# Preparation and ion exchange protocol for separating Pb and Cu from Au-Ag matrices according to Bendall 2003

The protocol is adapted from Bendall 2003 and is applicable to samples with an Au- and/or Ag-dominated matrix. It provides step-by-step instructions for the full procedure from weighing in the sample to the preparation of a pure Pb solution ready for mass spectrometry.

Abbreviations:

* MQ water: Ultrapure water („Milli-Q“ water)
* \*\*\* = triple-distilled

## References

Bendall C (2003) The Application of Trace Element and Isotopic Analyses to the Study of Celtic Gold Coins and their Metal Sources. PhD thesis, Goethe-Universität Frankfurt.

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| **Date:** | | **1** | **2** | **3** | **4** | **5** | **6** | **7** | **8** | **9** | **10** | **11** | **12** |
| Step | Sample name |  |  |  |  |  |  |  |  |  |  |  |  |
| Weighing and digestion | | | | | | | | | | | | | |
| 1 | Weigh sample into empty and bleached 10 ml Savillex beaker |  |  |  |  |  |  |  |  |  |  |  |  |
| 2 | Dissolve sample in 2 ml aqua regia  (1.5 ml 6N HCl\*\*\* and 0.5 ml 7N HNO3\*\*\*) |  |  |  |  |  |  |  |  |  |  |  |  |
| 3 | Ultrasonic bath for 60 min |  |  |  |  |  |  |  |  |  |  |  |  |
| 4 | Heat at 80 °C for 120 min on a hotplate |  |  |  |  |  |  |  |  |  |  |  |  |
| 5 | Ultrasonic bath for 60 min |  |  |  |  |  |  |  |  |  |  |  |  |
| 6 | Evaporate sample solution at 80 °C on a hotplate |  |  |  |  |  |  |  |  |  |  |  |  |
| Precipitate and remove Ag as AgCl | | | | | | | | | | | | | |
| 7 | Add 1 ml 6M HCl\*\*\* to dried sample from step 6, dissolve |  |  |  |  |  |  |  |  |  |  |  |  |
| 8 | Centrifuge |  |  |  |  |  |  |  |  |  |  |  |  |
| 9 | Decant liquid |  |  |  |  |  |  |  |  |  |  |  |  |
| 10 | Add 1 ml 6M HCl\*\*\* |  |  |  |  |  |  |  |  |  |  |  |  |
| 11 | Centrifuge |  |  |  |  |  |  |  |  |  |  |  |  |
| 12 | Decant liquid (containing Pb-Cu-Au) |  |  |  |  |  |  |  |  |  |  |  |  |
| 13 | Evaporate combined liquid from steps 9 and 12 at 80 °C on a hotplate |  |  |  |  |  |  |  |  |  |  |  |  |
| Cleaning the columns, load resin + clean | | | | | | | | | | | | | |
| 14 | Fill columns with 1N HBr |  |  |  |  |  |  |  |  |  |  |  |  |
| 15 | Fill column with resin: add resin/MQ water mixture to the column |  |  |  |  |  |  |  |  |  |  |  |  |
| 16 | Clean resin in columns: 6N HCl\*\*\* |  |  |  |  |  |  |  |  |  |  |  |  |
| 17 | Wash resin in columns: MQ H2O |  |  |  |  |  |  |  |  |  |  |  |  |
| 18 | Clean resin in columns: 6N HCl\*\*\* |  |  |  |  |  |  |  |  |  |  |  |  |
| 19 | Wash resin in columns: MQ H2O |  |  |  |  |  |  |  |  |  |  |  |  |
| 20 | Clean resin in columns: 6N HCl\*\*\* |  |  |  |  |  |  |  |  |  |  |  |  |
| 21 | Wash resin in columns: MQ H2O |  |  |  |  |  |  |  |  |  |  |  |  |
| 1st chromatographic column separation with DOWEX 1x8: Removing Au | | | | | | | | | | | | | |
| 22 | Condition columns with 0.5 ml 6N HCl\*\*\* |  |  |  |  |  |  |  |  |  |  |  |  |
| 23 | Change beaker |  |  |  |  |  |  |  |  |  |  |  |  |
| 24 | Dissolve dried Pb-Cu-Au solution from step 13 in 2x 0.5 ml 6N HCl\*\*\* |  |  |  |  |  |  |  |  |  |  |  |  |
| 25 | Load solution |  |  |  |  |  |  |  |  |  |  |  |  |
| 26 | Elute 4x with 0.5 ml 6N HCl\*\*\* |  |  |  |  |  |  |  |  |  |  |  |  |
| 27 | Evaporate liquid from steps 25+26 at 80 °C on a hotplate |  |  |  |  |  |  |  |  |  |  |  |  |
| 2nd chromatographic column separation with DOWEX 1x8: Removing Cu | | | | | | | | | | | | | |
| 28 | Dissolve dried Pb-Cu solution from step 27  in 1 ml 0.6N HBr\*\*\* |  |  |  |  |  |  |  |  |  |  |  |  |
| 29 | Condition column with 0.5 ml 0.6N HBr\*\*\* |  |  |  |  |  |  |  |  |  |  |  |  |
| 30 | Change beaker |  |  |  |  |  |  |  |  |  |  |  |  |
| 31 | Load the Pb-Cu solution in 2x 0.5 ml 0.6N HBr\*\*\* |  |  |  |  |  |  |  |  |  |  |  |  |
| 32 | Elute copper with 3x 0.5 ml 0.6N HBr\*\*\* |  |  |  |  |  |  |  |  |  |  |  |  |
| 33 | Change beaker |  |  |  |  |  |  |  |  |  |  |  |  |
| 34 | Elute lead with 4x 0.5 ml 6N HCl\*\*\* (Pb seperate) |  |  |  |  |  |  |  |  |  |  |  |  |
| 35 | Evaporate separately Pb and Cu solutions from steps 32 and 34 at 80 °C on a hotplate. |  |  |  |  |  |  |  |  |  |  |  |  |