadsheet and ensure the row, column, depth, and replicate numbers match.
  - Copy the data from the Quality Control Reference Materials into the same spreadsheet at the end of the homogeneity data.
  - Save the spreadsheet with your lab name and Ag RM number (e.g., XXXX AgRMY-Homogeneity Data yymmdd.xlsx).

**Figure 1**

After rolling, strip width has three columns and at least 32 rows of individual samples. This is a suggested sampling plan. Please implement as best you can.

1	2	3
4	5	6
7	8	9
10	11	12
13	14	15
16	17	18
19	20	21
22	23	24
25	26	27
28	29	30
31	32	33
34	35	36
37	38	39
40	41	42
43	44	45
46	47	48
49	50	51
52	53	54
55	56	57
58	59	60
61	62	63
64	65	66
67	68	69
70	71	72
73	74	75
76	77	78
79	80	81
82	83	84
85	86	87
88	89	90
91	92	93
94	95	96



Yellow filled pieces – taken for homogeneity testing

**Figure 2**

Diagram of samples taken for homogeneity testing re-arranged in a grid for testing and re-labelled for homogeneity testing.

The numbers identify rows, columns, top/bottom, e.g., **321: row 3, column 2, top 1; piece #50**. For this example, samples are taken from Figure 1 (original sample numbers in brackets from Figure 1). It is very helpful to use a marking pen to label each face with the code shown below.

## Top side

111 (4)	121 (5)	131 (6)
211 (25)	221 (26)	231 (27)
311 (49)	321 (50)	331 (51)
411 (73)	421 (74)	431 (75)
511 (94)	521 (95)	531 (96)

## Bottom side

113	123	133
213	223	233
313	323	333
413	423	433
513	523	533

## half depth from top side

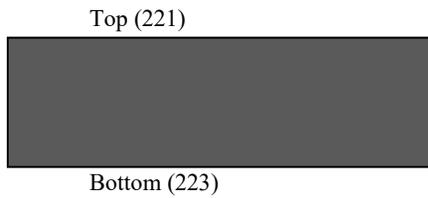
112	122	132
212	222	232
312	322	332
412	422	432
512	522	532

**Figure 3**

Diagrams of Steps: Sample Preparations, Labelling, and Analysis for Homogeneity Tests.

**Side view for Sample #26**

Sequence 1 Analysis of top and bottom surfaces of selected pieces



Sequence 2: Remove half the Ag from the top side (half depth) of the selected pieces. Analyse the new surface for each selected test piece in the order indicated in Table 1



**Table 2** Analysis Random Order Table: Homogeneity Test Sequence (example).

Replicate Set 1	Replicate Set 2
<b>Analysis of Top and Bottom</b>	
513	513
321	411
311	523
121	211
533	121
111	431
411	233
531	123
213	231
231	331
521	521
331	433
333	531
131	533
211	511
421	313
413	113
523	213
431	133
223	311
511	323
123	413
433	111
313	131
133	223
113	321
233	423
221	333
423	421
323	221
<b>Remove half depth from the top and analyse</b>	
322	332
422	212
132	532
332	132
312	422
532	222
112	232
522	412
222	512
412	122
512	522
432	312
122	432
212	112
232	322

Analyze QC reference materials at the beginning, end, and after every 30 determinations during the homogeneity test.