

## IRON OBJECTS FROM SOUTH INDIA

Numerous grave sites have been excavated in South India yielding a great number and variety of iron objects. Some of these sites like Brahmagiri, Maski and Piklihal can be dated to the second half of the first millennium B.C. The objects from these sites are the earliest known examples of iron from South India. However, none of these objects have been analyzed, so that one has absolutely no knowledge of the nature of the metal or its technology. It would be extremely useful if an analysis of some of these could be made, so that some knowledge may be gained about the metallurgical skills of the Iron Age people.

However, the objects will have to be obtained on loan from India. Negotiations are being made with the government of India and it is hoped that some samples will be available for such a study by the spring of 1966.

for George Dales  
7/65

Dr. Pritchard - Ruth Matson Fall 1965

Aim and scope of project: Arrange a typology of bronze weapons, tools, utensils, vessels, etc., found in the area of Syria and Palestine and covering the period through the end of the Bronze Age. If, as is probable, it proves too large a subject to include the entire period, concentrate attention on the LB period, both because this is the era of greatest abundance of bronze implements and because it will form a companion to Catling's work for the neighboring area of Cyprus. Figurines, which are really a topic in themselves, may fortunately be excluded since a dissertation on that subject has been completed just this past year by a Miss Ora Negbi in Israel. From this typology, it would be interesting to see if one can trace the chronological and geographical spread of forms, decorations, and techniques. This in turn may lead to answers to problems relating to trade and commerce in the Eastern Mediterranean, cultural origins and influences among the various peoples of Western Asia and the Aegean, development and spread of technology.

Procedure: First, a survey of the literature to make a catalogue of all published bronzes. Second, study as many as possible of these objects themselves to obtain more accurate and meaningful observations than those offered by publishers with no special interest in the subject. In addition to the typology of form and decoration, it will be important to make whatever observations are possible about the techniques of manufacture. This will surely entail study in museums through the Middle East... Jerusalem, Amman, Damascus, Aleppo, Beirut... as well as in Europe and the U.S.A. where artifacts have been removed from their country of origin. For the comparative work which will follow the preparation of a typology, it will probably be necessary to visit Cyprus, Turkey, Greece, Iraq, and possibly even farther afield.

Field equip.  
needed?  
Microscope?  
Sample  
Collection

As to method of observation, what I know of previous workers in this field indicates that it will be almost purely visual. Naturally, no method of analysis or observation which would destroy the objects is likely to be permitted. Maxwell-Hyslop has found radiographs of some use in determining doubtful cases of method of manufacture. Chemical or spectographic analysis, which could be done with very small samples of borings, would be a possibility, but Catling was advised that the trace elements which these tests would reveal are not sufficiently distinctive for any of the resources in our region to be worth the time and considerable money. I would suggest that if the typological categories succeed in pinpointing specific types to specific areas, and that is rather a far out chance, it would be useful to check by analysis of the content of the metals to see if there is any marked correlation between metal content and categories.

# W. B. Coleman & Co.

**METALLURGISTS • CHEMISTS • ENGINEERS**

METALLOGRAPHIC AND SPECTROGRAPHIC ANALYSES  
CHEMICAL AND PHYSICAL TESTING

BOILER AND COOLING WATER TREATMENTS  
FUEL TREATMENTS • ACID CLEANING

November 17, 1966

*next week  
12/19 ff.  
Cu  
Zn  
Sn  
Fe + Pb low*

Miss Elizabeth K. Ralph, Assoc. Director  
Museum Applied Science Center for Archaeology  
The University Museum, University of Penn.  
33rd & Spruce Streets  
Philadelphia, Penna. 19104

Dear Miss Ralph:

The following samples are too limited in surface area to scan:

- |            |                                 |
|------------|---------------------------------|
| E2535C     | 29-65-642                       |
| E4660      | 29-65-650                       |
| E9374      | 29-65-651 ✓                     |
| E9598      | 29-66-763 ✓                     |
| E9753D ✓   | 2-33-13-81                      |
| E10304 * ✓ | 4-33-13-80                      |
| E10335B    | E6 (only number on either side) |
| E11000     | 10-B-17528B                     |
| E11127 ✓   | 16-B-17356                      |
| E13156 *   | 24-B-17460                      |
- E 11660*

The above samples marked with an asterisk contain a surface suitable for X-Ray emission. This may be true of others also but it is only discernible where the samples are mounted in lucite.

We hope you are able to find larger sections to replace these fragments.

Very truly yours,

W. B. COLEMAN CO.

*Errol R. Brunhouse, Jr.*  
Errol R. Brunhouse, Jr.,  
X-Ray Spectroscopist



November 29, 1966

Mr. Errol R. Brunhouse, Jr.  
X-Ray Spectroscopist  
W.B. Coleman Col  
9th & Rising Sun Ave.  
Philadelphia 40, Pa.

Dear Mr. Brunhouse:

The following replacement samples are enclosed:

E 9598	From Egypt
E 11000	" "
E 10304	" "
29-65-65	" "
29-65-651	" "
29-66-763	" "
E 9753 D	" "
E 11127	" "
E 4660	" "
F 794	From Iraq
U 9904	" "
U 10794	" "

With the exception of E 11127 and E 4660 (actual objects), you may bend, polish, and do whatever you like to them. With E 11127 and E 4660 it is all right to polish a section or cut one off if you have to.

Sincerely yours,

Elizabeth K. Ralph

# W. B. Coleman Co.

*Did not search for  
As, Mn, Au, & Ag*

**METALLURGISTS • CHEMISTS • ENGINEERS**

METALLOGRAPHIC AND SPECTROGRAPHIC ANALYSES  
CHEMICAL AND PHYSICAL TESTING

BOILER AND COOLING WATER TREATMENTS  
FUEL TREATMENTS • ACID CLEANING

December 28, 1966

Museum Applied Science Center for Archaeology  
The University Museum, University of Pennsylvania  
33rd & Spruce Sts.  
Philadelphia, Pennsylvania 19104

Lab.: 883689  
Material: Copper Alloy

Attn: Miss E.K. Ralph, Assoc. Director

The submitted copper base samples were scanned by X-Ray Fluorescence in a qualitative and quantitative manner. The results are, in many cases, fairly accurate. For the remaining samples, the size and/or surface condition are the limiting factors and the classifications are indicated accordingly. In each instance the total percentage listed is an indication of the accuracy of the analysis.

Zinc as an alloying element was not found in any of the samples. The following results are tabulated accordingly:

Good - Excellent Surface

<u>Sample Number</u>	<u>Copper</u>	<u>Tin</u>	<u>Lead</u>	<u>Total</u>
E 9754	99.9 %	<.5 %	1 %	100+ %
34-51-1-435 *	93.5	1.5	1	96
29-85-322	88.7	8.7	<1	98
E9598	95.0	1.1	<1	96
E9753D	93.5	1.1	<1	95
29-65-642 *	91.0	4.7	1	97
29-65-650	90.5	5.4	1.5	97
6-38-13-38A	93.0	1.1	1	95
E11127	91.2	5.3	<1	97
E4660	95.0	1.5	1	98
29-85-111	94.8	<.5	<1	95
27-53-11-310	98.0	0.5	1	100+
9-30-12-272	87.8	7.5	1	96
29-85-187	94.8	0.8	1	97
E9586	99.9	<.5	<1	100+
E10885	94.8	0.8	1	97
E10342A	91.5	6.2	1	99
E9996	99.9	<.5	<1	100+
E9202	97.0	0.9	1	99



SINCE 1922

<u>Sample Number</u>	<u>Copper</u>	<u>Tin</u>	<u>Lead</u>	<u>Total</u>
E11116	89.0 %	7.9 %	1 %	98 %
E14224	97.0	0.6	1	99
E1055	90.3	7.5	1	99
36-31-17-265	90.6	7.1	<1	98
29-85-289	99.9	0.3	<1	100
E9747	94.5	1.1	<1	96
E9999	94.5	1.6	1	97
E10884	91.3	1.6	2	95
29-85-240	94.0	1.5	<1	96
E9588	93.0	1.6	1	96
E9997 *	91.7	1.9	1	95
<u>Fair Surface</u>				
E2900	88	4	1	93
E10304	80	5	<1	85
25-B-17460 *	64	6	1	71
8-31-17-267	84	2	1	87
20-31-17-186	63	9	1	73
S E13156	80	4 chisel	1	85
10-B-17528A	89	1	1	91
10-B-17528B	88	5	1	94
E11000	89	1	<1	90
6-38-13-38B	89	2	1	92
23-35-1-422	84	2	1	87
11-31-17-243	68	4	1	73
21-B-17480	77	6	1	84
E289	89	4	1	94
E13379	92	<.5	<1	92
35-31-43-510	80	4	1	85
33-B-16409	89	2	1	92
29-85-83	75	6	1	82
32-42-28	78	3	1	82
32-42-68	89	2	1	92
E9521 *	88	1	1	90
D E9749	91	2 adze	<1	93
E9736	92	2	<1	94
29-65-656 *	89	2	1	92
E10866	90	2	1	93
E11134	82	4	1	87
29-65-633	88	2	1	91
S E13144	90	2 chisel	<1	92
E2355	90	2	<1	92
29-65-629	76	5	1	82

Poor Surface

<u>Sample Number</u>	<u>Copper</u>	<u>Tin</u>	<u>Lead</u>	<u>Total</u>
E954	79 %	2 %	1 %	82 %
E12512B	72	3	1	76
29-66-763	70	7	<1	78
29-65-65	76	<.5	<1	77
29-65-651	76	<.5	1	77
29-85-366	76	5	<1	81
32-42-569	64	6	2	72

Dull Surface after Polishing

31-27-140	86	3.5	1	91
U9904	64.5	1	<1	66
U10794	78	1.5	<1	80
16-B-17356	71	10	1	82
E6	90	7	<1	97
31-53-11-299	71	2	1	74
15-31-17-254 **	50	5	1	56
3-33-13-82	75	2	1	78
32-53-11-302	81	2	1	84
1-33-13-78	79	2	1	82
30-53-11-308	74	2	1	77
12-B-17525 **	54	6	1	61
37-53-11-313B	79	3	1	83
29-31-43-497	83	4	1	88
7-31-17-352	89	4	1	94
37-53-11-313A	82	3	1	86
24-B-17276	79	4	1	84
19-35-1-479	88	4	1	93
18-31-17-217	68	12	2	82
14-B-17347	90	3	1	94
22-B-17330	66	15	<1	82
13-31-17-251	53	19	1	73
28-B-16426	89	2	<1	92

Good Surface - necessary  
to use oversized mask

29-85-171	57 %	5 %	1 %	63 %
5-33-13-77	76	3	1	80
17-B-17426	74	2	1	77
4-33-13-80	67	3	1	71
2-33-13-81	73	2	1	76
E2535C	70	5	1	76
E10335B	66	4	1	71

<u>Sample Number</u>	<u>Copper</u>	<u>Tin</u>	<u>Lead</u>	<u>Total</u>
N E9374	82 %	③ % <i>wire</i>	1 %	86 %
26-53-11-303	58	4	1	63
F794	74	1	<1	75

\* Sample did not quite fill mask opening causing low results  
\*\* Mask used is so small that background interferes with results.

NOTE: Number F794 has a small bright core surrounded by a large dull area.

*R. J. Stoult*

January 21, 1967

Mr. R. S. Stoudt  
W. B. Coleman Co.  
9th Street and Rising Sun Avenue  
Philadelphia, Pa. 19140

Dear Mr. Stoudt:

In order to compare the X-ray fluorescence analyses with published analyses of other objects done by emission spectroscopy, we should like to have the following samples analysed by emission spectroscopy:

E 12512 B  
E 14224  
E 10885  
E 10866  
E 10335 B  
✓ E 9374  
29-85-171

We should like to know particularly about the presence (or absence) of antimony, arsenic, bismuth, copper, iron, lead, magnesium, manganese, nickel, tin, and zinc. Please determine the elements present with the best quantitative precision possible.

A purchase order will be sent separately, and the samples are enclosed.

Many thanks for your help with these analyses.

Sincerely yours,

Elizabeth K. Ralph  
Associate Director

EKR:rs

enclosures

**W. B. COLEMAN & CO.**  
METALLURGISTS — CHEMISTS — ENGINEERS

**Key to Spectrographic Semi-Quantitative Analysis**

Major = Above 5.0% estimated

x.o = 5-10% estimated

Minor = 1.0 — 5.0% estimated

N. F. = Not found

.x, .ox, .oox, etc. — Concentration of the elements  
estimated to the nearest decimal place

Example: .ox = .01 to .09% estimated

High }  
Low } Pertain to the lowest ratio value (1)

Example: o.ox (low) = 0.01 to 0.05%

o.ox (high) = 0.05 to 0.09%

The numbers in parenthesis indicate the estimated ratio concentration  
of the same element among the various samples.

**W. B. COLEMAN CO.**  
**METALLURGISTS--CHEMISTS--ENGINEERS**

9TH ST. AND RISING SUN AVE.  
 PHILADELPHIA, PA. 19140

**REPORT OF ANALYSIS**

RECEIVED FROM  
 The University Museum-University of Penna.  
 33rd & Spruce Sts.  
 Phila., Pa. 19104

DATE February 2, 1967

LABORATORY NO. 885578/584

ANALYSIS OF Copper Alloy

Attn: Miss E.K. Ralph

SAMPLE IDENTIFICATION  
 See Below

REMARKS

P. O. NO.

**RESULTS**

Spectrographic Analysis:

	<u>E-12512B</u>	<u>E-14224</u>	<u>E-10885</u>	<u>E-10866</u>
Calcium	N.F.	N.F.	N.F.	N.F.
Cobalt	0.0X % (1)	N.F.	0.X % (10)	N.F.
Copper	Major	Major	Major	Major
Iron	Minor	Minor	Minor	0.X % (100)
Lead	Minor	0.X % (10)	0.0X (2)	0.0X (1)
Magnesium	0.0X (1)	N.F.	N.F.	N.F.
Nickel	0.0X (10)	0.0X (10)	0.X (100)	0.0X (10)
Silicon	0.0X (10)	0.00X (1)	0.00X (1)	0.00X (1)
Silver	0.0X (1)	0.0X (1)	0.0X (1)	0.0X (1)
Tin	Major	N.F.	0.X (10)	0.0X (1)
Antimony	0.X	N.F.	N.F.	N.F.
Arsenic	0.0X (1)	0.X (10)	0.0X (1)	0.X (10)
Bismuth	N.F.	N.F.	N.F.	N.F.
Manganese	N.F.	N.F.	N.F.	N.F.

*Aitken's photo no.*

W. B. COLEMAN CO.

BY \_\_\_\_\_

Spectrographic Analysis - Cont'd

	<u>E-10335B</u>	<u>E-9374</u>	<u>29-85-171</u>
Calcium	N.F.	0.00X % (1)	0.0X % (10)
Cobalt	N.F.	N.F.	0.0X (1)
Copper	Major	Major	Major
Iron	0.0X % (10)	0.00X (1)	Minor
Lead	0.X (10)	0.0X (1)	Major (low)
Magnesium	0.0X (1)	N.F.	0.0X (1)
Manganese	N.F.	N.F.	0.00X
Nickel	0.0X (10)	0.00X (1)	0.0X (10)
Silicon	0.00X (1)	0.00X (1)	0.X (100)
Silver	0.0X (1)	0.0X (1)	0.0X (1)
Tin	Major	N.F.	Major
Antimony	N.F.	N.F.	N.F.
Arsenic	0.0X (1)	0.0X (2)	0.0X (1)
Bismuth	N.F.	0.0X (3)	0.0X (1)

ELEMENTS CHECKED BUT NOT FOUND: Barium, Beryllium, Boron, Cadmium, Columbium, Gallium, Germanium, Gold, Molybdenum, Phosphorus, Platinum, Potassium, Strontium, Tellurium, Tungsten, Vanadium, Zirconium, Aluminum, Chromium, Sodium, Titanium, Zinc.

*RC Annan*

R.2  
Newark, Del. 19711  
3.12.67

Miss Elizabeth K. Ralph  
Assoc. Director,  
Applied Science Center for Archaeology.  
The University Museum  
University of Pennsylvania  
33rd. and Spruce Streets  
Philadelphia, Pennsylvania. 19104

Dear Miss Ralph:

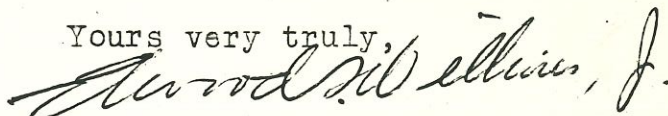
Following up our conversation at the conclusion of the February 18 Meeting of the Archaeological Society of Delaware; I have talked to my friend regarding his willingness to be of assistance to you in the analysis of your Roman coins. He has had considerable experience with the analysis of Roman coins by his improved x-ray fluorescence procedure. He has published on the subject and, as I told you, presented a paper at the Delaware Science Symposium at the University of Delaware on January 21 this year.

He has expressed his willingness to be of help to you and I suggest that you write to him directly.

He is: Dr. Giles F. Carter ✓  
Electrochemicals Department  
Building 335  
Dupont Experimental Station  
Wilmington, Delaware, 19898.

Should you decide to set up wet analysis procedures, I shall be glad to give what assistance that I can in that regard.

Yours very truly,



Elwood S. Wilkins, Jr.

March 14, 1967

Mr. Elwood S. Wilkins, Jr.  
R. 2  
Newark, Delaware 19711

Dear Mr. Wilkins:

Thank you very much for your kind letter of March 12th. We shall plan to contact Dr. Carter directly at some future date. At the moment, our conservationist is in the hospital so that this work here is a bit delayed.

I enjoyed seeing you in Wilmington and hope that we shall meet again soon.

Sincerely yours,

Elizabeth K. Ralph

March 14, 1967

Dr. Giles F. Carter  
Electrochemicals Department  
Building 335  
Dupont Experimental Station  
Wilmington, Delaware 19898

Dear Dr. Carter:

Mr. Wilkins has written that you have kindly offered to analyze some of our Roman coins by X-ray fluorescence.

At the moment our Roman coin problem seems to have disappeared, but from time to time, we do have a need for X-ray fluorescence analysis of particular objects. I hope that we may consult you and possibly submit some samples at a future date.

Sincerely yours,

Elizabeth K. Ralph

1422 Bucknell Road  
Wilmington, Del. 19803  
March 22, 1967

Miss Elizabeth K. Ralph  
Museum Applied Science Center for Archaeology  
University of Pennsylvania Museum  
33rd & Spruce Streets  
Philadelphia, Pennsylvania 19104

Dear Miss Ralph:

Thanks for your letter of March 14, 1967. As you may know I have analyzed quite a few Roman coins by x-ray fluorescence (over 300). So far I have not analyzed much in the way of other archaeological objects. However, recently I obtained a number of Sumerian metallic pieces that I'll work on soon. If you would like to talk over this work sometime, I would be glad to.

Sincerely,

*Giles J. Carter*

From M. Katzev  
rec'd. by EKR 11/18/67

## Carbonate Encrustation of a Nail from a Byzantine Wreck

John D. Milliman and Frank T. Manheim

### Introduction

Recent submarine archaeological excavations off the Turkish coast have resulted in the recovery of many encrusted metallic artifacts (1, 2). We have studied an encrusted nail recovered from a 7th century Byzantine wreck off the island of Yassi Ada, southwestern Turkey, at a depth of about 35 m of water (2). The concretion was obtained through the courtesy of Professor George Bass of the University of Pennsylvania. Nearly every iron object recovered from the wreck, primarily nails and the ship's anchor, was heavily encrusted with a calcareous cement. The nail had a concretionary coating approximately 0.95 cm thick. Most of the original nail had disappeared, leaving a hollow tube lined with hydrous iron oxide (Fig. 1). Some fibrous, woody fragments, perhaps remnants of the ship's planking, are incorporated in the encrustation.

A thin section of the nail and encrustation (Fig. 2) was studied. In addition, three samples at intervals of 0.00 to 0.20 cm, 0.25 to 0.50 cm, and 0.70 to 0.95 cm from the edge of the original nail were taken for further analysis. The inner sample (0.00 to 0.20 cm) was split, and one half leached with dilute HCL to remove soluble mineral species. The samples were then analyzed for mineral content by petrographic microscopy and by x-ray diffraction (Table I), using a Norelco x-ray diffractometer, and for chemical composition (Table II), using a precision emission spectrometric technique.

## Results

The inner half of the encrustation is <sup>dominated</sup> determined by a ground mass of an opaque mineral, probably limonite. Goethite was not detected by x-ray diffraction, but its amorphous equivalent is assumed to be present from microscopic observation and chemical analyses (Table II). Calcite with appreciable quantities of smaller ions in solid solution ("displaced calcite", recognized by the shift in the calcite diffraction pattern), siderite and magnetite are the primary crystalline minerals in the inner and middle layers; quartz, aragonite and pyrite are present in minor amounts (Table I). If the calcium and magnesium leached from the inner layer (Table II) represent dissolved carbonate only, a calculated 30 percent of the inner layer is displaced calcite. The leached Mg corresponds to 12 mole percent  $MgCO_3$  in the displaced calcite, in close agreement with the 13 to 14 percent estimated by the shift in the calcite diffraction pattern. Some iron may also be in the calcite lattice (4). The remainder of the leached sample is chiefly magnetite, siderite, and amorphous iron.

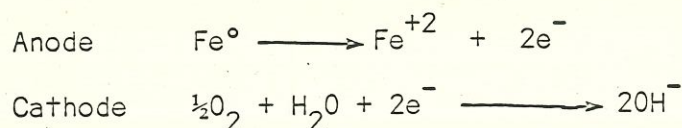
The outer half of the concretion is dominated by calcium carbonate. A cohesive but porous aragonite matrix encloses carbonate skeletal debris containing, in decreasing order, benthic foraminifera, mollusk, algal, and echinoderm fragments; elongate aragonite needles grow from many of the skeletons (Fig. 3). Aragonite and displaced calcite comprise over 90 percent of the total material. The remainder is chiefly quartz, limonite and pyrite.

## Discussion

The composition of iron and steel and its manufacturing methods changed relatively little in Europe and the Near East from primitive to late medieval times (5, 6). The Byzantine nail was probably a relatively pure wrought iron

similar to known Roman types. Because no metal or slag remains were found for metallographic study, and in view of the scarcity of data on trace metals in ancient iron implements, the present analyses can give little insight into the metallurgy or source of metals for the culture concerned. The unusually large lead trace in the inner layer of the concretion is probably not associated with the original nail itself. Since no alloying metal such as copper is present in significant amounts, the lead may come from an extraneous source of relatively pure metal.

The carbonate encrustation around the Byzantine nail appears to result chiefly from inorganic precipitation of aragonite (possibly some calcite), limonite and siderite to form a cementing matrix. A reasonable mechanism for this precipitation involves the corrosion of iron:



The rise in pH at the cathode and the consequent increased carbonate activity promotes supersaturation and inorganic precipitation of calcium carbonate. Soviet workers (7) have found that in electrochemical induration of continental sediments calcite and magnetite precipitate at the cathode, while limonite forms at the anode. Since the electrochemical cells in the corroding nail form at the molecular level, no physical differentiation can probably be made between anodic and cathodic precipitation. In sea-water solutions containing appreciable magnesium, the metastable calcium carbonate phase first to be precipitated (8) is likely to be aragonite, not calcite. A further confirmation of the inorganic origin of the carbonate is the fact that the ratio of the various skeletal species is roughly constant throughout the concretion, whereas the aragonite increases markedly from the inner to the outer layers.

Aragonite also has been observed as a cementing agent around recent iron scrap, such as cans, nails and barbed wire, in nearshore areas off the Netherlands (9). Encrustations found on other metal objects in the marine environment may have had a similar origin. Such induration need not be limited to corrosion of metals; oxidation of decaying matter may form concretions around organic nuclei (10).

On land and in fresh water, magnetite is nearly always observed in rust and corrosion products of iron, and more than 2000 years are needed in most environments before metallic iron and magnetite are completely converted to ferric iron (6, 11). In oxygenated sea water, reactions are much faster, and one might expect virtually complete oxidation of iron to take place within, say, 50 years (12). The continued presence of unstable siderite and magnetite after more than 1300 years on the sea floor is therefore noteworthy (13).

John D. Milliman

Frank T. Manheim

Woods Hole Oceanographic Institution

U.S. Geological Survey

Woods Hole, Massachusetts

INNER LAYER		OUTER LAYER	
0.00 to 0.20 cm	0.00 to 0.20 cm (leached)	0.25 to 0.50 cm	0.70 to 0.95 cm
(limonite)* (30)	(limonite)* (35)	(limonite)*	aragonite 50
displaced calcite 30	magnetite 25	displaced calcite	displaced calcite 40
siderite 15	siderite 20	siderite	quartz 5
magnetite 15	quartz 10	magnetite	(limonite)* (4)
quartz 5	pyrite 10	quartz	pyrite 1
pyrite 5		pyrite	
aragonite tr			

Table 1. Mineral species present in the concretion, as determined by x-ray analysis; numbers reflect relative percentages. \*Limonite abundance estimated from microscopic observation.

	INNER LAYER		OUTER LAYER
	0.00 to 0.20 cm	0.00 to 0.20 cm (leached)	0.70 to 0.95 cm
SiO <sub>2</sub>	4.8	10.1	6.5
TiO <sub>2</sub>	0.009	0.019	0.065
Al <sub>2</sub> O <sub>3</sub>	0.69	1.2	1.2
Fe <sub>2</sub> O <sub>3</sub> (tot.) <sup>1</sup>	49.4	67.4	5.4
MnO (tot.)	0.15	0.16	0.10
CaO <sup>1</sup>	17.7	1.5	46.0
MgO	1.8	0.53	3.5
Na <sub>2</sub> O	0.85	0.40	0.87
K <sub>2</sub> O	0.12	0.12	0.21
P <sub>2</sub> O <sub>5</sub>	0.15	<0.10	0.27
SrO	0.076	0.015	0.42
BaO	0.008	0.015	0.032
H <sub>2</sub> O <sup>-</sup> (120°C)	1.4	1.3	0.8
Ign. loss (1000°C)	24.2	18.5	35.5
Sum <sup>1</sup>	100	100	100
Pb	0.27	<0.02	0.07
Cu	0.008	<0.005	0.011
Zn	0.005	0.005	<0.005
Ni	0.011	0.014	0.011

Table II. Chemical composition of the inner (total and leached) and outer layers of concretion. Elements were determined on a Jarrell-Ash direct-reading emission spectrometer, using a high-voltage spark technique modified from the method of Landergren, et al. (3).

- Major constituent (Fe<sub>2</sub>O<sub>3</sub> or CaO) was determined by difference between the sum of the cation oxides, plus P<sub>2</sub>O<sub>5</sub>, SiO<sub>2</sub> and ignition loss, and 100.0. The ignition loss value adjusts for changes in oxidation state and loss of volatiles.

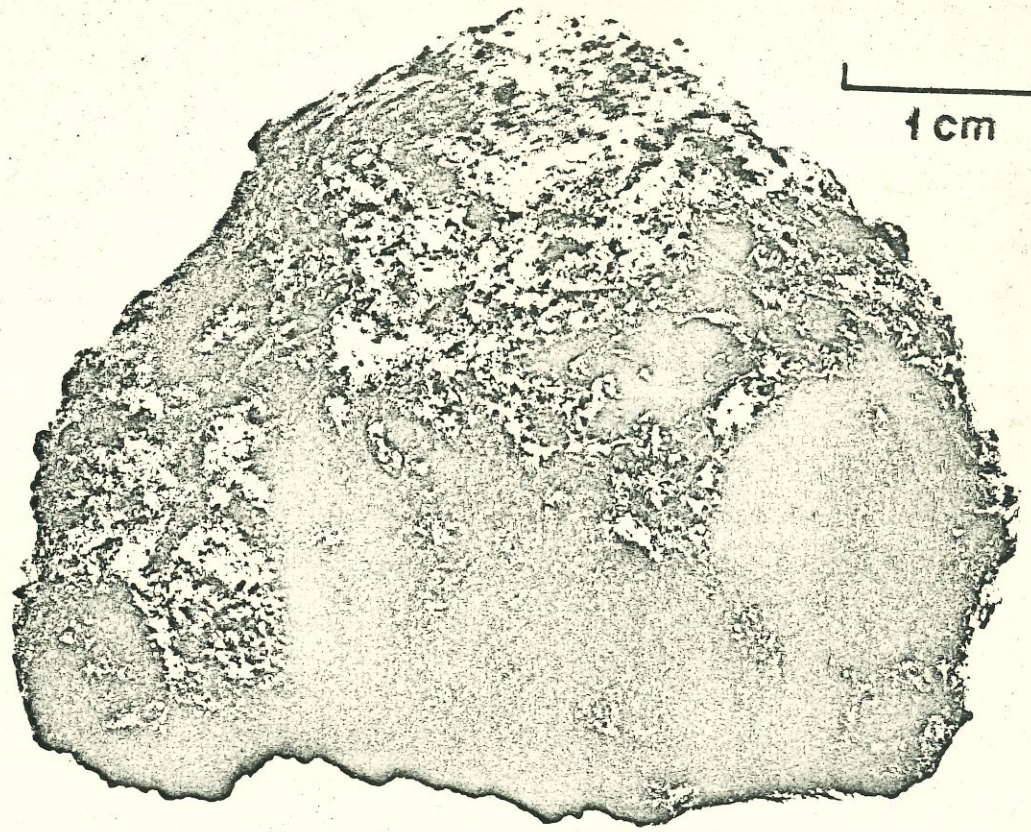
1. Throckmorton, Peter, 1960. National Geographic, v. 117:682-703; Throckmorton, Peter, 1962. National Geographic, v. 121:697-711.
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12. Woolacott, D., 1908. Trans. Nat. Hist. Soc. Newcastle, v. 3(2):
13. Contribution No. 1965 of the Woods Hole Oceanographic Institution.

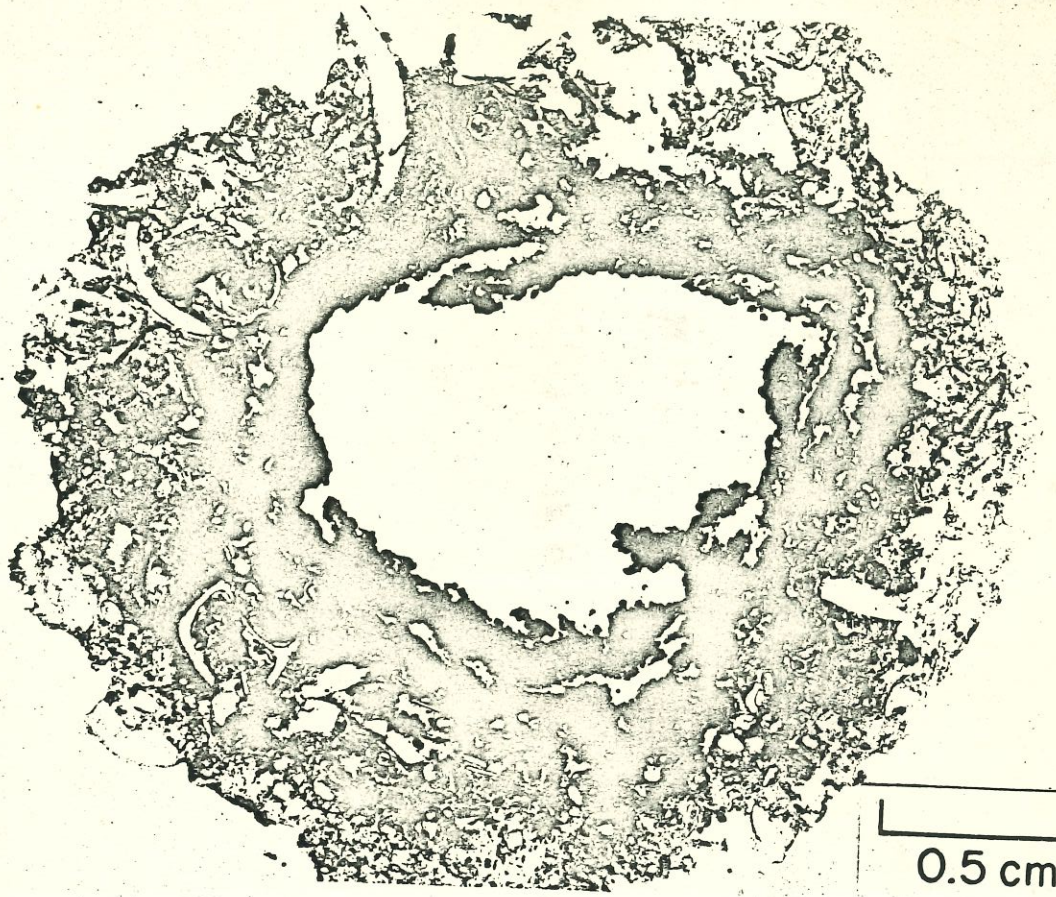
We thank J. L. Bischoff and K. O. Emery of the Woods Hole Oceanographic Institution, J. C. Hathaway, U.S. Geological Survey, G. F. Bass and M. L. Katzev, University of Pennsylvania, C. S. Smith of the Massachusetts Institute of Technology, Ernst Kitzinger, of the Institute for Advanced Studies, Princeton and John Gettens, Freer Gallery of Arts, Washington, D.C. for valuable discussions. Research was supported by the U.S. Geological Survey.

Figure 1. Encrusted nail. The dark tube on the right part of the encrustation outlines the original nail.

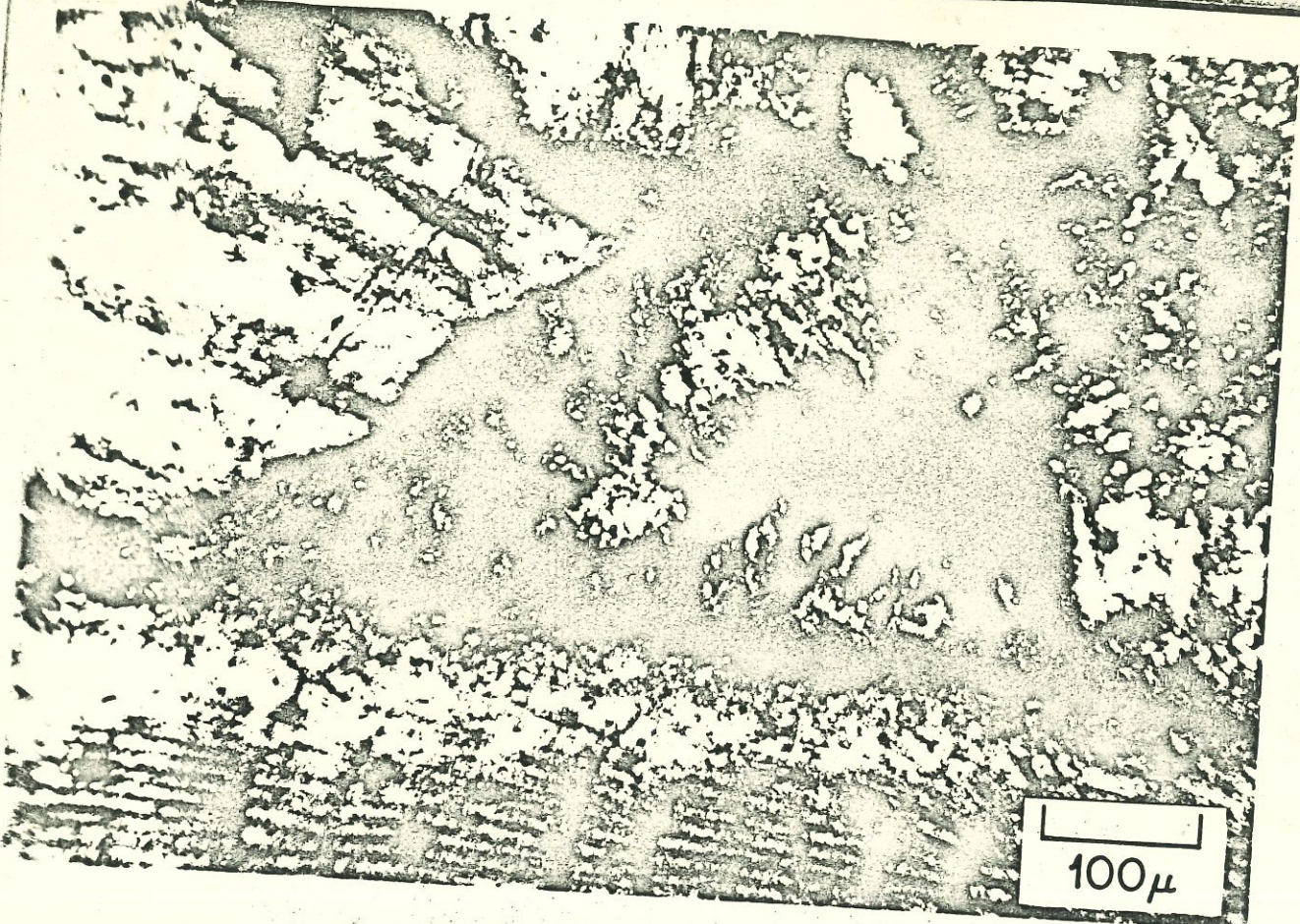
Figure 2. Cross section of the encrusted nail. Note the dark inner layer and the lighter, fossiliferous outer layer.

Figure 3. Aragonite needles growing on limonite-coated coralline alga (bottom) and mollusk (upper left) fragments.





0.5 cm



100 μ

Sent to Oxford

4/1/68

RESEARCH LAB - FOR ARCHAEOLOGY  
AND HISTORY OF ART.

Jordan Series

- |    |                                                                                               |
|----|-----------------------------------------------------------------------------------------------|
| 1  | 91 mg                                                                                         |
| 2  | 86 mg                                                                                         |
| 3  | 62 mg                                                                                         |
| 4  | 26 mg, 3 pieces                                                                               |
| 5  | 50 mg                                                                                         |
| 6  | 223 mg                                                                                        |
| 7  | 144 mg                                                                                        |
| 9  | 85 mg, from large diameter of longer of 2 parts                                               |
| 10 | 47 mg                                                                                         |
| 11 | 41 mg, from least corroded piece of 4 pieces                                                  |
| 12 | 140 mg, from larger of 2 parts                                                                |
| 15 | 41 mg                                                                                         |
| 17 | 42 mg, from largest piece                                                                     |
| 18 | 116 mg                                                                                        |
| 19 | 100 mg                                                                                        |
| 20 | 75 mg                                                                                         |
| 21 | 41 mg                                                                                         |
| 22 | 272 mg                                                                                        |
| 23 | 59 mg                                                                                         |
| 34 | 53 mg, section from back of knife; cutting edge missing; some deep corrosion left on one side |
| 46 | 206 mg, piece may contain some corrosion                                                      |

Totally destroyed in bakelite mountings: 13, 25, 26, 31

Sent to Oxford

Egypt

E 9996  
E 9586  
E 9997

Mesopotamia

31 - 17 - 186  
53 - 11 - 303  
31 - 17 - 243

E 14224  
E 10884  
29 - 65 - 651  
E 2235  
E 9699  
29 - 65 - 652  
E 9598

[JORDON]

Objects to be removed from the University Museum for testing  
by the Dept. of Metallurgical Engineering for research on  
bronzes by Ruth Matson

---

TELL ES-SA'IDIYEH

EL-JIB

46- { S549/Br.13	34-B78	
S574/Br.20	2-B93	62-30-230
39- S590/Br.24	18-B105	62-30-85
S729/Br.31	B109	62-30-67
S789/Br.33	5-B111	62-30-68
20- S811/Br.39	13-B114	62-30-69
38- S859/Br.43	B121	62-30-70
37- S864/Br.44	31-B125	62-30-73
S1008/Br.51	B134	62-30-211
22- S1027/Br.52	9-B142	62-30-212
21- S1028/Br.53	7-B317	62-30-290
17- S1031/Br.56	1-B333	62-30-286
S1128/Br.68	B342	
43- S1142/Br.69	3-B345	62-30-280
6- S1146/Br.70	10-B346	62-30-276
S1160/Br.72	11-B347	62-30-277
15- S1214/Br.77	B349	62-30-275
S1215/Br.78	12-B350	62-30-278
S959/J56	J63	
S1088/M275	J67	
	J73	
	23-J77	
	J78	62-30-81
	J90	62-30-76
	25-J91	
	19-J92	62-30-74
	26-J94	62-30-71

April 22, 1968

Mr. Mitchell, President  
Lehigh Testing Laboratories Inc.  
Wilmington, Delaware

Dear Mr. Mitchell,

Under separate cover we are sending you two samples from Jordan. They represent part of a program of metallurgical-archaeological studies which, we hope, will afford information about ancient metallurgical techniques and the technology of metal working in past times.

Please remove the corrosion from both samples and analyse the "pure" metal. One sample we think will weigh at least ten grams (J22), but the other (J6) will be undersized. These are the only two from this series which are large enough for wet analyses.

We should like to have the following analyses performed with as great precision as possible:

- 1) Emission spectrographic analyses of all elements present or detectable.
- 2) Wet chemical quantitative analyses of all major and minor elements which are present in quantities of 1.0% or more, that is, all elements which are present in greater than decimal percentages.

On the basis of previous analyses of ancient bronzes, the elements anticipated to be found are as listed, approximately in order of percentages: Cu, Sn, Pb, As, Sb, Ni, Bi, Fe, Zn, Ag, and Au. There may, however, be significant variations.

The Department of Metallurgy is sending a purchase order with the cost of these analyses estimated at \$75.00. If you should find that the cost is slightly greater in order to obtain maximum precision, we shall be glad to revise this estimate.

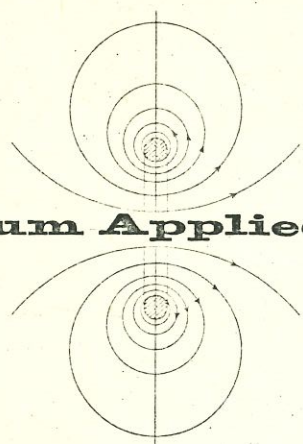
Sincerely yours,

Elizabeth K. Ralph

SKR/labn

Photomicrographs  
& comments

Beth's files



**Museum Applied Science Center for Archaeology**

Froelich Rainey, Director

Elizabeth K. Ralph, Associate Director

THE UNIVERSITY MUSEUM • UNIVERSITY OF PENNSYLVANIA  
33rd & SPRUCE STREETS • PHILADELPHIA, PENNSYLVANIA 19104  
594-7400 (Area Code 215) Cable Address "Antique"

302-772-4587

April 3, 1968

Mr. Norman A. Nielsen  
Building 304  
DuPont Company Experimental Station  
Wilmington, Delaware 19898

Dear Mr. Nielsen:

Under separate cover we are sending you 11 ancient bronzes (or coppers) for metallurgical examination. Six of these represent comparison series dating from 3000 to 2000 B.C. They are as follows:

Mesopotamia:

- |               |                                                            |
|---------------|------------------------------------------------------------|
| Museum Number |                                                            |
| 31-17-186     | (U.14238) Socketed axe from PB 1687 (former sample No. 20) |
| 53-11-303     | (3N 187) Pin, Nippur Ur III (former sample No. 26)         |
| 31-17-243     | (U.14244) Chisel, EDIII (former sample No. 11)             |

Egypt:

- |               |                                                     |
|---------------|-----------------------------------------------------|
| Museum Number |                                                     |
| E 9996        | Flat axe blade                                      |
| E 9586        | Square sectioned chisel (All date to 3rd millenium) |
| E 9997        | Flat adze blade                                     |

We hope that your studies will provide information about how the objects were made, heat treated, etc. and any other data which will shed light upon ancient metal-working techniques and changes.

In addition to the six objects, we are sending five others from Egypt. Some additional specific questions for these are as follows:

- E-14224. How were the holes made? Was the ridge made at the time of original hammering, or after annealing, or ?
- E-10884. How were the holes made? How was the riveting done?
- 29-65-651. Compare with E-10884.
- E-2235. How were the holes made, especially, in the mirror?

Mr. Norman A. Nielsen  
April 3, 1968  
Page 2

E-9598. How was it made and how was it heat treated?

We have sent 20 mg. or more of each object to the Research Laboratory for Archaeology and the History of Art at Oxford, England, for emission spectrographic analyses. We hope to have the results of these within 6 to 8 weeks and will forward copies to you.

We shall appreciate it if you will not remove any more material from the objects than is necessary for your studies, and please return the objects when you have finished. We have photographic negatives on file of all objects.

Many thanks for your kind collaboration.

Sincerely yours,

*Beth Ralph*

Elizabeth K. Ralph

cc. Dr. Maddin  
MASCA  
Dr. Robert Dyson  
Mr. James Weinstein

655 7358 **Lehigh Testing Laboratories** 302

P.O. Box 1241

WILMINGTON, DELAWARE 19899

NO. 179382 DATE 5/3/68

MATERIAL Non-Ferrous Alloy

RECEIVED FROM School of Metallurgy & Materials Science  
The University Museum  
33rd and Spruce Streets  
Philadelphia, Penna. 19104  
Elizabeth K. Ralph

SAMPLE MARKED PO#40944, J6

REMARKS

*Emission*  
Spectrographic Estimates of the Samples As Received

Copper	Major	Magnesium	0.002
Aluminum	0.02	Manganese	0.05
Antimony	0.2	Nickel	0.02
Arsenic	0.4	Silicon	0.05
Bismuth	0.05	Silver	0.2
Cobalt	0.2	Tin	Minor
Iron	0.3	Zinc	0.05
Lead	Minor (L)		

*wet* Chemical Analysis

Copper	89.40
Tin	6.70
Lead	2.25

Not found: Ba, Be, B, Cd, Ca, Cr, Cb, Ga, Ge, Au, In, Li, Hg, Mo, Pd, P,  
Pt, K, Ru, Na, Sr, Ta, Te, Tl, Ti, W, V, Zr:*J. E. Mitchell*

In the above analyses "X" is used to denote the relative percentage within one decimal place, i.e., 0.00X would indicate that the estimated percentage is between 0.001 and 0.009, 0.0X between 0.01 and 0.09, etc. The "X" followed by the words high or low indicate that the percentage is high or low in its particular category. Principal indicating above 50.0%, Major 10.0 to 50.0%, Minor 1.0 to 10.0%.

Research Lab for Archaeology  
6, Keble Road,  
Oxford,  
England.  
20<sup>th</sup> April 1968

Dear Beth,

I hope you don't mind the drop of formality the last series of our correspondence when we were working together on the Dr Pritchard project we wrote to each other as Beth and Anne. Anyway I enclose the analyses of the bronzes you asked me to do. I have been able to work all this week whilst the laboratory was closed so I have had a straight run through them. All the results are expressed as percent to two significant places. Where there is a blank in the analyses none of that element was detected. You will see that no gold at all was detected - the column is there to indicate that it was looked for.

2.

Do you want any of the material returned? The mounted samples you said were destroyable - the material in them was quite good and I have analysed them. The rest presented no problems - some were quite difficult to get small pieces off to make an accurate weighing of exactly 20 mg.

Now about the cost of the analyses - they have taken 32 hours to do the complete analysis and have cost £7 in materials ie 2 photographic plates, 6 complete rods of spectrographic carbons and their manufacture into cups. The only other expenditure was in travelling which was £4. To help cover some of these costs would it be alright for me to ask for £45? Honestly I just don't know what to say about this as I have never undertaken anything

3.

privately before.

If and when you do want another series analysing it would be best for you just to send them along - most of the year I can manage a couple of evenings extra in the week - and I would do them as quickly as possible. This time was lucky really having a complete week on my own to go at.

I hope that these results will be of use to you - if you have any questions on any of the work I shall be happy to help you further. I don't know whether you would like to know the limits of detection etc.

Sincerely yours.

Anne.



P. 1

Report of spectrographic analyses by Mrs. A. Millet  
Research Lab for Archaeology + the History of Art  
6 Keble Road  
Oxford, England

20 April 1968

All of the results are expressed as  
percent to two significant places. Where there  
is a blank none of that element was  
detected

No Au was found




P. 2

Report of spectrographic analyses by Mrs. A. Millet

20 April 1968

Research Lab for Archaeology & the History of Art  
6 Keble Road  
Oxford, England

Sample	Cu	Sb	Fe	As	Pb	Sr	Ni	Bi	Zn	Ag	Au
E 9598 (hawl)	99.2		0.051	0.39	0.039		0.24	0.023		0.027	
31-17-186 (socketed axe)	89.0		0.069	0.16		10.0	0.73	0.032		0.016	
31-17-243 (chisel)	88.1		0.17	0.33		10.8	0.49	0.023		0.012	
53-11-303 (pin)	86.9		0.39	0.52	3.2	8.3	0.13	0.028		0.54	
E 9699	95.6	0.18	0.28	3.5	0.058		0.016	0.022	0.30	0.084	
29-65-652	96.6		0.15	3.1	0.10	<0.1	0.030	0.022		0.026	
E 2235 <i>mirror</i>	97.6		0.33	1.8	0.075	0.11	0.032	0.011		0.026	
29-65-651 <i>mirror</i>	96.1		0.33	2.8	0.72		0.024	0.019		0.0068	
E 10884 <i>mirror</i>	96.4		0.058	3.3	0.23		0.031	0.020		0.013	
E 14224 	96.5		0.66	2.6	0.073		0.059	0.014		0.017	
Pa E 9997 (flat adze blade)	98.9		0.023	0.94	0.039		0.032	0.025		0.020	
E 9586 (spitax chisel)	98.9		0.18	0.69			0.21	0.025		0.018	
Pa E 9996 (flat axe blade)	98.1		0.079	1.7			0.038	0.019		0.030	
J. 1.	98.6		0.31	0.31	0.69		0.036	0.020		0.0067	
J. 2.	95.0		0.26	1.4	0.93	2.3	0.035	0.019		0.063	
J. 3	93.2		0.036	0.41	0.32	5.9	0.046	0.026		0.068	
J. 4	93.2		0.13	0.46	0.12	6.0	0.038	0.026		0.0081	
J. 5	91.5		0.055	0.30	0.10	7.9	0.040	0.023		0.0059	
J. 6	89.9		0.33	0.28	3.2	5.6	0.061	0.028	0.58	0.038	

Research Laboratory for Archaeology and the History of Art  
6 Keble Road  
Oxford, England

Sample	Cu	Sb	Fe	As	Pb	Sn	Ni	Bi	Zn	Ag	Au
J 7	96.1		0.045	0.80	0.23	2.7	0.079	0.024		0.034	
J 9	96.7		0.27	0.31	0.43	2.1	0.059	0.034		0.056	
J 10	91.2		0.012	0.32	0.61	7.7	0.048	0.043		0.033	
J 11	91.5		0.021	0.36	0.19	7.8	0.12	0.034		0.016	
J 12	98.4		0.51	1.1				0.022		0.0029	
J 15	90.5		0.077	0.11	3.2	6.1	0.062	0.033		0.024	
J 17	89.6		0.0096		6.7	3.6	0.035	0.023		0.047	
J 18	93.8		0.041	0.60	0.045	5.5	0.034	0.023		0.0085	
J 19	92.1		0.018	0.40	0.042	7.4	0.045	0.019		0.0099	
J 20	88.4		0.030	0.11	0.69	10.5	0.050	0.028	0.20	0.011	
J 21	91.6		0.068	0.28	0.39	7.5	0.060	0.024		0.042	
J 22	89.9		0.0047	0.23	2.0	7.7	0.038	0.032		0.039	
J 23	89.1			0.084	0.17	9.1	0.037	0.021		0.016	
J 34	96.0		0.37	1.2	0.11	2.2	0.064	0.028		0.030	
J 46	93.1		0.019	0.17	0.19	6.2	0.052	0.025	0.32	0.012	
mount. 13	90.8		0.084	0.62	0.11	8.3	0.067	0.025		0.013	
25	99.3		0.16	0.17	0.13	0.18	0.066	0.023		0.0069	
26	92.8		<0.005	0.44	0.050	6.7	0.037	0.024		0.0086	
31	97.0		0.057	0.19	0.027	2.7	0.024	0.021		0.0045	

PHONE 652-5168

PHONE 652-6224

**Lehigh Testing Laboratories**

P.O. Box 1241

WILMINGTON, DELAWARE 19899

NO. 179381 DATE 5/3/68

MATERIAL Non-Ferrous Alloy

RECEIVED FROM School of Metallurgy & Materials Science  
The University Museum  
33rd and Spruce Streets  
Philadelphia, Penna. 19104  
Elizabeth K. Ralph

SAMPLE MARKED PO#40944, J22

REMARKS

Spectrographic Estimates of the Sample as Received

Copper	Major	Magnesium	0.001
Aluminum	0.02	Manganese	0.05
Antimony	0.15	Nickel	0.2
Arsenic	0.3	Silicon	0.05
Bismuth	0.2	Silver	0.2
Cobalt	0.1	Tin	Minor (H)
Iron	0.2	Zinc	0.02
Lead	Minor (L)		

Chemical Analysis

Copper	87.30
Tin	8.27
Lead	2.84

Not found: Ba, Be, B, Cd, Ca, Cr, Cb, Ga, Ge, Au, In, Li, Hg, Mo, Pd, P,  
Pt, K, Ru, Na, Sr, Ta, Te, Tl, Ti, W, V, Zr

*J. E. H. H. H.*

In the above analyses "X" is used to denote the relative percentage within one decimal place, i.e., 0.00X would indicate that the estimated percentage is between 0.001 and 0.009, 0.0X between 0.01 and 0.09, etc. The "X" followed by the words high or low indicate that the percentage is high or low in its particular category. Principal indicating above 50.0%, Major 10.0 to 50.0%, Minor 1.0 to 10.0%.

'Aynhoe'  
28, Kings Moor Road,  
Stockton-on-the-Forest,  
YORK. YO3 9TY.

14<sup>th</sup> February 1969.

Dear Beth,

I enclose five sheets with 83 analyses which I know you are waiting for. Some of these look interesting - I hope they prove to be of interest anyway. How is the snow in your part of the world? Here we are just having another fall but as it is sunny I don't expect it will stay. I was in Oxford Monday and Tuesday of this week finishing off an Archaeometry article. It seems very strange to me to be no longer an everyday part of the laboratory. Work is progressing very well up here though. Fortunately none of the apparatus was affected by the move.

I am settling down in my new location

2.

well - I am getting to know York better and it really is beautiful. Of course there is much archaeology round about. The Minster is being well excavated at the moment before they fill the foundations in with concrete to save it from falling down. On one of your England visits will you come and visit us? By the way the money from your analyses I saved and now we have spent it all on a really luxurious carpet for our new home, it looks super.

Very many thanks for your patience for these last two batches, best wishes to you as always. Don't forget if you can come 'North' sometime, come here.

Yours

Anne.

Sample No.	Cu	Sb	Fe	As	Pb	Sn	Ni	Bi	Ag	Zn	Au.	
E 6	91.3	nd	0.090	nd	0.89	7.7	0.068	0.018	0.013	nd	nd	very corroded
E 289	91.5	"	0.27	"	0.12	8.1	0.038	0.018	0.021	"	"	
E 954	99.5	"	0.029	"	nd	0.38	0.045	0.020	0.0077	"	"	very corroded
E 1055	90.0	"	0.064	"	0.20	9.6	0.045	0.026	0.019	"	"	
E 2355	97.9	"	1.6	0.30	0.050	nd	0.041	0.027	0.020	"	"	
E 2535 C	88.9	0.092	0.019	nd	0.049	10.8	0.032	0.036	0.044	"	"	
E 2900	88.2	nd	0.14	0.62	4.6	6.4	0.042	0.024	0.036	"	"	
E 4660	98.2	"	0.31	1.3	nd	0.099	0.081	0.018	0.020	"	"	
E 9202	95.9	"	0.089	3.2	0.37	0.32	0.099	0.044	0.025	"	"	
E 9374	99.0	"	nd	0.87	0.036	nd	0.022	0.027	0.025	"	"	
E 9588	99.8	"	0.11	nd	nd	"	0.046	0.024	0.0085	"	"	
E 9521	99.6	"	0.041	"	0.21	"	0.052	0.032	0.020	"	"	
E 9736	98.5	"	0.13	1.0	0.10	"	0.089	0.040	0.063	"	"	
E 9747	99.8	"	0.041	nd	0.13	"	0.030	0.026	0.018	"	"	
E 9749	99.5	"	0.31	"	0.097	"	0.032	0.027	0.018	"	"	
E 9753 D	99.5	"	0.11	0.32	nd	"	0.071	0.020	0.014	"	"	

Sample No	Cu	Sb	Fe	As	Pb	Sn	Ni	Bi	Ag	Zn	Au	
E 9754	99.6	nd	0.13	nd	0.17	nd	0.043	0.027	0.021	nd	nd	
E 9999	99.5	"	0.037	0.33	nd	"	0.077	0.024	0.030	"	"	
E 10304	92.2	"	0.27	0.34	0.050	7.1	0.029	0.029	0.0066	"	"	
E 10342 A	88.4	"	0.068	0.36	0.089	11.0	0.039	0.021	0.024	"	"	
E 10866	95.8	"	0.20	3.7	0.10	nd	0.10	0.034	0.018	"	"	
E 10885	96.7	"	1.8	0.66	nd	0.24	0.53	0.022	0.015	"	"	
E 11000	98.9	"	0.46	0.52	"	nd	0.040	0.022	0.016	"	"	
E 11116	87.0	"	0.16	0.44	0.18	12.1	0.032	0.029	0.034	"	"	
E 11127	92.6	"	0.15	0.36	0.086	6.8	0.037	0.020	0.017	"	"	
E 11134	91.3	"	0.023	0.41	1.7	6.2	0.25	0.019	0.016	"	"	
E 12512	91.2	1.2	0.45	0.34	1.2	5.6	0.043	0.040	0.017	"	"	
E 13144	99.6	nd	0.23	nd	nd	nd	0.077	0.015	0.026	"	"	
E 13156	98.7	"	0.033	0.81	0.44	"	0.022	0.022	0.0073	"	"	
E 13379	99.6	"	nd	nd	0.38	"	0.019	0.028	0.027	"	"	



Sample No	Cu	Sb	Fe	As	Pb	Sn	Ni	Bi	Ag	Zn	Au
33 A	90.9	nd	0.32	nd	0.16	8.5	0.055	0.023	0.12	nd	nd
36	88.1	"	0.048	"	nd	11.7	<0.01	0.015	0.12	"	"
61	90.8	"	0.18	"	0.19	8.7	0.048	0.021	0.099	"	"
64	92.3	"	nd	"	nd	7.7	nd	0.022	0.053	"	"
72	91.7	"	0.041	"	0.14	8.0	0.042	0.019	0.057	"	"
104	90.4	"	0.030	"	nd	9.4	nd	0.024	0.11	"	"
105	91.9	"	0.061	"	3.1	4.9	"	0.025	0.033	"	"
257	90.1	"	0.093	0.42	0.23	8.7	0.078	0.0096	0.038	0.34	"
263	87.6	"	0.97	nd	0.098	11.3	nd	0.0082	0.032	nd	"
274	90.3	"	2.3	0.31	0.14	6.1	0.70	0.023	0.11	"	"
275	88.3	0.33	0.28	0.34	0.53	10.0	0.024	0.035	0.13	"	"
280	91.7	nd	nd	nd	0.46	7.7	0.058	0.018	0.061	"	"
282	92.0	"	0.040	"	0.11	7.7	nd	0.021	0.069	"	"
284	91.3	"	0.045	"	0.22	8.3	"	0.037	0.051	"	"
290	88.0	"	0.030	"	nd	11.8	"	0.019	0.12	"	"
291	78.3	"	0.016	"	15.0	6.4	0.049	0.041	0.13	"	"





## Jordanian Samples

		LTL	Ox
J-6	Cu	89.4	89.9
	Sn	6.7	5.6
	Pb	2.25	3.2
	Fe	0.3	0.33
	As	0.4	6.28
	Ni	0.02	0.061
	Bi	0.05	0.028
	Zn	0.05	0.58

		LTL	Ox
J-22	Cu	87.3	89.9
	Sn	8.7	
	Pb	2.84	
	Fe	0.2	0.0047
	As	0.3	0.23
	Pb	2.84	2.0
	Sn	8.27	7.7
	Ni	0.2	0.038
	Bi	0.2	0.032

HARDNESS TESTS - Jordanian samples (50x - 100 gr. load)

<u>Spec</u>	<u>KHN</u>	<u>Brinell</u>	<u>Rb</u>
1.	125-132-123	99-104-96	63-66-61
2.	95-99-123-121	73-76-96.5-95.5	38-42-61.5-60.5
3.	103-116-107-195-170-155	79.5-91.5-83 154-135-124	45.5-57.5-50- 89-82-77
5.	bad specimen		
6.	208-177-172	160-148-137	91-84-83
7.	115-160-162-100-102	91-128-128-77-79	57-79-79-43-45
9.	127-121-135-146-132-118	101-95.5-107-116.5 105-92	64-60.5-68-73.5 66.5-58
10.	124-114-106	98-90-82	62-56-49
11.	140-131-207-129	110.5-104-160-102	70.5-66-91-65
12.	135-138-137-124-119-115	107-109.5-109-98 94.5-91	68-69.5-69-62 59.5-57
13.	174-138-145-122-156-135-139	137-109.5-116-96 124-107-110	83-69.5-73-61- 77-68-70
15.	78-96-87		
17.	106-91.5-137-117	82-70.5-109-92	49-34.5-69-58
18.	113-108-104-88	<sup>89.5</sup> <del>98.5</del> -84-80-68	55.5-51-47-31
19.	132-143-121-116	104.5-114-95.5-91.5	66.5-72-60.5-57.5
20.	151-134-135	120.5-106.5-107	75.5-67.5-68
21.	137-128-146-127	109-101.5-116.5-101	69-64.5-73.5-64
22.	129-100-128	102-77-102	65-43-65
23.	154-224-237	124-195-184	77-99-97
25.	118-100-107	94-77-83	59-43-50
26.	129-96-157	102-74-126	65-39-78
31.	113-140-124	90-112-98	56-71-62
34.	182-158-158	142-126-126	85-78-78
37.	112-65-107-137	89-63-83-109	55-0-50-69
38.	bad specimen		
39.	137-91-145	109-70-116	69-34-73
43.	bad specimen		
46.	86.5-113-113	65.5-89.5-89.5	27-55.5-55.5

July 27, 1968

Mr. Norman A. Nielsen  
Building 304  
DuPont Company Experimental Station  
Wilmington, Delaware 19898

Dear Mr. Nielsen:

Under separate cover, I am sending you the mounted samples from Egypt, numbers:

E 9586  
E 9996 (2 samples)  
E 9997

and from Mesopotamia, numbers:

31-17-186  
31-17-243  
53-11-303

I have included a Xerox copy of the chapter entitled "Metallurgy of Some Ancient Egyptian Medical Instruments" from Archaeological Chemistry, and a list of our card-file references to metallography.

We appreciated your visit today, and are so pleased with the information you are obtaining.

With best regards,

Elizabeth K. Ralph

EKR:kw



## Applied Science Center for Archaeology

THE UNIVERSITY MUSEUM • UNIVERSITY OF PENNSYLVANIA  
33rd & SPRUCE STREETS • PHILADELPHIA 4, PENNSYLVANIA  
Froelich Rainey, Director EVergreen 6-7400 (Area code 215)  
Elizabeth K. Ralph, Associate Director  
EVergreen 6-0100 Ext. 8168 (Area code 215)  
Cable Address "Antique"

October 30, 1968

URARTIAN BRONZES: list of metal samples to be submitted for spectrographic analysis (sample numbers correspond with the final digits of the University Museum catalogue number of the object from which it was taken)

<u>Sample No.</u>	<u>Object from which taken</u>
1B	Fragment of a large hammered cauldron.
1Da 1Db	Bronze "patch" apparently for the above cauldron; some of the original vessel remains attached to the small rivets of the patch. 1Da is from the remains of the original vessel; 1Db is from the patch itself.
3B	Small cast leaping ram for attachment to cauldron.
4	Cast bull's-head attachment.
5	Small bull's-head attachment for unknown vessel.
6A	Small cast ibex on double plinth for attachment to an unknown vessel.
8	Shallow bowl.
11B	Rim fragment of a crushed bowl.
13	Crushed straight-sided bowl (?).
14B	Rim fragment of a fluted bowl.
21	Pointed helmet with engraved and hammered dec.
22	Fragment of a helmet with relief decoration.
24	Crushed helmet with two rows of rivet holes (?).
25	Round shield with relief decoration.
26	Quiver or scabbard decorated with reliefs.



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## URARTIAN BRONZES: list of samples for analysis (page 2).

- 27A strip with two iron rivets, possibly scale armor.
- 31A Cheek piece from horse harness.
- 32A Cheek piece with zoomorphic design.
- 33A Plain cheek piece similar to 32A.
- 36 Conical ornament for horse harness.
- 61 Bell made to dangle from harness.
- 64 Harness decoration with hammered decoration; could also be a shield boss.
- 72 Plain disc similar to 64.
- 104 Sheething for wooden throne or stool.
- 105 Fragment of a cast figurine; winged animal or deity (?); possible to decorate a piece of furniture.
- 257 Fragment of a bracelet ending in lion's head.
- 263 Fragment of a bracelet ending in snake's head.
- 274 "S"-shaped connecting ring.
- 275 Plain bracelet.
- 280 Segmented torque.
- 282 Heavy segmented ring.
- 284 Heavy ring with incised decoration.
- 290 Handle or part of a stand.
- 291 Metal sheathing (?).

Sample from ~~Iraq~~ <sup>Iraq</sup> ~~Jordan~~

Specimen no. is written on label of specimen - need correlation

20X - 200 gr Load

Spec.	K. H. N.	Brinell	R b
1.	132-129-129	106-102-102	67-65-65
2.	138-130-123	109.5-102.5-97	69.5-65.5-61.5
3.	111-107-119	87-83-94.5	54-50-59.5
4.	124-153-132	98-122-106	62-76-67
5.	172-158-169	137-126-135	83-78-82
6A.	111-119-104-94.5	87-94.5-80-72	54-59.5-47-37
6B.	107-122-116	83-96-91.5	50-61-57.5
7.	86.5-83-59	66-63.5- off-scale	28-24- off-scale
8.	102-96-108-109	79-74-84-85	45-39-51-52
9.	147-105-136	118-81-107.5	74-48-68.5
10A.	Bad Spec.		
10B.	107-115-112	83-91-89	50-57-55
11.	200-168-147-148	157-133-118-118	90-81-74-74
12.	Bad Spec.		
13.	Bad Spec.		
14.	149-156-145	120-124-116	75-77-73
15.	Bad Spec.		
16.	Bad Spec.		
17.	176-216-172-161	140-167-137-128	84-93-83-79
18.	Bad Spec.		
19.	Bad Spec.		
20.	Bad Spec.		

21.	143-128-110	114-102-86	72-65-53
22.	Bad Spec.		
23.	116-184-116-179-186	91.5-145-91.5-142-145.5	57.5-86-57.5-85-86.5
24.	Bad Spec.		
25.	131-150-157-150	104-120-126-120	66-75-78-75
26.	127-127	10-1-101	64-64
27.	87.5-87.5-91.3-87.5	68-68-70-68	31-31-34-31
28.	83.4-63.5-88.5-74.5	64-0.5-68-57	25-0.5-31-11
29.	Bad Spec.		
30.	135-143-143-141	107-114-114-112	68-72-72-71
31.	159-152-146-174	126-122-116.5-137	78-76-73.5-83
32.	94.5-107-120-130-122	72.5-83-95-103-96	37.5-50-60-65.5-61
33.	155-142-124-137-155	124-112-98-109-124	77-71-62-69-77
34.	143-139-137-146	114-110-109-116.5	72-70-69-73.5
35.	101-117-131-113-97-98.5	78-92-104-89.5-74.5-75	44-58-66-55.5-40-41
36.	106-98-100-99	82-75-77-76	49-41-43-42
37A.	135-157	107-126	68-78
37B.	Bad Spec.		

Miss Kramer's objects

	Fe	Pb	Ni	As	Sn	Ag	Si	Al	Co	Mg	Cr
Jamdet	--	0. x	0. x	0. x	--	--	--	--			
<u>Nasr</u>	--	Mi	--	0. x	--	--	--	--			
	--	--	--	Mi	--	--	--	--			
	Mi	0. x	--	Mi	--	--	--	--			
	0. x	Mi	--	0. x	Mi	--	--	--			
	--	Mi	--	0. x	--	--	--	--			
	0. x	--	0. x	0. x	--	--	--	--			
<u>Early Dyn</u>	0. x	--	0. x	0. x	Ma	--	--	--			
	0. x	--	0. x	0. x	--	0. x	--	--			
	0. x	--	0. x	0. x	Ma	--	--	--			
	0. x	--	--	--	Mi	--	0. x	0. x			
	0. x	0. x	--	0. x	--	0. x	0. x	--			
	--	--	0. x	. x	0. x	--	--	--			
	0. x	--	0. x	0. x	--	--	0. x	0. x			
	0. x	--	0. x	0. x	--	--	0. x	--			
Uruk	--	--	--	--	--	--	--	--			
Sargonid	0. x	--	0. x	0. x	--	--	--	--	0. x		
	0. x	--	0. x	0. x	--	--	--	--	0. x		
UR III	Mi	Ma	--	0. x	--	--	0. x	--	--		
	Mi	0. x	0. x	0. x	0. x	--	--	--	0. x		
Larsa	Mi	0. x	0. x	0. x	--	--	0. x	--	0. x	--	--
	Mi	0. x	0. x	0. x	--	--	Mi	0. x	--	0. x	0. x
	0. x	0. x	0. x	0. x	0. x	--	--	--	x	--	--
Old Babyl	--	0. x	--	--	Mi	--	0. x	--	--	--	--
	Mi	Mi	--	0. x	--	--	--	--	--	--	--
	Mi	0. x	0. x	--	0. x	--	0. x	--	x	--	--
Isin Larsa	Mi	Mi	0. x	0. x	0. x	--	--	--	x	--	--
				(0. xSb)							
Persian	Mi	Mi	--	(0. xSb)	Ma	--	--	--	--	--	--
Post-Akkad	0. x	0. x	--	0. x	Ma	--	--	--	--	--	--

## Further Notes on the Shaft-Hole Pick-Axe from Khurab, Makran

1968

C.C. Lamberg-Karlovsky

Metallurgical analyses and new dating evidence, resulting from recent archaeological excavations in southeastern Iran,<sup>1</sup> places a new perspective on the important shaft-hole pick-axe recovered from a burial by Sir Aurel Stein at Khurab.<sup>2</sup> This object has been the subject of separate articles by K.R. Maxwell-Hyslop<sup>3</sup> and F.E. Zeuner<sup>4</sup>. The axe-pick was found in a grave illustrated by Stein containing 32 complete vessels, mostly unpainted, two metal bowls similar in shape to those of ceramic, a complete alabaster jar and an agate bead tipped with gold ferrules at both ends. One small bowl was filled with human(?) bone fragments.<sup>5</sup>

Mrs. Maxwell-Hyslop has compared this shaft-hole pick-axe

1. De Cardi, B., 'The Bampur Sequence in the Third Millennium B.C.' Antiquity, Vol. XLI, No. 161, pp. 33-42, 1967; 'Excavations at Bampur S.E. Iran: A Brief Report' Iran, Vol. VI, 1968. A re-examination of the collections from Bampur and Khurab in the Peabody Museum, Harvard University will soon be published by the author.
2. Stein, A., Archaeological Reconnaissances in Southeastern Iran and Northwestern India. Macmillan, 1937. See Pl. XVIII, E, 1, 258, p. 121.
3. K.R. Maxwell-Hyslop 'Note on a Shaft-Hole Axe-Pick from Khurab, Makran' Iraq Vol. XVII, Pt. 2, pp. 161, 1955.
4. Zeuner, F.E., 'The Identity of the Camel on the Khurab Pick' Iraq, Vol. XVII, Pt. 2, pp. 162ff. 1955.
5. Stein, A., Op. cit. p. 121, Fig. 41, Pl. XVI, E, 1, 253, 254; Pl. XVIII, E, 1, 251, Pl. VI, E, 1, 252; Pl. X, E, 259.

to her types 9B and 15,<sup>6</sup> while also suggesting parallels for it with the metal rods of Hissar III which have human figures modelled at one end. Compared with the axe-heads with animals on the socket, as the well known example from the hypogeum at Til Barsip, or later Luristan examples, it is suggested that the Khurab blade is crudely fashioned and evidences little technical skill. Metallurgical analyses refutes the absence of a high standard of craftsmanship in the manufacture of this object. No convincing parallels have been found for this object to date.

Following the lead of Professor Piggott who dated the Khurab burials on the basis of far reaching and generalized parallels to around 2000 B.C.,<sup>7</sup> various authors have suggested dates between 2000-1800 B.C.<sup>8</sup> On the basis of recent work in this area their date (and the pick-axe) can be more securely suggested. At Khurab, Grave E, from which this object was recovered, contained cups almost identical to those recently excavated at Bampur in Period V context.<sup>9</sup> The appearance of a distinctive incised grey ware, in late Bampur IV and throughout V, of a type found also on Oman

6. Maxwell-Hyslop, K.R., 'Western Asiatic Shaft-Hole Axes' Iraq, Vol. XI, Pt. 1, Pl. XXXV, 15; Pl. XXXVIII, 5, 10, pp. 97ff, 104ff.
7. Piggott, S., Prehistoric India. Harmondsworth. 1950. p.218.
8. Maxwell-Hyslop, K.R., Op. cit. p. 161, 1955. See also Gordon, D.H. 'The Pottery Industries of the Indo-Iranian Border: A Restatement and Tentative Chronology' Ancient India, No. 10-11, 1954/55, pp.189ff.
9. De Cardi, B., Op. cit. 1968, p. 15 and the author's own study of the Bampur and Khurab collections in the Peabody Museum, Harvard. Similar cups are also evident in the Stein collection from Sutkagen Dor.

across the Persian Gulf<sup>10</sup> establishes the first stratigraphic link between the two sides of the Persian Gulf and sets a terminal date to the Bampur sequence; allowing us to suggest a date for Bampur V and thus by its ceramic parallels to Khurab Grave E.

Recent excavations on the island of Bahrain have uncovered a seal impression similar to a stamped seal tablet in the Yale Babylonian Collection.<sup>11</sup> This Yale impression is dated to the tenth year of Gungunum, King of Larsa, in southern Babylonia - that is 1923 B.C. ('middle chronology'). The Bahrain seal was found in a Barbar culture level, partially contemporary with the Umman-Nar culture of Oman, which as we have seen can be paralleled to Bampur V. The general evidence, thus, points to a date ca. 1900 B.C. for the terminus of the Bampur sequence,<sup>12</sup> and for the date of the Khurab shaft-hole pick-axe.

- 
10. De Cardi, B., Op. cit. Compare Pl. IVb;1-15 with G. Bibby, 'Arabian Gulf Archaeology' Kuml 1966, Fig. 11,12, 1967, and Thorvildsen, K., 'Burial Cairns on Umman-Nar' Kuml 1962, Fig. 20, pp. 191-219, 1963.
  11. For Yale Babylonian impression see Buchanan, B., 'A Dated Seal Impression Connecting Babylonia and Ancient India' Archaeology, Vol. 20, No. 2, pp. 104-107, 1967. For Bahrain seal see Bibby, G., 'Arabian Gulf Archaeology' Kuml 1965 p. 147,152, Note 1, 1967.
  12. The date of ca. 1900 B.C., or after, is further supported if the Bampur V parallels with Hissar III suggested by de Cardi, Op. cit. 1968, be accepted. It might be pointed out that the representation of a spoked chariot in Hissar III context suggests, as Childe long ago noted, a low date, after 2000 B.C., for the Hissar III settlement, see 'The First Waggon and Carts- from the Tigris to the Severn' Proceedings of the Prehistoric Society, Vol. 8, p. 184, 1951.

The report and technical analysis of the Khurab pick-axe was undertaken by Heather N. Lechtman of the Laboratory for Research on Archaeological Materials.<sup>13</sup>

Composition of the metal

Although the radiographs, Figs. 4, 5, 6,<sup>14</sup> and 7 indicate that the object is solid, the metal of which the camel and the socket were originally constituted has almost entirely corroded. Four drillings were made into the object, one in the body of the camel, two in the socket, and one in the blade to obtain metal samples for analysis. The positions of two of these sampling sites, the single drill hole made within the body of the camel to a depth of 7mm and one of the holes drilled completely through one side of the socket and into the socket cavity, are indicated by the two white arrows in Fig. 2. The second sample from the socket was removed from the opposite side of the object. None of these samples contained metal. The material removed was composed entirely of the mineral products of corrosion, primarily cuprite (cuprous oxide).

The blade of the pick, although extensively corroded, is

- 
13. Massachusetts Institute of Technology, Cambridge, Massachusetts.
  14. The radiographs reproduced in Figs. 4, 5, and 6 were made at the Eastman Kodak Company, Rochester, New York through the courtesy of Mr. Charles F. Bridgman.

still largely metallic, however. Samples of metal obtained from a depth of almost 1 cm within the blade were analysed by emission spectrography after all observable mineral had been removed from the drillings. The position of this drill hole, indicated by a white arrow in Fig. 3 and observable also as a round, black dot on the radiograph in Fig. 7, is approximately 3.5 cm below the point at which the blade meets the socket. The results of a qualitative analysis of two samples taken from this site are given in the following table.

Spectographic Analysis of Metal Drillings Removed from the Blade

Element	Percentage, by weight, of total sample
Ag	0.01-0.1
As	1.0-10
Bi	0.001-0.01
Cu	Major Component
Fe	1.0-10
Mg	0.001-0.01
Ni	0.001-0.01
Pb	0.1-1.0
Sb	0.1-1.0
Si	<0.0001
Sn	Not detected (detectable to 20ppm)

The metal is therefore a copper-arsenic alloy with antimony and lead as the major impurities. This composition is consistent with an early second millennium date for the object.

Method of Fabrication

The radiographs give every indication that the pick was cast solid in a single piece. The cavity in the socket was created by a core placed inside the mould before the metal was poured. From the position of the shrinkage zone shown in the radiographs, the mould could have been in either of two positions at the time that the molten metal solidified. These are given as positions A and B in Fig. 1. With the mould in position A, the metal would have been introduced through some sort of gate whose location was at the extreme end of the blade,

as shown in the diagram. The second, alternative position is that in which the entire mould was held vertically and the metal poured in from the top. In this case the gate was probably offset to one side of the blade's tip, an unusual displacement.

The evidence for both of these reconstructions is contained in the radiographs. Examination of the blade in Fig. 7 reveals a discontinuous line of high film density running almost precisely along the center line of the blade from the position of the drill hole to the extreme tip of the blade. This line, which corresponds to a region of low density within the metal and which is often referred to as a center line porosity, is characteristic of the final stages in the solidification of a cast metal ingot and represents inadequately fed shrinkage of the last portion of the molten metal to solidify. Porosity can also be caused by the evolution of small gas bubbles in the last of the molten metal. In this particular case it is difficult to estimate the relative extent to which each of these processes has contributed to the porosity indicated in the radiographs. On Figs. 4, 5, and 6 the line of porosity follows the geometric midline of the blade almost along its entire length, but it becomes much broader and more diffuse at the blade's tip and tends to bend toward one narrow side of the blade as indicated in drawings A and B of Fig. 1. The preponderance of porosity at the tip of the blade is due both to the increased amount of metal shrinkage at the place where the metal cooled last and possibly also to the presence of dross in the liquid which may have accumulated and floated to the top of the casting. The large, rectangularly shaped densities near the blade's tip which are particularly clear in the radiographs of Fig. 4 and 5 are

near the surfaces of the blade and are not associated with the center line shrinkage. The fact that the line of porosity tends to bend toward one side of the blade at its tip may be indicative of the fact that a hot sink of metal existed at this location. The last metal to solidify would do so at the place where the metal was hottest, presumably at the position of the gate. Thus the shrinkage would tend to shift over in the direction of this heated area. Either of the reconstructions given in Fig. 1 is consistent with the picture of the internal structure of the casting presented in the radiographs.

Small, almost spherical areas of high film density evident on the radiographs within the body of the camel also indicate porosities in the metal. These represent bubbles formed as the result of gas evolution within the metal itself or from the mould when it was struck by the hot metal. It should be noted that these bubbles tend to form a line along the horizontal where the body of the camel meets the socket of the pick. The socket contained a core made of a clay or a sand-clay mixture at the time of casting. Had the mould been in a vertical position, as in detail B of Fig. 1, it is not unlikely that, during the casting operation, the bubbles formed within the liquid metal of the camel's body were prevented, by the presence of this core, from continuing to move upward through the socket and, finally, from escaping by rising up along the length of the blade. The formation of the bubbles along this horizontal may, therefore, indicate the evidence for position B of Fig 1 as the actual casting position employed.

The shaft or blade of the pick has broken away from the socket at some time subsequent to excavation. The complete continuity of the corrosion products on the surfaces of the object on either side of the

break indicates that the object was intact while buried. The direction of the lines of fracture, which run almost horizontally across both sides of the object at the position of springing of the blade from the socket, is given by the dashed arrows in Fig. 2. The radiographs also reveal quite clearly the position and direction of the break which appears as a series of dark, horizontal lines in Figs. 4, 5, and 6. It is not extraordinary that the pick should have broken where it did. The radiographs indicate that, when the object was fabricated, inherent weaknesses in the form of hot tears may have existed in the metal precisely where the socket meets the much thicker blade. The formation of a hot tear in a casting, characterized by an actual parting of the metal, is not unusual in those portions of the casting where the mould changes abruptly from a relatively thin to a relatively thick size. Such regions are zones of potential weakness which, in the case of this object, may have facilitated the failure of the mineral precisely along such lines of weakness.

Reconstruction of the mould used in the fabrication of the pick is much more difficult. All that can be said with certainty is that it consisted of at least two parts, the outer material that formed the mould proper and the inner core which was necessary to create the socket cavity. Furthermore, there is some evidence that a single metal chaplet, in the shape of a cylindrical pin, was used to attach the core to the mould in order to hold it in place during the pouring of the metal. The position of this pin is outlined by a white circle in Fig. 2. The distance of the pin as measured along a horizontal from the rear of the pick is approximately 2.4 cm on one side of the object (Fig. 2) and approximately 2.3 cm on the opposite side of the socket indicating that the circular depression appearing in Fig. 2 bears

a close relationship to the corroded remnants of the pin which are still visible on the other side of the object. the roughly circular outline of the pin can also be seen in the radiograph in Fig. 5.. The core was also probably attached directly to the mould at both ends to give it increased rigidity during casting. There is, of course, the possibility that the pin described here as a chaplet may actually have been used to hold the pick to the wooden handle. Given the relatively small diameter of the pin, such a mechanical device would not have been adequate to secure the handle to the pick in a tool that was functional, but it might have served sufficiently well for an object only in ceremonial use.

The question remains as to whether the mould itself was a piece mould or whether it was a single structure built up over a wax model which was subsequently melted out of the interior in the usual manner of lost wax casting. The advantage of piece moulding is that many castings can be made from the original model; that of lost wax casting is the freedom it allows in the shaping of the wax model which may contain undercuts and intricacies of form easily reproduced in the casting. Careful examination of the pick has revealed no obvious flash lines in those places where it would seem most natural for mould joints to have existed. Since such lines are easily filed or scraped away, however, their absence on the finished object does not preclude their presence in the rough, as cast condition. The extent and unevenness of the corrosion of the object, as well as the fact that it has been mechanically cleaned in the past, make it difficult to detect any traces of such lines that may in fact still remain. If the mould was a piece mould, it could easily have been of the bi-valve

type with the parting line in the plane that bisects the narrow sides of the pick. Such a mould would draw easily from both sides of the model or, ultimately, from the casting, since the design of the object is such that there are no undercuts, and a simple two piece mould plus core would be adequate. The strongest argument in favor of lost wax casting is the absence of flash lines but, as was pointed out earlier, their absence is not conclusive evidence for the use of the cire-perdue process.

There are several roughly linear protruberances on the object that appear on the blade or just beneath the point at which the blade springs from the socket. Several of these are shown in Fig. 3. They may be due entirely to local differences in corrosion rates associated with cracks in the corrosion product. There is also the possibility, however, that they represent 'fins' of metal having formed when the molten metal ran into cracks in the mould, for they appear to be in no way associated with the parting lines of a piece mould. One of these protruberances, indicated by a white arrow in Fig. 3, can also be seen, in silhouette, in Fig. 2 extending from the bottom of the socket down along the rear, narrow side of the blade.

Although no traces of wood remain in the socket cavity, the corrosion of the inner surfaces of the cavity, especially on its upper surface, displays the fibrous structure associated with wood. It is evident that this corrosion formed while in contact with the original handle that must have been in place at the time of burial but which has since completely deteriorated. The method of assembly of the axe-pick and its handle is a matter of conjecture. The probability of the use

of a metal pin to hold the two together has already been mentioned. Examination of the outer surfaces of the socket revealed no obvious traces in the corrosion of an organic material, such as leather or a fibre cord, which might have been used to bind the axe to the handle. On the other hand, there is a very deliberate, wide, and deep groove with sharply delineated edges lying posterior to the hump of the camel and continuing diagonally along its side. This groove is part of the original casting and can be seen clearly in Fig. 2. The corrosion within the groove is slightly different from that of the surrounding areas. It is less coarse, lighter in color, and tends to give the appearance of having been formed in contact with some organic material which may have once lain within the groove, although no traces of such a material remain. The ridge may be nothing more than an anatomical detail of the camel's hump. It may be worthwhile considering the possibility either that it played some part in securing an organic lacing that was wound around both the pick and the handle or that the ridge was originally intended as the rendering of some portion of a trapping (see below). In the latter case it may or may not have been inlaid with an organic material representing such an element.

In F.E. Zeuner's discussion of the anatomy of this camel he does not mention this groove, although it is distinctly noticeable on the object.<sup>15</sup> This may be due to the fact that he did not inspect the object itself but only photographs all of which showed the opposite side of the camel reproduced in Fig. 2.<sup>16</sup> On the opposite

15. F.E. Zeuner, Op. cit. pp. 162-163.

16. For previously published photographs see D.H. Gordon, Op. cit., as well as F.E. Zeuner and K.R. Maxwell-Hyslop, see also V.G. Childe, New Light on the Most Ancient Near East, 4th ed. Pl. XXIXb.

side of the animal, in fact, the groove is decidedly less pronounced, although it does appear to continue in a shallower form. The speculations here as to the interpretation of this feature are simply several which are suggested by the technical examination.

Zeuner puzzled over the Bactrian or dromedary identity of the camel, "The Khurab beast has only one hump and thus might perhaps be regarded as a dromedary. But every other feature speaks against such interpretation."<sup>17</sup> It is his conclusion after balancing the contradictory evidence that it is a Bactrian camel. One might note that an earlier representation of the Bactrian camel can be seen on a sherd from Sialk.<sup>18</sup> Directly behind the above mentioned groove is a badly corroded area evident on Fig. 2. It is worthwhile considering the possibility that this area originally manifested the posterior hump of the camel, now eroded away. If we accept the posterior hump as eroded away through corrosion than originally the humps were separated by a groove which may have indicated some type of harness. If this interpretation be true it suggests the domestication of camel several centuries prior to the later part of the second millennium where evidence becomes abundant.

The metallurgical analyses and cultural context of this object, recovered from an archaeologically poorly defined area of Iran, rather confirms Professor Mallowan's recent comment "every part of Iran was in due course affected by the pace of technological development in other parts of Asia".<sup>19</sup>

17. F.E. Zeuner, Op. cit. p. 162.

18. From Sialk III-4 context. Ghirshman, R., Fouilles de Sialk, I, Pl. LXXIX, A2

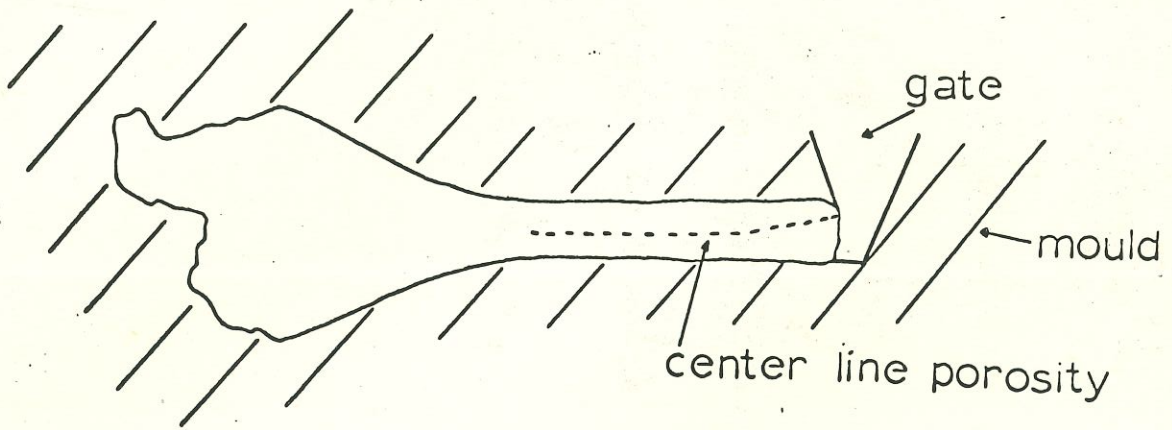
19. Mallowan, M.E.L., 'The Development of Cities' Cambridge Ancient History (fascicle 58). p. 56. 1967.

## Description of the Figures Illustrated

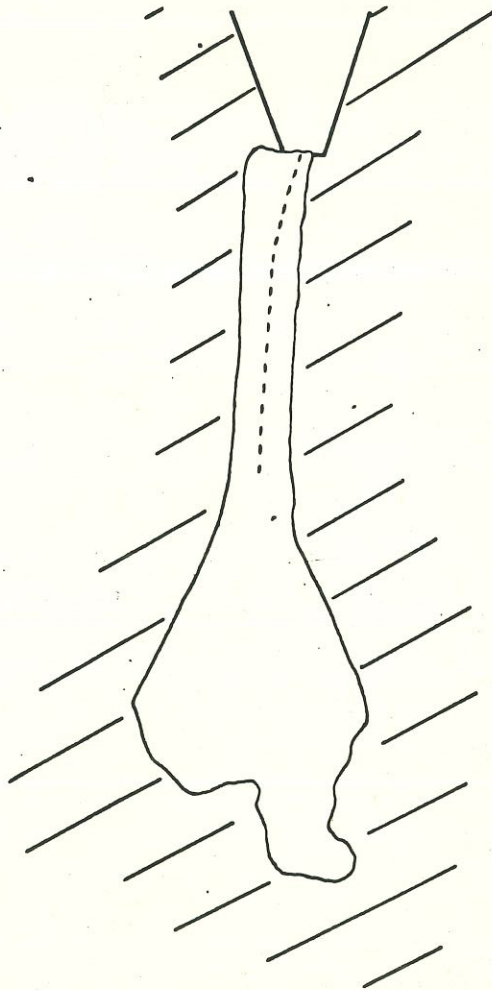
- Fig. 1 Two alternative reconstructions of the position of the mould during casting.
- Fig. 2 Detail of one side of the socket showing the positions of the pin, the sites of two sample drill holes, and the break between the socket and the blade.
- Fig. 3 Detail of the rear, narrow side of the blade showing the site of the metal samples removed for analysis and the linear corrosion protruberances, possibly related to the structure of the mould.
- Fig. 4. X-radiograph, side view (Courtesy of the Eastman Kodak Co.)  
Note particularly the porosity within the body of the camel, the center line porosity along the length of the blade, and the horizontal lines of the break.
- Fig. 5 X-radiograph, side view (Courtesy of the Eastman Kodak Co.)  
The roughly circular outline of the pin at the center of the socket can be seen. The bending of the center line porosity at the tip of the blade toward the front, narrow side is particularly clear.
- Fig. 6. X-radiograph, side view (Courtesy of the Eastman Kodak Co.)  
Considerable porosity exists in the upper portions of the blade, near the socket. Some evidence for the presence of hot tears in the metal where the blade meets the socket can be seen here as well as in Fig. 4.
- Fig. 7 X-radiograph, narrow end view  
Note the center line porosity along the length of the blade and the position of the drill hole where metal was removed for analysis.

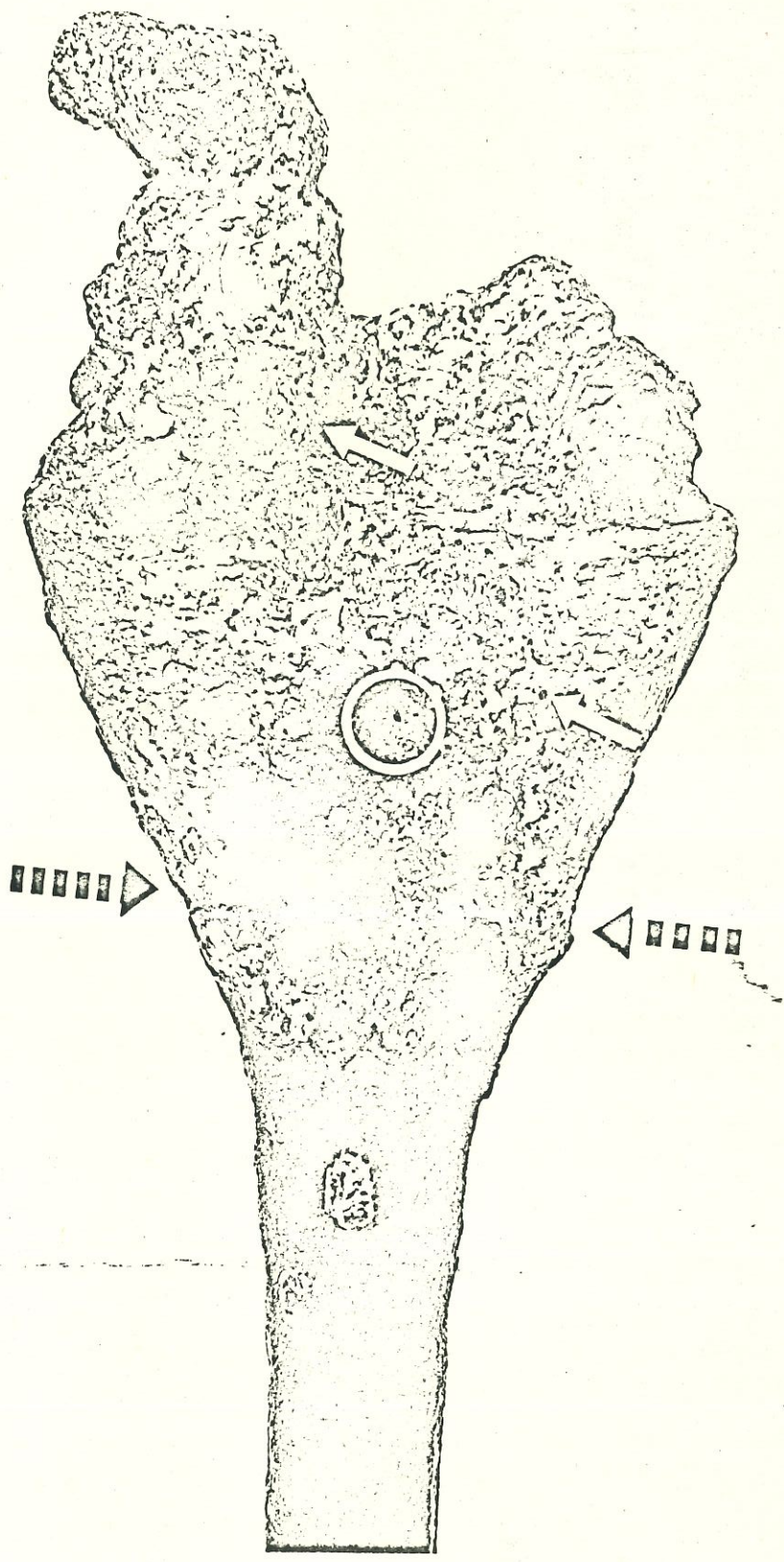
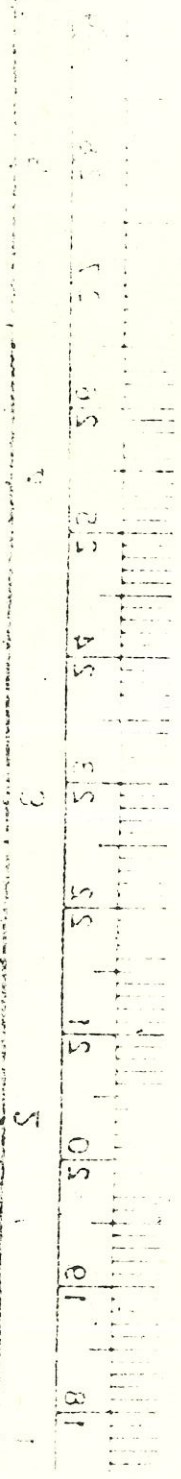
FIG 1

A



B





## NATIVE METALLURGY OF NORTHERN NORTH AMERICA

## PRELIMINARY REPORT

American Indian tools of native copper from Alaska and western Canada were submitted to metallographic and chemical analysis to relate the metal to its geological sources and to reconstruct the metal technology of various American Indian cultures. This study is being continued to include larger series from other museum collections, more analyses, and additional techniques.

The coppers are exceedingly pure, ~~and~~<sup>and</sup> the trace elements were at the limits of detection by X-ray fluorescence.

Emission spectroscopy sorted the coppers into three groups: those of the Central Eskimo which originate in the basalts of the lower Coppermine Basin; those of the interior Dene peoples, which originate in the basin of the Copper, Chitna, and White Rivers of Alaska; those of the Tlingit and Haida Indians of the Northwest Coast, the source of which has not yet been identified. Wet-bench chemistry of samples from the same specimens was conducted by chromatographic column and atomic adsorption techniques. This revealed ~~IDENTICAL~~<sup>the same</sup> branches of trace elements, but showed that their abundance was less than ~~10~~<sup>10</sup>% of that measured by spectroscopic methods. Samples are now being submitted to the neutron ~~activation~~ technique.

Metallographic studies of grain structure, hardness, and other details within sections of specimens sorted the tools into three technological groups. The Tlingit and Haida tools ~~were~~<sup>had been</sup> made by ~~sawing~~<sup>stone -</sup> slabs from a large piece of copper and ~~grinding~~<sup>by</sup> the slabs to shape. The workmen seem to have

been concerned with conserving ~~the~~ gross crystal patterns within the metal, and with their development as visible structure through chelation of copper surfaces with fish-oil. The Central Eskimo tools were formed in the same way, but cutting edges were then highly cold-forged to stress harden them, a refinement missing on the Northwest Coast tools. The Dene tools show shaping by forging with extensive annealing, with cutting edges stress hardened.

It is thus possible to define three different stone age technologies in native copper, each associated with a different copper source. Eskimo and Northwest Coast peoples worked copper by the same sawing and grinding techniques which they applied to jade and slate, their metal working being a simple extension of stone working. The Dene smithing is of a different character, having much in common with the technologies of metal age cultures.

John Witthoft

Frances Eyman

"Aynhoe"  
28, Kings Moor Road,  
Stockton on The Forest,  
YORK.

England,

3/2/69.

Dear Beth,

Thanks for your letter of January 22nd.  
I received the new series of Urceurian  
bronzes and the payment - thank you.  
I was able to do the analysis of  
them before I left Oxford in December.  
I have now got the microphotometer  
recalibrated and working here in York  
and have so far read and calculated  
some 40 of your samples. I shall  
send you the completed results of  
them all within the next 14  
days. I hope that this will be  
early enough to meet your deadline.  
I cannot stress too much how sorry  
I am that these have taken so  
long to finish - circumstances caught  
up with me.

With all best wishes.

Anne.

Report of spectrographic analyses by Mrs. A. Millet

Research Lab for Archaeology + the History of Art

6 Keble Road

Oxford, England

20 April 1966

All of the results are expressed as percent to two significant places. Where there is a blank none of that element was detected

No Au was found

The Winterthur Museum  
ANALYTICAL LABORATORY  
REQUEST FOR SERVICE

REQUEST NO. 260

DATE November 12, 1974

NAME Elizabeth Ralph, Jim Weinstein, University of Pennsylvania

REQUEST FOR ANALYTICAL SERVICES: (Describe what information is needed and how it will be used.)

Analyze 10 pieces of ancient Egyptian bronzes to determine composition and to compare with data obtained on emission spectroscopy equipment.

OBJECT (S) OR SAMPLE INFORMATION:

Accession No. \_\_\_\_\_

Check here if non-Winterthur piece:  X

Object Description: (State material, size, nature, etc.)

Date of object:  
Provenance: Egypt  
Maker:

REPORT

DATE November 12, 1974

LOCATION OF ORIGINAL DATA: Analytical Lab Notebook No. 64

INITIALS OF REPORTER: GJR/JHC *JHC*

DISCUSSION: Several of these pieces appear to be nearly pure copper (E13379, 29-65-647, 29-65-654, 29-65-655, 29-65-657, 29-65-661, D3049), with a few impurities present at the trace level. Three others are typical bronze compositions (E1055, 9266, 29-65-626). Note the unusually high arsenic content, 1.29 to 1.43% in the mirror 29-65-661. The silver content ranges from 0 to 0.12%.

The results of our X-ray fluorescence analysis of E13379 and E1055 cannot be compared to the emission spectrographic data brought with the samples for two reasons:

1. Our X-ray fluorescence brass-bronze standard contains no nickel and bismuth. Therefore we cannot give quantitative results for these elements.
2. Not all the elements determined by X-ray fluorescence analysis (i.e., zinc, arsenic) were determined by emission spectrograph.

cc: Charles Hummel  
Don Fennimore  
Mary Cash  
2 File

Returned: DATE \_\_\_\_\_

BY WHOM *JHC* 11-18-74

THE WINTERTHUR MUSEUM  
ANALYTICAL LABORATORY

page 1

	Cu	Mg	Fe	Ni	Zn	As	Pb	Au	Bi	Ag	In	Sn	Sb
A: Non-Winterthur R: 3009 Egyptian bronzes P: E 13379, side of chisel near middle for Elizabeth Ralph, U. of Pa.	98.99	0	.17	.11	0	0	.56	.07	0	0	0	.03	.01
A: R: 3010 P: E 13379, chisel near tapered end	96.89	.54	.54	.41	0	.02	1.13	.26	.01	0	0	.03	.02
A: R: 3011 P: Pure copper sheet (standard)	99.83	0	.03	0	0	.02	0	.10	0	0	0	0	0
A: R: 3012 P: E 1055 axe head unpolished	76.49	1.23	.96	.58	3.03	.23	2.89	.87	.04	.12	.08	12.34	.62
A: R: 3013 P: E 1055 axe blade unpolished, other side	76.88	1.21	.86	.63	3.12	.20	2.69	.80	.04	.11	.08	12.28	.58
A: R: 3014 P: E 1055 axe blade polished (same side as 3013)	77.44	1.17	.81	.56	2.97	.28	2.56	.86	.04	.11	.07	12.03	.58
A: R: 3015 P: 9266 knife blade, side without numbers	82.20	.82	.56	.35	2.36	.20	1.00	.56	.03	.10	.06	10.89	.47

A: Accession #  
R: Run #  
P: Part of Object

Non-Winterthur Bronzes (Egypt) Brass-Bronze Analysis  
11/12/74 Run # 3009-3015  
A.L. # 260 page 1 continued

THE WINTERTHUR MUSEUM  
ANALYTICAL LABORATORY

page 2

	Cu	Mg	Fe	Ni	Zn	As	Pb	Au	Bi	Ag	In	Sn	Sb
A: Non-Winterthur R: 3016 Egyptian Bronzes P: 9266 knife blade, side with numbers for Elizabeth Ralph; U. of Pa.	80.22	.86	.69	.52	2.58	.27	1.04	.67	.03	.12	.07	11.88	.56
A: R: 3017 P: 29-65-654 mirror side without numbers	98.35	.17	.11	0	.35	.22	.29	.17	0	.05	0	.12	.09
A: R: 3018 P: 29-65-654 mirror side with numbers	98.09	.12	.19	.07	.04	.61	.34	.26	0	.04	0	.06	.10
A: R: 3019 P: 29-65-655 mirror side without numbers	98.46	0	.28	.06	.25	.18	.41	.13	0	.06	0	.01	.04
A: R: 3020 P: 29-65-655 mirror side with numbers	98.68	.04	.12	0	.29	.11	.43	.15	0	.08	0	.01	.03
A: R: 3021 P: 29-65-647 mirror, convex side	98.31	.04	.45	.15	.05	.54	.14	.17	0	.02	0	.01	.01
A: R: 3022 P: 29-65-647 mirror, concave side	97.74	.23	.54	.09	0	.74	.24	.25	0	.02	0	.02	.01

A: Accession #  
R: Run #  
P: Part of Object

Non-Winterthur Bronzes (Egypt) Brass-Bronze Analysis  
11/12/74 Run # 3016-3022  
A.L. # 260 page 2 continued

THE WINTERTHUR MUSEUM  
ANALYTICAL LABORATORY

page 3

	Cu	Mg	Fe	Ni	Zn	As	Pb	Au	Bi	Ag	In	Sn	Sb
A: Non-Winterthur R: 3023 Egyptian Bronzes P: 29-65-644 razor, side with numbers for Elizabeth Ralph, U. of Pa.	97.70	.25	.27	.09	.19	.44	.70	.17	0	0	0	.02	.04
A: R: 3024 P: 29-65-644 razor, side without numbers	98.45	.07	.36	.12	0	.27	.40	.12	0	0	0	.02	.03
A: R: 3025 P: D 3049 side without numbers	99.50	0	.02	0	0	.34	.04	.04	0	.01	0	0	.02
A: R: 3026 P: D 3049 side with numbers	99.50	0	0	0	0	.35	.04	.07	0	.02	0	0	.02
A: R: 3027 P: 29-65-626 side without label	75.66	1.16	.94	.69	2.79	.19	1.83	.85	.04	.11	.08	14.42	.72
A: R: 3028 P: 29-65-626 side with label	78.33	1.06	.76	.56	2.22	.22	1.71	.70	.04	.10	.08	13.13	.60
A: R: 3029 P: 29-65-661 mirror	96.65	.18	.37	.05	.15	1.43	.48	.40	0	.04	0	.01	.08

A: Accession #  
R: Run #  
P: Part of Object

Non-Winterthur Bronzes (Egypt) Brass-Bronze Analysis  
11/12/74 Run # 3023-3029  
A.L. # 260 page 3 continued

THE WINTERTHUR MUSEUM  
ANALYTICAL LABORATORY

page 4

	Cu	Mg	Fe	Ni	Zn	As	Pb	Au	Bi	Ag	In	Sn	Sb
A: Non-Winterthur R: 3030 Egyptian Bronzes													
P: 29-65-661 mirror, other side for Elizabeth Ralph, U. of Pa.	97.43	.07	.18	.08	0	1.29	.41	.30	0	.04	0	.01	.08
A: R:													
P:													
A: R:													
P:													
A: R:													
P:													
A: R:													
P:													
A: R:													
P:													

A: Accession #	Non-Winterthur Bronzes (Egypt)	Brass-Bronze Analysis
R: Run #	11/12/74 Run # 3030	
P: Part of Object	A.L. # 260	page 4