

## ANALYSIS OF LATE BRONZE AGE GLASS AXES FROM NIPPUR—A NEW COBALT COLOURANT\*

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*A multidisciplinary study of a unique group of Late Bronze Age (LBA) ceremonial glass axe heads and other artefacts shows that these are the first significant group of glasses coloured with cobalt to be identified from the Near East. The axes were excavated from the site of Nippur, in present-day Iraq. Several are incised with the names of three kings, which dates the material to the 14th–13th centuries BC. Analysis by laser ablation inductively coupled plasma mass spectrometry (LA–ICPMS) indicates that the glass had high magnesia (MgO) and potash (K<sub>2</sub>O) associated with a plant-ash flux and was coloured blue by copper or a combination of copper and cobalt. These glasses are similar, but not identical, in major element composition to blue-coloured glasses manufactured in ancient Egypt and elsewhere in Mesopotamia in the same period. However, the Nippur cobalt- and copper-coloured glasses exhibit significantly different trace elemental compositions compared to Egyptian glass coloured with cobalt, showing that the ancient Near Eastern glassmakers had clearly identified and utilized a distinctive cobalt ore source for the colouring of this glass. Since it was previously thought that the only cobalt ores exploited in the LBA were exclusively of Egyptian origin, this new finding provides new insights on the origins of glass and how it was traded during the Bronze Age period.*

**KEYWORDS:** GLASS, LATE BRONZE AGE, COBALT, LASER ABLATION ICPMS, NIPPUR, KASSITE

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## INTRODUCTION

This paper describes a group of Late Bronze Age (LBA) glass objects with a unique elemental composition and cobalt colourant not previously identified amongst the glasses from the ancient Near East. These objects were excavated in 1890 at Nippur in southern Iraq (see Fig. 1) by the First Expedition (Peters 1897; Barag 1970). The glass was found in a box, together with an array of other small objects, belonging to a Parthian period building dated by the excavators to the late first or early second centuries AD. The most important finds contained in the box were over 100 fragments of glass axe heads (Peters 1897; Clayden 2011). Many of these glass fragments had texts inscribed on them in Akkadian, including the names of three Kassite kings, *Kurigalzu II* (1332–1308 BC, short chronology), *Nazi-Maruttash* (1307–1282 BC) and *Kashtiliashu IV* (1232–1225 BC) (Barag 1970). As discussed at length by Clayden (2011), the inscriptions on the ceremonial axes provide convincing philological evidence that these objects date to between the 14th and the 13th centuries BC, and are much earlier than other items in the box, as well as the building in which they were found. A logical interpretation for the history of the axes seems to be that they were uncovered during the building or renovation work at a later period and reburied on the site as part of a possible votive offering. What the glass axes were used for is equally unclear. They could not have been used functionally, but must have had important ritual significance. The collection is now in the University of Pennsylvania Museum of Archaeology and Anthropology in Philadelphia, USA.

The origins and production of glass in the ancient Near East have been the subject of intensive and ongoing investigation for several decades. It has been established that glass was first produced in a regular and controlled way in the middle of the second millennium BC (Barag 1962; Peltenburg 1987; Lilyquist and Brill 1993). The first glass was probably produced in the Near East in the region that is now northern Iraq and Syria. Egypt subsequently produced its own glass from at least the middle of the 15th century BC (Lilyquist and Brill 1993). Glass of this period was

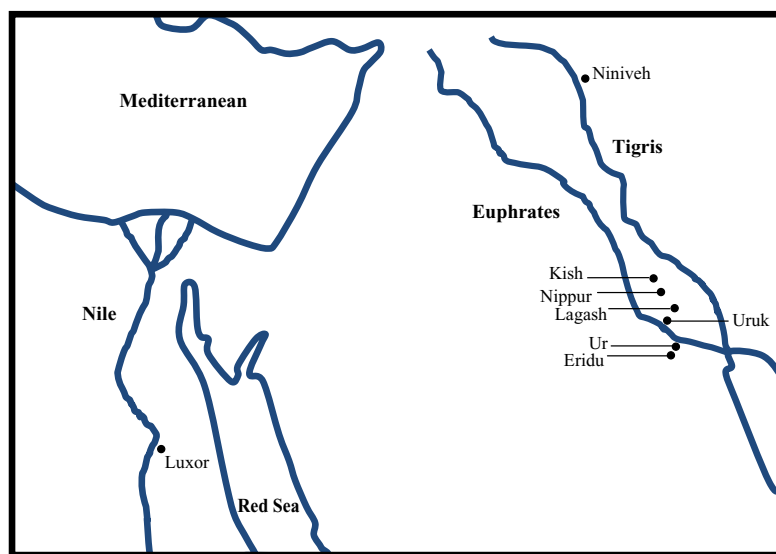


Figure 1 A map of the ancient Near East, showing the location of Nippur and other locales.

made with quartz pebbles and a plant-ash flux, and was nearly always coloured (Turner 1956b; Shortland 2000). The most common colour throughout the areas where this glass is found was various shades of blue, although white, yellow, pink, black, green, red, brown and colourless glass are also found, though less commonly (Kaczmarczyk and Hedges 1983; Lilyquist and Brill 1993).

The colour blue is the focus here since it is the colour of the Nippur glasses described. Blue glass in the LBA is coloured with either copper and/or cobalt, with cobalt being used extensively in dark blue glasses found in Egypt. Interestingly, this dark blue shade is absent from lighter blue glasses thus far identified as being produced in the Near East, where glasses coloured with copper were the norm (Henderson 1998; Shortland and Eremin 2006). In fact, no cohesive group of objects coloured with cobalt has been identified in the Near East, apart from rare, scattered finds which have been characterized as imports from Egypt.

When coloured using cobalt, Egyptian glasses have elevated levels of aluminium, manganese, nickel and zinc compared to any other LBA glasses coloured blue with copper (Sayre and Smith 1961). This elemental signature for Egyptian cobalt-coloured glass has been traced to an ore source in the Western Oases of the Egyptian desert, where cobalt-bearing alums have been found to have this specific elemental fingerprint (Kaczmarczyk 1986). All LBA glasses coloured with cobalt analysed prior to this analysis have been found to share this elemental composition and were therefore linked to this single cobalt source. This elemental signature has been used to document the trade of Egyptian glass to Tell Brak in northern Mesopotamia (Henderson 1998) and Mycenaean Greece (Walton *et al.* 2009), and also to identify the origin of blue ingots found on the LBA *Uluburun* shipwreck (Jackson and Nicholson 2010). As we describe below, the compositions of the glasses coloured with cobalt and copper presented here are considerably different from the glasses from Egypt and thus constitute a new type of cobalt-coloured glass unique to the ancient Near East.

#### MATERIALS

A total of 47 samples, enumerated in Table 1, were taken from the glass artefacts. Of these, 16 were definitely from glass axes, four were described as hair panels, two as rods, three as horns and the remainder were fragments believed to come from axes. The samples are denoted by the museum number, where they correspond to individual objects, such as the axes, horns and so on. These museum numbers are in varying formats, including B2496.*x* (where *x* can be a number or letter) and individual numbers such as B8695. Some axes are in fragments, and fragments with different numbers have been found to join each other. Such joins are noted in Table 1. Large numbers of fragments occur within three boxes labelled B2496/1, B2496/2 and B2496/3. Multiple fragments were removed from these and denoted by the box from which they came and a running sample number; for example, B2496/1(1) is fragment 1 from box B2496/1. It should be noted that sampling of the boxes of fragments was random, and it is not possible to determine how many objects are represented by these fragments or what the original objects might have been. Five of the fragments were opaque turquoise glass; all the others were dark blue translucent to semi-translucent glass.

#### METHODOLOGY

Samples were mounted in resin and polished using a series of diamond pastes with a 1 µm grit final polish. All samples were examined by laser ablation inductively coupled plasma

Table 1 Sample numbers and sample types, with optical descriptions

Museum number	Type	Texture
<i>Group 1</i>		
B2496/3(5)	Fragment	Fine white crystals
B2496/3(6)	Fragment	Scattered crystals, bubbles
B2486/1(3)	Fragment	White crystals, some alteration
B2496/2(2)	Fragment	Fine white crystals
B2496/3(10)	Fragment	White crystals
<i>Group 2a</i>		
B2496/3(3)	Fragment	Fine crystals
B2496/3(4)	Fragment	White crystals
B2496/3(7)	Fragment	
B2496/1(5)	Fragment	Scattered crystals, bubbles
B2496/2(1)	Fragment	Clear crystals, white crystals
B2496/2(5)	Fragment	Clear crystals, white crystals
B2496.2	Glass hair panel	White crystals, bubbles
B2496.8	Glass hair panel	Some crystals
B2496.10'	Glass rod?	Clear crystals, white crystals
B2496.22	Thin glass panel	Abundant fine crystals
B2496.27	Axe fragment	Clear crystals, white crystals
B2496.42	Glass rod?	Fine crystals, some coarser
B2496.43	Glass hair panel	Abundant crystals
B2496/3(12)	Fragment	Clear crystals, white crystals
B2496.37	Glass hair curl	Clear crystals, white crystals, bubbles
<i>Group 2a1</i>		
B8686	Axe fragment (inscribed <i>Kashtiliashu</i> )	Abundant fine crystals
B2496X	Fragment	Altered, hard to tell
B2496D	Glass horn	Very altered
<i>Group 2b</i>		
B3738	Axe fragment	Abundant elongate crystals
B4558	Axe fragment (inscribed <i>Nazi-Maruttash</i> )	Abundant fine crystals
B8681a	Axe fragment (inscribed <i>Nazi-Maruttash</i> )	Abundant dendritic crystals
B2496/1(1)	Fragment	Dendritic and elongate crystals
B2496/1(2)	Fragment	Dendritic crystals
B2496/1(4)	Fragment	Dendritic and elongate crystals
B2496/1(8)	Fragment	Dendritic crystals
B2496/3(1)	Fragment	Fine crystals
B2496Q	Fragment	Dendritic crystals
B2496/3(11)	Fragment	Fine crystals
B2496/3(13)	Fragment	Dendritic crystals
B2496/3(14)	Fragment	Fine dendritic crystals
B2496/3(15)	Fragment	Fine crystals
B2496/3(16)	Fragment	Fine crystals
B2496/3(17)	Fragment	Fine crystals, white crystals?
B8685	Axe fragment (inscribed <i>Nazi-Maruttash</i> )	Very altered, crystals present?
B2496G	Glass horn	Dendritic and coarse
<i>Group 2b1</i>		
B8762.a1	Axe fragment	Euhedral crystals
B4550/1	Axe (inscribed <i>Kurigalzu II</i> ), joins B4550/2 and B4544	Coarse euhedral crystals
B4550/2	Axe (inscribed <i>Kurigalzu II</i> ) joins B4550/1 and B4544	Coarse euhedral crystals
B4544	Axe (inscribed <i>Kurigalzu II</i> ), joins B4550/1 and B4550/2	Coarse euhedral crystals
B2496.5	Fragment	Coarse euhedral crystals
B2496/1(3)	Fragment	Coarse euhedral crystals

mass spectrometry (LA-ICPMS), scanning electron microscopy with energy-dispersive microanalysis (SEM-EDS) and X-ray diffraction (XRD). SEM-EDS was undertaken with a JEOL JSM-640LV scanning electron microscope at the Museum of Fine Arts, Boston, operated at 20 kV in high-vacuum mode on carbon-coated samples. XRD was performed using a Bruker D8 Multipurpose Diffractometer at the Massachusetts Institute of Technology. Samples were collected over a  $2\theta$  range of 30–90°, using Cu-K $\alpha$  radiation at 40 kV and 40 mA. The primary X-ray beam was collimated to approximately 0.5 mm using a monocapillary and the diffracted X-rays were detected using a GADDS detector. LA-ICPMS was undertaken at the Getty Conservation Institute, using a GBC Optimass 9500 Inductively Coupled Plasma Time-of-Flight Mass Spectrometer coupled to a New Wave Laser UP213 laser ablation, and an ablation spot of 60  $\mu\text{m}$  was used. Further details are given in a previous publication (Walton *et al.* 2009). Throughout the analysis by LA-ICPMS, Corning A standard was run to check for accuracy and drift. The major element values for Corning A were taken from Vicenzi *et al.* (2002). The results are shown in Table 2, and show good agreement for major elements.

Table 2 LA-ICPMS analyses of Corning A standard, made throughout the analytical runs. Accepted values are converted to ppm from the per cent oxides given in Vicenzi *et al.* (2002).  $\Delta$  is the difference between the analysed value and the accepted value, expressed as a percentage.  $\Delta^1$  is a comparable set of analyses done of Corning A by LA-ICPMS from Shortland *et al.* (2007)

	Mean (n = 22)	%RSD	Accepted	$\Delta$ (%)	$\Delta^1$
Na	103 994	2	106 083	-2	-
Mg	16 488	9	16 043	3	-
Al	5 720	16	5 291	7	-
Si	315 717	1	310 883	2	-
K	24 446	11	23 817	3	-
Ca	38 322	8	35 954	6	-
Ti	4 288	12	4 736	-10	-11
Fe	6 071	14	7 624	-26	-11
Cu	8 725	10	9 586	-10	-18
Mn	7 106	6	7 745	-9	-11
Cr	25	29	7	73	161
Co	1 236	8	1 337	-8	-11
Ni	215	16	157	27	2
Zn	469	16	402	14	2
Rb	84	10	91	-9	-11
Sr	788	9	1 184	-50	-27
Zr	41	14	37	9	8
Sn	1 302	11	1 812	-39	-34
Sb	12 285	11	13 174	-7	-
Ba	3 779	10	4 210	-11	-7
Pb	740	28	928	-25	-36
Bi	14	0	9	34	-13

## RESULTS

*Glass appearance*

As may be seen in Figure 2 (a), a hand specimen of lapis lazuli is compared with a Nippur glass fragment shown in Figure 2 (b). Based on the deep blue colour and the white mottling in the glass, the glassmakers were ostensibly producing a synthetic, yet visually convincing, analogue for the mineral lapis lazuli. While glass deterioration may account for the whitish/brown crust observed on the exterior surface of the glass in Figure 2 (b), the white within the glass interior is due to microcrystalline phases formed during the fabrication of the glass. In Figures 2 (c) and 2 (d), these microcrystalline phases may be observed in polished cross-sections of the glass in a SEM–EDS using the backscattered electron imaging mode (BSE).

Compositional analysis by SEM–EDS correlated to XRD analysis identified three crystalline phases: a major phase of combeite, a minor phase of calcium antimonate and traces of calcium–

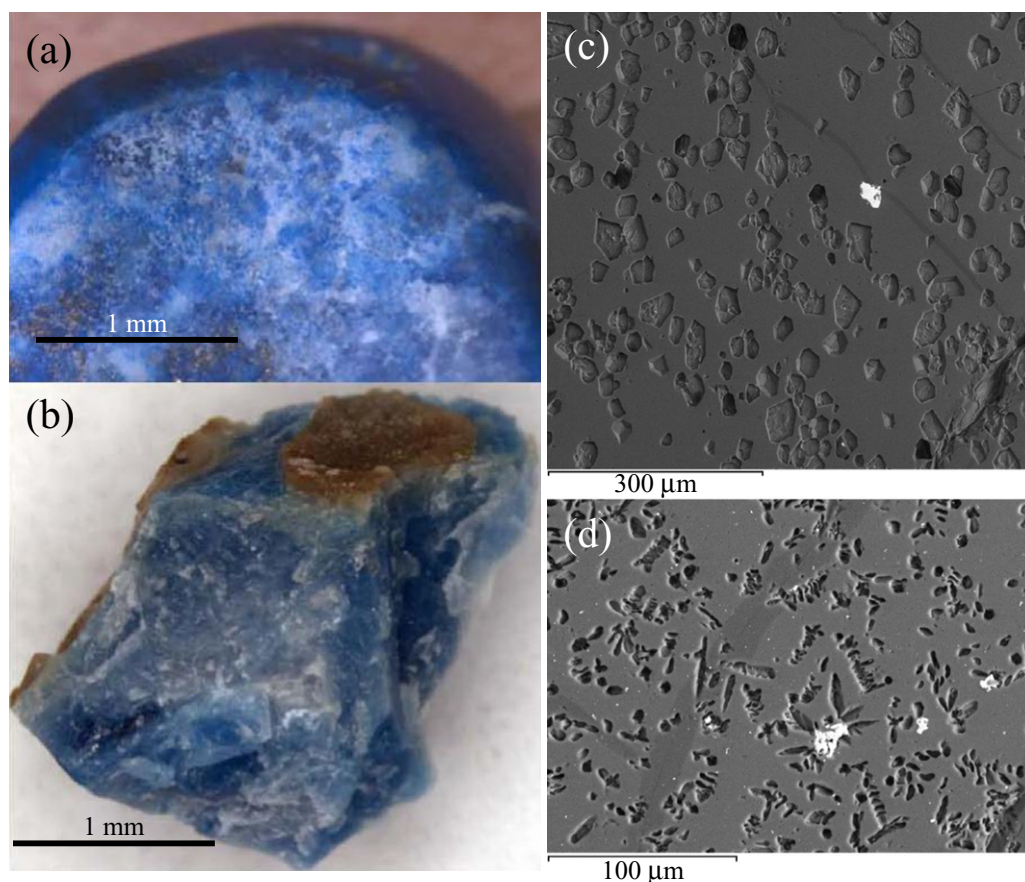


Figure 2 The appearance of the glass. (a) A lapis lazuli mineral specimen. (b) Nippur glass made in imitation of lapis lazuli. (c) A detail of euhedral combeite crystals in B2496/1(3), with a single crystal of calcium antimonite ( $\text{Ca}_2\text{Sb}_2\text{O}_7$ ) seen as a high Z-contrast particle. (d) A detail of dendritic crystals of combeite in B2496/3(16) as well as scattered calcium antimonite.



magnesium pyroxene (not shown in the images). Textural differences in the combeite phase divide the glass into two distinct groups. One group of glass is composed of coarse euhedral crystals of combeite, as shown in Figure 2 (c). The other group has finer dendritic and acicular crystals of combeite, as shown in Figure 2 (d). The textural distinction suggests variations in the cooling regime under which these phases formed.

The presence of combeite, which has the approximate formula of  $\text{Na}_2\text{Ca}_2\text{Si}_3\text{O}_9$ , is unusual for LBA glasses and is normally found in carbonatite volcanic rock (Fischer and Tillmans 1987; Dawson *et al.* 1989). The occurrence of this phase in these glasses and its intentional addition to the glass to impart the appearance of lapis lazuli are part of an ongoing investigation of these glasses and will not be discussed at length here.

### Glass composition

The major and trace element compositions of the Nippur glasses as determined by LA-ICPMS are presented in Table 3. The alkali levels of the glasses are usually between 15% and 20%  $\text{Na}_2\text{O}$ , with potash between 2% and 6% and magnesia mostly between 4% and 6%. Based on this compositional data, the Nippur glasses correspond to the typical technology of producing glass in the LBA from a mixture of plant ashes, quartz pebbles and lime (Turner 1956a; Lilyquist and Brill 1993). Due to possible issues of weathering in the lower soda glasses, it was decided that glasses with <10%  $\text{Na}_2\text{O}$  would not be included in averages (in bold) for the different groups described above, or plotted on the biplots. They are, however, included in the table for completeness.

As may be observed from Table 3, only five glass fragments were found to be coloured only with copper (1.7–2.3%  $\text{CuO}$ ) and are labelled as Group 1. Trace elements in these Group 1 glasses show they are composed of very little cobalt (<10 ppm Co) and lead (<20 ppm Pb) as well as low nickel (<220 ppm Ni) and arsenic (<100 ppm As). They are compositionally very similar, and all of them come from fragments of glass without any identifiable structure to suggest the type of object from which it came. Furthermore, no crystalline phases, other than calcium antimonite, were identified in these glasses.

The vast majority of the glasses (42 fragments) belong to a category coloured by both copper and cobalt, labelled as Group 2 in Table 3. In Figure 3, a bivariate plot of cobalt versus the sum of manganese, nickel and zinc, shows that the Nippur Group 2 fragments can be easily differentiated on the basis of the levels of these transition metals from Egyptian cobalt-coloured glasses (data extracted from Shortland *et al.* 2007), with the Nippur glasses containing substantially less of these trace elements. On average, the amount of cobalt in the Egyptian glasses is half that found in the Nippur glasses. Also, the Nippur glasses do not exhibit correlations between cobalt and aluminium, manganese, nickel and zinc as observed in the Egyptian glasses.

There are significant compositional variations within Group 2. As also indicated in Figure 3, the Nippur glasses divide into two groups on the basis of nickel content. Group 2a has Ni between 158 and 300 ppm and Group 2b has Ni between 442 and 835 ppm. Group 2a can be further differentiated from Group 2b on the basis of their respective Pb contents. As is shown in a plot of Pb versus Co (Fig. 4 (a)), Group 2a has lead levels at less than 700 ppm, whereas Group 2b has lead present at greater than 3000 ppm. Lastly, Group 2a has relatively low copper (<8000 ppm Cu) compared to Group 2b, which has copper levels greater than 9000 ppm.

A further distinction can be drawn within Group 2b between the majority of samples with low arsenic and high antimony (<160 ppm As, >3600 ppm Sb) and a smaller group with high arsenic and low antimony (>300 ppm As, 1845 < Sb < 2200 ppm), as can clearly be seen on the bivariate

Table 3 LA-ICPMS analyses of the glasses

Museum number	wt%										ppm												
	Na <sub>2</sub> O	MgO	Al <sub>2</sub> O <sub>3</sub>	SiO <sub>2</sub>	K <sub>2</sub> O	CaO	Ti	Fe	Cu	Mn	Cr	Co	Ni	Zn	As	Rb	Sr	Zr	Sr	Sb	Ba	Pb	Bi
<i>Group 1</i>																							
B2496/3(5)	17.02	5.24	0.59	63.45	3.73	5.68	165	2.444	13.848	324	11	4	148	153	68	11	388	27	12	16.868	72	16	0
B2496/3(6)	18.25	5.08	0.62	62.77	3.18	5.76	226	2.239	15.209	319	16	8	151	122	57	12	370	18	11	15.952	52	17	0
B2486/1(3)	18.20	5.28	0.60	63.05	2.79	5.70	381	2.469	14.051	301	10	6	144	101	60	9	414	24	10	17.073	71	15	0
<b>Average (three)</b>	<b>17.82</b>	<b>5.20</b>	<b>0.60</b>	<b>63.09</b>	<b>3.23</b>	<b>5.71</b>	<b>257</b>	<b>2.384</b>	<b>14.370</b>	<b>314</b>	<b>12</b>	<b>6</b>	<b>148</b>	<b>125</b>	<b>62</b>	<b>11</b>	<b>391</b>	<b>23</b>	<b>11</b>	<b>16.631</b>	<b>65</b>	<b>16</b>	<b>0</b>
<i>SD</i>	<i>0.70</i>	<i>0.11</i>	<i>0.02</i>	<i>0.34</i>	<i>0.47</i>	<i>0.05</i>	<i>112</i>	<i>126</i>	<i>734</i>	<i>12</i>	<i>3</i>	<i>2</i>	<i>4</i>	<i>26</i>	<i>6</i>	<i>2</i>	<i>22</i>	<i>4</i>	<i>1</i>	<i>597</i>	<i>12</i>	<i>1</i>	<i>0</i>
B2496/2(2)	9.15	5.69	0.66	70.92	2.75	5.92	169	2.676	15.834	374	23	5	216	134	78	9	414	40	13	19.408	72	20	0
B2496/3(10)	9.11	4.86	0.63	72.05	2.04	5.91	170	2.928	18.335	358	16	9	184	133	99	9	364	35	14	20.619	88	23	1
<i>Group 2a</i>																							
B2496/3(3)	18.17	4.57	1.02	65.04	3.30	4.78	265	4.668	7.410	265	25	1.273	277	150	80	30	236	17	30	9.774	38	20	0
B2496/3(4)	17.37	4.83	1.02	64.68	3.49	5.48	178	5.015	6.800	306	26	1.277	225	156	80	22	249	25	27	10.142	50	23	0
B2496/3(7)	17.34	5.23	1.27	63.97	2.90	6.43	294	4.471	6.133	287	8	1.232	245	90	92	26	299	38	33	9.001	72	18	1
B2496/1(5)	17.83	4.60	1.00	64.66	3.58	4.85	276	4.921	7.431	273	22	1.285	249	159	78	29	228	24	28	12.335	55	19	0
B2496/2(1)	15.53	4.60	1.65	65.97	3.69	5.51	247	5.714	4.310	249	32	1.626	175	88	88	28	251	16	16	10.858	68	49	1
B2496/2(5)	17.46	4.83	1.04	63.58	3.48	5.61	198	4.859	6.572	282	10	1.228	231	153	89	27	246	17	30	17.842	68	23	0
B2496.2	18.88	4.27	0.99	64.48	2.81	5.20	222	5.268	7.623	274	32	1.331	300	179	105	26	211	20	27	10.824	49	22	1
B2496.8	18.21	4.65	0.96	63.83	2.94	5.97	171	5.063	7.271	251	13	1.368	243	132	92	28	238	16	28	12.204	30	21	0
B2496.10'	17.86	3.88	1.43	63.66	4.98	5.13	250	5.358	4.662	217	16	1.640	202	74	96	29	224	29	19	11.058	61	60	1
B2496.22	12.84	4.85	1.07	68.49	3.30	6.17	208	5.643	7.803	293	29	1.568	251	137	91	26	257	22	27	9.124	49	19	0
B2496.27	18.92	4.14	1.06	64.08	3.02	5.56	197	5.071	7.367	256	20	1.343	242	122	93	33	247	16	32	10.104	69	18	0
B2496.42	18.78	5.25	1.02	63.27	2.43	6.31	228	5.068	7.056	294	29	1.552	158	101	72	24	325	16	2	7.872	60	15	0
B2496.43	15.35	4.77	1.01	65.85	3.10	5.92	198	5.379	7.387	293	21	1.436	282	130	107	27	235	12	32	15.984	52	20	0
B2496/3(12)	17.91	4.21	1.51	64.49	3.37	5.22	237	5.610	4.627	211	44	1.696	168	107	104	25	227	20	14	12.537	68	50	1
<b>Average (14)</b>	<b>17.32</b>	<b>4.62</b>	<b>1.15</b>	<b>64.72</b>	<b>3.31</b>	<b>5.58</b>	<b>226</b>	<b>5.151</b>	<b>6.604</b>	<b>267</b>	<b>23</b>	<b>1.418</b>	<b>232</b>	<b>127</b>	<b>90</b>	<b>27</b>	<b>248</b>	<b>20</b>	<b>25</b>	<b>11.404</b>	<b>56</b>	<b>27</b>	<b>0</b>
<i>SD</i>	<i>1.68</i>	<i>0.39</i>	<i>0.23</i>	<i>1.35</i>	<i>0.59</i>	<i>0.52</i>	<i>37</i>	<i>367</i>	<i>1.204</i>	<i>28</i>	<i>10</i>	<i>1.65</i>	<i>43</i>	<i>31</i>	<i>10</i>	<i>3</i>	<i>30</i>	<i>7</i>	<i>9</i>	<i>2.708</i>	<i>13</i>	<i>15</i>	<i>0</i>
B2496.37	17.10	3.95	0.97	60.10	8.59	5.96	201	4.757	7.231	243	22	1.239	223	146	97	29	226	16	31	11.737	44	22	0
<i>Group 2a1</i>																							
B8686	20.22	5.96	1.22	62.16	1.68	5.73	212	4.323	4.517	330	23	1.203	243	45	66	23	277	15	4	12.069	96	591	13
B2496X	14.66	6.20	1.27	66.55	1.78	5.89	525	5.062	5.023	312	30	1.257	278	65	71	29	289	8	3	15.131	90	655	10



Average (two)	17.44	6.08	1.25	64.35	1.73	5.81	369	4 693	4 770	321	26	1 230	260	55	68	26	283	11	3	13 600	93	623	12
SD	3.93	0.17	0.04	3.11	0.07	0.12	221	523	358	13	4	38	25	14	4	4	9	5	1	2 165	5	45	2
B2496D	2.23	8.22	1.59	77.57	3.40	4.07	297	7 784	5 315	554	41	1 993	285	145	36	22	401	24	4	4 876	81	321	4
<i>Group 2b</i>																							
B3738	15.76	5.00	1.09	61.69	3.42	9.57	242	6 484	9 442	308	27	2 298	667	93	102	23	355	14	7	3 648	92	3 255	51
B4558	11.77	5.86	1.02	68.85	3.49	4.91	202	5 406	11 906	325	44	1 722	650	140	102	30	291	13	8	7 463	88	4 260	68
B8681a	18.74	4.94	1.03	61.48	3.37	6.74	219	6 200	10 953	329	43	2 474	835	111	93	24	295	9	5	3 929	65	3 332	74
B2496/1(1)	18.56	5.23	0.82	60.10	2.28	8.54	526	4 897	12 493	324	22	1 953	620	111	159	24	412	13	60	9 146	109	4 906	78
B2496/1(2)	11.93	5.53	1.02	67.67	3.78	5.91	300	5 302	12 615	323	25	1 505	561	130	102	29	316	12	7	7 276	95	4 350	62
B2496/1(4)	18.91	5.22	0.83	60.50	2.49	7.44	485	4 980	13 118	337	23	2 008	686	92	144	28	378	11	61	9 366	108	5 103	81
B2496/3(1)	18.28	5.68	1.07	63.03	2.46	5.62	557	5 270	10 793	290	21	1 481	543	96	91	24	314	8	5	6 849	95	4 013	65
B2496Q	17.94	5.15	0.92	60.22	4.52	7.82	230	4 875	9 894	289	27	1 427	492	91	87	25	306	19	6	5 979	55	3 366	51
B2496/3(11)	15.82	5.42	1.15	63.26	3.75	6.70	235	5 090	11 339	306	31	1 477	537	111	109	25	335	15	7	7 552	104	3 810	48
B2496/3(13)	19.06	5.05	0.83	60.08	3.38	7.04	217	4 889	13 520	342	29	2 067	643	121	141	24	355	19	66	9 038	106	4 964	70
B2496/3(14)	18.36	5.40	0.99	61.12	3.61	6.82	218	4 999	10 721	306	18	1 479	534	104	86	22	333	13	6	6 641	98	3 728	54
B2496/3(16)	18.68	5.11	1.02	61.15	3.48	6.98	210	4 846	10 511	303	27	1 415	474	81	96	30	330	13	6	6 475	77	3 608	53
B2496/3(17)	18.55	5.22	0.96	61.86	3.52	6.31	199	4 888	10 306	289	23	1 422	442	112	89	28	293	15	8	6 630	75	3 615	57
Average (15)	17.29	5.27	0.98	62.27	3.39	6.89	293	5 180	11 295	314	27	1 707	579	109	105	26	329	14	18	6 900	90	3 982	61
SD	2.42	0.26	0.10	2.62	0.59	1.14	126	511	1 207	18	8	359	105	16	24	3	34	4	23	1 650	16	606	11
B8685	9.61	9.38	1.68	69.28	2.87	3.28	264	5 932	9 410	393	21	2 929	797	122	117	58	308	22	4	4 702	66	5 740	140
B2496G	9.44	5.45	1.15	69.50	4.26	5.84	238	6 074	13 022	350	30	1 702	579	134	114	23	313	21	6	7 407	75	4 543	59
<i>Group 2b1</i>																							
B8762.at	16.36	5.88	1.27	62.61	4.16	5.98	251	6 060	12 627	385	26	1 508	477	106	367	32	352	23	57	1 845	91	5 026	78
B4550/1	10.41	6.50	1.38	70.61	3.30	4.06	271	5 553	13 081	448	22	1 624	493	125	340	32	286	27	70	1 970	75	5 110	71
B4550/2	15.96	5.97	1.31	62.36	3.98	6.71	272	5 553	13 029	405	22	1 558	510	164	322	30	306	15	50	1 959	85	5 139	72
B4544	13.77	6.33	1.51	65.53	5.14	3.18	250	7 014	16 115	416	29	1 865	598	167	407	36	296	22	58	2 219	79	6 228	93
B2496.5	17.47	6.16	1.56	61.31	4.82	4.93	287	5 619	12 871	420	25	1 559	521	135	339	36	377	16	49	1 942	94	5 309	90
B2496/1(3)	15.64	6.22	1.51	61.86	5.55	5.44	274	5 961	12 957	425	21	1 457	485	163	316	25	359	23	70	1 959	89	5 253	76
B2496/1(7)	16.31	6.03	1.36	61.75	5.28	5.53	256	5 839	12 922	404	24	1 450	479	161	318	27	357	22	50	1 970	93	5 154	71
Average (seven)	15.13	6.16	1.41	63.72	4.61	5.12	266	5 943	13 372	415	24	1 574	509	146	344	31	333	21	58	1 978	86	5 317	79
SD	2.36	0.22	0.11	3.34	0.81	1.19	14	513	1 218	20	3	142	42	24	33	4	36	4	9	115	7	412	9

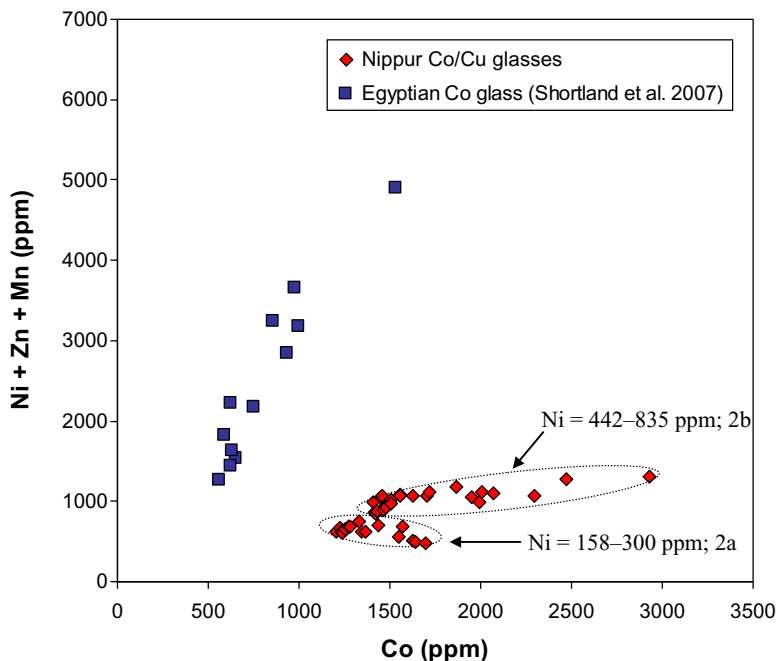


Figure 3 A bivariate plot of Co and the sum of the trace metals.

plot of As versus Sb in Figure 4 (b). The high-arsenic group is here called Group 2b1. There is also a possible distinction within Group 2a, as three samples have lead levels of 300–655 ppm and very low tin (<5 ppm), whilst the remaining 14 have much lower lead of <60 ppm and all but one have higher tin (up to 32 ppm). It is hence tempting to term the higher-lead samples a subgroup, Group 2a1, as indicated in Figure 4 (a).

Therefore, five distinct groups can be recognized and their characteristics are summarized in Table 4.

#### Correlations between groups

Taking the groups as a whole, several patterns emerge of elements that appear to be correlated. As can be seen in Figure 5 (a), one of the most striking concerns copper and tin. This appears to show two trends, both with a positive correlation. The first trend has a copper-to-tin ratio of around 1000 and includes all the copper-coloured Group 1 and Group 2a1 samples, all but three of Group 2b and one of Group 2a. The other trend has a copper-to-tin ratio of around 200 and includes all the other samples. This is one of the very rare examples where glasses from the same group follow different trends—other than this, they are very consistent.

The second correlation of interest shown in Figure 5 (b) concerns copper and lead. This shows that the copper of Groups 1 and 2a has very little lead, whilst the copper of Groups 2a1, 2b and 2b1 shows a positive correlation. The intercept with the axis is at about 3250 ppm copper, suggesting that these groups have two copper sources in them, one with no lead accounting for around 3250 ppm Cu, and a second with a consistent copper-to-lead ratio of around 2. Lead is also correlated with bismuth in all the glasses with significant lead, with a ratio of about 60:1, suggesting that a similar lead source might be involved in all the lead-bearing glasses.

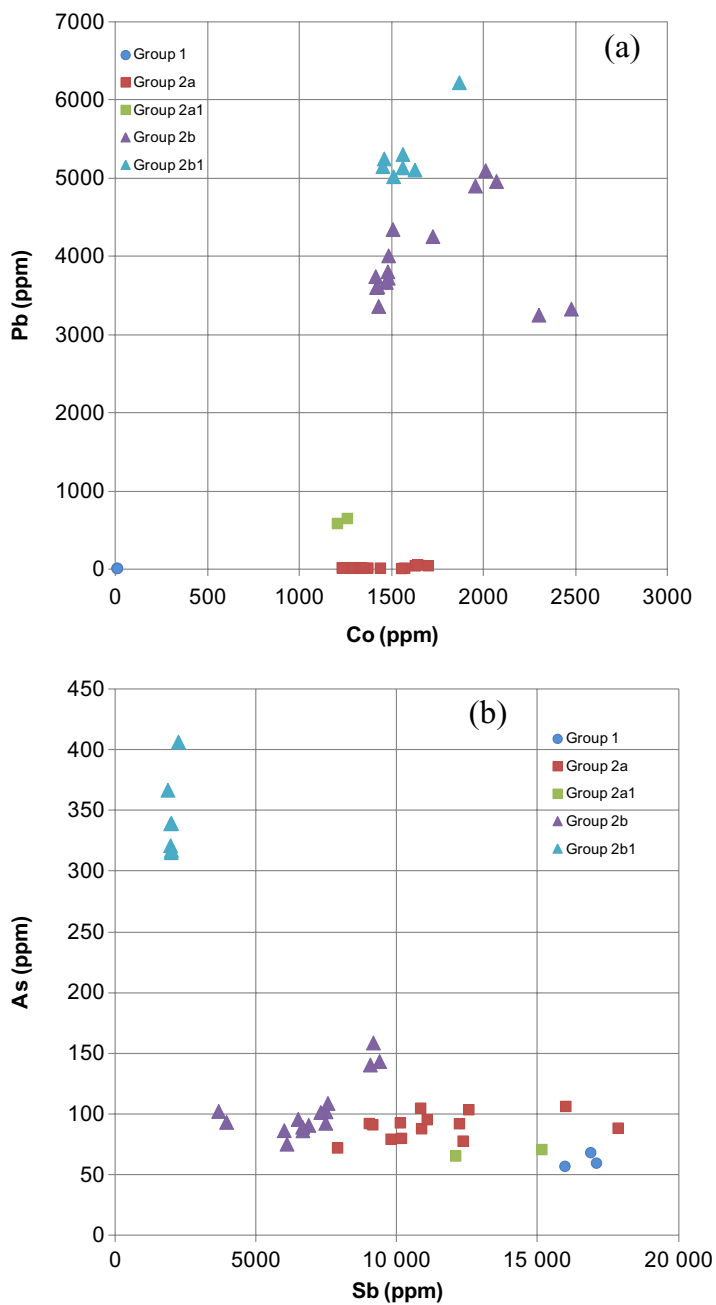


Figure 4 Bivariate plots of (a) Co versus Pb and (b) Sb versus As, showing the major compositional divisions in the Nippur glass data.

Table 4 A summary of the compositions of the various groups determined

Number:	Group 1: copper-coloured 5	Group 2a: Co + low Ni 15	Group 2a1: Co + low Ni 3	Group 2b: Co + high Ni + Pb 17	Group 2b1: Co + high Ni, Pb + As 7
Co	<10	1 200–2 000	1 200–2 000	1 400–3 000	1 400–1 900
Cu	>14 000	4 000–8 000	4 000–8 000	9 000–13 000	9 000–13 000
Ni	140–220	<300	<300	440–840	480–600
Sb	16 000–21 000	5 000–18 000	5 000–18 000	3 600–9 400	1 800–2 200
As	<100	<120	<120	<160	>300
Pb	<30	<60	300–700	3 000–6 000	3 000–6 000
Objects	Unknown Fragments	One axe, no inscription; one curl, two rods, two panels	One axe inscribed <i>Kashitlilashu</i> ; one axe, no inscription; one horn	Three axes inscribed <i>Nazi-Maruttash</i> ; one axe, no inscription; one horn	One axe inscribed <i>Kurigalzu II</i> ; one axe, no inscription

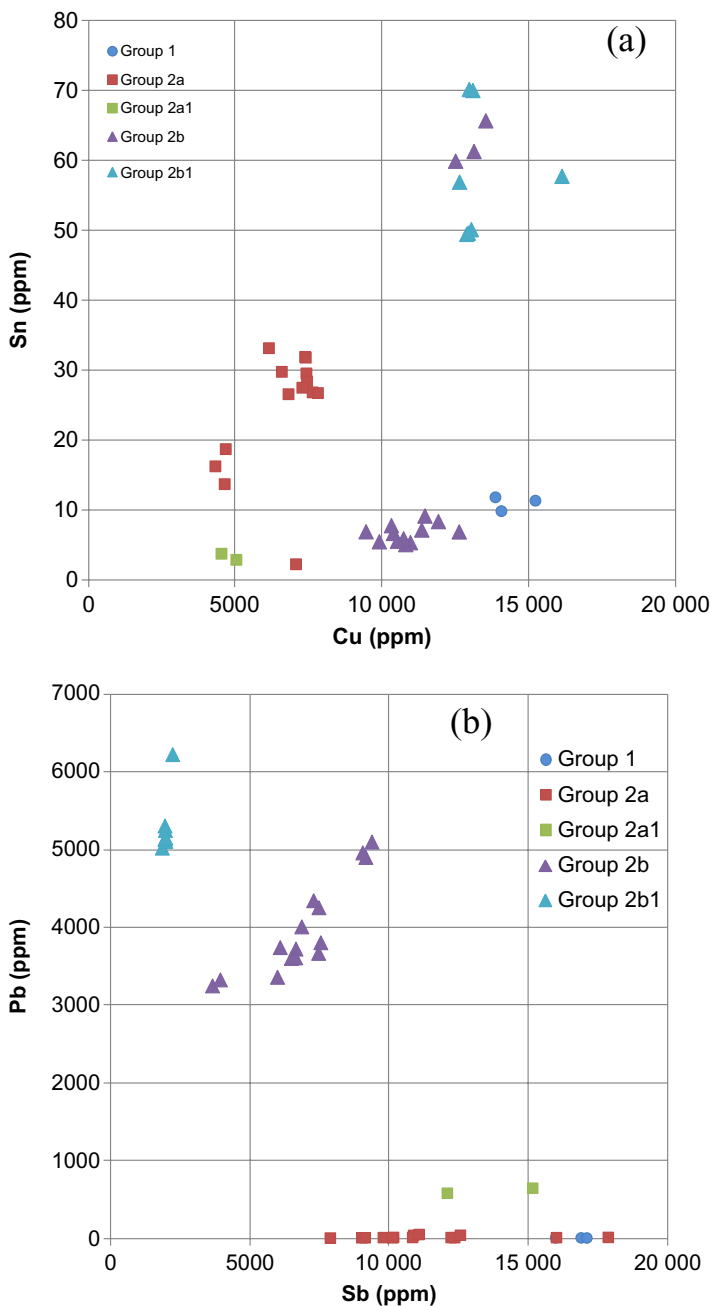


Figure 5 Correlations amongst groupings of Nippur glass fragments, (a) Cu versus Sn and (b) Sb versus Pb, showing the different copper sources used to produce these glasses.

A third correlation of interest is that between lead and antimony: Group 2b shows a weak positive correlation of lead and antimony, whilst—as can be seen in Figure 5 (b)—the other groups show no real correlation between the two.

In terms of artefact typologies, Group 1 consists entirely of fragments of uncertain type. All the recognizable axes and other pieces fall into Group 2. The distribution of artefacts between the different groups is shown in Table 3. Interestingly, although object types show no relationship to each compositional group, there might be a relationship with the inscribed objects. Although relatively few objects are inscribed, the different kings' names fall into different compositional groups. This is especially strong with the three different axes inscribed *Nazi-Maruttash*, all of which fall into Group 2b.

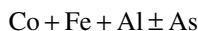
## DISCUSSION

### *Comparison with Egyptian co-coloured glasses*

As can be discerned from Table 5, and as discussed in the introduction, the Egyptian cobalt colourant derived from alum deposits located in the Western Oases of Egypt imparts a characteristic trace element signature to the glass. On average, Egyptian cobalt glasses, compared to copper glasses from the same sites, contain around 6000 ppm more Al, 800 ppm more Mn, 500 ppm Ni and 900 ppm Zn, with their average of 800 ppm Co. Although these are approximate values, and there is significant variation, these elements always occur in this colourant in significant quantities.

The cobalt-coloured Nippur glasses are very different in comparison to the Egyptian cobalt-coloured glasses, as was shown by the trace element data presented in Figure 3. In addition, they may be found to have the following unique characteristics. (1) The Nippur glass has higher levels of cobalt, around twice that seen in the average cobalt-coloured Egyptian glass. (2) Groups 2a and 2b have slightly higher aluminium contents than the Nippur copper-coloured glasses (Group 1), but do not have a correlation between these elements. Egyptian co-coloured glass, on the other hand, exhibit strong Al–Co correlations. (3) Unlike the Egyptian glasses, there is no significant difference in the manganese or zinc levels between the Nippur copper-coloured (Group 1) and the cobalt/copper-coloured glass (Group 2).

On the basis of these trace element data, it is found that the Nippur glasses contain a cobalt colourant that has the following trace element assemblage:



This is very different from the trace element assemblage associated with the Egyptian cobalt colourant:



It follows that the Nippur glasses appear to have been made out of a Co-bearing ore that is distinct from the well-characterized Egyptian source. On account of the elevated levels of iron in these glasses, it is speculated that cobalt may have been hosted in some iron-based mineral such as a jarosite or limonite. The exact nature of the ore used to produce these glasses and the location of the deposit are still very much outstanding questions that need to be addressed by further research.



Table 5 A summary of the average compositions of the various groups compared to contemporary glass from Egypt and other Near Eastern sites (Shortland et al. 2007)

Location:	Nuzi	Tell Brak	Malkata	Kassite axes	Kassite axes	Kassite axes	Kassite axes	Malkata										
Colour:	Cu blue	Cu blue	Cu blue	Cu blue	Co blue (2a)	Co blue (2b)	Co Blue (2b1)	Co blue										
Number:	14	11	4	3	2	15	7	12										
	SD	SD	SD	SD	SD	SD	SD	SD										
Al	2 518	1 052	2 413	1 469	4 330	3 020	3 194	95	6 069	1 091	6 595	194	5 192	513	7 488	598	11 017	3 019
Ti	155	48	165	82	558	363	257	112	226	37	369	221	193	126	266	14	607	219
Mn	220	68	165	80	191	86	314	12	267	28	312	13	314	18	415	20	990	425
Fe	1 609	573	1 862	1 174	3 199	2 063	2 384	126	5 151	367	4 693	523	5 180	511	5 943	513	3 599	1 304
Co	5	3	11	20	23	30	6	2	1 418	165	1 230	38	1 707	359	1 574	142	814	278
Ni	17	7	23	18	17	16	148	4	232	43	260	25	579	105	509	42	517	251
Cu	10 441	5 167	7 620	4 354	8 882	5 383	14 370	734	6 604	1 204	4 770	358	11 295	1 207	13 372	1 218	1 220	1 386
Zn	18	4	18	7	73	76	125	26	127	31	55	14	109	16	146	24	981	471
As	36	19	55	45	31	20	62	6	90	10	68	4	105	24	344	33	6	4
Rb	13	4	15	7	8	2	11	2	27	3	26	4	26	3	31	4	4	1
Sr	393	65	424	96	657	179	391	22	248	30	283	9	329	34	333	36	572	288
Sn	28	43	15	10	925	638	11	1	25	9	3	1	18	23	58	9	44	39
Sb	2 063	5 028	13 245	11 540	1 854	2 998	16 631	597	11 404	2 708	13 600	2 165	6 900	1 650	1 978	115	1 211	1 400
Ba	32	8	41	22	52	14	65	12	56	13	93	5	90	16	86	7	42	9
Pb	18	23	86	200	43	49	16	1	27	15	623	45	3 982	606	5 317	412	34	26
Bi	0	0	1	1	0	0	0	0	0	0	12	2	61	11	79	9	0	0

### *Cu-coloured glasses*

The copper-coloured glasses have very few other elements associated with the copper. The tin levels are low, only in the range of 3–61 ppm Sn, which is far below the hundreds of ppm of tin seen in contemporary Egyptian glasses, where the colourant is thought to be bronze or bronze scale (Table 5; other data from Shortland *et al.* 2007). The low tin seen here is very similar to the tin levels seen in glasses from Nuzi and Tell Brak, and probably represents trace levels of tin impurities within the copper source. It should also be noted that other elements commonly associated as trace components of colourants—for example, iron and manganese—are slightly high in comparison to glasses from Nuzi and Tell Brak. Of even higher concentration in the Nippur glasses are nickel and zinc, which are around 8–10 times the values of those from the other two Near Eastern sites.

### *Base glass composition*

The Nippur glasses also possess some unique features related to their base glass composition that bear further elaboration. In terms of those elements not associated with the colourants—that is, the major elements, silicon, sodium, potassium, magnesium and calcium—the two strategies of colouring the Nippur glass by either Cu or Co/Cu are indistinguishable other than the lower rubidium content in the copper-coloured glasses. This difference in rubidium suggests some variation in raw materials between the copper- and cobalt-coloured glasses, either the plant ash or the silica source. However, the base colourless glass for all the cobalt-coloured glass is essentially identical, which implies that the same raw materials and techniques in terms of silica source and plant-ash flux were used. Significantly, none of the glasses in this study show the correlations between elements such as aluminium, titanium, chromium and others seen in all other LBA glasses from Egypt and the Near East analysed so far (Shortland *et al.* 2007). This very much sets them apart, perhaps forming a separate glass type, but the reason for this is unclear.

### *The archaeological implications of this research*

The Nippur glass axes are both the first Mesopotamian LBA glasses clearly shown to be coloured with cobalt— all previous analyses of glass from Mesopotamia having indicated copper as the colourant for blue glasses— and the first LBA glasses shown to utilize a different cobalt source from that used for Egyptian cobalt-coloured glass. Therefore, the major archaeological inference of our data is that the technology to create glass objects coloured with cobalt existed in Mesopotamia and Egypt at around the same time. This finding has implications for understanding how semi-precious materials (e.g., man-made glass and other vitreous materials) were exchanged in the ancient world, as well as how technological expertise was disseminated to local craftsmen throughout the Mediterranean.

Another interesting finding, as observed from Figure 2, is that the Nippur glasses represent the clearest example of ancient glass manufactured to deliberately mimic lapis lazuli. Deliberate imitation of precious stones has been previously suggested from ancient glass-making texts and inventories of trade and tribute, all of which differentiate ‘lapis from the mountains’ (*uqnû ša šadî*)—believed to be the mineral lapis lazuli—from ‘lapis from the kiln’ (*uqnû ša kuri*)—interpreted as ancient dark blue glass (Oppenheim 1970). However, most LBA dark blue glass resembles lapis only in the colour and lacks any textural similarity. Due to the unique appearance

of the Nippur glass, it is believed that this is the first blue coloured glass thus far identified that may be considered a synthetic lapis lazuli produced in Mesopotamia, thus persuasively linking the archaeological and textual records.

#### CONCLUSIONS

The Nippur glass provides an important and interesting window into glass-making in the Near East. Glass in the Near East is typically very poorly preserved and good analyses are very hard to obtain, but the excellent preservation of most of this glass provides a substantial amount of reliable data to assess. The glasses are typical LBA plant-ash glasses and are split into two major colouring strategies: a small number coloured with copper and the remainder with a combination of copper and cobalt. The copper glasses are in many ways similar to contemporary glasses from Nuzi and Tell Brak, but with higher levels of colourants used, and significant trace levels of nickel and zinc. The cobalt/copper glasses are of a unique composition. This is the first non-Egyptian cobalt colourant to be fully characterized.

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