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**
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 HNO_3***)								
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								
	1^{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 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								
	2^{nd} 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.								