## 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 Agdominated 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.

| Date: | $\mathbf{1}$ | $\mathbf{2}$ | $\mathbf{3}$ | $\mathbf{4}$ | $\mathbf{5}$ | $\mathbf{6}$ | $\mathbf{7}$ | $\mathbf{8}$ |
| :--- | :--- | :--- | :--- | :--- | :--- | :--- | :--- | :--- |
| Step | Sample name |  |  |  |  |  |  |  |

Weighing and digestion

| 1 | Weigh sample into empty and bleached 10 ml <br> Savillex beaker |  |  |  |  |  |  |  |  |
| :--- | :--- | :--- | :--- | :--- | :--- | :--- | :--- | :--- | :--- |
| 2 | Dissolve sample in 2 ml aqua regia <br> $\left(1.5 \mathrm{ml} 6 \mathrm{~N} \mathrm{HCl**}\right.$ and $\left.0.5 \mathrm{ml} 7 \mathrm{~N} \mathrm{HNO}_{3}{ }^{* * *}\right)$ |  |  |  |  |  |  |  |  |
| 3 | Ultrasonic bath for 60 min |  |  |  |  |  |  |  |  |
| 4 | Heat at $80^{\circ} \mathrm{C}$ for 120 min on a hotplate |  |  |  |  |  |  |  |  |
| 5 | Ultrasonic bath for 60 min |  |  |  |  |  |  |  |  |
| 6 | Evaporate sample solution at $80^{\circ} \mathrm{C}$ on a hotplate |  |  |  |  |  |  |  |  | Precipitate and remove Ag as AgCl


| 7 | Add 1 ml 6M HCl*** to dried sample from step 6, <br> dissolve |  |  |  |  |  |  |  |  |
| :--- | :--- | :--- | :--- | :--- | :--- | :--- | :--- | :--- | :--- |
| 8 | Centrifuge |  |  |  |  |  |  |  |  |
| 9 | Decant liquid |  |  |  |  |  |  |  |  |
| 10 | Add 1 ml 6M HCI*** |  |  |  |  |  |  |  |  |
| 11 | Centrifuge |  |  |  |  |  |  |  |  |
| 12 | Decant liquid (containing Pb-Cu-Au) |  |  |  |  |  |  |  |  |
| 13 | Evaporate combined liquid from steps 9 and 12 at <br> $80^{\circ} \mathrm{C}$ 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 <br> 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 |  |  |  |  |  |  |  |  |

$1^{\text {st }}$ 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 <br> step 13 in $2 \times 0.5 \mathrm{ml} 6 \mathrm{~N} \mathrm{HCl}{ }^{* * *}$ |  |  |  |  |  |  |  |  |
| 25 | Load solution |  |  |  |  |  |  |  |  |
| 26 | Elute $4 \times$ with $0.5 \mathrm{ml} 6 \mathrm{~N} \mathrm{HCl}{ }^{* * *}$ |  |  |  |  |  |  |  |  |
| 27 | Evaporate liquid from steps $25+26$ at $80^{\circ} \mathrm{C}$ on a <br> hotplate |  |  |  |  |  |  |  |  |

$2^{\text {nd }}$ chromatographic column separation with DOWEX 1x8: Removing Cu

| 28 | Dissolve dried $\mathrm{Pb}-\mathrm{Cu}$ solution from step 27 in $1 \mathrm{ml} 0.6 \mathrm{~N} \mathrm{HBr}{ }^{* * *}$ |  |  |  |  |  |  |  |  |
| :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: |
| 29 | Condition column with $0.5 \mathrm{ml} 0.6 \mathrm{~N} \mathrm{HBr}{ }^{* * *}$ |  |  |  |  |  |  |  |  |
| 30 | Change beaker |  |  |  |  |  |  |  |  |
| 31 | Load the $\mathrm{Pb}-\mathrm{Cu}$ solution in $2 \times 0.5 \mathrm{ml} 0.6 \mathrm{~N} \mathrm{HBr} * * *$ |  |  |  |  |  |  |  |  |
| 32 | Elute copper with $3 \times 0.5 \mathrm{ml} 0.6 \mathrm{~N} \mathrm{HBr} * * *$ |  |  |  |  |  |  |  |  |
| 33 | Change beaker |  |  |  |  |  |  |  |  |
| 34 | Elute lead with $4 \times 0.5 \mathrm{ml} 6 \mathrm{~N} \mathrm{HCI***} \mathrm{(Pb} \mathrm{seperate)}$ |  |  |  |  |  |  |  |  |
| 35 | Evaporate separately Pb and Cu solutions from steps 32 and 34 at $80^{\circ} \mathrm{C}$ on a hotplate. |  |  |  |  |  |  |  |  |

