

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

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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.



Date:		1	2	3	4	5	6	7	8	9	10	11	12
Step	Sample name												
			Weig	ghing and	digestio	n		-1		ı			
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												
	·	Clea	ning the	columns,	load res	in + clea	n						<u> </u>
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 chromato	graphic	column	separatio	n with DC	WEX 1	k8: Remo	ving Au					<u> </u>
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.						