Objects from the Ur collection of the British Museum Sampling and Analytical Investigations

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Introduction

Our core project on the study of the artifacts from the Royal Tombs of Ur was developed in collaboration with the University of Pennsylvania Museum for Archaeology and Anthropology (Penn Museum, curator R. Zettler) which, like the British Museum, holds approximately a quarter of the excavation material from Charles L. Woolley's campaigns at Ur. The major part of the finds is in the National Museum of Baghdad. Initially, the focus was mostly on the objects from the Ur collection of the Penn Museum, Philadelphia. In Philadelphia, the great chance arose to analyze, non-destructively, the most prominent of the precious artifacts, but also it was permitted to sample a great number of the artifacts by drilling or clipping in order to carry out high-resolution analysis in our laboratories. In total, approximately 170 metal objects, 50 pigments and 6 calcite-alabaster vessels were sampled personally by Sabine Klein and Andreas Hauptmann at the Penn Museum.

In addition to the material from the Penn Museum, which was already accessible in generous volume, access was requested to the British Museum to its portion of the Ur material from the Royal Cemetery. The aim was to supplement the material from Philadelphia, more precisely the different material groups such as the gold, the silver and the copper-based objects. The focus was on the metallic objects at the British Museum rather than on the stone material from Ur.

The first visit to London to inspect the Ur material in the British Museum's collection was in November 2013. The first contact was Susan La Niece, Senior Metallurgist at the Department of Scientific Research and afterwards there was an appointment with Sarah Collins, Curator of the Early Mesopotamia collections, Middle East Department. After the first orientation, an application was prepared to conduct scientific analysis of British Museum material, in this case the sampling of selected objects from the Royal Tombs, and finally permission was granted and the sampling was undertaken at the British Museum in December 2014. Usually, this permission can only be received if the project can offer analytical techniques that are not held at the British Museum.

Selection of samples

In particular, permission was granted by the British Museum to sample 7 gold, 10 silver and 16 copper-based objects from the Royal Tombs of Ur. The British Museum does not permit researchers to undertake the sampling themselves. Thus, the sampling was carried out by Susan La Niece, but with the other authors presence in order to discuss the optimal sampling strategy for each of the objects.

As for the sampling, this was performed either by drilling with drill bits (1 mm) or by clipping off small pieces from the objects. For the analyses, a sample of c. 10 mg of fresh material was required. The first step was remove of the surface corrosion layer to reach the fresh material. The total depth of the drilled hole was about 5 mm. The drilling can only be applied when the material is appropriate in thickness and stability, and has to be carried out on areas of the objects that are hidden to the viewer (e.g. bottom, rims and broken edges). The hole can be filled after sampling – if requested – with colored resin or bees wax (In this case it was not requested). Alternatively, a tiny bit of the metal was cut from an existing edge of the object whenever it appeared less destructive than drilling or if drilling was not possible as in the case of sheets or very flat metal objects.

The previously approved application required that the samples taken remain the property of the British Museum, and shall be returned to the British Museum afterwards.

Details of objects sampled

The following metal objects from the Ur collection (Early Dynastic III) were pre-selected by Sarah Collins and Susan La Niece as potentially suitable for sampling. Af-

Table 1. Complete sample list of objects from Ur. The sampling was done at the British Museum by Susan La Niece on December 11, 2014.

Inv. Nr.	Ur-#	Grave	Object	Sample Position
Gold Objects				
1928-10-10-294		PG 800	Gold Ribbon	Clipped from Edge
1928-10-10-293	U 9983	PG 800	Boat-Shape Earring Fragment	Clipped from Sheet Area
1928-10-10-292	U 9983	PG 800, Body 7	Boat-Shape Earring Fragment	Clipped from Sheet Area
1928-10-10-80		PG 800	Large Boat-Shape Earring	Clipped from Pin
1929-10-17-51		PG 1054	Gold Ribbon	Clipped
1935-01-13-437	U 10586A	PG 789	Gold Ribbon	Clipped from Edge
1928-10-10-166		PG 800	Leaf from Headdress	Clipped from Edge
Silver Objects				
1928-10-10-132(A)		PG 800	Tumbler, Crushed, Undecorated	Clipped from Rim & Drilled Sample
NN-B		PG 800	Tumbler Fragment, Decorated	Clipped from Edge (Not Rim)
NN-C		PG 800	Tumbler Fragment, Decorated	Clipped from Edge (Not Rim)
1928-10-10-124		PG 789	Pin with Lapislazuli Head	Drilled Sample
1929-10-10-562		PG 1234	Pin Shaft (Head Separate)	Drilled Sample
1929-10-17-63		PG 1133	Bowl with Electrum Handles	Clipped Sample from Body
1928-12-12-149		PG 1276	Hair Ring	Clipped Sample
1928-10-10-133		PG 800	Stopper for Skin Vessel	Clipped Sample from Twisted Wire
1935-1-16-17		PG 789 (Probably)	Silver Ox Harness	Clipped from Edge
1928-10-10-131		PG 800	Sieve	
Copper and Copper-Based Objects				
1928-10-10-331		PG 777	Tubular Lance Head	Drilled Sample from Rim
1928-10-09-316		PG 580	Barbed Spearhead	Drilled Sample from Tip
1928-10-10-338		PG 789	Narrow Spearhead	Drilled into Side
1928-10-10-400		PG 800	Table with Leg	Drilled into one of the Legs
1928-10-10-320		PG 789	Spearhead	Drilled into Side
1929-10-17-632(A)		PG 1054	Stack of Bowls and Strainers	Drilled Sample from Large Bowl
1929-10-17-632(B)		PG 1054	Stack of Bowls and Strainers	Drilled Sample from Handle of Small Vessel
1929-10-17-632(C)		PG 1054	Stack of Bowls and Strainers	Loose Piece from the Body of Small Bowl
1928-10-10-399		PG 800	Ladle Handle	Drilled Sample from Handle
1928-10-10-302		PG 800	Adze Head	Drilled Sample
1928-10-10-398		PG 800	Adze Head	Drilled Sample, into Previous Drill Hole
1928-10-09-242	U 9337	PG 580	Lance/Javelin Head	Drilled Sample
1928-10-09-245	U 9337	PG 580	Lance/Javelin Head	Drilled Sample
1928-10-09-247		PG 580	Lance/Javelin Head	Drilled Sample
1928-10-09-249		PG 580	Lance/Javelin Head	Drilled Sample
1928-10-09-299		PG 580	Chisel	Drilled Sample

ter examination, sampling positions were agreed upon for drilling at an unobtrusive spot with a 1 mm diameter drill or clipping at damaged edges as appropriate.

The following listing gives an overview of the items which were originally selected for sampling and what was finally sampled in the British Museum (see also Table 1):

GOLD – sheet metal items

1. Gold ribbon fragment 1928,1010. 294, PG 800, body 7,
2. Gold boat-shaped earring fragment, 1928, 1010. 293, PG 800 (U9983),
3. Gold boat-shaped earring 1928,1010. 292, PG 800 Body 7 of Puabi's attendants - thin gold foil, crushed and damaged,
4. Gold boat shaped earring 1928,1010. 80, PG 800, Queen's attendant (with a second earring filled with bitumen),
5. Gold ribbon 1929,1017. 51, PG 1054,
6. Gold ribbon 1935,0113. 437, PG 789,
7. Leaf from gold and lapis collar / headdress - 1928, 1010.166, PG 800 Puabi attendant body.

In the course of the sampling, it was decided that the sampling of a pre-selected small, single earring 1928, 1009.165, PG 337 (thin gold foil) would cause unacceptable damage to the object. It was therefore excluded from sampling. This also applies for several of the leaf-shaped gold pendants that were previously intended for sampling which were found to be heavily torn and damaged; they were not sampled.

SILVER OBJECTS

1. Silver tumbler fragment of Royal Tomb style 1928,1010.132 crushed and fragmentary PG 800
2. Tumbler fragment, decorated, unnumbered,
3. Tumbler fragment, decorated, unnumbered,
4. Silver pin with lapis head from Kings Grave 1928,1010. 124, PG 789. Broken shaft tip and poor condition,
5. Silver pin shaft 1929,1017. 562, PG1234 head broken off shaft,
6. Silver bowl with electrum handles 1929,1017. 63, PG 1133 (Tray 66)- bent in and broken on one side,
7. Silver hair ring 1928,1010. 149 (121465), PG 1276,
8. Silver stopper (for vessel in G56) with twisted wire 1928,1010. 133, PG 800,
9. silver ox harness fragment, 1928, 1016. 17,
10. Silver sieve on mount ex gallery 56 1928,1010. 131, PG 800

In the course of the sampling, it was decided that a silver ox harness encased in wax 1935,0116. 36, PG 789 Kings Grave (probably) - radiograph film 6735 in CSR - was too deeply corroded and fragile to be sampled.

COPPER AND COPPER-BASED OBJECTS

1. Tubular lance head 1928,1010. 331, PG 777 (NB also interesting technology of rivets),
2. Barbed spearhead 1928,1009. 316, PG 580,
3. Narrow spearhead 1928,1010. 338, PG 789,
4. Table with animal legs1928.1010. 400, PG 800,
5. Spearhead 1928, 1010. 320, PG 789 (strange chemical treatment),
6. Stack of bowls and strainer (very corroded but heavy)1929,1017. 632, PG 1054, three samples,
7. Ladle handle 1928.1010. 399, PG 800,
8. Copper-alloy adze head 1928,1010. 302 PG 800 - in G56,
9. (Small, green) copper-alloy adze head 1928,1010. 398 PG 800 - in G56,
10. Small lance / javelin heads (there are 4 we could select just 3?) 1928,1009. 242 - in G56,
11. Ditto – 1928, 1009.245,
12. Ditto – 1928,1009. 247,
13. Ditto – 1928,1009. 249,
14. Chisel 1928,1009. 299, PG 580 - in G56

Elemental composition

The British Museum generally favors proper sampling and subsequent high-resolution analysis rather than non-destructive analysis. Where non-destructive methods (e.g., portable or micro-XRF) only give the main and minor elemental composition, and this also only for the surface of the objects, the analysis by mass spectrometry on samples taken by drilling from the inner core of the objects can give exact information about the body, i.e., the true composition of the objects. Lead isotope analysis provides insight to the provenance of the metals used for the production of the objects. In consequence, it gives information such as about connection routes, trading of materials and interaction within the city of Ur. It also contributes to further enlarge the reference database of lead isotope ratios and build a new one for osmium isotope ratios, which is not available at present.

The results presented concentrate on the sampled copper-based objects. Silver and gold objects are still under study; further results will be presented separately. Elemental analysis was performed in Bochum on dissolved samples with a mass spectrometer (Table 2). They reflect relatively pure copper objects and objects made of a copper-tin alloy.

Table 2. Elemental analysis of the copper-based (copper and copper-tin) samples from Ur (BM) by Mass Spectrometry (solution). [Inv.Nb. = Inventory number, norm. = normalized to 100%, detected = total detected]

Sample	Inv. Nb.	Cu	Sn	Pb	Ni	As	Fe	Ag	Sb	Zn	Co	S	Te	Hg	Bi	P	Se	norm.	detected
		in weigth%					in ppm												
Copper objects																			
5004_14	1928-10-10-331	99.68	0.010	0.072	0.005	0.127	0.106	1400	200	470	2	860	5	6.3	35	20	25	100	94.62
5006_14	1928-10-10-338	98.27	0.041	0.386	0.260	0.761	0.284	730	310	20	120	860	70	4.3	110	20	110	100	99.85
5008_14	1928-10-10-320	98.57	0.006	0.082	0.077	0.973	0.288	670	380	15	15	400	40	1.5	45	6	25	100	100.77
5011_14	1929-10-17-632(C)	96.67	0.023	0.029	0.130	2.740	0.411	270	230	35	40	250	35	<1	15	5	80	100	100.00
5014_14	1928-10-10-398	97.96	0.028	0.061	0.176	1.302	0.471	490	500	30	340	520	10	<1	55	20	20	100	102.11
5015_14	1928-10-09-242	99.03	0.007	0.016	0.237	0.366	0.345	120	180	45	150	3000	35	<1	40	4	90	100	101.07
Leg of a table																			
5007_14	1928-10-10-400	96.57	0.010	1.751	0.169	0.894	0.607	610	1200	45	50	450	15	2.1	70	6	75	100	100.63
Copper-tin objects																			
5005_14	1928-10-09-316	91.66	5.648	0.519	0.294	1.041	0.840	1000	1200	80	35	1400	15	3.4	50	80	15	100	81.68
5009_14	1929-10-17-632(A)	89.68	9.376	0.049	0.399	0.357	0.136	35	85	30	35	1400	40	2.2	25	20	110	100	95.28
5010_14	1929-10-17-632(B)	92.50	6.545	0.154	0.236	0.272	0.289	70	170	80	20	630	35	4.1	45	45	25	100	84.60
5012_14	1928-10-10-399	86.94	12.909	0.012	0.089	0.02	0.033	95	85	10	15	1200	45	<1	10	65	20	100	68.71
5013_14	1928-10-10-302	88.87	10.695	0.006	0.021	0.181	0.222	620	150	40	7	560	4	<1	8	25	20	100	99.24
5016_14	1928-10-09-245	89.45	9.505	0.368	0.103	0.229	0.348	290	340	25	15	190	15	1.2	60	20	45	100	96.15
5017_14	1928-10-09-247	91.49	7.593	0.314	0.160	0.280	0.158	280	470	15	25	75	15	<1	65	3	10	100	99.82
5018_14	1928-10-09-299	90.92	7.450	0.351	0.237	0.553	0.493	380	590	35	80	730	30	1.9	70	3	50	100	101.22
5019_14	1928-10-09-250	89.78	9.766	0.029	0.026	0.189	0.207	460	200	120	15	350	6	1.1	20	15	15	100	100.54

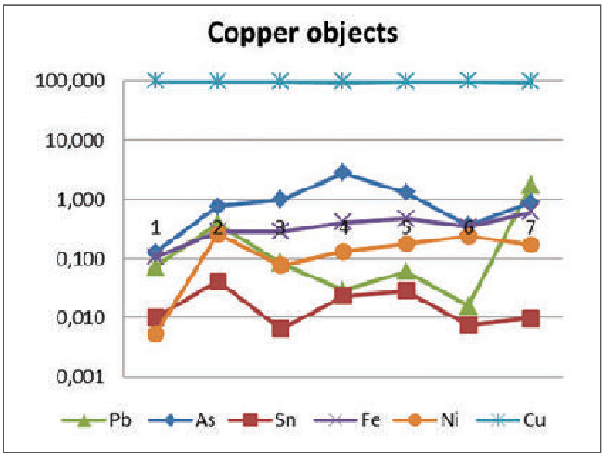

Figure 1. Elemental composition of objects made of copper. Objects are a lance head, spearheads, an adze head, a sample of a stack of bowls and strainers, a lance / javelin head and also a leg of a copper table. Values are in wt. %. Note that arsenic is variable and exceeds 1 wt. % in two samples (one adze head and one sample of the stack of bowls and strainers) and that one sample contains a high lead concentration (Leg of table, nb. 7 in diagram). Arsenic does not correlate with nickel.

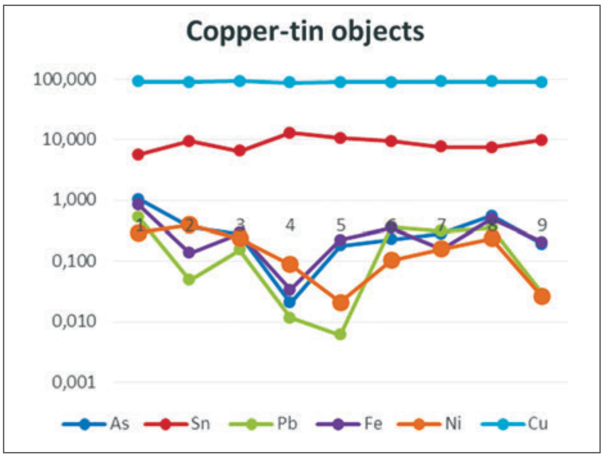

Figure 2. Elemental composition of objects made of copper-tin alloys. Objects are a spearhead, 2 samples from the stack of bowls and strainers, an adze head, a ladle handle, a chisel and lance / javelin heads. Values are in wt. %.

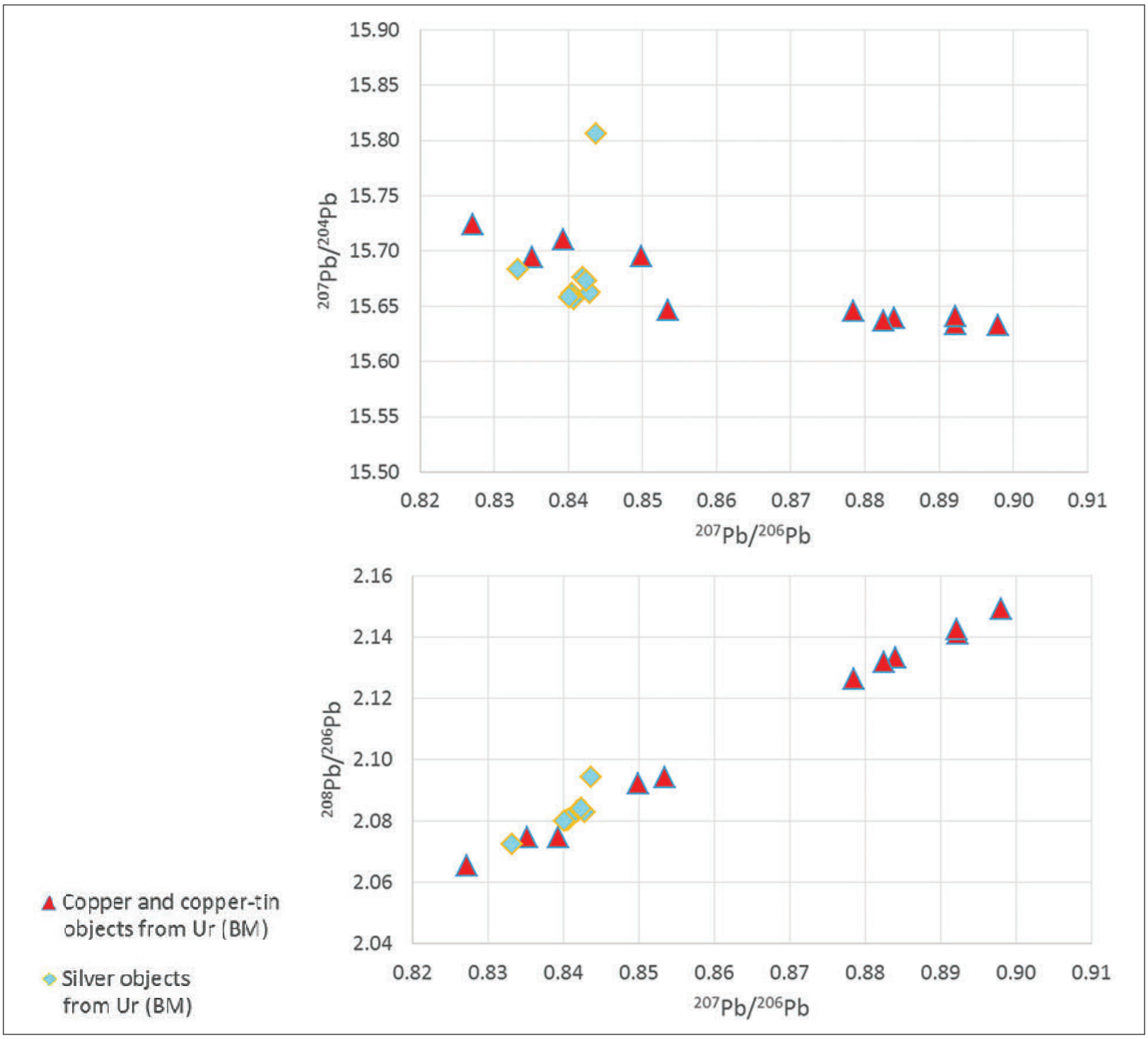

Figure 3. Binary diagram of lead isotope ratios of the copper-based objects and the silver objects sampled from the British Museum's collection (BM). The linear trend line defines the copper based objects.

Table 3. Lead isotope results for copper and copper-tin and silver objects from Ur (BM) by multicollector mass spectrometry (Frankfurt, from solution).

Sample	Inv. Nb.	$^{205}Tl/^{203}Tl$	2s	$^{206}Pb/^{204}Pb$	2s	$^{207}Pb/^{204}Pb$	2s	$^{208}Pb/^{204}Pb$	2s	$^{207}Pb/^{206}Pb$	2s	$^{208}Pb/^{206}Pb$	2s
Blank HNO_3		2.4148	0.0467	25.599	50.992	21.901	44.184	55.107	108.854	0.8464	0.1094	2.1511	0.1824
NIST 981 certified			**16.936**	**0.001**	**15.489**	**0.001**	**36.701**	**0.003**	**0.9146**	**0.0004**	**2.1670**	**0.0001**	
Std981-4		2.4139	0.0008	16.932	0.012	15.485	0.013	36.681	0.034	0.9146	0.0003	2.1664	0.0008
Std981-2		2.4093	0.0010	16.932	0.013	15.485	0.014	36.679	0.041	0.9146	0.0003	2.1664	0.0008
Std981-1		2.4091	0.0006	16.931	0.011	15.485	0.011	36.681	0.026	0.9146	0.0002	2.1664	0.0006
Silver samples													
4994-14	1928-10-10-132/A	2.4088	0.0008	18.623	0.013	15.657	0.011	38.749	0.034	0.8407	0.0003	2.0808	0.0011
4995-14	1928-10-10-132/B	2.4075	0.0011	18.619	0.012	15.676	0.012	38.788	0.033	0.8419	0.0003	2.0833	0.0008
4996-14	1928-10-10-132/C	2.4098	0.0010	18.582	0.022	15.662	0.017	38.703	0.050	0.8429	0.0003	2.0829	0.0010
4998-14	1929-10-10-562	2.4094	0.0006	18.823	0.016	15.683	0.014	39.012	0.036	0.8332	0.0002	2.0725	0.0006
4999-14	1929-10-17-63	2.4084	0.0010	18.637	0.015	15.658	0.013	38.765	0.038	0.8402	0.0003	2.0801	0.0010
5000-14	1928-10-10-149	2.4085	0.0012	18.605	0.016	15.673	0.016	38.776	0.046	0.8424	0.0002	2.0842	0.0009
5001-14	1928-10-10-133	2.3955	0.0783	18.732	0.595	15.806	0.749	39.239	2.460	0.8437	0.0134	2.0942	0.0661
5002-14	1935-1-16-17	2.4093	0.0008	18.634	0.021	15.662	0.019	38.765	0.047	0.8405	0.0002	2.0804	0.0008
Copper samples													
5006-14	1928-10-10-338	2.4119	0.0010	18.793	0.016	15.695	0.014	38.992	0.039	0.8351	0.0002	2.0749	0.0009
5008-14	1928-10-10-320	2.4104	0.0013	18.718	0.020	15.710	0.020	38.839	0.053	0.8393	0.0003	2.0748	0.0011
5014-14	1928-10-10-398	2.4086	0.0018	18.334	0.021	15.647	0.022	38.394	0.061	0.8534	0.0004	2.0942	0.0015
Leg of table													
5007-14	1928-10-10-400	2.4127	0.0007	18.468	0.018	15.695	0.014	38.640	0.038	0.8498	0.0002	2.0924	0.0006
Copper-tin samples													
5005-14	1928-10-09-316	2.4116	0.0011	17.523	0.015	15.634	0.015	37.521	0.039	0.8922	0.0004	2.1413	0.0010
5009-14	1929-10-17-632(A)	2.4092	0.0016	17.531	0.021	15.641	0.022	37.565	0.060	0.8922	0.0004	2.1427	0.0014
5010-14	1929-10-17-632(B)	2.4102	0.0010	17.410	0.017	15.633	0.017	37.421	0.052	0.8980	0.0003	2.1494	0.0012
5016-14	1928-10-09-245	2.4124	0.0007	17.693	0.019	15.640	0.018	37.742	0.047	0.8840	0.0002	2.1333	0.0006
5017-14	1928-10-09-247	2.4120	0.0009	17.720	0.017	15.638	0.017	37.775	0.048	0.8825	0.0003	2.1320	0.0010
5018-14	1928-10-09-299	2.4118	0.0008	17.810	0.015	15.646	0.013	37.870	0.032	0.8785	0.0003	2.1264	0.0008
5019-14	1928-10-09-250	2.4128	0.0012	19.011	0.015	15.724	0.016	39.269	0.047	0.8271	0.0003	2.0657	0.0010

1) Copper objects

Copper metal is represented by 6 of the samples from various types of objects: a lance head, spearheads, an adze head, a sample of a stack of bowls and strainers and a lance / javelin head (Figure 1). The objects are composed of: Cu = 96.8 - 99.7 wt. %, Sn low ≤ 0.03 wt. %, Pb low ≤ 0.3 wt. %, Arsenic (As) is variable with 0.13-2.74 wt.%, Ni ≤ 0.4 wt.%. One sample from the cast leg of a copper table had an elevated lead concentration with 1.75 wt.% Pb, Sn 0.01 wt.%, As 0.9 wt.% Fe low ≤0.06 wt.%. Despite the view that the copper adze head is supposedly a cast product, and thus must contain lead for the improvement of the casting conditions, the object contains an unexpectedly low lead concentration.

2) Copper-tin objects

This category is represented by 9 samples with a composition of Cu 92.7 - 87 wt. %, Sn variable 5.7 - 13 wt. %, As low ≤ 0.55 wt. % (one exception 1.04 wt. %), Pb low ≤ 0.5 wt. %, Fe low ≤ 0.84 wt. %, Ni 0.4-0.03 wt. % (Figure 2). Objects of this category are a spearhead, 2 samples from the stack of bowls and strainers, an adze head, a ladle handle, a chisel and lance / javelin heads.

Lead isotope composition

Lead isotope analysis was done on the copper-based samples and also the silver (Table 3). The copper-based samples plot more widely but string along a linear trend line (Figure 3) independently from the main element composition (pure copper, with arsenic, alloyed with tin). The silver samples form a homogeneous isotope group. One sample is anomalous: the twisted wire from a stopper for a skin vessel (1928-10-10-133, PG 800. The vessel is of silver, too, but imitating a skin bottle). Element analysis of the silver objects may help to clarify this difference.

Comparison with minerals from potential source regions

Most significant is the consistency of the lead isotope signature between copper and silver of the objects from Ur, and other similarities are observable for the lead isotope ratios of both the copper-based and the silver objects. Good matches are given with the a) the copper-based objects sampled from the British Museum's collection, b) copper objects from Ur published by Begemann, et al. (2010) and c) to the green copper-rich pigments from the Royal Tombs, which are presented in a parallel study (see pigments, Hauptmann, et al., this volume). Additionally, silver vessels and ingots from Troy (Romer and Born, 2009) are included in the comparison, but these are only similar in the ^{207}Pb / ^{206}Pb vs. ^{208}Pb / ^{206}Pb diagram, but do in fact show differences in the ^{204}Pb lead isotope (Figure 4).

Comparison was made with reference data of copper ores, predominantly from potential sources in Oman and Turkey. Figure 5 includes lead isotope reference data sets from a larger reference database than available in Frankfurt (own collection of reference data and incorporation of the database from F. Begemann & S. Schmitt-Strecker, Mainz). The copper ore deposits of Oman and Turkey overlap exactly in the region of the diagram where the major group of data points plot for copper objects and for silver objects from Ur. Begemann, et al. (2010) argue that many of the copper and bronze artifacts are consistent with the copper ore deposits of the Samail Ophiolite Complex of Oman. Oman was previously also suggested by our own interpretation for the green pigments (see pigments, Hauptmann, et al, in this volume). In combination with these previous observations, Oman gives strong evidence for being the raw material supplier here. However, Turkey cannot be completely excluded at present as an alternative candidate.

Conclusions

Copper was used at Ur in a wide spectrum of compositions. As has been proven previously on Ur material, the copper comprises either of pure copper metal or it is arsenic-containing or tin-containing (eg. Craddock, 1984). It is open to discussion in which cases deliberate alloying had taken place and what numerical limitation has to be drawn between naturally occurring "impurities" and deliberate addition of alloying material. The authors are convinced that compositional differences can also be caused by introduction of elements (such as arsenic, and possibly tin) through the natural mineral composition of polymetallic ores in many cases. The question arises, whether the typically postulated limit of 1 - 2 % of an element to indicate a natural impurity rather than a deliberate addition is still tenable.

Lead isotope analysis of the objects from Ur from the British Museum's collection testify to the similarity of copper-based objects with previous analysis (Begemann, et al., 2010) as excavated from various graves. Also, the lead isotope signature of the green copper-rich pigments found in the Royal Tombs appear to be identical. Surprisingly even the silver objects analyzed here appear to

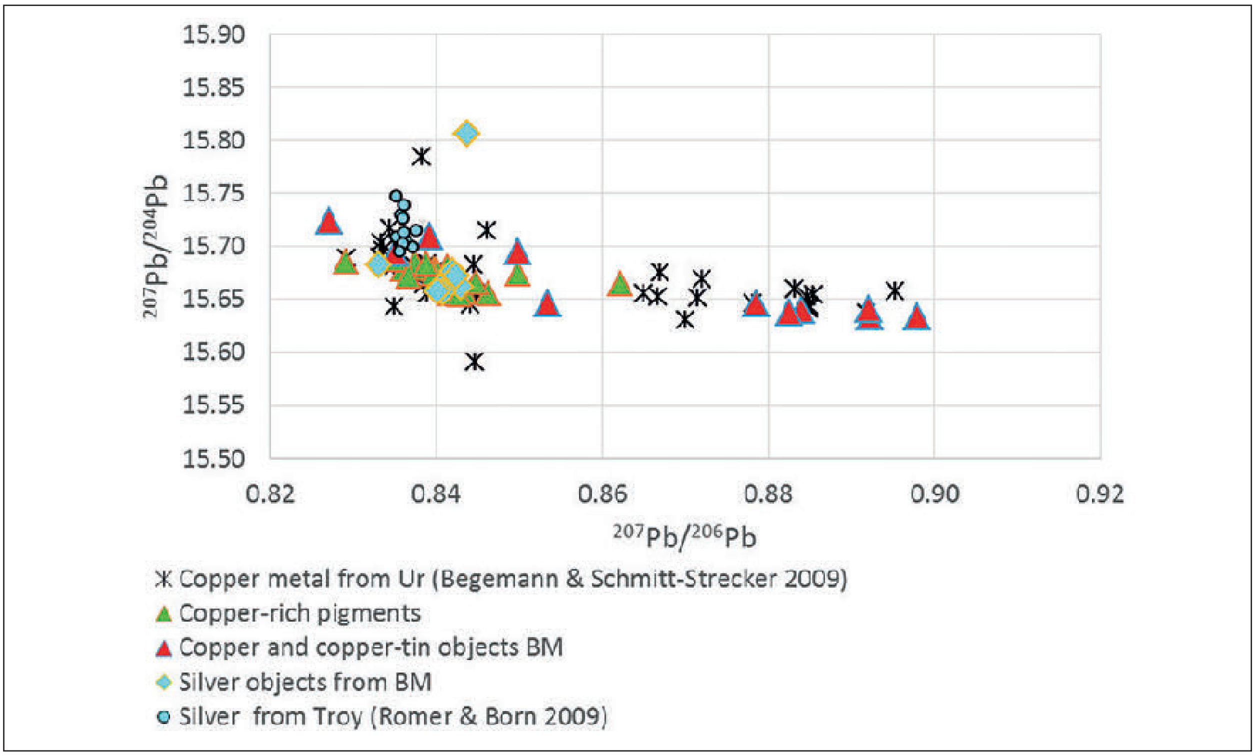

Figure 4. Binary diagram of lead isotope ratios of the copper-based and silver objects sampled from the British Museums collection (BM). The linear trend line defines the copper based objects. For comparison, copper metal from Ur (Begemann and Schmitt-Strecker, 2009) and silver vessels and ingots from Troy (Romer and Born, 2009) are plotted in the diagram.

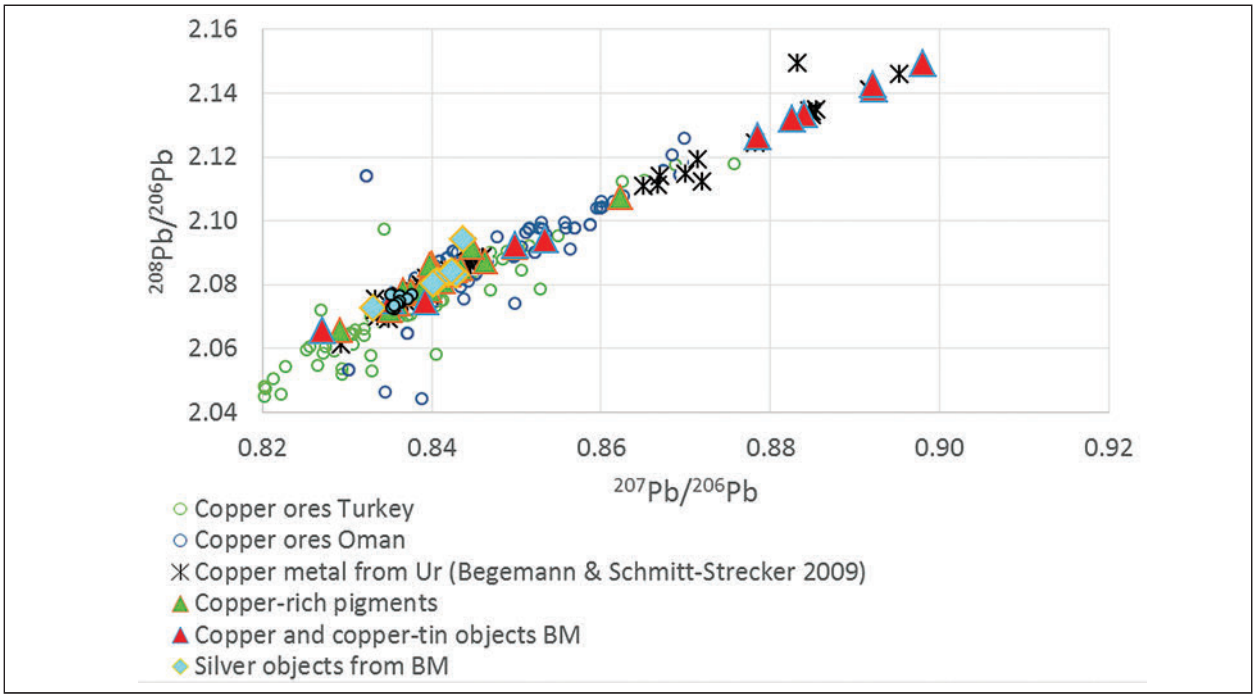

Figure 5. Binary lead isotope diagram. Comparison of objects from Ur (BM) with reference data of copper ores from Oman and Turkey.

be identical to the source or sources of a portion of the copper.

The matching lead isotope ratios point to a strong geochronological relationship between copper and silver sources, which means that silver might have originated from an identical source region to some of the copper metal, or at least from sources of similar geological age. Oman may be a potential candidate source region of the raw materials used for some of the copper objects, as there is a growing corpus of evidence suggesting this (Begemann, et al., 2010); however, Turkey may have been a source for the raw materials used for both copper and silver objects. A detailed examination of the trace elements in conjunction with the lead isotope data is still in progress and will be useful in sorting out the probable metal sources of individual objects.

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